Ceramic Micropalaeontology

The analysis of microfossils in archaeological ceramics with

special reference to its application in the southern Aegean

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Volume 1

Thesis submitted for the degree of Doctor of Philosophy

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September 1999

"A number of approaches are available which may be adopted to extend the power of ceramic petrology, but some of them require a considerable investment of analyst time for improvements which are far from guaranteed and which may be marginal. Careful consideration is advocated before they are applied."

Freestone (1991, 407).

Abstract

Within the scientific analysis of archaeological ceramics, four principal aims can be specified: description, classification, the reconstruction of ceramic technology and the determination of provenance. In order to achieve these, sophisticated methods of thin section analysis have been developed which permit the retrieval of detailed information about the nature of the rock and mineral inclusions as well as the textural features of the ceramic micromass. One important group of inclusions which occur in many archaeological ceramics are the organic or mineralised remains of various microscopic animals and plants, collectively referred to as microfossils. Microfossils are studied in detail only rarely by ceramic petrographers, however they contain information pertaining to the geological age and palaeoenvironment in which their host sediment was deposited, and as such can be used to characterise and provenance the raw materials of ceramic manufacture. Whilst holding great potential for the analysis of archaeological pottery, there are also a variety of problems associated with these types of inclusions, such as their alteration and removal by various processes during the production and post-depositional history of ceramics.

Specialist analyses of microfossils in archaeological ceramics are small in number and biased towards the investigation of diatoms from the Neolithic to Iron Age pottery of north-west Europe. This thesis represents the first comprehensive study of the occurrence and utility of all microfossils in archaeological ceramics and is divided into two main sections. The first comprises a detailed account of the occurrence, preservation, methods of analysis, behaviour upon firing, and utility of all groups of microfossils in archaeological ceramics. This reappraisal is followed by several individual case studies from the Bronze Age of Crete and elsewhere in the Mediterranean which utilise calcareous microfossils to address a variety of archaeological questions of varying geographical scale and detail concerning ceramic provenance and technology.

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Abbreviations

MA	Million years
BP	Before present
EM	Early Minoan
MM	Middle Minoan
LM	Late Minoan
LO	Last occurrence
FO	First occurrence
FCO	First common occurrence
LCO	Last common occurrence
acme	Period of high abundance
NN	Neogene nannoplankton zones (Martini 1971)
CN	Neogene nannoplanton zones (Okada and Bukry 1980)
DSDP	Deep Sea Drilling Project
ODP	Ocean Drilling Project
PPL	Plane polarised light microscopy
XP	Cross polarised light microscopy
SEM	Scanning electron microscopy
AAS	Atomic absorption spectrophotometry
ICP	Inductively coupled plasma spectrometry
INAA	Instrumental neutron activation analysis
OES	Optical emmision spectrometry
XRD	X-ray diffraction
IGME	Institute of Geology and Mineral Exploration, Athens

Acknowledgements

In the four years which it has taken to produce the following thesis I have been fortunate to receive assistance, advice and even inspiration from many sources, some of which are outlined below.

The original impetus for a reappraisal of the analysis of microfossils in archaeological ceramics came from Dr Peter Day of the Archaeology and Prehistory Department, University of Sheffield and Dr. Nicolette Hine, formerly of the Centre for Palynology, University of Sheffield, who enlisted me for the task. Both have been a great source of guidance throughout the years, especially Dr. Day, to whom I owe a special debt of gratitude for the many different ways in which he has assisted me, which have often been beyond his obliged duties as my supervisor.

This project would not have been possible without the generous financial assistance of the Natural Environment Research Council (NERC) who covered not only my fees and living expenses, but funded several crucial fieldwork seasons in the Aegean. I would like to thank Mrs. Janice Black and all other staff at NERC who dealt with my studentship (No. GT4/95/280) and ensured the prompt reimbursement of my often confusing expense budget.

Numerous specialists from several fields have offered their expert advice on various aspects of this report and I would like to acknowledge them accordingly. In the analysis of calcareous nannofossils I have received the assistance of Dr. Jeremy Young of the Natural History Museum, London, who as editor of the journal *Marine Micropalaeontology* gave me the chance to publish my first writings in the form of a small review of the application of micropalaeontology to archaeology (Quinn 1997). He, and several other members of the British Micropalaeontological Society Nannofossil Working Group, especially Dr. Jackie Burnett and Dr. Paul Bown have offered their advice and guidance on several occasions as well as allowing me to keep in contact with the field of micropalaeontology when I have been increasingly involved with archaeology. Other nannopalaeontologists who have assisted me on occasion include Dr. Katharina Von-Salis and Dr. Carla Müller, not forgetting my supervisor Dr. Hine, who kindly read my lengthy discussions of taxonomy and

1 Introduction

The main aims of the compositional analysis of archaeological ceramics are the characterisation and classification of pottery samples, which in turn can be used to illustrate issues of provenance and of ceramic technology (Riley 1982). There are several approaches possible within this subject, each focussing on different characteristics of archaeological ceramics, for example the determination of chemical composition, the observation of microstructure in the scanning electron microscope (SEM) or mineralogical analysis by X-ray diffraction (XRD). One popular and relatively low-tech form of compositional analysis, ceramic petrography, uses the inclusions and the appearance of the clay micromass within thin sections of pottery samples to interpret the nature of the raw materials and technology used to transform them into ceramics. Various types of inclusion occur in ceramics, including single mineral grains and fragments of sedimentary, igneous and metamorphic rocks made up of agglomerations of one or more minerals. In order to identify potential sources of raw materials for ceramic manufacture and thus the provenance of the pottery in question, petrographers relate the nature of these inclusions to the established local and regional geology of an area, as well to as comparative sediment and ceramic samples.

One distinctive group of inclusions which occur in archaeological ceramics from various parts of the world are the microscopic, organic or mineralised remains of various single and multi-celled organisms (Figure 1.2). These conspicuous structures, which are referred to as 'microfossils' because of their small size and biological origin, occur in many types of marine and non-marine sediments deposited during the

ERA	PERIOD
Cenozoic	Quaternary
	Neogene
	Palaeogene
	65 MA
Mesozoic	Cretaceous
	Jurassic
	Triassic
	245 MA
Palae- ozoic	Permian
	Carboniferous
	Devonian
	Silurian
	Ordovician
	Cambrian
	570 MA
Proterozoic	

Figure 1.1. The geological column. MA = Million years.

Group of microfossils		Biological affinities, ecology geological range and size.
Foraminiferal tests Chapter 6		Internal, chambered, often coiled, usually calcareous skeletons of single-celled animals (Foraminifera), which inhabit marine environments and can have a planktonic or benthic life habit. Range = Cambrian to Recent. Size = usually < 2 mm
Ostracod valves Chapter 7		External, hinged, bivalved calcareous shells of minute crustaceans (Ostracods), which inhabit most aqueous environments including lakes, rivers and the deep sea, and can have a planktonic or benthic life habit. Range = Cambrian to Recent. Size = 0.7-5 mm.
Radiolarian tests Chapter 8		Internal siliceous skeleton of single-celled planktonic animals (Radiolaria), which inhabit marine environments. Range = Cambrian to Recent. Size = usually < 500 μm
Diatom frustules Chapter 8		External, box-like, two-part siliceous skeleton of single-celled plants (Diatoms) which inhabit all marine and non-marine aqueous environments, including soil, and can be benthic or planktonic. Range = Early Jurassic to Recent. Size = 10-100 μm.
Calcareous nannofossils Chapter 5		External, calcareous plates (coccoliths) of single-celled, planktonic marine plants (Coccolithophores), as well as various minute calcareous structures of unknown origin (nannoliths). Range = Late Triassic to Recent. Size = usually <10 μm.
Dinoflagellate cysts Chapter 9		External, pylomate, organic resting cysts of single-celled celled marine plants. Range = Late Triassic to Recent. Size = usually < 100 μm.
Pollen and spores Chapter 9		Organic, often spherical, reproductive structures of multicellular land plants which occur in marine and non-marine aqueous sediments. Range = Silurian to Recent. Size = usually < 200 μm.

Figure 1.2. The biological affinities, ecology, geological range and size of the main groups of microfossils.

last c. 500 million years (Figure 1.1). Microfossils have been studied intensively for many years in an offshoot of geology called micropalaeontology, where specific forms are used to date and correlate rocks as well as to determine the palaeoenvironment in which their host sediment was deposited. This property of microfossils, which has been utilised heavily during the latter half of this centrury in the search for oil and gas, is well-suited to the characterisation of the raw materials of ancient ceramic manufacture.

However, detailed analyses of microfossils in archaeological ceramics have so far been undertaken on an *ad hoc* basis, perhaps due to the highly specialised nature of micropalaeontology which does not lend itself to routine application within the petrographic description of archaeological ceramics, and the lack of collaboration between micropalaeontologists and archaeological scientists. The studies which do exist have been sporadic and are heavily biased towards the analysis of siliceous microfossils from Neolithic, Bronze Age, Iron Age and Medieval pottery from north-west Europe.

The following thesis represents the first detailed account of all groups of microfossils in archaeological pottery and attempts to construct the foundations of a new approach to the subject of 'ceramic micropalaeontology'. In Chapter 2 a thematic review of previous detailed analysis of microfossils in archaeological ceramics is presented. This serves to illustrates the way in which microfossils have been used with varying success to ascertain different levels of information about ancient ceramic production, as well as highlighting some of the inherent problems associated with the analysis of microfossils in archaeological pottery. These problems are expanded in Chapter 3,

which looks in detail at the production sequence and history of ceramic artefacts and identifies several potential sources of contamination and alteration of microfossil assemblages, some of which are investigated experimentally in Chapters 5 to 9. Chapter 4 presents an overview of the geological history of the Aegean and identifies those sediments which are of concern to the analysis of microfossiliferous archaeological ceramics from this region and have been studied in further detail in Chapter 10.

The bulk of this thesis is represented by Chapters 5 to 9, which deal with each of the main types of microfossils in turn; outlining their occurrence, preservation and the methods by which they can be studied in archaeological ceramics, their transformation during the process of firing (based upon experimental work and previous literature) and the potential they offer to the scientific analysis of ancient pottery. In addition, Chapters 5 to 7 contain a review of the numerous biostratigraphic schemes which have been presented for late Neogene calcareous nannofossils, foraminifera and ostracods in the Mediterranean which are utilised in the analysis of these calcareous microfossils in Chapter 11. Because of the difficulties which are involved with the biostratigraphy of calcareous nannofossils for this time period in the Mediterranean, it has been necessary to undertake a thorough review of the occurrence and utility of several less conventional members of this microfossil group. This review is presented in Appendix II.

Chapter 11 applies the methodology outlined in the main body of this thesis to the analysis of calcareous microfossils from pottery of the Bronze Age and later archaeological periods in the Mediterranean. Each of the five case studies which are

presented involve the application of micropalaeontology to coherent archaeological questions. These confirm and refine interpretations of ceramic production and exchange previously formulated through other techniques of ceramic compositional analysis. The information produced through the analysis of microfossils in these examples has direct relevance to the understanding of ancient societies. It offers a new level of detail regarding questions of ceramic production, notably the location of production centres and the choice and manipulation of raw materials by potters in the past. As it is these very questions which increasingly are being posed by archaeologists, micropalaeontological analysis of ceramics is seen to be a potentially powerful tool within archaeological scientific analysis.

The various strands which are presented in this thesis have been brought together in the final chapter, in order to define the 'subject' of ceramic micropalaeontology and provide a coherent starting point for future work.

2 Previous studies on microfossils from archaeological ceramics

2.1 Introduction

As mentioned in the previous chapter, microfossils are a relatively common component of archaeological ceramics from many parts of the world. It is now necessary for us to review the way in which both archaeologists and micropalaeontologists have dealt with the occurrence of these distinctive non-plastic inclusions by summarising the approach to the description of microfossils within the subject of ceramic petrography, and by reviewing the previous detailed analyses of microfossils in archaeological ceramics.

2.2 Microfossils in ceramic petrography

Before we review the previous micropalaeontological investigations of archaeological ceramics, it is necessary for us to consider the way in which archaeologists have dealt with the occurrence of microfossils in pottery. As an illustration, this section will focus on two examples where microfossils have been encountered in the petrographic analysis of ancient ceramics; the work of Riley (1981) and Whitbread (1995).

During his petrographic examination of Late Bronze Age coarse-ware stirrup-jars from Mycenae, Riley (1981) encountered chert inclusions containing radiolaria in several of the 37 samples which he analysed. Riley did not describe these microfossils in detail, or utilise their occurrence in his petrographic classification of the stirrup-jars; as all three samples were placed in separate fabric groups. More significant,

however was the concurrence of foraminifera and metamorphic rock fragments in ten of the 37 stirrup-jar thin sections. Riley classified these as his Fabric B on the basis of the two distinctive types of inclusions. In outlining this fabric, Riley (1981, 336) attempted a rudimentary description of the foraminifera which he had seen in thin section, by noting their abundance, "5 per square millimetre" and their size, "0.4 mm across". This information which is of very little value was not utilised by Riley in his interpretation of the fabric. He also mentioned that "the foraminifera are well-preserved" and used this to suggest that his Fabric B was constructed from 'recent sediments', mixed with a clay containing low-grade metamorphic fragments (1981, 336). Although Riley's identification of clay mixing was correct, his interpretation of the geological age of the microfossiliferous component of Fabric B, was not. Well-preserved foraminifera can occur in very ancient sediments, as well as in fired pottery manufactured from such deposits, and despite the occurrence of an overall correlation between geological age and preservation, one is never used as a guide to the other.

Twelve of Riley's stirrup-jar samples were also analysed with optical emission spectrometry (OES) by Catling *et al.* (1980). These authors suggested that seven of the samples may have a Cretan origin. Although Riley (1981, 338) commented that "this is neither fully confirmed nor can this be rejected on petrographic grounds", he cited the occurrence of foraminifera in fine pottery and their association with phyllite in coarse ware pottery from the LM IB period at Knossos. By comparing the results of Catling *et al.* (1980) with Riley's petrographic groups, it can be seen that the two Fabric B samples which were analysed by OES have a different chemical composition.

The petrographic correlation between the coarse-ware pottery from Crete and Mycenae, which Riley indicated, was significant, however, as the author noted himself, "further examination of pottery and clays" (1981, 339) was needed before any conclusions could be made on a petrographic basis. He nevertheless considered the geology of Crete to be compatible with such an interpretation due to the occurrence of "outcrops of phyllite in both western and eastern Crete as well as parts of central Crete" (1981, 339), but did not include the foraminifera in this consideration. Neogene sedimentary deposits containing foraminifera occur extensively on Crete (Chapter 4, Figure 4.1), however the geological age of these marine sediments has been well established by various authors, e.g. Zachriasse (1975) and Spaak (1983), and nowhere on the island have Recent sediments containing foraminifera been found. The term 'Recent', in a geological sense means the present. In which case, Riley's (1981) interpretation of the raw materials used in the construction of his Fabric B ceramics was incorrect. A better interpretation of the geological age of the raw materials of ceramic manufacture through the generic and specific identification of the foraminifera, was achieved in the later work of Riley (1983) and Riley *et al.* (n.d.).

Riley's (1981) analysis of the Late Bronze Age coarse-ware stirrup-jars from Mycenae is a good example of how ceramic petrographers, both successfully and unsuccessfully, have utilised the microfossils occurring in thin sections of archaeological pottery. In classifying the stirrup-jars, Riley (1981) treated the microfossils as he would any other inclusion, and considered their occurrence together with the rest of the fabric. In this respect, his decision to unite the samples of his Fabric B by the occurrence of foraminifera and metamorphic rock fragments, yet

separate samples 10968, 5357 and 10970 which contained radiolarian chert, was correct. Riley's description of the microfossils occurring in the pottery samples however, was far too simple and of little use, except perhaps to convey to the reader the approximate size of the foraminifera. His comment on the state of preservation of these microfossils was significant but the subsequent interpretation of the nature of the raw materials of the Fabric B ceramics, which Riley (1981) based upon this, was incorrect.

In his monograph on ancient Greek transport amphorae, Whitbread (1995) encountered microfossils in ceramic thin sections from Rhodes, Kos, Chios, Lesbos, the Kassandra peninsula, the Sithonia peninsula, Paros and Corinth. In his detailed fabric descriptions of this pottery, Whitbread included the microfossils in his coarse fraction, as a separate type of inclusion, or as part of another inclusion (e.g. radiolarian chert). He also indicated the semi-quantitative frequency of the microfossil specimens within this fraction of the fabric, and identified, wherever possible, the broad microfossil group to which they belong (i.e. foraminifera; radiolaria).

Thus, Whitbread utilised the occurrence of microfossils in a similar manner to Riley (1981), as a means of characterising his fabric groups (where they occurred consistently alongside other distinctive inclusions, for example in the case of the Chian amphora Fabrics), to subdivide fabric groups (e.g. Classes 1 and 2 of his Corinthian type A' Fabric), or to interpret their sporadic occurrence as variation within fabric groups characterised by other types of inclusions (e.g. Koan Fabric Class 3).

In describing the microfossils which occur in this pottery, Whitbread (1995, 152) did little more than Riley (1981) in that he usually included a rough indication of their size, e.g. "frequently about 0.2 mm in size". He also described those specimens which he was unable to attribute to a broad microfossil group, in terms of their overall shape, e.g. "spherulitic structures (possible radiolaria)", and other dimensions, e.g. "ovoid tests, about 0.02 mm thick, with a single chamber, about 0.6 mm in diameter" (Whitbread 1995, 287 and 162). This latter description was of two microfossils which he encountered in a single thin section of the Lesbian amphorae Fabric; the specimens in question, which are illustrated in Whitbread's monograph (1995, Fig. 4.4, 159), are considered here to be ostracod valves.

However, in the interpretation of the occurrence of microfossils in Greek amphorae, Whitbread (1995) made some significant conclusions pertaining to the provenance and technology of these ceramics through the analysis of comparative sediment samples. This can be demonstrated by his analysis of amphorae from Corinth. Whitbread's type A and type A' Class 1 fabrics contained radiolarian mudstone inclusions, chert fragments with radiolaria and isolated radiolarian tests in thin section, and his type B Fabric (Classes 1 and 3) contained chert with rare to very rare "traces of radiolaria" (1995, 290). The occurrence of radiolarian mudstone in Corinthian amphorae has been noted by others, including Farnsworth (1964) who attributed these and other rock fragments to the local geology of Corinth. Whitbread identified the source of this temper as the shale-sandstone-radiolarite formation which occurs on the flanks of the Acrocorinth and Penteskouphi mountains near to Corinth, and studied comparative samples, which revealed that these deposits were very similar to the inclusions which "are typical of Corinthian ceramic Fabrics" (1995, 330).

The two examples presented above, are illustrative of the way in which archaeological scientists have dealt with the occurrence of microfossils in thin sections of ceramics from the Aegean, the way they have utilised them to characterise and classify pottery, as well as to indicate the provenance of raw materials used in their manufacture. As outlined in the previous chapter, microfossil assemblages contain useful information pertaining to the geological age and depositional environment of their host sediment. In order to extract this information it is necessary to analyse them in detail and identify the taxa which they contain. So far, this has been beyond the capability of most archaeological scientists. However, there have been several studies in which micropalaeontologists have applied their expertise to the study of ceramics, often in collaboration with archaeological scientists and archaeologists, in order to attain more detailed interpretations of the microfossil assemblages in ancient pottery. These are presented in Figure 2.1 and reviewed in the following section.

2.3 Detailed analyses of microfossils in archaeological ceramics

There have been numerous detailed analyses of microfossils in archaeological ceramics since the 1950's. However, the publication of these has been sporadic and heavily biased towards the analysis of diatoms from the pottery of north-west Europe (Figure 2.1). Nevertheless, there have been several, published and unpublished investigations of other microfossil groups occurring in archaeological pottery from other parts of the world, e.g. foraminifera in ceramics from the Mediterranean (Riley 1983; Troja et al. 1996; Alaimo et al. 1997; MacGillivray et al. 1988; Riley et al. n.d.), calcareous nannofossils and pollen in ceramics from England (Burnett and

AUTHOR(S)	DATE	REGION	PERIOD	MICROFOSSIL GROUPS
Davis	1951	England	Iron Age	Foraminifera
Edgren	1966	Finland	Sub-Neolithic	Diatoms
Foged	1968	Norway	Neolithic	Diatoms
Edgren	1970	Finland	Sub-Neolithic	Diatoms
Jansma	1977	Netherlands	Neolithic, Iron Age, Medieval	Diatoms
Alhonen and Matiskainen	1980	Finland	Sub-Neolithic	Diatoms
Alhonen et al.	1980	Finland	Sub-Neolithic	Diatoms
Alhonen and Väkeväinen	1981	Finland	Sub-Neolithic	Diatoms
Jansma	1981	Netherlands, England	Neolithic, Iron Age, Medieval, Saxon	Diatoms
Gibson	1983a	England	Neolithic, Bronze Age	Diatoms
Gibson	1983b	England	Neolithic, Bronze Age	Diatoms
Riley	1983	Crete	Bronze Age	Foraminifera
Matiskainen and Alhonen	1984	Finland	Sub-Neolithic	Diatoms

Figure 2.1 Part 1. Catalogue of published and unpublished detailed analyses of microfossils in archaeological ceramics.

AUTHOR(S)	DATE	REGION	PERIOD	MICROFOSSIL GROUPS
Jansma	1984	Netherlands	Neolithic, Iron Age	Diatoms
Håkansson and Hulthén	1986	Germany, Sweeden	Neolithic	Diatoms
Håkansson and Hulthén	1988	Sweeden	Neolithic	Diatoms
MacGillivray *et al.*	1988	Crete	Bronze Age	Foraminifera
Jansma	1990	Netherlands	Neolithic	Diatoms
Troja *et al.*	1996	Sicily	Neolithic, Bronze Age	Foraminifera, Nannofossils
Stilborg	1997	Denmark	Iron Age	Diatoms, Foraminifera
Alaimo *et al.*	1997	Sicily	Punic	Foraminifera, Ostracods
Riley *et. al.*	unpublished	Crete, Greece, Sicily	Bronze Age	Foraminifera
Burnett and Young	unpublished	England	Bronze Age	Nannofossils
Hunt	unpublished	England	Iron Age	Organic microfossils
De La Fuente	unpublished	Argentina	Inka	Diatoms
De La Fuente and Macchiavello	unpublished	Argentina	Inka	Diatoms

Figure 2.1 Part 2. Catalogue of published and unpublished detailed analyses of microfossils in archaeological ceramics.

Young, n.d.; Hunt, 1996), as well as a few analyses of diatoms in ceramics from elsewhere (Gibson 1983a and b; De La Fuente and Martinez Macchiavello 1997).

These studies have utilised the information contained in microfossil assemblages, with varying success, to classify ceramics and interpret their provenance and technology, as well as aspects of clay choice. The following review is presented in terms of these archaeological questions.

2.3.1 Classification

On the simplest level, the presence, absence and the nature of microfossil assemblages in archaeological ceramics have been used as a means of characterising and classifying pottery sherds, as well as interpreting pottery typology (Battarbee 1988). Microfossils are usually very distinctive where they occur in ceramics and are therefore an obvious type of aplastic inclusion with which to classify samples. The different ways in which microfossils have been used to characterise and group pottery are outlined below.

2.3.1.1 Presence/absence

In Section 2.2 we reviewed the method by which two petrographers (Riley 1981; Whitbread 1995) utilised the presence of specific microfossils in ceramics, to classify their samples. In these examples, the consistent occurrence of foraminifera (Riley 1981) and radiolaria (Whitbread 1995), in addition to distinctive rock and mineral inclusions, were used to correlate thin sections of archaeological pottery and establish

petrographic groups. More sporadic occurrences of microfossils were also noted in other thin sections, however, these were not used as a means of classification, due to the lack of correlation between the microfossil specimens and the other petrographic characteristics of the samples. Instead, these microfossiliferous samples were interpreted as variation within other fabric groups. This 'weighting' of the presence/absence of microfossils, by its contextualisation rather than an exclusive concentration upon it is essential in classifying archaeological ceramics. However, such an approach has not been taken in some more specialist micropalaeontological analyses of ceramics (e.g. Jansma 1977).

Jansma (1977; 1981; 1984; 1990) has isolated diatoms from Dutch Neolithic, Iron Age and Medieval pottery (Section 8.4.1). At the Neolithic site of Zandwerven, Jansma (1977) analysed small numbers of sherds belonging to the stratigraphically differentiated Vlaardingen (VL) and Protruding Foot Beaker (PFB) 'cultures'. His results indicated that the VL sherds contained diatoms and the PFB sherds were barren. This correlation between the presence/absence of diatoms in the archaeological pottery samples, and the 'cultural' phase of the Dutch Neolithic to which they belonged, would appear to be significant, and on this basis Jansma (1977) ascribed a different provenance to the two groups of ceramics (Section 2.3.2.1). However, at two neighbouring archaeological sites from which Jansma (1977) analysed 'culturally' contemporaneous Neolithic pottery samples, the VL sherds were barren (Leidschendam) and the PFB sherds contained diatoms (Voorschoten).

The problem with Jansma's (1977) interpretation of the VL and PFB ceramics at Zandwerven is that, as indicated by the evidence from Leidschendam and

Voorschoten, the presence/absence of diatoms may not necessarily reflect a difference in the nature of the raw materials used in the manufacture of this pottery. By analysing more of these pottery sherds and comparing the presence/absence of diatoms with their petrography, it may have been possible to separate the two typological groups of pottery on a compositional basis. The presence/absence of diatoms in the archaeological ceramics from successive 'cultures' could in fact be an artefact of firing. This is clear as, in his six restrictions pertaining to the diatom analysis of pottery, Jansma (1977, 77) stated that "the silica of which the thecae are formed cannot endure temperatures above 1000°C" (Section 8.3) and he considered this possibility in accounting for the absence of diatoms in Neolithic sherds from the Dutch sites of Drente, Utrecht and Noord-Brabant.

2.3.1.2 Composition of the microfossil assemblage

There are several ways in which the *composition* of microfossil assemblages in archaeological ceramics have been used as a means of classifying samples. These are: a classification based entirely upon the presence/absence and abundance of specific taxa in the assemblage (Alhonen *et al.* 1980; Alhonen and Matiskainen 1980; Matiskainen and Alhonen 1984; De La Fuente and Martinez Macchiavello 1997), and a classification based upon an interpretation of the microfossil assemblage in terms of the palaeoenvironment (Jansma 1984), or the geological period (Troja *et al.* 1996) in which the raw materials of pottery manufacture were deposited.

De La Fuente and Martinez Macchiavello (1997) isolated diatoms from 16 Inka pottery sherds which were collected during a surface survey in the Chascuil region,

Catamarca, Argentina. Their quantitative analysis indicated that all but one of the samples contained siliceous microfossils which, however, were not very abundant and were comprised of many fragments. On the basis of the relative abundance of the diatom taxa which were liberated from the samples, De La Fuente and Martinez Macchiavello (1997) classified them into five groups, characterised by the dominance of particular taxa. For example Group 1, which was composed of eight sherds, was dominated by the species *Fragilaria brevistriata* (12.5 - 59 % of the assemblages).

As presented, this classification appears relatively legitimate. However, by referring to the actual quantitative data upon which they are based, it is possible to see that some of these authors' diatom groups contained but a single sherd (Groups IV and V), with only 20 or so diatoms, many of which were unidentifiable fragments. For example, the sherd in Group IV, which was supposedly characterised by the dominance of *Aulacoseira granulata* and *Melosira sp.*, contained one specimen of the former, two of the latter and only one other identifiable diatom valve (*Nitzschia sp.*).

In addition to being rather tentative, the five groups which De La Fuente and Martinez Macchiavello (1997) proposed on the basis of their diatom analysis have little meaning, as the authors did not interpret the various floras with which they characterised their groups. Although it is clear that certain diatoms dominated the assemblages of some of the groups, e.g. *Fragilaria brevistriata* in Group 1, there are numerous taxa which were present in several groups, and thus clearly some kind of interpretation is needed.

Another restriction of this study might be its reliance upon diatoms for the classification of the sherds. De La Fuente and Martinez Macchiavello (1997) did not

present any information on either the typology, petrography or chemistry of the Inka ceramics from the Chaschuil region. In the absence of any sound interpretation of the diatom classification, additional data is required in order to confirm or contest these groups.

A similar problem is clear in work by Alhonen *et al.* (1980), Alhonen and Matiskainen (1980) and Matiskainen and Alhonen (1984), in which they proposed a diatomological classification of Sub-Neolithic pottery from the Finnish site of Nikasuo. At this 'Comb Ceramic period' dwelling site (c. 5500-4500 BP), some 17,000 sherds were excavated. These belong to three different stylistic groups: the Typical Comb ceramics (subdivided into three subgroups on the basis of their predominant motif), the Late Comb ceramics, and the Pit and Comb ceramics, of which only a single sherd was encountered.

Alhonen *et al.* (1980) analysed chemically a total of ten sherds from these three stylistic groups and subgroups, as well as studying the nature of their diatom floras. Only very small numbers of diatom specimens were liberated from the ten sherds (2-11). Nevertheless, Matiskainen and Alhonen (1984, 153) claimed that there was a "distinct correspondence between the species and the stylistic classification". However, in Group B of the Typical Comb ceramics, the species *Cocconeis scutelum*, which also characterised Group C, was only represented by one specimen out of a total assemblage of four or five diatoms. In addition, the two samples which belong to this group have but one other feature in common, which is that they both contained a single specimen of the genus *Pinnularia*. The Pit and Comb sherd also contained two diatoms specimens, of the species *Cocconeis scutelum*, however, the authors did not

acknowledge the obvious correlation between this sample and those of their Typical Comb ceramic Groups B and C.

In his review of the application of diatoms to archaeology, Battarbee (1988, 637) presented the work of Alhonen *et al.* (1980) as a rare example in which "the considerable potential of diatom analysis as an aid in typological studies of pottery" has been exploited. Alhonen *et al.*'s (1980) classification of the sub-Neolithic pottery from the Finnish site of Nikasuo, is certainly one of only a few studies in which diatom analysis has been compared to stylistic groupings of archaeological pottery. However, there are several problems with this work and the interpretations which are drawn from it. The main criticisms concern the small number of samples which were analysed and the basic non-interpretative method of classification which Alhonen *et al.* (1980) based upon low abundance diatom floras (as in De La Fuente and Martinez Macchiavello 1997 above).

It appears as if Alhonen *et al.* were convinced that the diatom floras and stylistic classification of the Nikasuo pottery samples should match, and then set out to prove this by utilising a few somewhat tentative links, e.g. the concurrence of two related specimens in Group B. Perhaps a better approach, would have been to compare the diatom floras of each of the pottery samples in an objective manner and re-evaluate the stylistic classification in the light of this.

To their credit, they carried out chemical analysis of the pottery samples from Nikasuo by atomic atomic absorption spectrophotometry (AAS). However, the results of this analysis revealed that "the raw materials of the vessels classified according to features of ornament and form differ from each other except in Group C" (Alhonen *et*

al. 1980, 201), which they interpreted in terms of variation in coarse sand temper added to the pottery. It might be suggested that what was needed in this study was thin section petrography, which may have been used to confirm the presence and composition of any temper, as well as to characterise the base clay to which this was added. In this way, it may have been possible to compare the AAS results of, for example, Group C, and assess the validity of the crude correlations between the stylistic classification and the diatom floras.

2.3.1.3 Interpretation of microfossil assemblages in terms of geological age or palaeoenvironment

A more sophisticated approach to the micropalaeontological classification of archaeological pottery samples might be the interpretation of their microfossil assemblages in terms of either geological age or palaeoenvironment. In this way, the raw materials of ceramic manufacture can be compared more reliably to the locally-available sources of clay or temper, in order to infer provenance (Section 2.3.2). Two previous analyses which exemplify this approach are Jansma (1984) and Troja *et al.* (1996).

Jansma (1984) studied 10 Late Neolithic sherds with different types of decoration, from the site of Aartswould in the Netherlands. On the basis of his qualitative diatom analysis (Section 8.4.2), he sub-divided the ceramics into two groups. The larger group contained numerous sherds with assemblages dominated by marine diatoms, such as *Cymatosira belgica*, *Melosira sulcata* and *Raphoneis surirella* with comparable numbers of broken and unbroken specimens per species. The smaller

group on the other hand, contained only one sherd, which had a diatom flora dominated by freshwater species of the genera *Eunotia* and *Tabellaria*. This sherd also possessed a 'zigzag' style of decoration, which was distinctively different from that of the other nine samples.

This study represents a good, straightforward example of how the palaeoenvironmental interpretation of microfossil assemblages can be used to group archaeological ceramics by the nature of their raw materials. On the basis of the contrasting environmental tolerances of the dominant diatom taxa in the 'zigzag' sherd and the other nine Late Neolithic pottery samples from Aartswould, Jansma (1984) was able to infer a different provenance for the two groups (Section 2.3.2.1).

A biostratigraphic approach to classifying ceramics in order to determine provenance was attempted by Troja *et al.* (1996) in their integrated analysis of Neolithic to Bronze Age pottery samples from Milena, Sicily. They analysed foraminifera and calcareous nannofossils in thin section, and defined two microfossiliferous groups on the basis of their preservation, abundance and composition. Group 1 contained 13 samples with relatively rich associations of planktonic and benthic foraminifera (identified to family and genus level) and calcareous nannofossils (identified to genus and species level). However, Group 2, which was composed of an equal number of sherds, was characterised by a low diversity of poorly-preserved planktonic foraminifera and very rare calcareous nannofossils (both identified to genus level), as well as the unidentifiable remains of diatoms.

Troja *et al.* did not present assemblage descriptions of the individual samples in their analysis. Instead, they summarised the nature of the microfossil assemblages in each

group and therefore, it is not possible to assess the reliability of their classification. Nevertheless, on the basis of their combined assemblage descriptions, the samples of Group 1 appear to be contemporaneous. Those of Group 2, however "are characterised by different associations of microfauna" (Troja *et al.* 1996, 126), and the only feature which the microfossil assemblages of these samples have in common is their low abundance, low diversity and poor preservation.

The authors compared their two microfossil groups with other compositional data, but did not interpret this in any way. As a result, they failed to recognise a problem with their proposed two-fold micropalaeontological classification. The x-ray diffraction (XRF), instrumental neutron activation analysis (INAA) and inductively coupled plasma spectrometry (ICP) analysis of the pottery sherds from Milena indicated that the samples of Group 1 could not be distinguished in terms of their chemical composition from those belonging to Group 2. This would seem to suggest that the samples belonging to the two groups may have been constructed from similar raw materials; a possibility which was also reflected in the petrographic analysis of this study. Nevertheless, Troja *et al.* (1996) did not integrate their micropalaeontological results with this chemical and petrographic data, but retained the two groups, which were assigned to different geological periods and then correlated to field samples of local clay sources (Section 2.3.2.5).

An alternative interpretation of Troja *et al.*'s (1996) material, from the sparse data which they presented, is that the pottery samples in Group 2 are poorly-preserved examples of the microfossiliferous fabric which characterises the sherds of Group 1. This interpretation is supported by several points, which are outlined below.

1. All foraminifera and calcareous nannofossil taxa identified in the assemblages of Group 2 are also present in Group 1.

2. There is no reason why the microfossil assemblages of Group 2 cannot be attributed to the same geological stage as those in Group 1 (Section 2.3.2.5).

3. The microfossil assemblages in Group 1 are well-preserved, whereas those in Group 2 are poorly-preserved.

4. These two groups were not confirmed by the chemical and petrographic analyses.

5. Troja *et al.* inferred that some of the pottery had been affected by groundwater (1996, 120).

Despite the numerous techniques which were employed in the study of Troja *et al.* (1996) and the multidisciplinary nature of the team which analysed the archaeological pottery samples from Milena, the numerous lines of evidence were not considered an integrated whole, which may have brought about a different conclusion.

<u>2.3.2 Provenance</u>

The determination of provenance has long been the primary focus of ceramic compositional analysis (Bishop *et al.* 1982), including the study of microfossils in archaeological pottery (Battarbee 1988). The nature of microfossil assemblages in archaeological pottery samples have been utilised in a variety of ways in order to indicate ceramic provenance on various geographical scales, as outlined in Sections 2.3.2.1 to 2.3.2.5 below.

2.3.2.1 Local versus non-local

Perhaps the simplest interpretation of ceramic provenance is that of local versus non-local; this is the distinction between archaeological pottery which was manufactured close to its site of excavation, using local raw materials, and that which was produced elsewhere and imported. This type of provenance interpretation has been achieved by several analyses of diatoms from archaeological ceramics, where the dissimilarities between the floras of pottery sherds and local raw materials have led to confident conclusions (Battarbee 1988). As with the micropalaeontological classification of archaeological ceramics (Section 2.3.1), local and non-local provenance interpretations have been established via the presence or absence of microfossils, the composition of their assemblages, or through the palaeoenvironmental or biostratigraphic interpretation of the microflora and fauna.

<u>Presence/absence</u>

In Section 2.3.1.1 Jansma's (1977) diatom analysis of PFB and VL sherds from the Dutch Neolithic site of Zanderwerven was reviewed. In this work he utilised the presence/absence of diatoms to indicate dissimilarities between the raw materials of these stratigraphically and stylistically separate ceramics. As a result of this distinction between the diatomaceous VL sherds and the barren PFB samples, Jansma (1977) postulated a different provenance for the two groups of pottery. The site of Zanderwerven is "situated on Holocene deposits far from Pleistocene deposits" (Jansma 1977, 82), a fact which was used to infer that the local clays contained diatoms. Based upon this assumption, Jansma (1977) concluded that the PFB pottery

sherds from Zanderwerven, or the raw material from which they were produced, was imported into the site, and the VL pottery was produced locally.

This local versus non-local provenance interpretation of the VL and PFB was rather ambitious, as it lacked the analysis of representative field samples, or a consideration of the firing technology of these ceramics. Local non-diatomaceous clays sources may have been present in the region surrounding the archaeological site of Zanderwerven, or alternatively the barren PFB pottery samples may have originally contained diatom specimens, which were subsequently removed during the process of firing. In fact, the latter scenario was considered by Jansma as a possible explanation for the absence of diatoms in the contemporaneous Neolithic pottery which he analysed from the archaeological sites of Drente, Utrecht and Norrd-Brabant.

Furthermore, Jansma's contention that the diatomaceous VL sherds were local to Zanderwerven was equally unsubstantiated, as Holocene sediments containing diatoms cover much of the Netherlands and therefore, the pottery could have been produced at any other Neolithic site which is situated close to clays of this type. One possible means of confirming his interpretation, would have been to analyse the composition of the diatom floras in terms of palaeoenvironment, a method which Jansma later utilised in his subsequent study of the PFB sherds from the site of Aaartswould (1984), which is outlined below.

Palaeoenvironmental interpretation

A more sophisticated approach to the determination of local and non-local ceramics has been attempted by Jansma (1984) in his quantitative analysis of ten PFB sherds

from the Neolithic site of Aartswould, Netherlands, which is introduced in Section 2.3.1.3. Aartswould is situated near to the coast, a fact which was used to infer that "the clay in the neighbourhood would have been deposited in a predominantly marine environment" (Jansma 1984, 533), without the analysis of any representative samples of locally available raw materials, which he previously viewed as being essential (Jansma 1977).

By analysing the composition of the diatom floras within the ten PFB sherds, Jansma (1984) was able to separate the 'zigzag' decorated sherd, with its dominantly non-marine diatoms, from the other nine samples which contained marine diatom assemblages. On the basis of this valid distinction between the zigzag sample and the other pottery in his analysis, Jansma postulated that the vessel from which this sherd originated, was imported and the remaining sherds "probably came from pots made with locally available clays" (1984, 533).

Although the agreement between the stylistic and environmental differences of the zigzag sample and the other PFB sherds was significant, there are several problems with the provenance interpretation which Jansma (1984) then made. Firstly, as diatomaceous marine clays accumulate in great quantities in macrotidal coastal areas such as the Netherlands, the nine PFB pottery sherds which contained marine diatoms could have been manufactured at any other Neolithic coastal site which contains contemporaneous pottery. Secondly, his opinion that non-marine sediments were not available at Aarstwould, which is implied by his contention that the zigzag sherd was non-local, was not substantiated by clay sampling or reference to pre-existing geological knowledge. It is possible that non-marine diatomaceous clays existed in the

area surrounding the site of Aartswould in the form of fluvial or lacustrine deposits, or older sediments. Lastly, Jansma (1984, 533) stated that the zigzag sherd is part of "a separate group within the PFB culture", however, he did not indicate whether this distinction was stratigraphic or geographic. If the latter is the case, then a consideration of the other occurrences of this type of pottery could have been used to support its suspected non-local origin.

2.3.2.2 Coastal areas versus inland

Jansma's (1984) conclusion that the zigzag sherd which he analysed from the coastal site of Aarstwould was non-local because it contained a diatom assemblage which was dominated by freshwater taxa (Section 2.3.2.1), led him to postulate that it had been imported from somewhere inland. This broad interpretation of coastal versus inland provenance, which is based upon the salinity tolerances of the dominant diatom taxa in pottery samples and the proximity of their site of excavation relative to the sea, is an extension of the local/non-local approach, and has been used in other diatom analyses of archaeological pottery from northern Europe (e.g. Stilborg 1997).

Jansma (1984) applied the same method in his study of some Iron Age pottery from the inland site of Hooidonske Akkers, near Son en Breugel, the Netherlands. In this example, nine sherds were analysed qualitatively and could be divided into two groups on the basis of the salinity tolerances of their diatom taxa. In Group A, to which five of the nine sherds belonged, more than 50% of the total number of diatom species present (13-36) were indicative of a freshwater environment (e.g. *Fragilaria construens* and *Gomphonema angustatum*). The flora of the four sherds belonging to

Group B, on the other hand, were dominated by a comparable percentage of marine diatom taxa such as *Melosira sulcata, Melosira westii, Podosira stelliger* and *Rhaphoneis amphiceros*. On the basis of this division, Jansma concluded that there was a strong possibility that the pottery of Group A was produced locally, and that the sherds belonging to Group B "were imported from the coastal area, a distance of 150 km" (1984, 536).

As with Jansma's non-local, inland provenance interpretation of the zigzag sherd from Aartswould, this conclusion is too simplified, as the absence of marine sediments at Hooidonske Akkers was not established by the analysis of representative clay samples. Jansma's contention that marine clays "are not available in the surroundings" (1984, 536), appears to be an assumption. Considering the low-lying nature of the Netherlands, as well as the periodic flooding of the land by the sea (Jansma 1977), the occurrence of Pleistocene or Holocene marine clays cannot be discounted without detailed fieldwork or the consideration of published geological reports.

Other criticisms of Jansma's (1984) analysis of the Iron Age pottery from Hooidonske Akkers, include its reliance upon the qualitative diatom technique, which fails to distinguish between allochthonous and autochthonous specimens, and its disregard for other aspects of the pottery sherds, such as typology, petrography and chemistry, which may have been used to confirm or disprove the micropalaeontological classification.

By considering local and regional geology in the analysis of microfosssil assemblages from ceramics, rather than the proximity of the archaeological site relative to the coast (e.g. Jansma 1984; Stilborg 1997) and analysing representative samples of locally

available sources of raw material for direct comparison, much more specific provenance interpretations can be made, as outlined by in Sections 2.3.2.3 to 2.3.2.5 below.

2.3.2.3 Biostratigraphic macroprovenance

By comparing the microfossil assemblages of archaeological pottery in terms of the geological age or palaeoenvironment of the raw materials of ceramic manufacture, with the local and regional geology in the area of excavation, more reliable provenance interpretations can be sought. These can be classified in terms of their geographical precision, into macro, meso, and micro-scale provenance interpretations. Macro-scale provenance interpretations are those in which it is possible to assign a particular sample or group of pottery to a large area (e.g. east versus west Mediterranean). A mesoprovenance interpretation would indicate the region within this broad area from it is likely to have originated, e.g. north versus south, or one valley versus another, and if a specific geological formation or sedimentary deposit with an isolated geographical occurrence, can be identified as the likely source of the raw material from which the ceramics were manufactured, then this is a microprovenance interpretation. A good example in which the biostratigraphic interpretation of microfossils in archaeological ceramics has been used to determine macroprovenance, is the work of Riley *et al.* (n.d.), which is outlined below.

Riley *et al.* (n.d.) restudied the foraminifera in the ten Late Helladic coarse-ware stirrup-jar thin sections from the House of the Wine Merchant described by Riley (1981), and were able to disprove the previous, tentative provenance interpretations

(Section 2.2). Riley's (1981) conclusion was that the stirrup-jars originated from an area with low-grade metamorphic rocks and microfossiliferous sediments, such as Crete. This proposition is in agreement with the chemical composition of some of the samples, as analysed by optical emmision spectrometry (OES), and the evidence of subsequent studies on coarse ware pottery (Catling *et al.* 1980; Day 1995a).

Riley *et al.* (n.d.) were able to identify some of the foraminifera specimens in these stirrup jar thin sections to genus and species level, and made a biostratigraphic interpretation of the microfossiliferous sediment used in the manufacture of the pottery. The presence of the stratigraphically significant planktonic foraminifer *Globorotalia inflata* (Section 6.6.2.2) in one of the samples clearly indicated that the raw material of this sherd was deposited during the latest Pliocene. The Neogene sedimentary succession of Crete has been well studied in terms of its microfossil assemblages and stratigraphy (Section 4.2), and nowhere on the island have Late Pliocene sediments, containing *G. inflata* been found. The stratigraphic extent of Neogene strata is also well known in many other areas of the Aegean and the eastern Mediterranean, and the distribution of latest Pliocene marine sediments, prompted Riley *et al.* (n.d.) to suggest that the stirrup jars could have originated from the Peleponessus, the Ionian Islands or Rhodes. This broad macroprovenance interpretation was of course based upon the assumption that all of the stirrup-jars samples were manufactured from the same raw materials, as the species *G. inflata* was only identified in one of the sherds.

The presence of the same stratigraphic marker species, was also used in a similar manner by Riley *et al.* (n.d.) to indicate a local source for examples of a

microfossiliferous Late Bronze Age wheel-made decorated ware from Trebisacce, southern Italy. This pottery was originally suspected to be Cretan in origin, on the grounds of its high technological quality, fine decoration and clear Aegean motifs. However, the discovery of *G. inflata* in the dominantly planktonic assemblage of foraminifera, disproved this hypothesis. The occurrence of latest Pliocene sediments with a high plankton/benthos ratio near to Tresbiacce on the other hand, indicated to Riley *et al.* (n.d.) that the pottery was local to the region. This contention is in agreement with the subsequent compositional analysis performed by Jones and Vagnetti (1989), who suggested that itinerant craftsmen were operating in southern Italy during the Late Bronze Age.

Another study in which the foraminifera of Bronze Age Mediterranean ceramics have been used to indicate large scale provenance, is that of MacGillivray *et al.* (1988). Here, micropalaeontology was applied alongside petrography, AAS and INAA analysis to the problematic Dark Faced Incised Ware pyxides (DFIW), which were excavated at Knossos, Crete. The typological homogeneity of this pottery group was confirmed by all of the techniques listed above, and a Miocene or Pliocene age for the raw material was provided by the generic identification of foraminifera specimens in thin section.

On the basis of this broad biostratigraphic interpretation, MacGillivray *et al.* (1988) suggested that the DFIW may have originated from Aegina, Melos, Thera or Crete. However, by the mineralogical comparison of the sherds with contemporary pottery as well as the depositional environment of the Neogene sediments in these areas, they concluded that the pyxides had a source in "central or eastern Crete where

Miocene/Pliocene deposits occur" (MacGillivray *et al.* 1988, 93). This study is a good example of how micropalaeontology can be used in association with other techniques of characterisation in order to macroprovenance ceramics, and is re-investigated and compared to the micropalaeontological analysis of other Bronze Age ceramics from Knossos, in the present report (Section 11.3).

2.3.2.4 Environmental and biostratigraphic mesoprovenance

During the deglaciation of the Weichselian ice-sheet which covered much of north-west Europe during the last ice age, extensive marine and non-marine sediments were deposited in the 'Baltic Ice Lake' and the various stages of the Baltic Sea, which followed. These deposits occur extensively in Finland and can be divided into four units (Yoldia; Ancylus; Mastogloia; Littorina), on the basis of their diatom floras, which reflect the different environmental conditions in the successive stages. The diatom associations of these sediments have been documented by Alhonen (1971; 1979), and a palaeoenvironmental diatom stratigraphic scheme has been established for this time period, based upon the occurrence of specific taxa.

The Holocene clays of the Yoldia, Ancylus, Mastogloia and Littorina stages are one of the best available natural sources of raw material for the manufacture of ceramics in Finland, as evidenced by their use in the modern ceramics industry, and Neolithic pottery production (Alhonen and Väkeväinen 1981; Alhonen and Matiskainen 1980; Matiskainen and Alhonen 1984). These authors analysed the diatom floras of ten 'Early Comb Ceramic' type sherds from six archaeological sites in the Åland Islands,

an archipelago off the coast of Finland, and related these to the four-fold Holocene diatom stratigraphy in order to infer the macroprovenance of this pottery. Of the ten samples, eight contained diatoms. These were not abundant enough to be analysed in detail using a quantitative approach, but nevertheless indicated that "a freshwater clay was used as their raw material" (Matiskainen and Alhonen 1984, 150).

By comparing the floras in the 10 Early Comb Ceramics, to the diatom assemblages which characterise the four stages of the Baltic Sea in Finland, Alhonen *et al.* (1980) noted that the samples could be correlated with the sediments of the Ancylus Lake period. Despite geological observations in the field (by Cleve-Euler 1935 and Glückert 1978), no Ancylus stage sediments were found to occur on the Åland Islands. Matiskainen and Alhonen considered this to indicate that "ready-made pots or their raw materials were transported to the area from the Finnish mainland", where deposits of these clays occur (1984, 151). At the time of Early Comb ceramic occupation (6400-5800 BP), Åland consisted of but a few small islands and was inhabited by seal hunting populations, who, according to Alhonen and Matiskainen (1980, 47), "carried raw clay with them, so that part of the vessels may have been manufactured on the islands".

Despite the qualitative nature of Alhonen and Väkeväinen's (1980) diatom analysis, their contention that the Early Comb ceramics from Åland had a non-local source in an area where non-marine Ancylus clays occur, is "strengthened by the presence of similar ceramics with a similar diatom flora at Kokemaki" (Battarbee 1988, 639), a contemporary site on the mainland. This evidence, however conflicts with Alhonen and Matisakinen's (1980) opinion, that the seal hunters may have transported clay

with them. If the Neolithic inhabitants of Åland did indeed transport unfired Ancylus clay from the Finnish mainland, then this would conflict with the opinion of Alhonen *et al.* (1980, 203), that "raw material for clay vessels most readily available in the environment of the Stone Age dwelling sites was taken from the clay in the surface sediments of the terrain".

2.3.2.5 Environmental and biostratigraphic microprovenance

The utilisation of locally available clay sources by ancient potters is suggested by Jansma's (1977) analysis of pottery sherds from the stratigraphically/chronologically differentiated Bell Beaker (BB) and Vlardingen (VL) 'cultures' at the Neolithic site of Vlardingen in the Netherlands. Here, he compared the diatoms in both groups of pottery to the stratigraphic succession of clay which was deposited at this site. The diatom assemblages of the VL sherds compared well to the brackish and freshwater diatom flora of a clay which filled a creek bed at Vlardingen, whereas the BB pottery which was dominated by allochthonous marine and brackish diatom taxa, could be related to a clay which was deposited just prior to the BB habitation of the site, during the flooding of the land by the sea. The "striking and completely convincing" (Battarbee 1988, 638) correlation between the sherds and the Holocene sedimentary succession at Vlardingen, agrees with the assumption of Alhonen *et al.* (1980) which is outlined above, and represents "a classic example of what may be achieved by diatom analysis under favourable circumstances" (Jansma 1977, 82).

A less convincing example in which micropalaeontological analysis has been used to microprovenance the raw materials utilised in pottery manufacture to a specific local clay source, is the work of Troja *et al.* (1996). These authors compared the calcareous microfossils in thin sections of their two groups of Neolithic to Bronze Age pottery from Milena, Sicily (Section 2.3.1.3) to the assemblages contained within "some samples of clay outcropping in the Milena area" (Troja *et al.* 1996, 124). The sediments in question were deposited in the post-orogenic basin of Caltanisseta and include the Lower-Middle Miocene Terravecchia formation and the Lower Pliocene Trubi formation, which are separated by Messinian gypsum, clays and limestones. Troja *et al.* did not indicate the geographic or stratigraphic location of these clay samples which they analysed, however, it appears that samples CL1 and CL2 came from the Terravecchia formation and CL3 from the Messinian clays, as these were dated to the Serravalian, Tortonian and Messinian respectively, on the basis of their planktonic foraminiferal associations.

The planktonic foraminifera and scanty calcareous nannofossil assemblages of Group 1 were dated to the Serravalian stage of the Middle Miocene and it was suggested that they were "analogous to those found in the clays CL1 and CL2" (Troja *et al.* 1996, 126). The pottery of Group 2 on the other hand, contained much poorer microfossil assemblages which were considered to be Messinian in age, and were therefore assumed to be "manufactured from the Messinian clays (CL3) of the Milena area" (1996, 126). On the basis of these correlations, and the similarity between the major element analysis of the pottery and that of Middle and Upper Miocene clays, the authors then proposed that the Neolithic and Bronze Age pottery of Milena was manufactured from local raw materials.

There are several points of debate concerning this analysis. Firstly, the microfossils within the pottery of Group 1 are not analogous with those of clay samples CL1 and CL2. They contain specimens of similar genera, but as the foramininfera were identified to a different taxonomic level in the two types of material, and no calcareous nannofossils were analysed from the clay samples, a comparison cannot be made. Furthermore, the Middle Miocene, Serravalian date which the authors assigned to this pottery based upon its calcareous nannofossil assemblage, is incorrect. The few long-ranging taxa which were identified in the floras of Group 1 are more consistent with the Late Miocene, early Tortonian Stage. Troja *et al.'s* (1996) biostratigraphic assignment of the pottery samples in their second Group 2 however, is even less substantiated, as the assemblages of this group contain only scanty foraminifera, identified to genus level, and one calcareous nannofossil taxon. No other evidence was presented in this work, except for their spurious geological assignment, with which to correlate the pottery of Group 2 with clay sample CL3.

Secondly, as outlined in Section 2.3.1.3, the two micropalaeontological pottery groups were based upon different criteria, and the samples in Group 2 are likely to be poorly preserved examples of Group 1. This view is supported by the "homogeneous petrographic characteristics" of the pottery samples from Milena (Troja *et al.* 1996, 118), the failure of their chemical analysis to distinguish between the two groups of pottery, and the observation that some of the samples were affected by post-burial alteration.

Calcareous nannofossils have also been utilised by Burnett and Young (n.d.) in order to correlate pottery samples with specific geological units. These authors analysed the

calcareous nannofossils in a single sherd of pottery, from a Bronze Age boat which was excavated at Dover, England. Their biostratigraphic interpretation of the diverse calcareous nannofossil assemblage which was contained within this crudely-fired sample indicated that the vessel from which the sherd originated was constructed from Early Cretaceous, Albian Stage clays.

By considering the Cretaceous geology of this part of the south coast of England, the authors identified the Gault Clay Formation as the most likely candidate for the source of this material. The Gault Clay Formation outcrops on the coast at Folkestone, near Dover, as well as further west at Eastbourne. Burnett and Young (n.d.) therefore decided that it was reasonable to infer that the pot was produced locally. However, the Gault Clay also occurs on the other side of the English channel, near Wissant, France. Thus, considering the context in which the sherd was found (i.e. a boat), it is not possible to determine on which side of the channel the raw material of the original vessel was constructed, without further evidence.

Another group of microfossils which are very useful for biostratigraphy and have been used to relate pottery to specific geological units in England, are organic microfossils, or palynomorphs. Hunt (1996) treated ten oxidised and ten reduction-fired Iron Age sherds from North Furzton, near Milton Keynes, England, with hydrofluoric acid (Section 9.3) in order to liberate organic-walled microfossils. Seven of the reduced sherds contained recognisable palynomorphs, however, all of the oxidised samples were barren. The assemblages from the productive sherds were composed of pollen, spores, dinocysts, woody tissue, stem and leaf fragments, and amorphous organic matter. These were not consistent between the various samples, and on the basis of

some stratigraphically important taxa, Hunt (1996) suggested several possible sources for the raw materials which were utilised in the construction of this pottery.

One of the reduction-fired pottery samples from North Furzton contained many long-ranging Mesozoic dinoflagellate taxa including *Gleicheniidites senonicus* which first appeared in the Middle Jurassic. The presence of this taxon, as well as the high abundance of organic matter in this sample indicated to Hunt (1996) that the raw material used in the manufacture of the original vessel was procured from the Callovian, Oxford Clay Formation which outcrops not far from the site of excavation.

Stratigraphically significant dinoflagellate cysts, such as *Parvocysta sp.*, *Nannoceratopsis senex* and *Pareodinia ceratophora*, which occurred in another sample are indicative of a Bajocian age source such as the Fuller's Earth beds, which occur in the Cotswold Hills, some distance from North Furzton. However, this sample also contained abundant Quaternary tree pollen of the genera *Pinus* and *Tillia*, as well as grass pollen and *Spirogyra*, a green algae which inhabits shallow stagnant pools and ditches. This Quaternary component of the sample suggested to Hunt (1996) that Recent alluvium may also have been used in the manufacture of the vessel from which the sherd originated. On the basis of these two conflicting lines of evidence, Hunt (1996) suggested that the clay in this sherd could be a mixture of an alluvial clay and the Jurassic Fullers Earth. This suggestion, which could have been investigated further by using thin section petrography, pertains to the technology of the ceramic manufacturing process, and represents another way in which the scientific investigation

of pottery, including the analysis of microfossils, can be used to retrieve archaeological information (Section 2.3.3).

2.3.3 Technology

As illustrated in the example above (Hunt 1996, Section 2.3.2.5) it is possible to interpret technological aspects of the manufacturing process used in the production of ancient pottery samples, through the analysis of their microfossil assemblages, as well as the relationship between the microfossils and the other components of the fabric in thin section (Riley 1981, Section 2.2). There have been several detailed micropalaeontological analyses which have attempted to infer aspects of ceramic technology, including clay mixing, tempering and firing. These are reviewed in Sections 2.3.3.1 to 2.3.3.3 below.

2.3.3.1 Clay mixing

In addition the work of Hunt (1996), the micropalaeontological analysis of ceramics has been used to interpret the mixing of one or more clays for the production of a single vessel or group of pottery, by Jansma (1977), Gibson (1983) and Matiskainen and Alhonen (1984). In these examples, the concurrence of diatoms with conflicting ecological tolerances (e.g. marine and freshwater) has been interpreted in terms of the admixture of two clays, from different depositional environments.

Matiskainen and Alhonen (1984), in their analysis of Early Comb ceramics from Åland (Section 2.3.2.4), discovered that one of the sherds which they analysed

contained a broad range of diatom species, some of which were not indicative of the Ancylus Lake clays which were suspected to be the source of the raw material used in the other samples. The authors believed this to indicate that "lake mud was mixed with the raw clay either inadvertently or as tempering" (Matiskainen and Alhonen 1984, 151).

A mixed diatom flora, containing taxa which are indicative of different environmental conditions, can also occur in a single source of raw material and the pottery manufactured from it, as a result of the transportation and the reworking of specimens from one deposit into another. Diatom valves that have been re-sedimented in this way can be damaged, and it is therefore possible to distinguish between these allochthonous components of the diatom assemblage and the *in situ* species which are indicative of the original depositional environment, by counting the proportion of broken and complete valves from each taxon (Section 8.4.2).

In Jansma's (1977) quantitative analysis of the diatom flora in a single PFB sherd from the Dutch Neolithic site of Hekelingen, a mixture of marine, brackish and freshwater taxa was recorded. By counting the numbers of broken and unbroken individuals of each ecological group, Jansma discovered comparable percentages of unbroken marine-brackish and brackish-fresh species, which he then interpreted as "a mixture of slightly more marine with slightly more brackish-fresh clays" (1977, 83).

The occurrence of conflicting microfossil assemblages need not be a consequence of re-sedimentation or clay mixing, but can be produced by the addition of temper, as speculated by Gibson (1983a). Evidence for the contamination of diatom assemblages

by the process of tempering was discovered by Jansma (1984; 1990), and is outlined below.

2.3.3.2 Tempering of non-plastic inclusions

In Jansma's (1984) analysis of nine Iron Age sherds from the site of Hooidonske Akkers, near Son en Breugel in the Netherlands, the samples belonging to his Group A, which were dominated by freshwater diatoms, contained small numbers of marine species. These species, which were clearly not deposited *in situ* in the original clay, were interpreted as evidence for the "addition of small amounts of grit, derived from sherds tempered with ground sea shells" (Jansma 1984, 536). In making a two-fold technological inference, Jansma implied that the Iron Age sherds which he analysed from Son en Bruegel were tempered with ground pottery (i.e. grog), and that in the pottery which formed the grog, ground sea shells which contained diatoms, were used as temper.

Although this situation is theoretically possible, Jansma's (1984) reasons for inferring such a scenario were unclear, as with his interpretation of shell temper in one sherd of Bell Beaker pottery from the former island of Schokland, in the Netherlands (Jansma 1990). There did not appear to be any other evidence for the occurrence of the grog or shell temper in either study, and no attention was paid to the proportions of broken and unbroken valves of each ecological group of diatoms in either study. In his latter example, Jansma (1982) inferred the provenance of the shell temper in the BBC (Bell Beaker Culture) pottery which he analysed, without confirming its presence by hand specimen or petrographic analysis. However, the latter two techniques are essential in

the determination of ceramic technology, and must be utilised in combination with micropalaeontological analysis in order to establish the existence and nature of clay mixing or tempering.

2.3.3.3 Firing

One of the most important technological aspects of ceramic manufacture is firing. Firing temperature and atmosphere are the two most widely investigated properties of this process, and can be estimated by the analysis of numerous physical and chemical characteristics of archaeological ceramics. Unfortunately, microfossils in ceramics do not appear to be well-suited to the determination of ancient firing technology as they are commonly degraded and even destroyed upon heating. However, some authors have attempted to utilise various groups of microfossils in this way, the most important of which is the work of Hunt (1996) on Iron Age pottery from Milton Keynes, England (Section 2.3.2.5).

Hunt made the significant discovery that, out of ten oxidised and ten reduction-fired ceramics, only the latter contained any organic matter. He did interpret this outcome, however, its true importance in terms of the differential effect of the two firing atmospheres on organic microfossils in ceramics has been elucidated in the present report (Section 9.4.8). As well as utilising the palynological associations which were isolated from the reduction-fired sherds as a means of provenancing this pottery (Section 2.3.2.5), in this study Hunt also attempted to determine the maximum temperature at which they were originally fired.

Organic microfossils undergo a well-documented colour change upon heating in geological contexts (Section 9.4.2). This process, which usually takes place in sedimentary rocks which are deeply buried, has been used by geologists to approximate the maximum temperature to which a organic-rich rocks have been subjected. In order to do this, it is necessary to compare the colour of the thermally altered palynomorphs with experimental standards, e.g. Staplin's (1969) 'Thermal Maturity Index'. Hunt (1996), compared the colour of the reduction-fired palynomorphs which he isolated from his Iron Age sherds with this scheme and concluded that the ceramics had been fired at a consistent temperature of around 400°C (equivalent to 2+ and 3 on Staplin's scale).

Although Hunt's estimations of firing temperatures from the thermal alteration of organic matter were pioneering, the figure of 400 °C which he suggested, is likely to be an underestimation. This is evidenced by the much slower rate of thermal alteration of palynomorphs in the absence of oxygen, which has been revealed in the present report (Section 9.4.9.2). At 400 °C very little vitrification of the clay minerals in the ceramic will have taken place, and the pottery will therefore have been very poorly fired.

Several authors have estimated or investigated the firing temperature at which diatom frustules are destroyed in ceramics (Jansma 1977; 1981; Håkansson and Hulthén 1986; Gibson 1983b, Section 8.3). The range of firing temperatures which have been ascertained indicate that the process which leads to the destruction of diatoms in ceramics is very complex, and may be related to several factors other than temperature. Despite the attention which this process has received, the nature of

diatom assemblages in archaeological ceramics have been used only rarely to infer the temperature or degree of firing.

Upon discovering that several samples of Neolithic pottery from the Dutch sites of Drente, Utrecht and Noord-Brabant did not contain diatoms, Jansma (1977) suggested that the vessels from which these sherds had originated, were manufactured from the non-diatomaceous local boulder clay, or freshwater clays containing very few diatoms which were then destroyed upon firing. This latter interpretation appears to have been purely hypothetical, as Jansma (1977) did not analyse any representative local sediment samples, and presented no evidence for the use of diatomaceous clays at these sites. In the same study, Jansma (1977, 77) established that "the silica of which diatom thecae are formed cannot endure temperatures above about 1000 °C". Therefore, his suggestion that the Neolithic sherds from Drente, Utrecht and Noord-Brabant may have once contained diatoms, which were removed through firing, implies that this pottery was fired to very high temperatures. This, if correct, conflicts with Jansma's own contention that "in most cases, the prehistoric pottery of our regions was baked at rather lower temperatures" (1977, 77).

Another group of siliceous microfossils which have been used to infer the temperature of firing in ceramics, are sponge spicules. These thin, rod-shaped structures are a common component of archaeological ceramics from some parts of the world (Linné 1957, Bolivia; Keech McIntosh and Macdonald 1989, Mali) and may be destroyed due to the physical strain caused by firing over 800 °C (Brissaud and Houdayer 1986). The latter authors have used this to suggest that the sponge-tempered pottery which they analysed from the river Niger, was fired to a maximum temperature not

exceeding 800°C, and Linné proposed that his archaeological ceramics were "not exposed to a strong heat" (1957, 156) and may therefore have been fired in an open hearth.

In what was the earliest detailed analysis of microfossils in archaeological ceramics, Davis (1951) isolated and identified several shell fragments and a one foraminifer (*Nubeculinella sp.*) from a single Iron Age sherd excavated at the site of Chinnor in the Chilterns Hills, England. He speculated that the vessel from which this sherd originated was "only partly fired, for there are no signs of calcination or decay in the fossil-shell fragments" (1951, 148). This simple interpretation of the degree of firing based upon the preservation of calcareous micro and macrofossil shell fragments which are present in the pottery, was significant in that it is one of the rare attempts in which calcareous microfossils have been used to infer firing technology.

2.3.4 Clay choice

The nature of microfossil assemblages in archaeological ceramics, have been utilised, infrequently, to infer the actual clay choices which potters made in the past. This subject has been approached from three perspectives, which are, the preference of one clay over another, the utilisation of several clay sources by sedentary potters, and changes in clay choice which take place over time (Sections 2.3.4.1 to 2.3.4.3). These types of interpretations represent a progression from the identification of potential clay sources and the technology utilised in the manufacture of archaeological pottery, towards an understanding of the ecology of ceramic production (Matson 1965) and the adaptation of populations to changing resources (Arnold 1985).

2.3.4.1 Preference for one clay source over another

In all of their analyses of Finnish Sub-Neolithic pottery, Alhonen *et al.* (1980), Alhonen and Matiskainen (1980) and Matiskainen and Alhonen (1984) discovered, through the analysis of diatom floras, that clays of the Ancylus, and to a lesser extent, the Yolida Stage of the Baltic Sea, were used as a raw material. In comparison, the marine diatom floras of the Littorina Sea stage were not present in any of the sherds which were analysed. Alhonen *et al.* speculated that these clays were not utilised by Finnish Neolithic potters because of either the "salinity or humic consistency of the clay" (1980, 204), which apparently made it unsuitable for the manufacture of ceramics.

In the later versions of their Finnish pottery analysis Alhonen and Matiskainen (1980) and Matiskainen and Alhonen (1984) presented the data of Okko (1957) and Romu (1977; 1978) on various parameters of the Littorina, Ancylus and Yoldia Stage clays. This confirmed the high salt and humus content of the Littorina clays, and led Alhonen and Matiskainen (1980) to conclude that the sediments from this marine stage of the Baltic Sea were avoided in antiquity because of these characteristics.

In support of this interpretation, Alhonen and Matiskainen (1980) and Matiskainen and Alhonen (1984) noted that the Littorina stage clays from Finland were not used in the modern clay and brick industry of this region. They then however, made the ambitious proposition that "the motives of selecting the source of material were the same in prehistoric times as they are in modern ceramic industry" (Alhonen and Matiskainen 1980, 49).

Although, the correlation between the selective use of the sediments from the various stages of the Baltic Sea by Sub-Neolithic potters, and the different concentrations of salt and organic matter in these clays is significant, and ethnographic evidence has indicated that highly saline clay sources are avoided by modern potters (Matson 1965, 210), Alhonen and Matiskainen's (1980) interpretation that the motives involved in this preference for one clay over another were purely compositional, was unwise. Other evaluations of the clay choices made by ancient potters (e.g. Whitbread 1995), as well as ethnographic studies of ceramic manufacture (e.g. Day 1989), have indicated that the most suitable types of raw materials are not always used for the production of pottery.

The clay choices made by ancient potters may also have been strongly influenced by tradition, the ownership of specific deposits of raw materials and even taboo (Stilborg 1997). With this in mind, it is unlikely that the motives for selecting clays for the manufacture of Sub-Neolithic pottery were the same as those for modern ceramic production in Finland, as suggested by Alhonen and Matikainen (1980). The fact that Littorina stage clays were not utilised in either processes is not likely to be a coincidence, but a less ambitious interpretation is preferable, such as Matiskainen and Alhonen's view that "the raw clays used in prehistoric pottery seem to match the quality requirements of modern clay and brick technology quite well" (1984, 156).

Jansma (1981) also interpreted aspects of clay choice, in his diatom analysis of pottery sherds from a kiln dated to the 12th century AD in the town of Haarlem, near Amsterdam, the Netherlands. He was able to correlate the raw material of the samples with one of "two sites with easily extractable clay open for exploitation" (Jansma

1981, 159), on the basis of the ecological tolerances of their dominant diatom taxa. Jansma's interpretation indicated that the potters working at this kiln site procured their clays from the site of Velserbroek Polder (5 miles from Haarlem), rather than from the closer source at Bakenes. By way of an explanation, he noted that the clay from Bakenes was rich in organic matter and was therefore "more suitable for the products of the kiln" (1981, 159).

Although the diatom composition of the sherds which were analysed by Jansma (1981) was more consistent with that of the clay samples which he collected from Velserbroek Polder, than the assemblage which was recorded in the sediments of Bakenes, it may not be necessarily correct to infer that the raw material utilised in the manufacture of these ceramics was procured from this site, as other sources of clay may have been available in this area in the past.

Diachronic variation is a very important factor which must be considered in the compositional analysis of ceramics (Bishop *et al.* 1982). This is particularly true when considering the clay sources which were available for the manufacture of pottery, as the existence and nature of suitable raw materials can change considerably over time (Jones 1986). The utilisation of different clay sources over time has been addressed in several analyses of microfossils from archaeological pottery, and is discussed in Section 2.3.4.3 below.

2.3.4.2 The utilisation of several clay sources by sedentary potters

The utilisation of different, microfossilifeous clay sources by sedentary potters, has been suggested by Stilborg (1997) in his interdiscipliniary study of Iron Age pottery

from Denmark. Using the micropalaeontological analysis of Hannelore Håkansson, Stilborg (1997) defined two microfossiliferous groups of pottery from the Gudme-Lundeborg region. These were the 'D-ware', which contained diatom frustules, and the 'F-ware' which was characterised by the presence of foraminifera. Both types of microfossilifeous fabric groups occurred at various Iron Age sites in this region, however Stilborg (1997) considered the 'F-ware' pottery to have been produced at the coastal site of Lundeborg, due to the occurrence here of clays with foraminifera. He also linked the 'D-ware' to the site of Brudager cemetery, where the oldest examples of this fabric group were found.

The discovery at the nearby Brudager settlement of a single sherd which was composed of a mixture of "silty fossil bearing clay and a silty clay containing substantial amounts of diatoms" (Stilborg 1997, 224), led this author to infer that the same potters had produced both the D-ware and the F-ware. These potters were suspected to be from the inland site of Brudager, and appeared to have produced both D-ware and F-ware vessels "both at home and at Lundeborg trading site" (Stilborg 1997, 248). The utilisation of different clay sources by sedentary potters for the production of the same type of ceramics has also been documented by other workers (e.g. Matson 1965).

2.3.4.3 Change in the choice of raw materials over time

In Jansma's (1977; 1981) diatom analysis of Neolithic BB and VL sherds from the site of Vlardingen, the Netherlands, the floras within these pottery samples could be related to two contemporaneous clays, deposited near to the site during each phase of

habitation or 'culture' (Section 2.3.2.5). This differential utilisation of local clay sources by succeeding 'cultures', illustrates very well the importance of time in assessing the raw materials which were available to the potter. Although different clay sources were procured at different times for the production of ceramics at Vlardingen, it appears that that "raw material for clay vessels most readily available in the environment" (Alhonen *et al.* 1980, 203), of this site were used. A different situation, in which the potters of succeeding populations or 'cultures' utilised the same clay sources, was highlighted by Jansma's (1990) diatom analysis of pottery from the former island of Schokland. Nevertheless, as at Vlardingen, the occupants' motives for selecting raw material may have been similar at different periods during its archaeological history.

The utilisation of different sources of clay through time at the same archaeological site, has also been interpreted by Matiskainen and Alhonen (1984). These authors claimed to have used diatom analysis to confirm the stylistic groupings of some stratigraphically differentiated pottery sherds from Kymi, Finland (Section 2.3.1.2). From this they inferred that "different sources of clay were used at different times throughout the period of occupation of the site, and that in accordance with stylistic tradition, a certain sediment was chosen as the source of the raw clay" (Matiskainen and Alhonen 1984, 153).

Alhonen *et al.*'s (1980) original comparison between the diatom assemblages of the ten sherds which were analysed, was problematic (see Section 2.3.1.2 for a review), and the subsequent inference regarding the clay choice at Kymi through time, which they then based upon it is even less substantiated. Matiskainen and Alhonen (1984)

did not analyse any local clay samples at Kymi, or relate the different assemblages which they used to confirm the stylistic groupings to the diatom stratigraphy of the Baltic Sea (Section 2.3.2.4), which they discussed at length. In this case, none of the 'different sources of clay' were actually identified, and the pottery may not have been local. In addition, the AAS analysis of the same ten pottery sherds, failed to confirm the stylistic/diatom classification and was not interpreted independently. Considering the lack of evidence for the utilisation or different raw materials during different periods at Kymi in the two versions of this study, as well as the absence of potential local sources, Matiskainen and Alhonen's (1984) statement on the relationship between clay choice and stylistic tradition is ambitious.

A more substantiated example in which microfossils have been used to indicate changes in clay choice over time, is the work of Riley (1983) and Riley *et al.* (n.d.) from Knossos, Crete. By analysing thin sections of Knossian fine-ware sherds from different stages of the Late Minoan period, these authors were able to identify a change in the composition of the pottery and supposedly the raw material from which they were constructed, occurring between LM IIIA and LM IIIB. Riley noted that the "key change is the presence of sponge spicules, which occur regularly and only in this period", which he interpreted as either a change in the source of the raw material used for the manufacture of Knossian fine-wares "within the general region or a new horizon within the same clay bed" (1983, 285).

This evidence appeared to indicate that the pottery of LM IIIB was produced with different raw materials than that in LM IIIA, though Riley's (1983) conclusion as to the context of this change was vague. However, by re-assessing the micro and

macrofossils in these thin sections, Riley *et al.* (n.d.) were able to qualify the change between LM IIIA and LM IIIB, in terms of the geological date of the raw materials. Using the presence of the stratigraphically significant foraminifer *Globorotalia margaritae* in some of the LM IIIA fine-ware thin sections from Knossos, they assigned the raw material of these samples to the Early Pliocene *Globorotalia margaritae* Zone (Section 6.6.2.2), the sediments of which occur extensively in the Knossos region. In addition, Riley *et al.* (n.d.) were able to relate the raw materials of some samples to specific, well-studied outcrops of Early Pliocene sediments on the basis of certain associations of benthic foraminifera, such as *Nodosaria spp., Bolivina spathulata* and *Uvigerina cylindrica.*

The LM IIIB thin sections, however, appeared to contain different associations of foraminifera, with, for example, a greater number of benthic foraminifera and radiolaria, in addition to the high abundance of sponge spicules which was noted by Riley (1983). Riley *et al.* (n.d.) considered this to indicate that the source of the raw materials in these samples came from middle Pliocene sediments. Several occurrences of laminated, sediments of this age occur in the Knossos region, which clearly indicated to Riley *et al.* (n.d.), that a stratigraphic shift in the site procurement of the raw materials used for the production of fine wares at Knossos took place between LM IIIA and LM IIIB. The authors did not specify whether they considered this shift to represent a change in the geographical site of procurement, or in terms of the level from which raw materials were obtained within the same outcrop. Both situations may have been possible, as the authors indicated that Lower to middle Pliocene sediments occur in the Knossos region at various localities, as well as within a single outcrop (e.g. at Finikia).

2.4 Discussion

As illustrated in Section 2.3 above, there have been several detailed, published and unpublished, studies on various groups of microfossils in Neolithic to Medieval pottery from different areas of the world since the 1960's, by micropalaeontologists, often in collaboration with archaeological scientists or archaeologists. These analyses have used, with varying success, the presence/absence of microfossils, the overall composition of the microfossil assemblages, as determined by the generic or specific identification of specimens in thin sections or digested residues of archaeological pottery sherds, and the geological age or palaeoenvironmental interpretation of these assemblages, to describe, classify and provenance ceramics and investigate aspects of ceramic technology and clay choice. Many of these topics have been addressed by other, more conventional techniques of ceramic analysis, however several of the examples which have been outlined in the above discussion demonstrate the way in which microfossils can be used to further such interpretations.

The main criticisms of these previous micropalaeontological analyses of archaeological ceramics concern their lack of consideration of the other characteristics of the pottery being analysed, such as archaeological context, typology, petrography and chemistry, a poor understanding of the nature of ceramics and how they differ from sediment samples, a lack of raw material prospection or the consideration of published geological reports in provenance interpretations, and a highly generalised view of the behaviour of ancient potters.

3 Processes affecting microfossil assemblages in archaeological ceramics

3.1 Introduction

In some of the analyses outlined in the previous chapter, it was suggested that the original microfossil assemblages of the raw materials which were utilised for the manufacture of ceramics, may have been altered during the processes of clay preparation (Jansma 1977; 1982; 1990; Gibson 1983b; Matiskainen and Alhonen 1984; Battarbee 1988; Stilborg 1997) and firing (Jansma 1977; Gibson 1983b; Hunt 1996; Brissaud and Houdayer 1986). In addition, the microfossil assemblages present in archaeological ceramics may have been further affected after use, in the post-depositional environment (Troja *et al.* 1996), or as a result of sample preparation for scientific analysis (Gibson 1983b; Håkansson and Hulthén 1986; Battarbee 1988). These examples represent rare attempts at understanding the nature of microfossil assemblages in archaeological ceramics, in terms of a few of the potential processes and sources of bias which can have a detrimental affect on their abundance, state of preservation, and the level of information which can be sought through their analysis.

There is a great deal of uncertainty surrounding the exact nature of the original raw materials utilised in the manufacture of ceramics (Riley 1984), the various stages involved in its transformation into a finished artefact, the conditions to which archaeological ceramics were subjected during use, as well as those acting upon it in the burial environment. Nevertheless, through the petrographic study of ceramic thin sections and other techniques of analysis, comparison with ethnographic examples, and experimental reconstruction, an insight can be gained into technological aspects

of the pottery production process (Whitbread 1995), as well as the nature of post-firing transformations. It is within this framework that the following chapter anticipates the possible effects of a variety of processes, from clay procurement to artefact curation and sample preparation, on the nature of the microfossil assemblages in archaeological ceramics and the interpretations which can be based upon them (Figure 3.1).

3.2 The original microfossil assemblage

Potters utilise many types of raw materials, some of which may contain microfossils. The microfossil assemblages in such raw materials are likely to vary considerably in terms of abundance, diversity and preservation. Therefore, the nature of the microfossil assemblages even in the untreated raw materials is varied and of course, has a direct affect on the state of those which can be observed in samples of archaeological pottery.

Two secondary sources of raw material which may be utilised in the manufacture of ceramics are river clays (Maggetti 1982; Day 1991) and weathered slope deposits. In an area such as Crete, where fluvial systems erode thick deposits of marine strata, the sediments which are deposited within their bed and banks, can contain reworked microfossils from many sources (Ayyad et. al. 1991; Hunt, 1996; Quinn et al. 1998, Section 11.6 of this report). Loose secondary deposits which accumulate at the base of outcrops from the weathering of microfossiliferous sediments also contain mixed microfossil assemblages, which vary according to the nature and extent of the sediments above. When interpreting the microfossil assemblages of archaeological

ceramics constructed from these secondary sources of raw material, it may be difficult
to apply the precise biostratigraphic and palaeoenvironmental methods of determining
provenance which are outlined in Sections 5.9.2, 6.7.4, 7.7.4, 8.5.3 and 9.3.2.
However, if potential deposits of secondary sediments can be sampled and analysed
for direct comparison, then it may be possible to identify those clay sources which
were likely to have been used in antiquity, as demonstrated in Section 11.6.

In situ surface sediments contain variations in the abundance, diversity, preservation
and reworking of microfossils, both vertically and laterally. Therefore, it is possible
that two vessels constructed from raw materials obtained at the same outcrop but
at different times may contain markedly different microfossil assemblages (Riley
1983; Rosenfeld and Stibbe 1997).

Figure 3.1. Possible sources of contamination and alteration of microfossil assemblages in
archaeological ceramics: 1. Procurement, 2. Contamination during preparation, 3. Intentional
clay mixing, 4. Alteration of assemblage during firing, 5. Alteration as a result of usage, 6.
Alteration and contamination during burial, 7. Alteration and contamination as a result of poor
sample curation and thin section or smear slide preparation.

ceramics constructed from these secondary sources of raw material, it may be difficult to apply the precise biostratigraphic and palaeoenvironmental methods of determining provenance which are outlined in Sections 5.9.2, 6.7.4, 7.7.4, 8.5.3 and 9.5.2. However, if potential deposits of secondary sediments can be sampled and analysed for direct comparison, then it may be possible to identify those clay sources which were likely to have been used in antiquity, as demonstrated in Section 11.6.

In situ marine sediments contain variations in the abundance, diversity, preservation and reworking of microfossils, both vertically and laterally. Therefore, it is possible that two vessels, constructed from raw materials, obtained from the same outcrop but at different levels, may contain markedly different microfossil assemblages (Riley 1983; Riley *et al.* n.d; Stilborg 1997).

3.3 Contamination during raw material procurement

The nature of the original microfossil assemblages in the raw materials which were used for the manufacture of ceramics, depends not only upon their origin, but also how they were obtained. It is in this initial stage of the pottery production sequence that contamination of the microfossil assemblage can first take place. The tools with which the raw materials are obtained can be a potential source of contamination, as they may contain material from other deposits with different microfossil assemblages, which can then be transferred during the process of digging or transporting the raw materials. Of course, as large quantities of raw materials are procured for the production of pottery, the effect of this form of contamination is likely to be small.

Another form of contamination which may occur during the process of procuring sediments for the production of pottery, is the unintentional incorporation of neighbouring material. The action of extracting clays from an outcrop is likely to loosen the sediment/rock above, which they were supporting, and some of this may become incorporated within the excavated material. If the source of the raw material is an outcrop of microfossiliferous marine sediments, then this form of accidental contamination is likely to result in a mixed microfossil assemblage, containing taxa which are indicative of different ages or environments.

3.4 Contamination and damage during clay storage and preparation

During the storage and preparation of raw materials for the production of pottery, there are various scenarios in which the alteration and contamination of their microfossil assemblages may take place. The potters workshop can contain raw materials from more than one source (Blitzer 1984, 146), which may have different microfossil assemblages, and it is therefore likely to be an area of high risk for contamination.

During the preparation of microfossiliferous raw materials for the manufacture of ceramics, the actions of sun-drying, crushing, sieving, levigation, foot trampling and working clay can potentially affect the nature of their microfossil assemblages. Foreign material may be introduced from the tools which are used, the surface upon which the operation is performed (often the ground), and even the air within or outside the potters workshop (in the case of very small or light groups of microfossils such as pollen, spores and calcareous nannofossils). In their interpretation of

agricultural information from the identification of organic microfossils in sun-dried Egyptian mudbricks, Ayyad *et al.* (1991) indicated that the Nile silt used in their manufacture, may have received pollen and spores during various stages of its preparation.

Whilst the quantity of foreign material which may be introduced during clay preparation is likely to be small, it may however be significant, especially if the original raw material did not contain microfossils to begin with (Battarbee 1988). Crushing and working microfossiliferous raw material for the manufacture of ceramics may also damage the microfossil specimens within (Gibson 1983b; Battarbee 1988; Håkansson 1997). This is demonstrated experimentally in Section 5.4.6.1 of the present report, and may affect the accuracy with which the assemblage composition of the finished artefact can be determined (Gibson 1983a).

In the ethnographic literature there are some accounts of the systematic 'ageing' or 'souring' of clays. This involves their storage for some time, during which the natural process of bacterial action helps to improve the workability of the material (Glick 1936; Leach 1976). Rice (1987, 119) states that the process of ageing may be assisted by "adding small amounts of acidic substances", a practice which would surely result in the degradation of any calcareous microfossils within the clay.

3.5 Intentional clay mixing and tempering

Ethnographic study has indicated that it is common for modern potters to mix two or more types of raw material in order to produce a paste which has the specific properties that they require (Arnold 1971; Voyatzoglou 1974; Blitzer 1984), and

numerous detailed analyses of ceramics have revealed that such practices were widespread in antiquity (Riley 1981; Jones 1984, 872; Day 1989). Intentional clay mixing and tempering poses major complications in analytical studies of ceramics in terms of "clay source determination, technology and, to a lesser degree, fabric characterisation" (Whitbread 1995, 375). Similarly, the mixing of two or more microfossiliferous clays (Jansma 1977; Matiskainen and Alhonen 1984; Stilborg 1997; Hunt 1996) as well as the contamination of microfossil assemblages by the addition of temper (Jansma 1982; 1990; Gibson 1983b; Battarbee 1988) can produce a confusing assemblage. This may be difficult to discriminate from other sources of contamination or even reworking in the original sample. Thin section petrography is very important for the determination of intentional microfossiliferous clay mixing (Stilborg 1997) or the addition of microfossiliferous temper (Whitbread 1995, 349), as is a consideration of any plant macrofossils occurring in palynological residues from ceramics (Hunt 1996; Ayyad *et al.* 1991).

More than one type of raw material can be used in the manufacture of a single vessel for the construction of its various parts, e.g. one clay may be used for the body of the vessel and another for the handle (Riley 1982; Wilson and Day 1994). Where complete vessels are available for analysis, it is necessary to be aware of this scenario, and design an appropriate sampling strategy (Section 5.3.2.2).

One less obvious way in which microfossils may be introduced into the raw materials used for the manufacture of ceramics, is by the intentional addition of sea water during clay preparation. It is common for potters to mix dry powdered clay with a suitable quantity of water in order to produce a paste of the desired consistency (Rye

1981, 36), and where pottery was produced close to the coast, sea water may have been used for this purpose. Several extant groups of planktonic microfossils occur in coastal and estuarine waters, as well as lakes, and small numbers of these may well be incorporated into pottery in this way. It may be possible, in some cases, to identify this potential source of contamination by the biostratigraphic interpretation of microfossil assemblages. However, the use of marine and lake water in the manufacture of pottery may pose serious problems for palaeoenvironmental analysis of extant quaternary diatoms. This scenario may explain the occurrence of small numbers of marine diatom species in the dominantly fresh water assemblages, which were noted by Jansma (1982; 1990, discussion in Section 2.3.3.2).

3.6 Alteration of the microfossil assemblage during firing

The behaviour of calcite during the firing of ceramics has been studied in some detail, and a range of estimates have been proposed for the temperature at which it undergoes the chemical transformation outlined below.

$$CaCO_3 \xrightarrow{600\text{-}900\ °C} CaO + CO_2 \uparrow$$
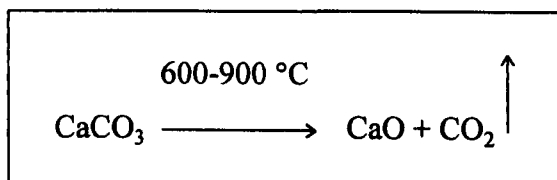
The different critical temperatures which have been recorded by various authors (ranging from 650 °C to as high as 900 °C), indicate that the threshold at which calcite undergoes this reaction depends upon other factors in addition to temperature (Rice 1987, 98). The temperature gradient, the duration and atmosphere of firing, as well as

the nature of the clay and calcite itself, are also likely to influence the behaviour of calcareous matter in ceramics during firing.

Several groups of microfossils are composed of calcite, and it is therefore important to consider how these are affected by the process of firing. In this report, experiments have been conducted on the behaviour during the firing of ceramics of calcareous nannofossils (Section 5.4) and palynomorphs (Section 9.4) and a comparison has been made between SEM estimates of firing temperatures and the nature of foraminifera and ostracods in archaeological ceramics (Section 6.4). In addition, the current state of knowledge regarding the behaviour of diatoms during firing has also been reviewed (Section 8.3).

The details of these investigations and their implications are discussed in full in the sections referred to, however it is worth noting here that, in general, the process of firing degrades the quality of micropalaeontological information contained within archaeological ceramics. It is therefore, one of the more crucial factors in the chain of events which transform the original microfossil assemblage of the raw material into what is seen under the microscope.

3.7 Alteration of the microfossil assemblage as a result of usage

After firing, the microfossil assemblages in archaeological ceramics may have been further degraded during usage. Ceramic vessels and other objects had a very large range of applications in ancient societies (Georgiou 1986), some of which may have altered the composition of their raw materials. Two common types of utilitarian

ceramic vessels which may have be altered as a result of their intended use, are cooking and storage vessels.

3.7.1 Cooking wares

In antiquity, certain vessels which were used for the preparation of foodstuffs, such as cooking pots and frying pans may have been subjected to heat over a fire (Riley 1984). If these types of vessels were manufactured from microfossiliferous raw materials, then such treatment may have led to the alteration or even the destruction of their microfossil assemblages, depending upon the temperature and duration of heating. When a pot is heated from below, the outside becomes much hotter than the inside (Arnold 1985, 23), and consequently, the microfossil assemblages in various parts of the vessel may be affected by varying degrees. This phenomenon has clear implications for the sampling of microfossiliferous ceramics. The effect of heating nannofossiliferous ceramics, as well as the possible differential alteration of calcareous nannofossil assemblages in cooking vessels during usage, are discussed in Section 5.5.

3.7.2 Storage wares

The degree to which the microfossil assemblages in ceramics may or may not be altered in storage wares during usage, is likely to depend upon the commodities which they contained. Weakly acidic substances such as vinegar and wine (Callender 1965; Riley 1981) are important in this respect, and oil-based products (Jones 1984, 842;

Tournavitou 1995, 79) may also have an effect. Investigations into the effect of these types of commodities on calcareous nannofossil assemblages in ceramics are presented in Section 5.5.

3.8 Alteration and contamination in the burial environment

After use, in the burial environment, there are numerous soil processes that may alter the composition ceramics (Jones 1984; Schiffer 1987), including the nature of any microfossils contained within (Troja *et al.* 1996). The post-depositional alteration of ceramics can affect their colour (Rice 1987, 345), trace element content (Franklin and Vitali 1985), thermoluminescence (Freestone *et al.* 1985), as well as leeching and depositing major elements within artefacts. Sodium, barium and calcium are particularly susceptible to dissolution and re-deposition by soil water, "especially when the latter two are present as carbonates" (Bieber *et al.* 1976, 73). Freeth (1967) points out that the calcite in fired pottery is converted to calcium hydroxide, which is highly soluble, and can be selectively leeched from ceramics after burial (e.g. Peacock 1968), and re-deposited elsewhere (e.g. Prag *et al.* 1974; Middleton and Woods 1990).

A process which may inhibit the post-depositional alteration of ceramics is the development of 'diffusion barriers' (Franklin and Vitali 1985). In order to establish the extent to which the trace element composition of ancient ceramics may be altered during burial, Franklin and Vitali conducted experiments into the effect of a broad spectrum of simulated soil solutions on fired briquettes, and discovered that "after an initial dissolution ... from the briquette surfaces, reaction and/or alteration products provide diffusion barriers that protect the briquette from further chemical attack"

(1985, 10). However the analysis of Freestone *et. al.* (1985, 161) on the behaviour of phosphates in buried ceramics did not agree with the existence of Franklin and Vitali's (1985) 'diffusion barriers', and these authors commented that the subject of the post-depositional alteration of ceramics during burial is "confused and controversial". Whether diffusion barriers develop in buried ceramics or not, is a subject of speculation, however it is likely that soil water does alter the nature of ceramics in many ways depending upon its temperature, chemistry and pH as well as the mineralogical composition of the ceramics in question and their degree of vitrification (Freestone *et al.* 1985).

These post-depositional processes are likely to have affected the nature of any calcareous microfossils in some archaeological ceramics, and under certain circumstances may completely destroy assemblages. The partial dissolution or overgrowth of calcareous microfossil specimens makes their identification more difficult and will therefore hinder the interpretation of the geological age or palaeoenvironment of the raw materials used in ceramic manufacture, as well as any provenance interpretations based upon this (see interpretation of Troja *et al.* 1996, Section 2.3.1.3). Siliceous microfossils however, are resistant to chemical weathering (Stilborg 1997), and organic microfossils are virtually indestructible except by oxidation.

Another way in which the nature of microfossil assemblages in ceramics can be biased during burial, is by contamination from the surrounding sediment. When excavated, archaeological pottery can contain burial deposits of their surrounding material (Rye 1981), and the incomplete removal of this material from the surfaces of a vessel or

sherd may leave a contaminant residue on the artefact, if the sediment in which it was buried contains reworked microfossils or is a relatively recent microfossiliferous deposit (a possible scenario at coastal sites). This subject has been addressed by Jansma (1981; 1984), in terms of the contamination of diatom assemblages by 'foreign clay particles', which can be removed by brushing sherds prior to preparation (Section 8.4.1). Caution is also required during the sampling of archaeological ceramics for the study of calcareous nannofossils in order to avoid contamination from secondary deposits, as outlined in Section 5.3.2 and Figure 5.4.

3.9 Alteration of the microfossil assemblage as a result of poor sample curation

As mentioned above, most archaeological ceramics, when newly excavated are encrusted with sediment. During excavations in the Aegean and elsewhere it is often standard practice to disperse or dissolve this material by immersing the artefacts in water or weak acid (Jones 1986, 37). Such a practice could have a serious effect on calcareous microfossils in ceramics, and although many vessels which have been treated in this way do contain microfossils, it is possible that some alteration of the assemblage could have taken place. It may be possible to detect the presence of larger calcareous microfossils in ceramics which have been altered in this way by the occurrence of distinctive shaped voids in thin section (Section 6.3, Figure 6.4; Section 7.3). However, calcareous nannofossils can be dissolved from ceramics without leaving a trace (Sections 5.4.8 and 5.5.2), and it may be that some sherds which appear barren under the microscope may have contained a calcareous nannofossil assemblage prior to acid treatment.

In order to isolate siliceous microfossils, such as diatoms, from archaeological ceramics using the 'flotation method' which is outlined in Section 8.4.1, it is often necessary to fragment the samples by hand (Jansma 1981), with pliers (Jansma 1984) or in a pestle and mortar (De La Fuente and Martinez Macchiavello 1997), depending upon their degree of vitrification. The latter two techniques, which are applied to medium and highly fired ceramics appear, to damage the diatom specimens contained within (Håkansson and Hulthén 1986). The fragmentation of specimens in this way can severely hinder species identification and will therefore affect the diatom 'profile' which is recorded, as well as the accuracy of interpretations based upon this (Gibson 1983a; Håkansson and Hulthén 1986).

Diatom assemblages in archaeological ceramics can also be damaged by several other processes taking place during the history of the clay and the finished artifact (Stilborg 1997), including the transportation and reworking of specimens in the original sedimentary environment (Jansma 1977), maceration and clay working in the pottery production process (Gibson 1983a), and the affects of high firing (Gibson 1983b). It may therefore be very difficult to distinguish between these types of alteration, and fragmentation produced by the deliberate crushing of diatomaceous sherds during sample preparation (Battarbee 1988). A possible solution to this problem, which has been suggested by Gibson (1983b) is to fragment well-lithified pottery sherds using ultrasonic vibration, however this method has not been proven to be successful.

Specimens may also be further damaged during the subsequent chemical treatment (Håkansson and Hulthén 1986) and centrifuging (Battarbee 1988) of crushed sherds in the flotation method of diatom preparation (Section 8.4.1). In their account of this

procedure, which involves heating the broken pieces of pottery at 50 °C in a c.10 % solution of hydrogen peroxide for up to ten days, Håkansson and Hulthén (1986) state that diatom colonies (Section 8.1.1) disintegrated and some larger species became broken.

One last form of contamination which may take place after the excavation of ceramics, is the transferral of microfossiliferous material from one sample to another during thin section preparation. This final source of error is particularly applicable to calcareous nannofossils, because of their small size, and can be avoided by making 'smear slides' from small scrapings of the original sherds using the method described in Section 5.3.2.

4 The Geology of Crete and the southern Aegean

4.1 Introduction

In the preceding chapter we anticipated the way in which several natural and human-induced processes potentially can alter the nature of the raw materials which ancient potters utilise for the manufacture of ceramics. In this context we also discussed various types of clay sources, as well as the occurrence of natural variability in sedimentary rocks. Both of these are fundamental to the subject of ceramic petrography in that they help to separate pottery into different fabric groups, which can then be related to the nature of specific geological deposits. With this in mind, it is imperative in any scientific analysis of archaeological ceramics, to consider the nature of the local and regional geology (Bishop *et al.* 1982). A comprehensive geological field study is beyond the scope of any such project, especially when dealing with a large and complex area such as the Aegean. However, in the following chapter we aim to review the current state of knowledge of geology of Crete, so that specific rock types and sedimentary formations can be identified and sampled for further analysis (Chapter 10). The present report is concerned primarily with the extensive Neogene sediments of Crete and other islands in the southern Aegean (Figure 4.1).

4.2 The late Cenozoic geological evolution of Crete and the southern Aegean

Structurally, the Aegean region represents a "young, small ... but geodynamically extremely active part of the Alpine-Himalayan orogenic system" (Berckhemer 1978, 21). This orogenic system, which contains the Alpine-Hellenide-Tauride mountain chain, was initiated during the closing of the Tethyan ocean which existed between Eurasia and Africa, Arabia, India and Australia. In the eastern Mediterranean the African continental plate is being subducted beneath the fragmented Eurasian plate as they converge in a roughly N-S direction at the Hellenic trench, south of Crete (Figure 4.1). This subduction process has resulted in continued tectonic and volcanic activity within the Aegean region throughout the Cenozoic until the present day, and is responsible for the complex geology of the region.

Crete, Karpathos, Rhodes, Kassos and Kythira, which form the southern Aegean island arc (a chain of islands running in a curve across the eastern Mediterranean north of the boundary between the African and Eurasian plates; Figure 4.1) were all originally part of the 'southern Aegean landmass', during the Oligocene. This consisted of a complex nappe pile of allochthonous Tethyan rocks, emplaced upon autochthonous Permian-Oligocene rocks by the converging plates. Today these rocks form the basement of the islands in the southern Aegean as well as some of the Cycladic islands, which may also have been a part of the southern Aegean landmass.

The basement consists of a complex arrangement of volcanic, metamorphic and metasedimentary rocks which are highly deformed, and can be divided into discrete units by the tectonic contacts which were involved in their emplacement. The biggest remnant of the southern Aegean landmass exists on Crete and is referred to as the
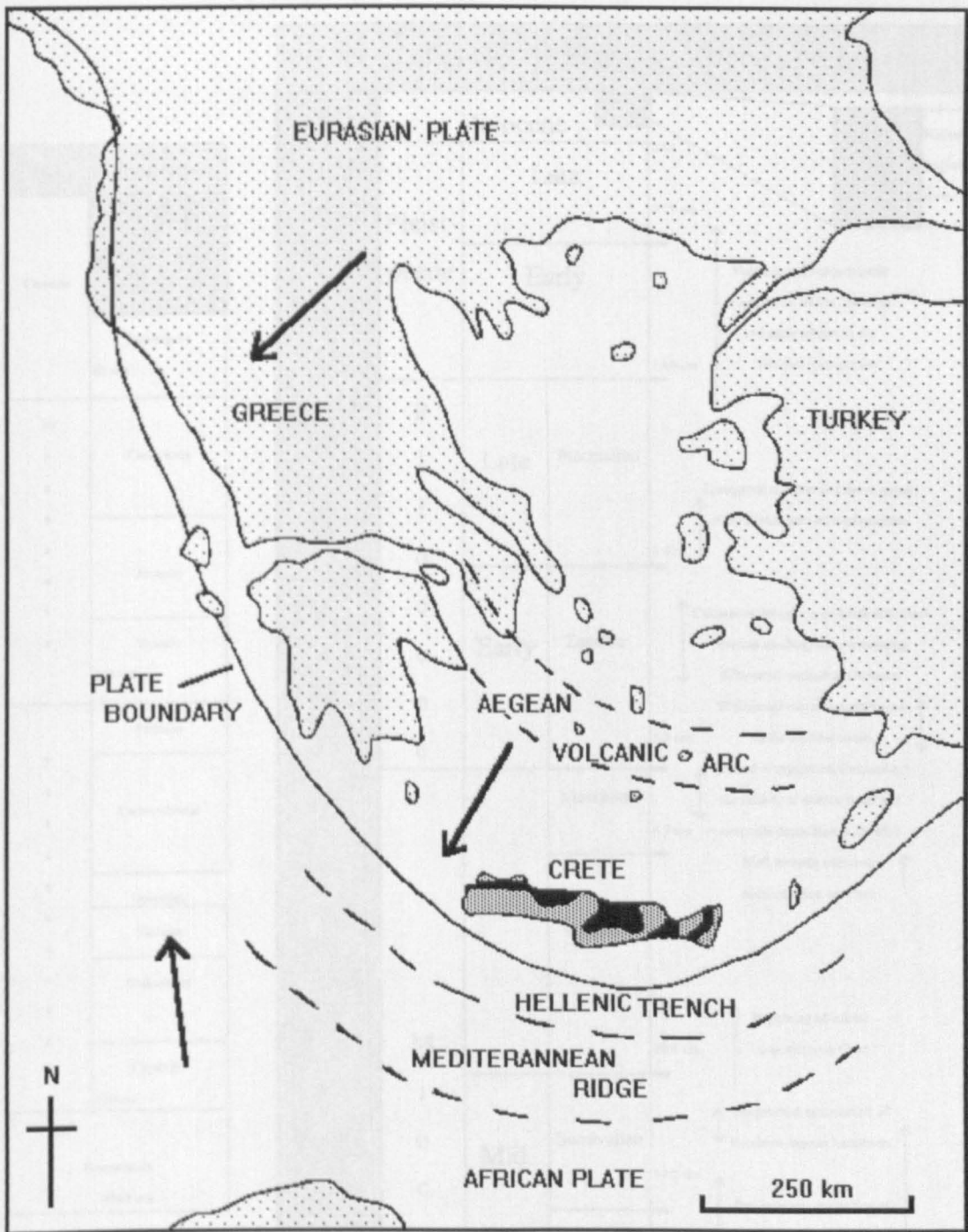
Figure 4.1 The gross geological configuration of the southern Aegean area (arrows represent the direction of plate movement), and the distinction on Crete between Neogene and pre-Neogene rocks (Neogene = grey; pre-Neogene = black). After Hall *et al.* (1984) and Meulenkamp (1971).

ERA	PERIOD
Cenozic	Quaternary
	Neogene
	Palaeogene
	65 ma
Mesozoic	Cretaceous
	Jurassic
	Triassic
	245 ma
Palaeozoic	Permian
	Carboniferous
	Devonian
	Silurian
	Ordovician
	Cambrian
	570 ma
	Proterozoic
	4560 ma

Period	Epoch	Stage	Dates and Events
Quaternary	Holocene		
	Pleistocene	Late	0.01
			0.75 ma
		Early	1.64 ma
Neogene	Pliocene	Late	Piacenzian
			Emergence of Crete and development of the islands present configuration.
			3.40
		Early	Zanclian — Calcareous deposition on Crete with marl breccias resulting from continuing differential vertical movements.
			5.2 ma — Widespread marine transgression in the Mediterranean.
	Miocene	Late	Messinian — Period of regression, increase in the salinity of marine water and evaporite deposition in the Med.
			6.7 ma — Shift towards carbonate sedimentation on Crete.
			Tortonian
			10.4 ma — Beginning of marine deposition on Crete.
		Mid.	Serravalian — Structural fragmentation of Southern Aegean Landmass.
			14.2 ma
			Langhian 16.3 ma — Post orogenic deposition of coarse non-marine clastics on Crete.
		Early	Burdigalian — Deposition of small marine sequences in the Cycladic islands.
			21.5 ma
			Aquitanian
			23.3 ma
	Oligocene		

Volcanic and volcaniclastic eruptions on Melos, Santorini and other islands in the 'Hellenic volcanic arc'

323 BC	Hellenistic period	
3000 BC	Begining of Bronze Age	
7000 BC	First settlers on Crete	

Figure 4.2 The geological column, with geological and archaeological events on Crete. Dates after Harland et. al. (1989).

Cretan 'pre-Neogene' (to distinguish it from the contemporaneous Neogene sedimentary rocks which cover the other third of the island; Figure 4.1). The pre-Neogene forms a block faulted backbone running E-W across the length of the island and contains several mountain ranges which reach a height of 2000 metres above sea-level. The distinction on Crete and many other Aegean islands between the pre-Neogene basement (of the southern Aegean landmass) and the Neogene sediments which were deposited on and around them (Figure 4.1), is crucial to the present report. This is because the pre-Neogene is generally non-microfossiliferous, whereas the Neogene sediments contain abundant microfossils of marine and non-marine origin. Whilst it is not uncommon for ancient and modern potters to have used pre-Neogene sediments as a raw material for the manufacture of ceramics (Day 1991), their lack of microfossils, means that the pre-Neogene rocks of Crete and the southern Aegean are not discussed further in the present report. However, it is worth noting that the erosion of the pre-Neogene basement may have provided a great deal of the clastic material which is contained within the thick Neogene deposits that unconformably overlie them.

Detailed field mapping and the study of numerous sections within a framework of biostratigraphic data by Greek geologists such as M. Dermitzakis and stratigraphers of the University of Utrecht, since the 1960's, has revealed a great deal of information regarding the nature of the Neogene deposits of Crete, as well as aiding the reconstruction of the late Cenozoic techno-sedimentary history of the Aegean as a whole. Micropalaeontology has played a very important role in this work, both as a means of dating important events and in the interpretation of palaeoenvironment. A result of the partnership between stratigraphy and micropalaeontology, led primarily

by workers from Utrecht, has been the proposal of many biostratigraphic schemes for Crete and the Aegean based on various microfossil groups. These are discussed in detail elsewhere (Sections 5.6, 6.6 and 7.6).

Good accounts of the late Cenozoic geological evolution of Crete and the Aegean can be found in the publications of Meulenkamp (1971; 1985) and Meulenkamp *et al.* (1979), upon which the following summary is based. In addition, detailed considerations of the structural configuration of the southern Aegean and the geotectonic processes involved in the emplacement of the Cretan nappe can be found in Berckhemer (1978) and Hall *et al.* (1984).

In the Oligocene to Middle Miocene, whilst Crete, Rhodes, Karpathos, Kassos and Kythira were part of the southern Aegean landmass (or 'southern Aegean block' Meulenkamp 1985), the Cyclades area was "at least partly covered by the sea" Meulenkamp (1971, 11). This resulted in the deposition of small sequences of marine sediments on some of the islands. On the southern Aegean block sedimentation was in the form of post-orogenic, coarse, non-marine clastics. These Middle Miocene coarse sandstones and conglomeratic fluvial sediments are common on Crete, for example in the Ierapetra region, where they form the conglomeratic fans of the Mithi Formation, which lies in front and on top of the allochthonous pre-Neogene relief (Fortuin 1978).

Some time near the end of the Middle Miocene (approximately 13 million years BP), a reorganisation of the various Aegean microplates, related to the onset of the Hellenic subduction process, resulted in a structural fragmentation of the southern Aegean landmass and a general subsidence in this area, linked with an uplift in the Cyclades to the north. This was a reversal of the situation which had existed since the Late

Oligocene (Meulenkamp 1971). The sea first invaded Crete in the Ierapetra basin, a NE-SW graben-like depression (Fortuin 1978) which is perhaps the largest transverse fault in the southern Aegean island arc and seems to have been initiated in the late Serravalian. This resulted in the deposition of Crete's first marine sediments, the marly clays of Fortuin's upper Males Formation.

The fragmentation of the southern Aegean landmass took place mainly via north-south and east-west trending faults which defined the Neogene palaeogeographic configuration, as well as the present contours of the island (Meulenkamp 1985). The result was a transformation of the land area into a complex of islands (horsts) and basins (graben), which were flooded by the sea. Erosion of the uplifting land supplied large amounts of material to the rapidly subsiding basins, in which clastic marine sequences of Late Miocene, Tortonian age accumulated, whilst biogenic sedimentation took place on the shoals and around the islands.

Differential tectonic movements took place during this time. These produced variations in the thickness of marine sediments between basins and resulted in the uplift and erosion of earlier Neogene sediments in some places. In the Ierapetra basin, tectonics played an important role in determining the nature of the Tortonian sedimentation, resulting in a complicated sedimentary pattern (Fortuin 1978), in which breccias (Prinia complex) and turbidites (Makrylia Formation) were deposited.

An overall shift towards carbonate sedimentation seems to have taken place in Crete and the surrounding area during the transition from the Tortonian to Messinian. This may have resulted from the combination of a reduction in the supply of clastic material (perhaps linked with a reduction in the uplift of horst blocks) and a decrease

in the overall subsidence of the landmass, producing a general shallowing in the grabens. The area became a "mosaic of many partly land-locked basins" (Meulenkamp *et al.* 1979, 147), with widespread deposition of bioclastic limestones and calcareous marls in shallow warm waters.

Within the Messinian, gypsum evaporite deposits were laid down throughout the Mediterranean as a result of the 'Messinian salinity crisis'. This period of regression and increase in the salinity of marine waters, was the consequence of restricted 'basinal' connections within the Mediterranean, and a general cooling of the climate (Thomas and Van Der Zwaan 1981). In many parts of Crete, gypsum occurs as discrete beds, intercalated within marls, or as thick marl breccias. Everywhere it is laterally discontinuous and the thickness varies in relation to the subsidence and supply of clastic material at the time. For example, in Khania (west Crete) the Messinian gypsum beds can be up to two metres thick, whereas in the area south of Iraklion (north-central Crete), the gypsum of equivalent age may reach a thickness of four metres.

Whilst the early Messinian sedimentary and structural history of Crete was similar over most of the island, the late Messinian period was characterised by different palaeogeographic and sedimentary conditions in the various basins (Meulenkamp *et al.* 1979). For example, following an intra-Messinian period of uplift which affected many parts of Crete, renewed subsidence took place in the Khania region, whereas in north Iraklion, Rethymnon and Sitia, marl breccias unconformably overlie lower Messinian evaporites and calcareous marls. This latter situation suggests that the "post-early Messinian period of uplift and erosion was apparently not followed by

renewed subsidence of any significance" (Meulenkamp *et al.* 1979, 147). The upper Messinian of Crete is a complex mixture of fluvial, brackish and shallow marine sediments with varying degrees of synsedimentary deformation, and reflects a period of tectonic instability expressed by the differential relative movement of small crustal blocks.

During the Early Pliocene, a widespread marine transgression took place throughout the Mediterranean, due to an overall rise in sea-level (Meulenkamp *et al.* 1979). It was the widest transgression that had affected the area during the whole of the late Neogene and resulted in an invasion of the Messinian archipelago of islands, tropical seas and land-locked lagoons. Open marine conditions were extensive on Crete in the Early Pliocene, and only the large pre-Neogene masses of the Lefka Ori and Idi mountains remained above sea-level (Meulenkamp 1971). Sedimentation was dominantly calcareous, with reefs developing on the islands (horsts). In central and eastern Crete, Early Pliocene marl breccias are a testament to continuing differential, vertical movements which spread to other areas and caused the removal of much Lower Pliocene material from the rising blocks (Meulenkamp 1985). These earth movements heralded the onset of a major tectonic phase which culminated in the gradual emergence of Crete throughout the mid-Late Pliocene (Fortuin 1978), and gave rise to the island's present configuration.

The youngest marine deposits on Crete, are the middle Pliocene sediments of the north-central Crete (Meulenkamp 1985), which record a regressive sequence caused by the uplift of the island. At the time of Crete's emergence from the sea, marine deposition was continuing in some areas of the southern Aegean, such as Karpathos,

as well as in the southern Cyclades (Melos), and to the east (Ionian Islands). However, in the central Cyclades continental conditions still existed, and the sea did not reach this area again until the Pleistocene (Meulenkamp 1971).

4.3 Discussion

Of the two main groups of rocks which constitute the geology of Crete, only the autochthonous, microfossiliferous, late Neogene marine sediments are of concern to the present report. The allochthonous pre-Neogene basement rocks upon which these marine sediments lie, were heavily metamorphosed during their emplacement, and as a result are non-microfossiliferous.

Due to continuing variations in the tectonic movement of the various pre-Neogene crustal blocks of Crete during the late Neogene period, as well the overall rise and fall of sea level in the Mediterranean sea, several types of calcareous marine sediments were deposited on the island, including marls, clays, shallow water limestones, marl-breccias and gypsum. These late-Neogene sediments occur in many parts of the Crete, but tend to be restricted to coastal areas as well as the low-lying land between the various chains of high mountains (Figure 4.1). As a result of many years of intensive study by various stratigraphers and micropalaeontologists, the nature of the Cretan Neogene sedimentary record, and consequently, the late Cenozoic geological history of the island is well understood (Section 4.2). The lithology, micropalaeontology, geological age and geographic distribution of these sediments are outlined in several reports (e.g. Freudenthal 1969; Meulenkamp 1969; Gradstein 1973; Fortuin 1977) as well as numerous maps, published by the Institute of Geology and Mineral

Exploration (IGME), Athens. These have been utilsed in the selection of field samples in the present report and the detailed description of the various late Neogene sedimentary formations of the north-central Crete, the Ithmus of Ierapetra and the south coast of the Island which are presented in Chapter 10.

Micropalaeontology has been instrumental in understanding the late Neogene sediments of Crete, through the interpretation of palaeoenvironment, as well as the geological age in which the various marine formations were deposited. Consequently, several late Neogene biostratigraphic schemes have been proposed for the eastern Mediterranean (e.g. Zachraisse 1975; Spaak 1983; Theodoridis 1984; Driever 1988), based upon the numerous groups of microfossils which occur in the late Serravalian to middle Pliocene sediments of Crete. These studies have been utilised in the present report, and are reviewed in detail in the following chapters.

5 Calcareous nannofossils

5.1 Introduction

The late Neogene microfossiliferous marine sediments of Crete appear to have been used by Minoan potters as a source of raw materials (Riley 1981; 1983; MacGillivray *et al.* 1988; Riley *et al.* n.d), and consequently, much of the archaeological pottery excavated on the island contain calcareous or siliceous microfossils. Before attempting to utilise these distinctive inclusions to analyse the archaeological ceramics of Crete and elsewhere in the Mediterranean (Chapter 11), it is necessary to discuss each group of microfossils, in terms of their morphology, biological affinities and geological applications, their occurrence within the study material, the way in which they are affected by the firing process, their utility for the biostratigraphic and palaeoenvironmental interpretation of the raw materials of ceramic manufacture, as well as the methodology which is proposed for their use in ceramics. This chapter focuses on the most commonly used group of microfossils in the present report; calcareous nannofossils.

Calcareous nannofossils are a heterogeneous group of microfossils containing various organically precipitated calcium carbonate structures which are usually < 10 μm in size. These minute calcite bodies are common in fine-grained pelagic sediments of the Late Triassic to Recent from many areas of the world. The calcareous nannofossils can be divided into two main groups; coccoliths and nannoliths. Coccoliths, the most common type of calcareous nannofossil, are ornate calcite plates which form an external test or 'coccosphere' in the coccolithophorid algae (Figure 5.1). The

'coccolithophores' are unicellular, biflagellate, autotrophic, marine phytoplankton belonging to the Haptophyte (golden-brown) algae, which are common in the world oceans today, and have been one of the most important producers of carbonate sediment since the Cretaceous period.

Coccoliths are morphologically very diverse, but can be divided into holococcoliths and heterococcoliths on the basis of their calcite ultrastructure. Holococcoliths, which are produced on the outside of the cell, are constructed of many equidimensional rhombohedra of calcite (Figure 5.1). As such they are delicate and are less likely to be preserved in the fossil record than heterococcoliths. The other type of coccoliths, heterococcoliths, are produced within the cells of coccolithophorid algae and are constructed of larger, complex shaped, often interlocking, calcite crystals to form a more robust structure (Figure 5.1). Heterococcoliths, are most common type of calcareous nannofossils in the geological record and exhibit great morphological variation, however, some of the more common shapes include: tiered discs (placoliths), un-tiered discs (muroliths), tiered discs with asymmetrical flanges (helicoliths) and basket or vase shaped structures (lopadoliths), see Figure 5.1.

The other major group of calcareous nannofossils, the nannoliths, are those structures possibly produced by coccolithophorids which lack the typical features of heterococoliths and holococcoliths. These include the star-shaped *Discoaster*, horseshoe-shaped *Ceratolithus* and the five-sided plate-like *Braarudosphaera* (Figure 5.2) as well as a great plexus of variously-shaped calcite structures which have an unknown origin, but are likely to have been the remains of coccolithophores or some other group of marine plankton, due to their cosmopolitan distribution and association

with coccoliths. Nannoliths can be rod-shaped: *Microrhabdulus*, bladed: *Triquetorhabdulus*, triangular: *Lithostromation*, sub-spherical: *Schizosphaerella*, as well as some bizarre and seemingly amorphous shapes: *Marthasterites* (Figure 5.2).

Calcareous nannofossils are classified on the basis of morphology, ultrastructure and crystallography using binomial nomenclature, similar to that applied to living organisms, in order to define genera and species which characterise particular periods of their c. 200 MA geological history. In this way, calcareous nannofossils are an extremely useful tool for biostratigraphy, and it is this potential, first discovered in the 1950's and utilised extensively in the exploration for natural resources as well as the Deep Sea Drilling (DSDP) and Ocean Drilling (ODP) Projects, which has fuelled the extensive study of calcareous nannofossils in the latter half of this century. Research into the biology of living calcareous nannoplankton has greatly aided the understanding of calcareous nannofossils, especially in the Cenozoic and Quaternary. Although there are a few conflicts between the study of living and fossil forms, for example, in terms of classification, the two are complimentary.

Detailed accounts of the morphology, terminology, classification, evolution and biostratigraphy of calcareous nannofossils can be found in Perch-Nielsen (1985a, b), Haq (1983a, b), Siesser (1993), Lord (1982), Aubry (1984-1990), Farinacci (1969-1979), Bown (1987), Theodoridis (1984), Young and Bown (1997a, b and c), Young *et al.* (1997) and Bown (1998), upon which the above account is based. Numerous reports which deal with the current state of knowledge on the biology of living coccolithophores can be found in Winter and Siesser (1994).
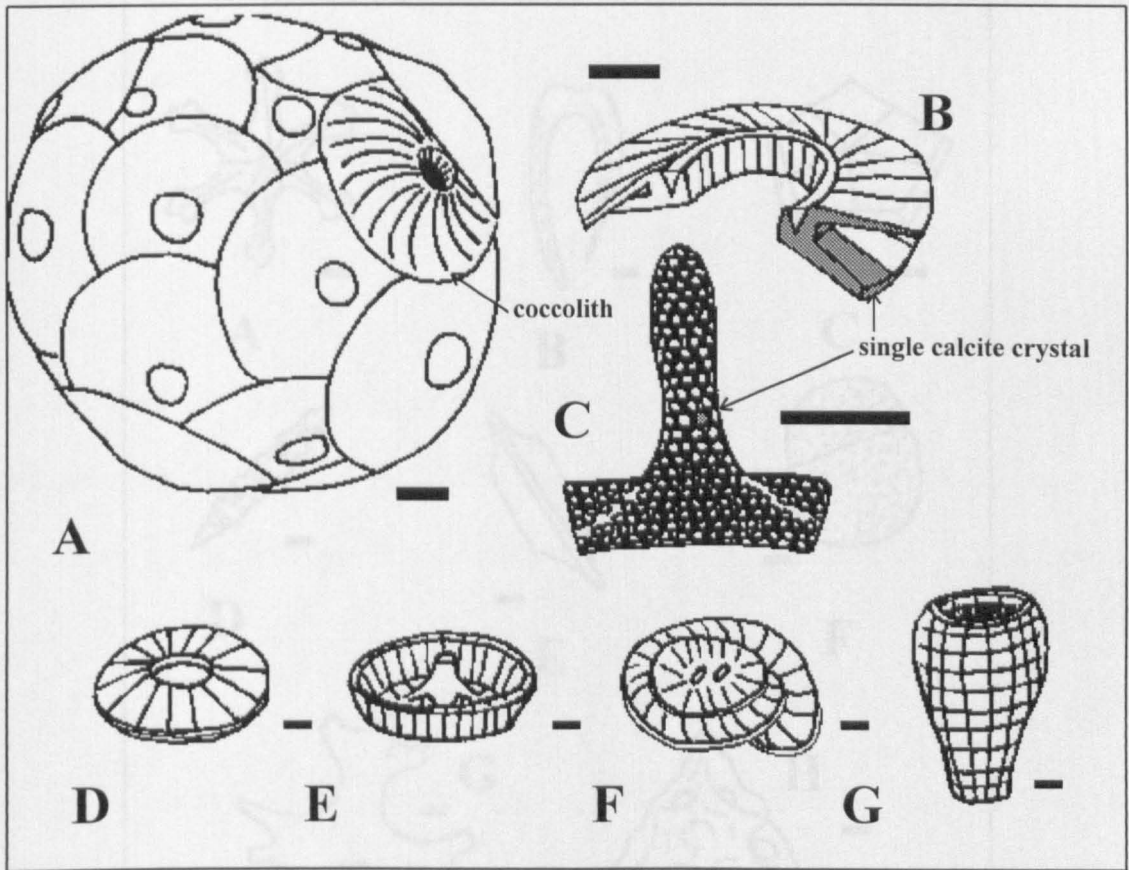
Figure 5.1. A coccosphere (A), the calcite ultrastructure of heterococcoliths (B) and holococcoliths (C), and the range of morphology in heterococcoliths: placolith (D), murolith (E), helicolith (F) and lopadolith (G). Scale bars: A, B and D-G = 1 μm, C = 5 μm. After Aubry (1990), Theodoridis (1984), Winter and Siesser (1994) and Young *et al.* (1997).

5.2 The occurrence of Calcareous Nannofossils in Bronze Age archaeological ceramics from Crete

Calcareous nannofossils are a significant component of ceramics from the Bronze Age of Crete, as well as pottery from other archaeological periods and different parts of the world (Quinn et al. 1995; Cetin et al. 1999; Bennett and Young ...

Calcareous nannofossils can occur as several components of a ceramic: in the groundmass, within other inclusions, as part of calcareous slips and paints applied to the exterior of the vessel, or in secondary residues attached to the fabric during burial in the archaeological record (Figure 5.1). This report focuses on the study of calcareous nannofossils in the groundmass, and calcareous slips and paints; measures are taken during sampling in order to keep calcareous nannofossils from the other two contexts (Section 5.3.2).

Calcareous nannofossils only occur in the groundmass of calcareous ceramics (those which retain a substantial portion of their carbonate content in isolated coccoliths and nannoliths, or in whole or partial fragmented coccospheres; Plate 5.3). The preservation and abundance of calcareous nannofossils varies greatly between individual pottery samples as a result of the nature of the original raw materials of ceramic production as well as the various processes which may have altered the assemblage during the history of an artefact (Chapter 3). In general, the calcareous nannofossil assemblages of archaeological ceramics are rather poorly preserved compared to those in ...

Figure 5.2. The range of morphology in nannoliths. *Discoaster* (A), *Ceratolithus* (B), *Braarudosphaera* (C), *Microrhabdulus* (D), *Lithraphidites* (E), *Schizosphaerella* (F), *Marthasterities* (G) and *Lithostromation* (H). All scale bars = 1 μm. After Aubry (1988), Perch-Nielsen (1985a and b) and Young *et al.* (1997).

5.2 The occurrence of Calcareous Nannofossils in Bronze Age archaeological ceramics from Crete

Calcareous nannofossils are a significant component of ceramics from the Bronze Age of Crete, as well as pottery from other archaeological periods and different parts of the world (Troja *et al.* 1996; Quinn *et al.* 1998; Burnett and Young n.d.).

Calcareous nannofossils can occur in several components of a ceramic; in the groundmass, within other inclusions, as a part of calcareous slips and paints applied to the exterior of the vessel, or in secondary residues attached to artefacts during burial in the archaeological record (Figure 5.3). This report focuses on the study of calcareous nannofossils in the groundmass and calcareous inclusions, and measures are taken during sampling in order to avoid calcareous nannofossils from the other two contexts (Section 5.3.2).

Calcareous nannofossils mainly occur in the groundmass of calcareous ceramics (those which contain abundant fine calcium carbonate), either as isolated coccoliths and nannoliths, or more rarely as whole coccospheres (Figure 5.3). The preservation and abundance of calcareous nannofossils can vary greatly between different pottery samples as a result of the nature of the original raw materials of ceramic production as well as the various processes which may have altered the assemblage during the history of an artefact (Chapter 3). In general, the calcareous nannofossil assemblages of archaeological ceramics are rather poorly-preserved compared to those in geological samples. Ceramic calcareous nannofossil assemblages are often dominated by robust and solution-resistant taxa, as well as those which may have been abundant in the assemblage of the original raw material.

Figure 5.3. Calcareous nannofossils in thin sections of Bronze Age archaeological ceramics from Crete. A discoaster in the groundmass of sample Kn 84/30 (A), a horseshoe-shaped nannolith (*Amaurolithus*) in a secondary deposit at the edge of sample Kn 95/400 (B), a complete coccosphere (C) and an isolated coccolith (D) in a calcareous slip or paint on sample Kn 95/400 (C). A and B = plane polarised light (PPL), C and D = crossed polars (XP). Field of view = 50 μm.

In the archaeological ceramics which have been analysed in the present report, calcareous nannofossils commonly occur in association with other calcareous microfossils such as planktonic foraminifera, benthic foraminifera, ostracods, and much less commonly with siliceous microfossils. Those containing calcareous nannofossils often have significant amounts of oxidised amorphous organic matter, as seen in smear slides with PPL (Section 5.3.2). However, very few identifiable palynomorphs have been found associated with calcareous nannofossils in the present report.

5.3 Methods of studying calcareous nannofossils in archaeological ceramics

5.3.1 Introduction

Calcareous nannofossils can be observed in thin sections of archaeological ceramics with a high power (1000 x) transmitted light microscope. Studying thin sections at these high magnifications necessitates the use of an oil-immersion objective which rides on a thin film of oil between the lens and the slide, therefore any ceramic thin sections observed in this way should be coverslipped in order to avoid damaging them. In ceramic thin sections, calcareous nannofossil specimens are often obscured by the clay matrix or other inclusions (Figure 5.3), as they are usually < 15 μm in size, whilst most ceramic thin sections are ground to a thickness of 30 μm. For this reason it is necessary, when searching for calcareous nannofossils in ceramic thin sections, to traverse around the edge of the section where it is usually less thick (Figure 5.5). Negotiating the irregular periphery of a ceramic thin section in this way is difficult

under high magnifications of 1000 x or more, therefore a mechanical microscope stage is required.

Whilst it is possible to observe calcareous nannofossils in ceramic thin sections, only small numbers of specimens are visible and their identification is difficult. Troja *et al.* (1996) studied calcareous nannofossils in thin sections of Neolithic to Bronze Age pottery from Milena, Sicily (Sections 2.3.1.3 and 2.3.2.5), and their poor assemblage descriptions exemplify the difficulties of such work.

A distinct disadvantage in studying calcareous nannofossils in thin sections of archaeological pottery, as opposed to larger calcareous microfossils, is the risk of contamination. The extremely small size of calcareous nannofossil specimens makes them very easy to transfer unnoticed from one place to another. Too small to be seen by the naked eye, thousands of these minute calcite structures can be contained within a sample of sediment the size of a crumb. The standard procedure for the preparation of geological samples for the analysis of calcareous nannofossils, which is outlined below, requires extreme cleanliness, however contamination can still occur from nannofossil specimens suspended in the air. During the preparation of ceramic thin sections very few precautions are taken in order to avoid the transferral of fine material, produced by grinding, from one section to another. Thin sections are cut and ground using the same apparatus. In the final stage of the process it is common for several sections to be polished in a slurry of carborundum grit and water, a procedure which is likely to result in contamination. Most slides are washed after polishing before a coverslip is added to them, however it is common to find often large amounts

of carborundum (a grey translucent mineral), in voids or around the edges of thin sections, indicating that the cleaning process failed to remove all contaminants.

As there is no record of the exact procedures used in the production of the extensive ceramic thin section collections which exist, and it is likely that most were prepared in a fashion similar to, or with less care, than that described above, the analysis of calcareous nannofossils in thin sections of archaeological pottery is not to be relied upon.

An alternative method of studying calcareous nannofossils from archaeological pottery, which was utilised by Burnett and Young (n.d.) in their analysis of a Bronze Age pottery sherd from Dover, England (Section 2.3.2.5) and is adopted in the present study, is by the preparation of 'smear slides'. This technique is virtually identical to the standard procedure used to prepare geological samples for the analysis of calcareous nannofossils. It involves scraping a small quantity of powder from the original sherd or vessel onto a microscope coverslip, spreading it out with water and adhering this onto a glass slide (Section 5.3.2). Smear slides often contain hundreds of calcareous nannofossils, which are separated from the clay matrix and associated minerals, and can be more easily identified and measured (Figure 5.5). In this way, a detailed analysis of the composition of calcareous nannofossil assemblages from even very small samples of archaeological pottery can be made (Quinn *et al.* 1998; Burnett and Young n.d.; Appendix II).

5.3.2 Procedures for preparing calcareous nannofossil smear slides of archaeological ceramics

The preparation of calcareous nannofossil smear slides from geological samples and archaeological material must take place in a clean, dust free environment, preferably away from other laboratory work and raw sediment samples. During the preparation of smear slides, it is advisable to treat calcareous nannofossils as if they were a highly contagious virus; cleaning and replacing the equipment between different sediment/pottery samples and repeating the operation if any contamination is suspected. In order to determine whether airborne contamination may have taken place during the slide making process, it is necessary to leave a coverslip somewhere in the vicinity of the operations, and mount this face-down on a glass slide at the end of the preparation. If this 'control slide' contains a significant contaminant nannoflora then the corresponding batch of smear slides should be discarded and repeated, taking greater care.

5.3.2.1 Equipment

Geological or archaeological samples in sealed bags: Standard microscope slides: Large square or rectangular glass coverslips: Optical adhesive (e.g. 'Norland', 'Entellan'): Small beaker: Small disposable phials: Disposable pipettes: Distilled water: Flat toothpicks: Tissues: Self adhesive slide labels: Permanent marker pen: Knife: Labcoat: Hotplate: Fume cupboard: Sink with soap and a scrubbing brush.

5.3.2.2 Procedure

1. Wipe down the area in which the operation is to take place, as well as the surface of the hot plate, wash hands, scrub and dry the knife.

2. Set the hot plate on a medium heat (150-200 °C), so that a wetted coverslip placed on its surface dries in about ten seconds. Lay out tissue or paper in the fume cupboard on which to place the coverslipped slides for the adhesive to set. On the work surface, lay out tissue and on this place one labelled microscope slide, one coverslip, one toothpick and the clean knife. Fill the clean beaker with distilled water, leave a pipette in it and place this nearby. Leave the adhesive and a pipette near the hotplate or in the fume cupboard.

3. Lick one side of the coverslip and place it on the tissue with the moist side up. Open the sample bag and manipulate the sample so that the desired surface is protruding from it. With geological samples it may be useful to snap a rock fragment (whilst in the bag) in order to produce a fresh surface from which to scrape. If this is not possible, or when scraping a sample of pottery, it may be necessary to scrape away the surface layer in one area, onto the tissue in order to reveal a fresh surface below. With the knife, scrape the desired surface above the moist coverslip until a small quantity of powder is deposited upon it. Put the knife back on the tissue, seal the sample bag and place this to one side.

When scraping a sample of pottery, it is necessary to first inspect the sherd in order to decide from which surface the scraping should be made (Figure 5.4). Ideally, the scraping should be taken from the centre of a fresh broken surface in order to avoid any slips or paints, as well as secondary deposits which may not been completely

removed after excavation (Section 3.8). If a thin section has been taken from the sherd then it will contain a freshly cut surface and a sample should be taken from this, after scraping away the surface layer in order to remove any contamination which may have been left by the cutting process (Section 5.3.1).

If a complete vessel is available for sampling, then there are two points to consider when deciding where to scrape. Firstly, if the vessel is likely to have been used for cooking (this can be determined by its typology or characteristic deposits inside or on the base exterior) then it is inadvisable to take a sample from the base, because the calcareous nannofossils are likely to be poorly-preserved in this region due to the effect of heat during usage (Section 5.5). Secondly, if different types of clay have been used for the construction of various parts of the vessel, e.g. one for the body of the artefact and another for the handle (Section 3.5), then smear slides should be prepared from the main body or all of its various components.

4. Add a drop of distilled water to the powder, being careful not to touch it with the pipette. Pick up the coverslip between finger and thumb, and using the tip of the toothpick mix the water and sediment together. Spread this out over the coverslip by using the edge of the toothpick, then transfer the coverslip to the hot plate with the smear-side up.

5. Whilst the smear is drying, put two or three drops of adhesive onto the labelled slide using a pipette. Lift the dry coverslip from the hotplate and gently lower it with its smear-side down, onto the slide. This part of the operation requires practice in order to avoid trapping air bubbles between the slide and the coverslip. If air bubbles

VESSEL

Core

Internal
Surface

Seondary
Deposit
External
Surface

Body

Broken or SHERD
cut surface

Slip

Decoration

DETAIL
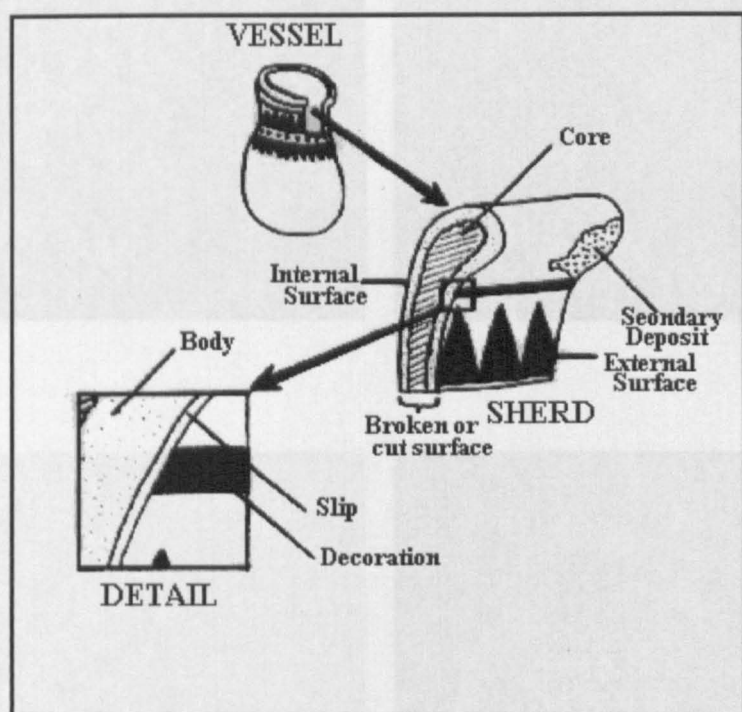
Figure 5.4 Sampling a sherd of archaeological pottery for calcareous nannofossil smear slides. A scraping should be made from the body of the sherd on a freshly cut or broken surface.

are present, it is possible to coax them to the edge of the slide using another toothpick
to apply pressure on the glass.

Place the coverslipped slide on the tissue paper in the fume cupboard to dry. Some

A

B

C

D

every 30 minutes or so, as this can begin to boil. If a coverslip breaks on the hot plate
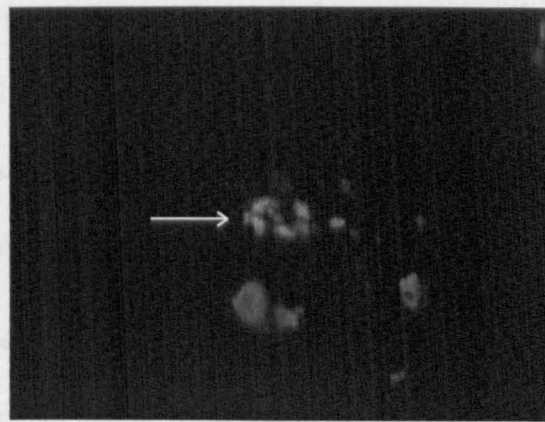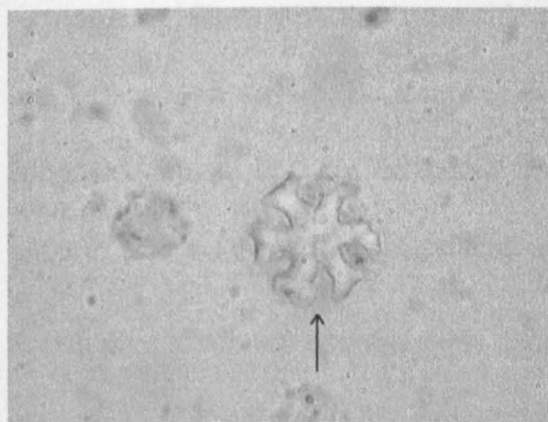
separate the coarse fraction in water. This is done by scraping the sample into a
labelled phial, adding distilled water, shaking the mixture and leaving it to settle for a
few seconds. The calcareous nannofossils should remain in suspension near the
surface while the larger quartz grains will sink rapidly to the bottom. Using a pipette

Figure 5.5 Calcareous nannofossil specimens at the edge of ceramic thin section Kn 84/30 (A and
B) and in a calcareous nannofossil smear slide of sample Kn 95/376 (C and D). A, B and D = XP,
C = PPL. Field of view = 50 μm.

are present, it is possible to coax them to the edge of the slide using another toothpick to apply pressure on the glass.

6. Place the coverslipped slide on the tissue paper in the fume cupboard to dry. Some brands of adhesive, e.g. 'Norland', require ultraviolet light to set, and this should be done in a dark place, using eye protection.

7. Between each sample, discard the tissue and toothpick, wash the knife, wash hands and repeat the procedure. It is worth changing the distilled water and discarding the pipette approximately every ten samples, as these can become contaminated. When using caustic adhesives such as 'Entellan' it may be necessary to use a new pipette every 30 minutes or so, as this can begin to melt. If a coverslip breaks on the hot plate or jumps and lands with the smear-side down, then turn down the heat and wipe the hot surface with a cloth. Be aware of, and take measures to avoid other sources of contamination, such as dirt on sample bags and labcoats, and always think about the order in which the operations should be carried out.

8. Samples which are slightly sandy can be difficult to smear and the adhesive may not spread efficiently leaving large areas of air bubbles. In this case it is useful to separate the coarse fraction in water. This is done by scraping the sample into a labelled phial, adding distilled water, shaking the mixture and leaving it to settle for a few seconds. The calcareous nannofossils should remain in suspension near the surface while the larger mineral grains will sink rapidly to the bottom. Using a pipette, draw off the upper surface of the water and transfer this onto a moistened coverslip on the hot plate. A lower heat is required for this process as spitting may take place if the liquid is dried to quickly. A thin calcareous residue will be left on the dry coverslip,

which will hopefully contain calcareous nannofossils, and should be mounted as before.

5.4 Investigations into the behaviour of calcareous nannofossils in the firing of ceramics

5.4.1 Introduction

One of the most important processes which may affect microfossil assemblages in ceramics is firing (Section 3.6). In order to understand the way in which calcareous nannofossils behave during firing, several experiments are presented in which nannofossiliferous clays were experimentally fired to various temperatures, in oxidising and reducing atmospheres, for different durations. It was suggested in Section 3.6 that variations in these three factors may affect the way in which microfossil assemblages are degraded during the process of firing, and the following experiments reveal useful information with regard to the importance of each of these, as well as the threshold at which calcareous nannofossils are destroyed.

5.4.2 Material

Several kilograms of nannofossiliferous clay were procured during a British Micropalaeontological Society, Nannofossil Working Group, field excursion to the Boulonnais, northern France on 9.3.1997. The material which was collected came from the middle Cretaceous (Albian) Gault Clay Formation, which occurs at the coast near to Strouanne. These sediments contain a very rich, well-preserved calcareous

nannoflora, with reasonably high species diversity, which was ideal for the purpose of these investigations. In addition, the Gault Clay Formation is well-suited to the production of ceramics and appears to have been utilised as a raw material by ancient potters (Burnett and Young n.d., Section 2.3.2.5).

5.4.3 Processing

The Gault Clay was broken up into small (c. 5 cm^3) pieces and allowed to dry in buckets for a period of one week. The dry pieces of clay were then ground to a fine powder using a large pestle and mortar and a 1 mm sieve. The implications of this process are discussed in Section 3.4. The ground sediment was then mixed with a suitable volume of tap water, in a large beaker, to produce a malleable clay paste, which was transferred to ice cube trays and left to dry in a warm place for three days. After this period, the dry cubes of clay had contracted and could easily be removed from the containers, to serve as equidimensional briquettes for the experiments. In order to drive out any remaining water in preparation for firing, the briquettes were then heated in an oven at 30 °C for one day.

5.4.4 Details of the firing process

The firings took place at the Department of Archaeology and Prehistory, University of Sheffield and the Laboratory of Archaeometry at the Demokritos National Centre for Scientific Research, Athens. Three factors were altered between the various firings.

These were, the maximum temperature of firing, the length of firing and the atmosphere in the kiln.

5.4.4.1 Maximum temperature of firing

Gault Clay briquettes were fired at a maximum temperature of 600, 700, 800, 900, 1000 and 1100 °C, for one hour, with constant rates of temperature rise and fall (Fig 5.6). These temperatures were chosen in accordance with the range which was attained by potters in the Bronze Age of Crete, as well as the range at which the thermal alteration and decomposition of calcium carbonate takes place during the firing of ceramics (Rice 1987, 98).

5.4.4.2 Length of firing

At a constant maximum temperature of 600 °C, briquettes were fired for six different durations of 60, 120, 180 and 240 minutes respectively, as illustrated in Figure 5.6. A temperature of 600 °C was chosen in accordance with the lowest estimates for the temperature at which calcium carbonate begins to decompose during the firing of ceramics.

5.4.4.3 Atmosphere of firing

The experimental firings which are described above, were performed in air (an oxidising atmosphere), and a mixture of 4 % H_2 : 94 % Ar per volume (a reducing
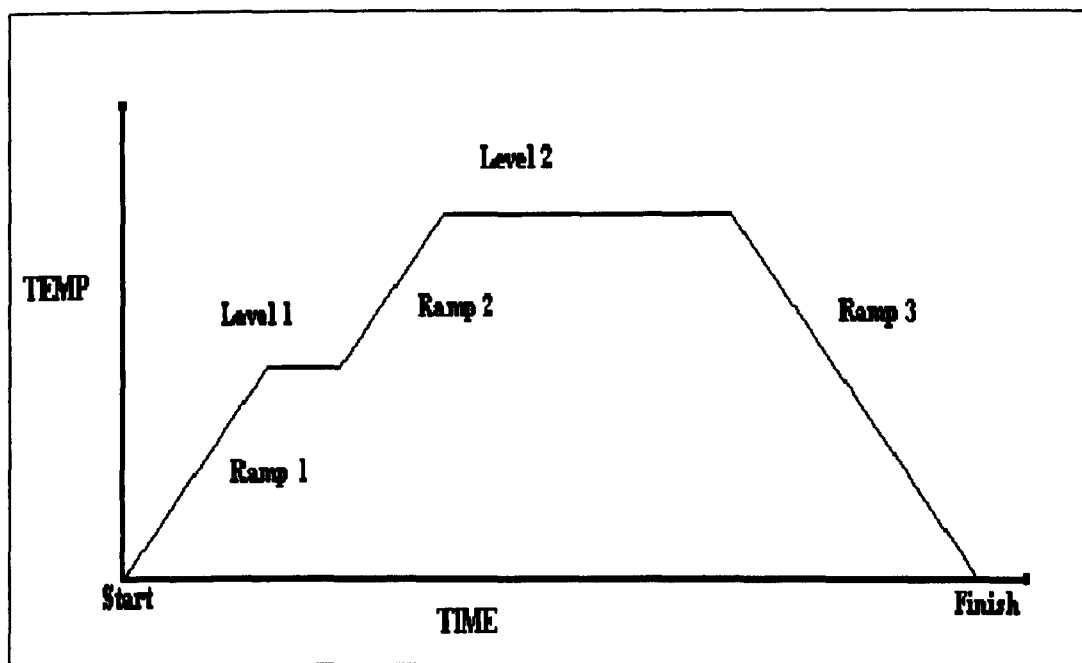
Figure 5.6. Details of the firing programme for experiments into the effect of the maximum temperature and the length of firing on calcareous nannofossil assemblages in ceramics. The temperature and duration of 'level 2' were varied between firings and all other details of the programme were kept constant.

atmosphere). This produced twelve oxidised and twelve reduced Gault Clay samples for analysis.

5.4.5 Calcareous nannofossil analysis

Standard calcareous nannofossil smear slides were produced from each of the 24 fired briquettes, using the method described in Section 5.3.2. Smear slides of the unfired (processed and unprocessed) clay were also prepared in this way and studied in detail, in order to serve as a control with which to compare the fired calcareous nannofossil assemblages.

In all slides, 200 calcareous nannofossil specimens were identified in random fields of view and their overall preservation was noted. In addition, a record was made of the total number of calcareous nannofossil specimens in 25 random fields of view along a single traverse of each slide, in order to document changes in the overall abundance of calcareous nannofossils between successive firings. The results of this analysis are presented in Figures 5.7 to 5.12, and discussed below.

5.4.6 Results

5.4.6.1 The unfired calcareous nannofossil assemblage

The middle Cretaceous (Albian) Gault Clay contains a very diverse, well-preserved calcareous nannoflora. A high rate of species turnover in calcareous nannofossil populations at the time of deposition (Bown *et al.* 1992), combined with the

favourable facies of the Gault Clay, are the reasons for its exceptional assemblage, which is ideal for the purpose of these experiments.

Many different broad structural groups of calcareous nannofossils are represented in the diverse nannoflora of the Gault Clay. These include imbricating placoliths of the family Ellipsagelosphaeraceae, such as *Watznaueria, Cyclagelosphaera and Manivitella*; non-imbricating placolith species of the genera *Biscutum, Polypodorhabdus, Prediscosphaera, Cretarhabdus* and *Discorhabdus*; loxolith murolith coccolith taxa such as *Zeugrhabdotus, Chiastozygus, Staurolithities, Rhagodiscus* and *Eiffellithus*; protolith type muroliths of *Stradnerlithus, Scapholithus* and *Rotelapillus;* and nannoliths, such as species of the genera *Eprolithus, Lithraphidities* and *Nannoconus*. The two most abundant calcareous nannofossil species in the assemblage are *Watznaueria barnesae* and *Zeugrhabdotus erectus*, which together constitute over half of the total nannoflora.

In order to evaluate the effect of clay maceration on the calcareous nannofossil assemblage of the Gault Clay, smear slides were prepared from a sample of the raw sediment, and an unfired processed briquette. In both samples, 200 specimens were identified in random fields of view as in the fired samples (Section 5.4.5), but in addition, a count was made of the number of broken and unbroken Watznaueria specimens in 50 fields of view. By comparing the number of complete and incomplete specimens of this genus in the raw and processed clays it was possible to gauge the degree to which this process damaged the calcareous nannofossils. The species of *Watznaueria* are rather robust (Perch-Nielsen 1985a), however, as they are the most abundant group of calcareous nannofossils in the Gault Clay and can easily be
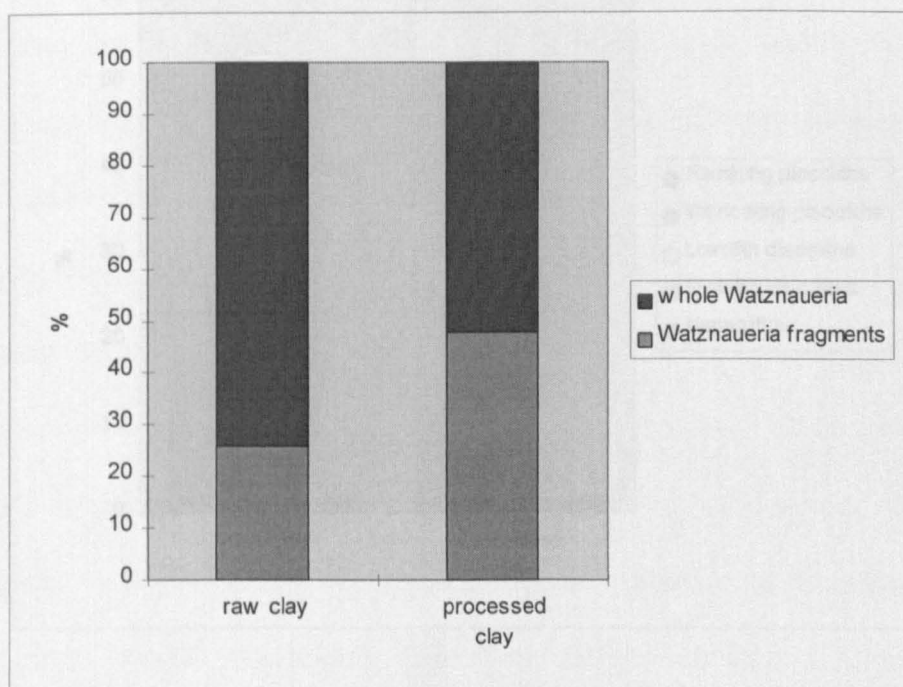
Figure 5.7 The proportion of complete versus broken specimens of *Watznaueria barnesae* in processed and unprocessed Gault Clay.

60

50

40

% 30

20

10

0

Raw clay   Processed
clay

Radiating placoliths
Imbricating placoliths
Loxolith discoliths
Protolith discoliths
Nannoliths

Figure 5.8 The relative abundance of the various calcareous nannofossil structural groups in processed and unprocessed Gault Clay.

recognised from small fragments, the members of this genus were chosen in preference to other, more delicate, less abundant taxa which are not as readily identifiable when broken.

From the results of this analysis, presented in Figure 5.7, it can be seen that the process of clay maceration (Section 5.4.3) significantly alters the ratio of broken to unbroken calcareous nannofossil specimens. An increase in the proportion of broken *Watznaueria* specimens from approximately 25 % to approximately 50 % was recorded as a result of the clay processing technique. The relative abundance of the various calcareous nannofossil structural groups (Figure 5.8) indicates that an increase in the proportion of imbricating placoliths, a decrease in the proportion of the other coccolith groups and no change in the relative abundance of the nannoliths, took place during clay processing. The increase in the relative abundance of the imbricating placoliths is interpreted as being a consequence of the resilience of the genus *Watznaueria* to the physical process of maceration, compared to the more delicate murolith and radiating placolith taxa such as *Zeugrhabdotus, Rhagodiscus, Staurolithities, Biscutum* and members of the family Retecapsaceae. These exhibit a reduction in relative abundance after processing. It is worth noting that, in addition to physical resilience, the identifiability of the various nannofossil taxa from fragments clearly affects their relative abundances between macerated and un-macerated samples. The results of this experiment indicate that the maceration of raw nannofossiliferous clay alters the ratio of broken to unbroken specimens and the relative abundance of the various taxa.

5.4.6.2 The firing atmosphere

From the results of the experiments presented in Figures 5.9 to 5.12, it can be concluded that firing in a reducing atmosphere has a more severe affect on the calcareous nannofossil assemblage of the Gault Clay, than firing at equivalent temperatures in an oxidising atmosphere. This is indicated by the survival of calcareous nannofossils in briquettes fired to a higher temperature in an oxidising atmosphere, than those fired under reducing conditions, as well as the different relative abundances of the various nannofossil groups in briquettes fired to identical temperatures in the two different atmospheres (compare Figures 5.11 and 5.12). If an increase in the relative abundance of imbricating placoliths is assumed to indicate an increase in the severity of firing, then a comparison of the proportion of this group in oxidised and reduced Gault Clay briquettes, which were fired to a maximum temperature of 700 °C supports this interpretation.

5.4.6.3 Maximum temperature of firing

Calcareous nannofossils were present in the smear slides of Gault Clay briquettes fired up to temperatures of 700 and 800 °C under reducing and oxidising atmospheres respectively. After this, the samples were barren and low in calcite (Figures 5.9 and 5.10).

Distinct changes took place in the composition of the calcareous nannofossil assemblage with increasing temperature, as represented in Figures 5.9 to 5.12. The general trend is that of a decrease in the overall abundance of calcareous nannofossils (Figures 5.9 and 5.10), as well as a reduction in the diversity of the assemblage
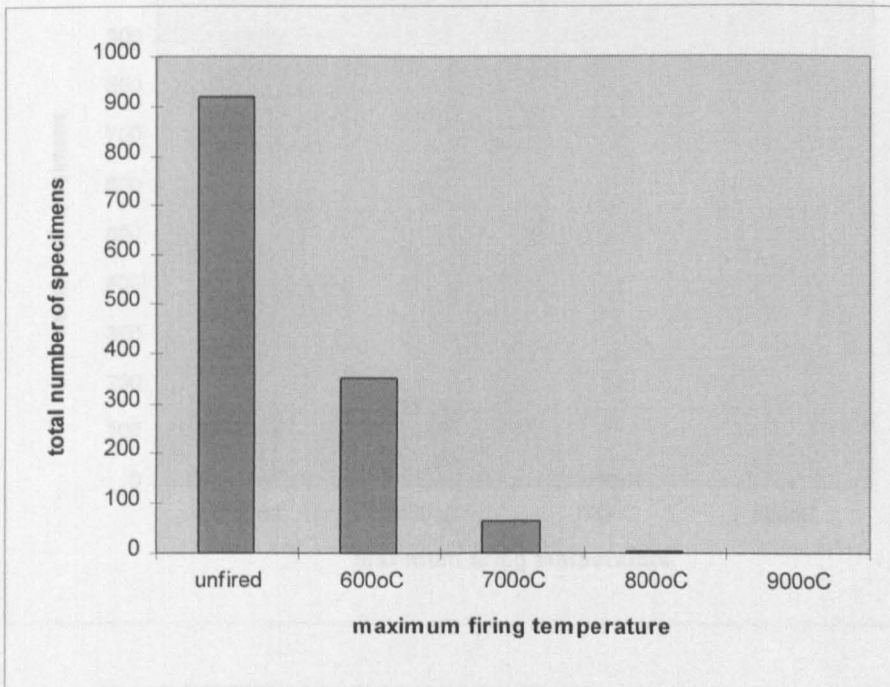
Figure 5.9 The overall abundance of calcareous nannofossils in Gault Clay fired at different temperatures in an oxidising atmosphere, as represented by the total number of specimens in 25 random fields of view.
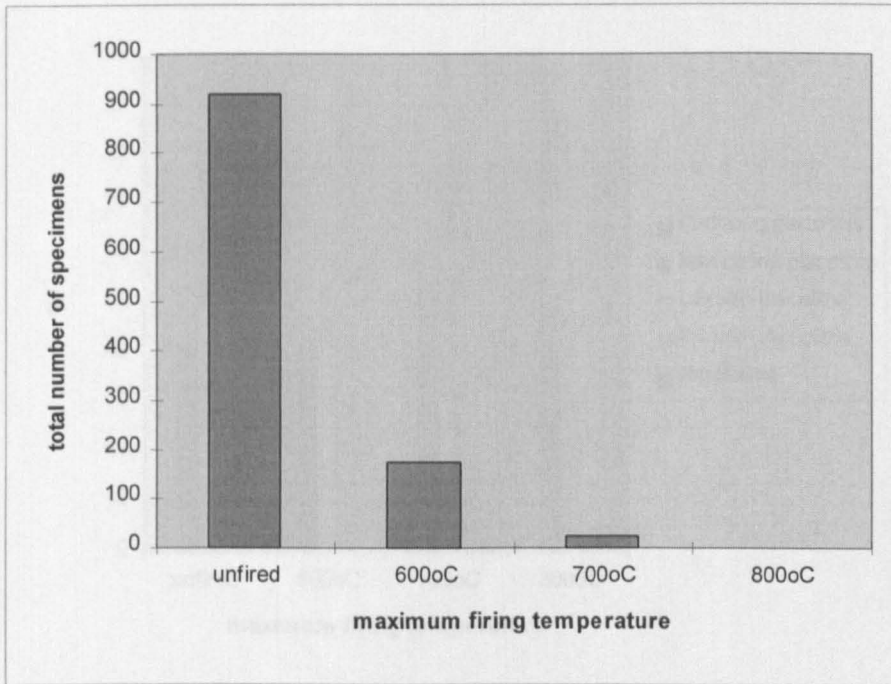
Figure 5.10 The overall abundance of calcareous nannofossils in Gault Clay fired at different temperatures in a reducing atmosphere, as represented by the total number of specimens in 25 random fields of view.
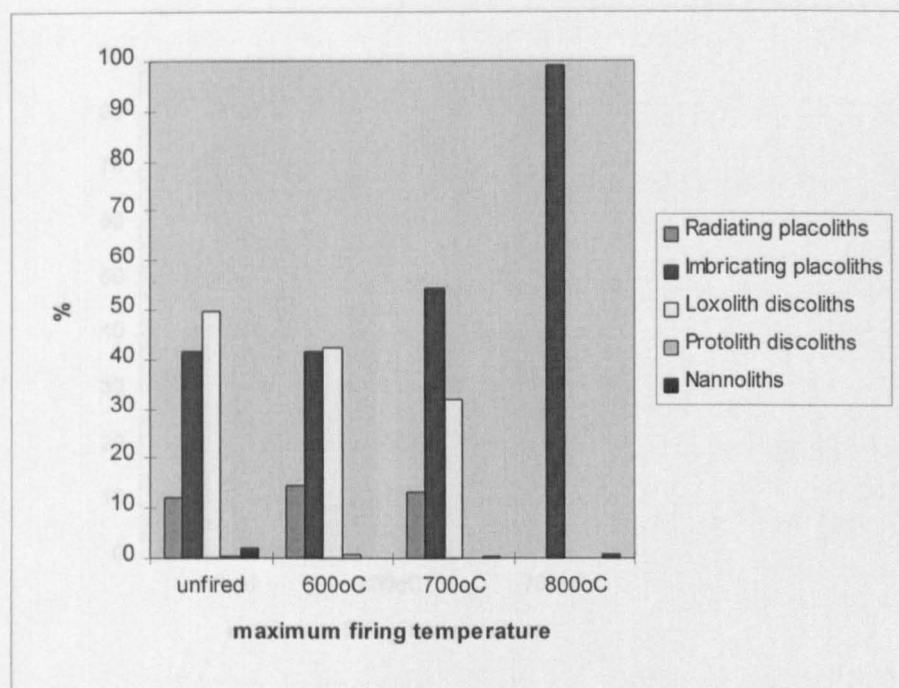
Figure 5.11 The relative abundance of the various calcareous nannofossil structural groups in Gault Clay fired at different temperatures in an oxidising atmosphere.

through a decrease in the relative abundance of the more delicate taxa, or those which were poorly represented in the original nannoflora, and a relative increase in the abundance of the more robust taxa, e.g. *Watznaueria*, which eventually dominate the assemblage.

The changes in the relative abundance of the various structural groups of calcareous nannofossils which took place with increasing maximum firing temperature are roughly comparable to those seen in the previous experiment (Section 5.4.5.2), i.e. a decrease in the abundance of delicate radiating and imbricating placoliths.

5.4.6.4 Length of firing

The changes in the relative abundance of calcareous nannofossils and the relative abundance of the various structural groups of structural groups in Gault Clay briquettes with increasing firing duration (Figures 5.13 and 5.14) follow the trends seen in the previous experiments. There is a constant decrease in the overall abundance of calcareous nannofossils with increasing firing duration, and an increase in the proportion of imbricating placoliths relative to radiating placoliths and microliths.

It can be concluded from the results of this experiment, that longevity, in addition to the maximum temperature of firing, has an effect on the nature of the calcareous nannofossil assemblage in ceramics. The alteration resulting from a 100 °C increase in maximum temperature is more severe than that produced by sustaining this

Figure 5.12 The relative abundance of the various calcareous nannofossil structural groups in Gault Clay fired at different temperatures in a reducing atmosphere.

through a decrease in the relative abundance of the more delicate taxa, or those which were poorly represented in the original nannoflora, and a relative increase in the abundance of the more robust taxa, e.g. *Watznaueria*, which eventually dominate the assemblage.

The changes in the relative abundance of the various structural groups of calcareous nannofossils which took place with increasing maximum firing temperature are roughly comparable to those seen in the processing experiment (Section 5.4.6.2), i.e. a decrease in the abundance of all groups except the imbricating placoliths.

5.4.6.4 Length of firing

The changes which took place in the overall abundance of calcareous nannofossils and the relative proportion of the various calcareous nannofossil structural groups in Gault Clay briquettes with increasing firing duration (Figures 5.13 and 5.14), follow the trends seen in the previous experiments. There is a constant decrease in the overall abundance of calcareous nannofossils with increasing firing duration, and an increase in the proportion of imbricating placoliths, relative to radiating placoliths and muroliths.

It can be concluded from the results of this experiment, that longevity, in addition to the maximum temperature of firing, has an effect on the nature of the calcareous nannofossil assemblages in ceramics. The alteration resulting from a 100 °C increase in maximum temperature is more severe than those produced by sustaining this temperature for one hour. However, the nature of these changes with regard to the
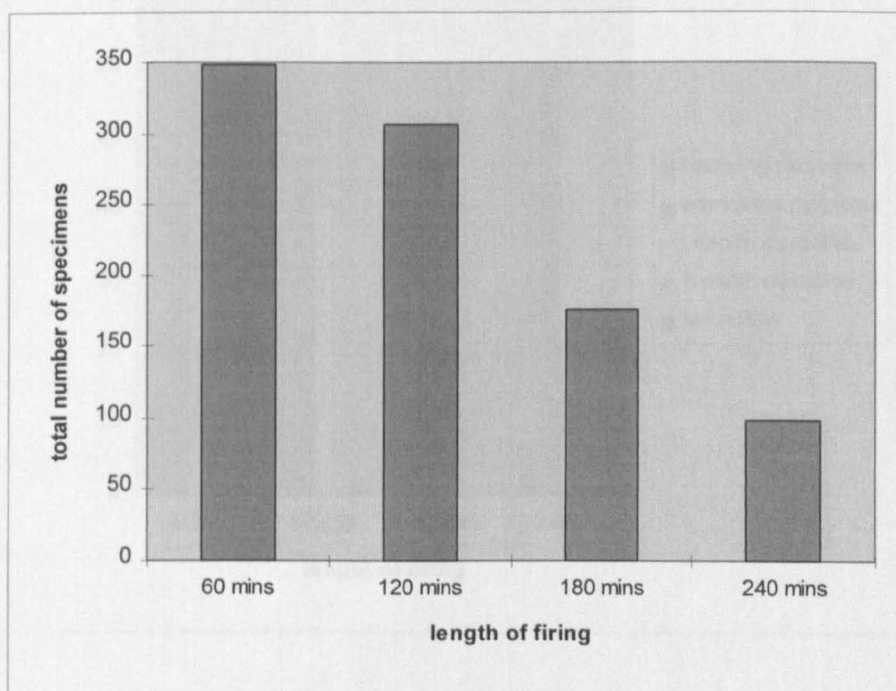
Figure 5.13 **The overall abundance of calcareous nannofossils in Gault Clay fired for different durations at 600 °C in an oxidising atmosphere, as represented by the total number of specimens in 25 random fields of view.**

Figure 5.14 The relative abundance of the various calcareous nannofossil structural groups in Gault Clay fired for different durations at 600 °C in an oxidising atmosphere.

overall abundance of calcareous nannofossils and the relative abundance of the various structural groups of calcareous nannofossils, are the same in both cases.

5.4.7 Comparison with existing archaeological data

Calcium carbonate ($CaCO_3$), which can be present in many forms within the raw materials used for ceramic manufacture, may be altered upon firing, to CaO. After firing, the CaO, which has an affinity for water, re-hydrates by absorbing moisture from the air and becomes $Ca(OH)_2$. This process, which is accompanied by a release of heat and a volume increase, presents a serious problem where large calcium carbonate inclusions are present in ceramics, as their expansion can damage the fired pottery to varying degrees, ranging from the 'lime popping' of the vessel surface to its complete destruction.

Various estimates, ranging from as low as 600 °C to as high as 900 °C, have been proposed by different authors as the critical temperature at which the decomposition of calcium carbonate begins during the firing of ceramics (Rice 1987, 98). This range reflects the complexity of the firing process, in which other factors such as the duration of firing and the firing atmosphere, as well as the clay and calcite composition may also influence the threshold at which this process takes place (Fisher 1927; Tite and Maniatis 1975; Maniatis *et al.* 1983).

The level at which calcareous nannofossils were destroyed during the experimental firing of Gault Clay in this study (between 700 and 900 °C, Figures 5.9 and 5.10) is in agreement with the temperature range which has been proposed for the decomposition of calcite. The differences in the overall and relative abundance of calcareous

nannofossils in Gault Clay fired for different lengths of time at a temperature of 600 °C (Figures 5.13 and 5.14), confirm the above suggestion, that time is also an important factor in the alteration of calcium carbonate during firing. With this in mind, the degree to which calcareous nannofossil assemblages are degraded during the firing of ceramics, is likely to be the consequence of a combination of the maximum temperature of firing, as well as the duration of the firing process, i.e. the 'degree of firing', 'equivalent firing temperature' or 'work heat' (Roberts 1963; Nelson 1984).

Firing Gault Clay briquettes in a reducing atmosphere was found to have a more severe affect on their calcareous nannofossil assemblage than firing them to the equivalent temperatures in an oxidising atmosphere (Figures 5.9 and 5.10). Calcareous nannofossils were destroyed at lower temperatures in the absence of oxygen, which indicates that their alteration takes place at a higher temperature or proceeds less rapidly, in an oxidising atmosphere. This discovery contradicts with the findings of Laird and Worcester (1956, 555), who stated that, the undesirable effects of lime spalling or 'lime blowing' may be avoided by "firing limestone-containing bricks in a reducing atmosphere". If, as these authors appear to suggest, the absence of oxygen during firing inhibits the decomposition of calcite, then another mechanism may be responsible for the degradation and eventual disappearance of calcareous nannofossils in a reducing atmosphere, at around 700-800 °C in these experiments.

No lime spalling was observed in any of the Gault Clay briquettes, even several months after firing. This may be explained by the fact that the calcium carbonate in the Gault Clay briquettes (largely calcareous nannofossil specimens), was extremely

fine (usually < 10 μm), so that the disruptive force produced by its re-hydration after firing, which is proportional to the square of the radius of the particle (Möller 1908), was not very significant.

In the Gault Clay briquettes fired above 800 and 900 °C in a reducing and an oxidising atmosphere respectively, very little calcium carbonate was present (as seen in the calcareous nannofossil smear slides of these samples). This indicates that the fine calcium carbonate in these briquettes may have reacted with the clay minerals to form calcium silicates. Butterworth (1956) claimed that the calcium carbonate 'melts' and is converted into other substances at temperatures over 1000 °C, a higher figure than indicated by the present study. However, Tite and Maniatis (1975) suggested that the process is accelerated in a reducing atmosphere or when the calcite is very fine. These latter authors experimentally fired Gault Clay (as well as several other British clays), and discovered that the first indications of vitrification can occur at a temperature of 840 °C in an oxidising atmosphere, and lower still in a reducing atmosphere.

Therefore, it appears that the calcareous nannofossil specimens in the Gault Clay briquettes were degraded by the conversion of their $CaCO_3$ to CaO and its re-hydration after firing, at temperatures as low as 600 °C. Because of their small size, the re-hydration of the altered calcareous nannofossil specimens after firing, did not exert sufficient a force on the ceramic briquettes to cause any noticeable structural damage. At higher temperatures (c. 700-800 °C in a reducing atmosphere and c. 800-900 °C in an oxidising atmosphere), the calcareous nannofossils appear to have reacted with the surrounding clay minerals. As a result, the briquettes which were

fired above these temperatures, were barren and did not contain any calcium carbonate. This process seems to have taken place at lower temperatures than those quoted by other authors (e.g. Butterworth 1956), and may be explained by the very fine nature of the calcium carbonate in the Gault Clay (usually < 10 μm). In addition, the lower temperature which was recorded for the reaction of calcium carbonate with the clay matrix of the Gault Clay in the reduction firing experiments, is in agreement that the suggestion of Tite and Maniatis (1975) that this process is accelerated in the absence of oxygen. In which case, the discovery by Laird and Worcester (1956), that lime spalling can be combated by firing ceramics in a reducing atmosphere, may be due to the premature reaction of calcium carbonate with the clay minerals, rather than a slower alteration of $CaCO_3$, in the absence of oxygen.

There exist several other approaches which may be adopted in order to avoid the re-hydration of CaO in fired ceramics (and therefore the undesirable affects of spalling), in addition to those suggested by Laird and Worcester (1956). The most commonly cited method is the addition of salt to the ceramic paste during its preparation. Salts may already be present in significant amounts in some natural clays which are utilised by potters (Arnold 1971). However, more commonly salts or salt water must be added to the clay to prevent spalling in this way (Rye 1976). Upon firing, the salts react with the $CaCO_3$ preventing its transformation into CaO (see equation below). The resulting compounds are relatively stable after firing and have no affect on the ceramic body.

$$CaCO_3 + 2NaCl \longrightarrow CaCl_2 + Na_2CO_3$$

In order to determine what affect the addition of salt has on the behaviour of calcareous nannofossils during firing, two additional firings were made, with saline Gault Clay briquettes. In the preparation of these briquettes, macerated Gault Clay was mixed with tap water saturated with household table salt. The briquettes were fired at maximum temperatures of 800 and 900 °C in an oxidising atmosphere for one hour. These temperatures were chosen in order to determine whether the destruction of calcareous nannofossils at these temperatures in the previous experiments, was a result of the reaction of their $CaCO_3$ with the clay minerals of the Gault Clay (as suggested above), or due to their conversion to CaO and its subsequent re-hydration after firing.

The analysis of smear slides, prepared from the resulting briquettes indicated that the addition of salt had no affect on the behaviour of calcareous nannofossil in Gault Clay which was fired to these temperatures. A very poorly preserved calcareous nannofossil assemblage, dominated by the genus *Watznaueria* was observed in the briquettes fired to 800 °C, and the higher-fired samples were barren. These results seem to support the suggestion that the it is the reaction of $CaCO_3$ with the clay minerals of fired Gault Clay, rather than the post-firing re-hydration of CaO, which is responsible for the disappearance of calcareous nannofossils which was observed between 800 and 900 °C in the above experiments.

5.4.8 Discussion and consequences for the analysis of calcareous nannofossils in ceramics

The results of these experiments have several consequences for the analysis of calcareous nannofossils from archaeological ceramics. Firstly, it is clear that calcareous nannofossils can be removed from ceramics during firing, at temperatures as low as 800 °C (Figure 5.10). This discovery casts doubt over the use of the presence/absence of calcareous nannofossils as a means of classifying ceramics. Although the temperatures at which calcareous nannofossils were destroyed during the firing of Gault Clay briquettes (< 900 °C), may not be representative for all nannofossiliferous ceramics, these figures may indicate that a significant proportion of medium-fired archaeological ceramics may have once contained calcareous nannofossils, but were rendered barren as a result of high firing (Section 11.2).

The differential alteration of the various groups of calcareous nannofossils in the fired Gault Clay samples, is suspected to be related to several factors, including their resilience to the physical stresses induced during and after firing, and the ease with which they can be identified in poorly-preserved assemblages. This discovery, has serious implications for the direct comparison of calcareous nannofossil assemblages from archaeological ceramics fired at different temperatures, as well as the applicability of quantitative biostratigraphic dating techniques (e.g. the Pliocene subzonal scheme of Driever 1988, Section 5.6.2.2) in the absence of conventional marker species.

The degradation of calcareous nannofossil assemblages by progressive firing, which has been demonstrated in these experiments, results in a continual decrease in the

quality of information, which can be attained by their study. The main potential of calcareous nannofossils for the study of ceramics is their biostratigraphic utility (Sections 5.9), which can be used to determine the precise geological date of the raw materials of ceramic manufacture (Troja *et al.* 1996; Burnett and Young n.d.). The latter authors identified the raw material of a crudely fired Bronze Age sherd as the Early Cretaceous (Albian) Gault Clay by analysing its diverse, well-preserved calcareous nannofossil assemblage. However, in comparison, the heavily degraded assemblage of the Gault Clay briquettes, fired to 700 °C in an oxidising atmosphere, in the experiments presented above (Figure 5.9), cannot be interpreted biostratigraphically with anywhere near the same level of precision.

If calcareous nannofossils in ceramics are affected by the transformation of their $CaCO_3$ into CaO and its post-firing re-hydration to $Ca(OH)_2$ as suggested above, then they are also likely to experience an associated size increase. This could affect the applicability of biometric biostratigraphic events, such as the important 'small *Reticulofenestra* interval' of Young *et al.* (1994), which is a period of the Late Miocene (Tortonian), during which the maximum diameter of coccoliths belonging to the nominate genus did not exceed 7 µm (Section AI.8.2.1), or Driever's (1988) size increase in large 'reticulofenestrid' coccoliths which takes place in the Early Pliocene (Section AI.8.3.1).

In order to determine the extent to which the size of calcareous nannofossil specimens are affected by this volume expansion, 100 complete specimens of the species *Watznaueria barnesae* were measured in random fields of view from the smear slides of unfired Gault Clay and briquettes fired to a maximum temperature of 700 °C in an

oxidising atmosphere. A mean of the various measurements in both samples, indicated that if a size increase had occurred, as a result of the volume expansion of these calcareous nannofossils after firing, it was very small and not of concern.

An estimate of the maximum temperature in the firing of ancient ceramics can be attained by studying various physical, mineralogical or chemical properties (see Rice 1987, 427-434 for a review). These 'archaeothermometric' techniques have distinct advantages and disadvantages, and are usually only applicable to the determination of specific temperature ranges, nevertheless they offer a means of attaining a rough figure of firing temperature.

Despite the progressive alteration of calcareous nannofossil assemblages during the firing of ceramics, which has been demonstrated in the above experiments, their analysis has very little potential as a method for archaeothermometry, due to the nature of the changes which take place as well as the lack of knowledge about the state of the original calcareous nannofossil assemblages of archaeological ceramics. Nevertheless, the presence/absence of this group of microfossils may well be used to make simple inferences about the 'degree of firing' in certain circumstances. By considering the thresholds which were determined for the disappearance of calcareous nannofossils in oxidation and reduction fired Gault Clay (Section 5.4.6.3), it may be possible to infer that certain samples of archaeological ceramics were fired below a specific maximum temperature, depending upon the atmosphere of firing.

Likewise, in coherent fabric groups which contain fossiliferous and non-fossiliferous (barren) samples it may be possible to use the presence/absence of calcareous nannofossils to indicate those samples which were lower, and those which were
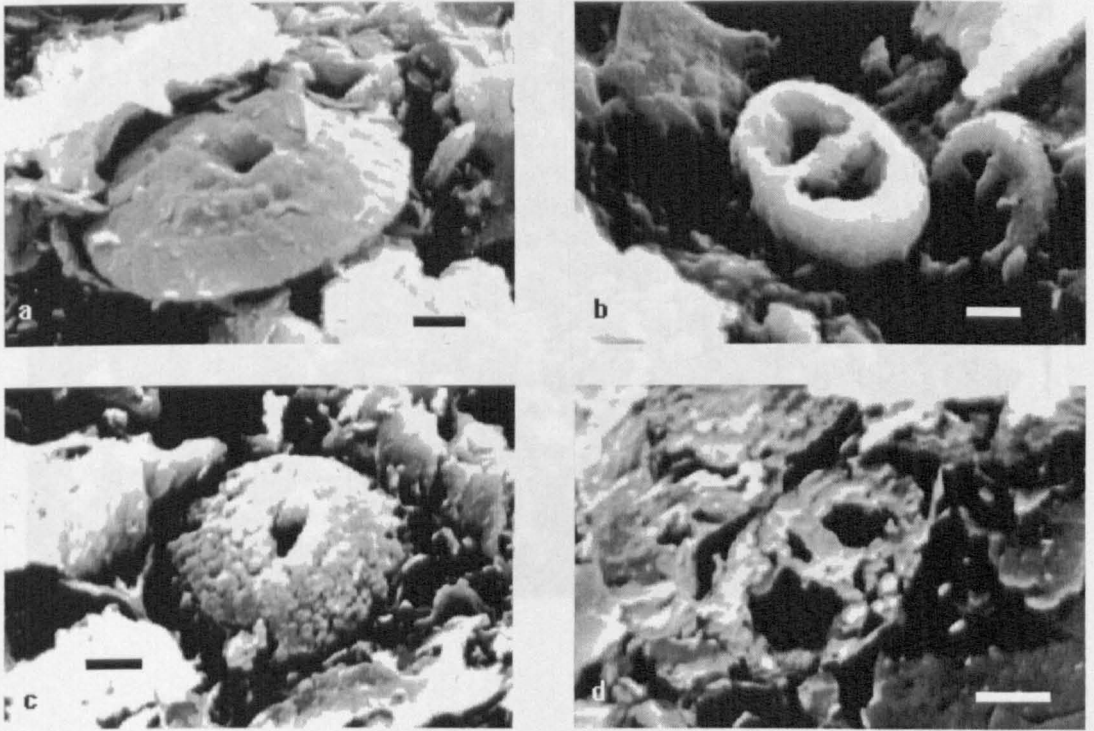
Figure 5.15 The degradation of calcareous nannofossils during the experimental firing of Gault Clay, as seen in the SEM. *Watznaueria barnesae* (a. and c.) and *Zeugrhabdotus erectus* (b. and d.) from Gault Clay fired at 600 °C (a. and b.) and 800 °C (c. and d.). Scale bars = 1 µm.

Figure 5.16 The broken surface of a Gault Clay briquette fired to 600 °C in an oxidising atmosphere. Compare the clay vitrification and abundance and preservation of calcareous nannofossil specimens to that in Figure 5.17 below.

Figure 5.17 The broken surface of a Gault Clay briquette fired to 800 °C in an oxidising atmosphere. Compare the clay vitrification, and abundance and preservation of calcareous nannofossil specimens to that in Figure 5.16 above.

higher fired (Section 11.2). In this case, it may again be possible, by referring to the firing atmosphere (as determined macroscopically) to indicate a broad temperature range for nannofossiliferous and barren samples (e.g. < 800 °C or > 800 °C). However, as indicated by the above experiments, other factors in addition the maximum temperature of firing (e.g. the duration of the firing process and the firing atmosphere) are also important in determining the level at which calcareous nannofossils are destroyed, and therefore such broad inferences are likely to be less than accurate.

5.5 Investigations into the behaviour of calcareous nannofossils during the use of ceramics

5.5.1 Introduction

In order to determine how calcareous nannofossil assemblages in archaeological ceramics may have been altered during usage, a set of simple experiments were carried out using fired Gault Clay briquettes. In Section 3.7, it was suggested that the direct heating of cooking wares and the corrosive properties of such commodities as oil, wine and vinegar, contained within storage wares, may affect the nature of calcareous microfossil assemblages in these types of ceramics.

Three Gault Clay briquettes, fired to 600 °C in an oxidising atmosphere, were placed in malt vinegar (pH 3), white wine and olive oil respectively, for one day, then dried in an oven at 30 °C. In addition, one briquette was held directly above a naked flame for a period of 30 minutes and then allowed to cool. Calcareous nannofossil smear slides were prepared from the four treated briquettes, these were analysed in the

manner which is described in the firing experiments (Section 5.4.5), and the results were compared with those of the untreated, fired Gault Clay.

5.5.2 Results

The treatment of fired Gault Clay briquettes with vinegar resulted in the removal of all calcareous nannofossil specimens. This is to be expected, given the acidic nature of vinegar which readily dissolves calcareous matter. Immersing samples in olive oil and wine was found to have little affect on the calcareous nannofossil assemblage of the Gault Clay.

It was surprising to find that the surface layer of the Gault Clay briquette which was held above a naked flame, contained a reasonably well-preserved calcareous nannofossil assemblage. By comparing the quantitative analysis of this sample with that of the untreated Gault Clay briquette (Figures 5.15 and 5.16,) it can be seen that the direct heating reduced the overall abundance and altered the relative proportions of the various groups of calcareous nannofossils contained within the briquette. Whilst a reasonably well-preserved assemblage was contained within the surface layer of the briquette, which became red-hot during the experiment, frequent episodes of continual heating such as that which may have taken place in antiquity are likely to have had a more severe effect on calcareous nannofossil assemblages in cooking vessels.
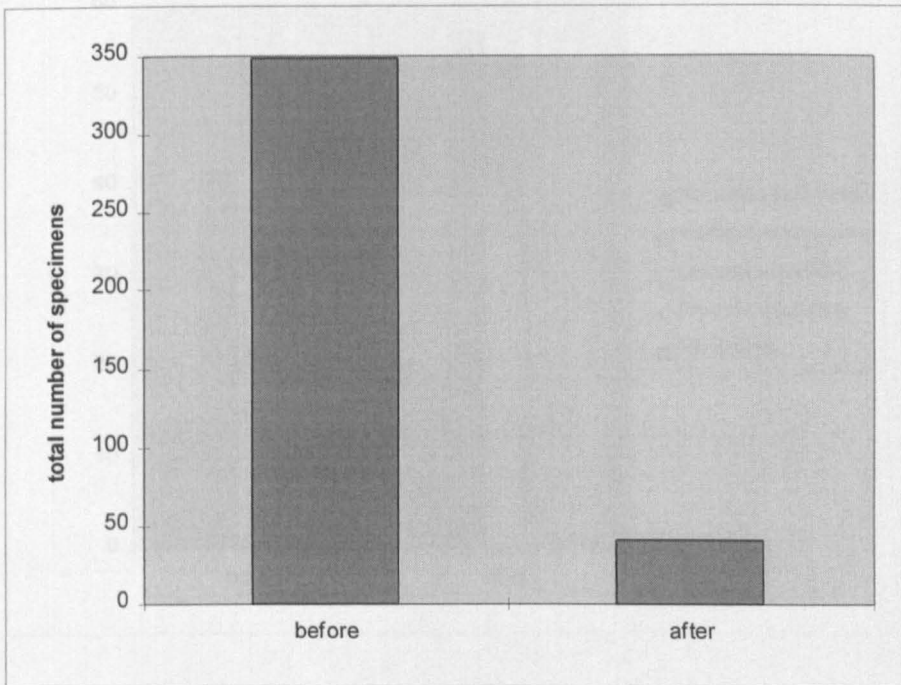
Figure 5.18   The overall abundance of calcareous nannofossils in Gault Clay fired at a temperature of 600 °C for 1 hour, before and after direct heating above a naked flame for 30 minutes, as represented by the total number of specimens in 25 random fields of view.

5.6 Calcareous Nannofossil biostratigraphy of the Mediterranean Neogene

5.6.1 Introduction

In order to interpret the geological age of the raw materials utilised in the manufacture of archaeological ceramics through the analysis of their calcareous nannofossil assemblages, it is necessary to review the various biostratigraphic zonations which have been proposed for this group of microfossils, and identify those schemes which are most suitable for our purposes. As outlined in Chapter 1, we are concerned, in the present report, with the nannofossil zonations which occur extensively on Crete. There are numerous zonations for the Neogene. The most important conventional calcareous nannofossil schemes, together with their limitations, are briefly considered below.

5.6.1 Applicability of the standard zonations

The world-wide 'standard' zonation for the entire Cenozoic Era is the compilation of Martini (1971), which includes 21 nannoplankton zones for the Neogene (NN 1-NN 21) and is based upon the data of Bramlette and Wilcoxon (1967) and Gartner (1969). An alternative standard zonation for this time period is that of Okada and Bukry (1980), which is a refined and coded version of the zonal and subzonal schemes of Bukry (1971, 1973, 1975), established for the routine examination of DSDP material (Schmidt 1973).

Figure 5.19 The relative abundance of the various calcareous nannofossil structural groups in Gault Clay fired at a temperature of 600 °C for 1 hour, before and after direct heating above a naked flame.

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are most suitable for our purposes. As outlined in Chapter 4, we are concerned, in the

present report, with the late Neogene sediments, which occur extensively on Crete.

There are numerous difficulties involved in the application of the 'standard'

calcareous nannofossil zonations for this time period in the Mediterranean, as outlined

below.

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(Schmidt 1973).

Amaurolithus primus (Bukry and Percival 1971) Gartner and Bukry (1975)

Catinaster calyculus Martini and Bramlette (1963)

Catinaster coalitus Martini and Bramlette (1963)

Ceratolithus acutus Gartner and Bukry (1974)

Ceratolithus rugosus Bukry and Bramlette (1968)

Discoaster calcaris Gartner (1967)

Discoaster druggii Bramlette and Wilcoxon (1967)

Discoaster hamatus Martini and Bramlette (1963)

Discoaster kugleri Martini and Bramlette (1963)

Discoaster neoerectus Bukry (1971)

Discoaster quinqueramus Gartner (1969)

Sphenolithus belemnos Bramlette and Wilcoxon (1967)

Triquetorhabdulus carinatus Martini (1965)

Figure 5.20 Neogene calcareous nannofossil species which are rare, absent or atypically developed in the Mediterranean (Theodoridis 1984).

The zonations of Martini (1971) and Okada and Bukry (1980) are both based upon oceanic sediments and utilise extensively, the Last Occurrences (LOs) of calcareous nannofossil species (Raffi and Rio 1979). As such, they are not universally applicable, but provide a framework for finer, local subdivisions (Perch-Nielsen 1985b). Nowhere has this been more true than in the Mediterranean. Here, the use of the standard nannofossil zonations is "more or less restricted to several intervals" (Bizon and Müller 1977, 382). For example, Theodoridis (1984, 47) noted that "of the 14 index species Bukry (1975) used for the subdivision of the Miocene, eight are absent, atypical or extremely rare in the Mediterranean region" (Figure 5.20), and Raffi andRio (1979) were only capable of applying Bukry's scheme on a broad zonal level in their Pliocene DSDP material from Site 132 (Tyrrhenian sea). The Neogene interval of the standard zonations can also be difficult to apply elsewhere (Bizon and Müller 1977; Young 1990), for example, in the north-east North Atlantic (Martini 1979) and the Norwegian-Greenland Sea (Müller 1976).

The main reason for the limited applicability of the standard zonations in the Neogene of marginal seas and high latitude sediments is their reliance upon the open-marine, low-latitude species of *Discoaster* and the deep-water genera *Ceratolithus* and *Amaurolithus*. In general, representatives of *Discoaster* and *Ceratolithus/Amaurolithus* are rare (< 1 %), in Mediterranean Neogene calcareous nannofossil assemblages (Driever 1988), and many important species, such as *Discoaster neoerectus, Discoaster quinqueramus* and *Discoaster hamatus* appear to be absent. Another difficulty is the atypical development of certain taxa, particularly discoasters, in the Mediterranean, as compared to the open-ocean (Theodoridis 1984). This causes "the uncertain classification of forms to one or another species" (Bizon

and Müller 1977, 382), and is likely to be a result of different ecological conditions and a "more regional development of nannofossil assemblages" (Müller 1978, 743).

Further difficulties arise when attempting to apply the standard zonations to the Mediterranean on-land record, where discoasters and ceratoliths are even more scarce (Rio 1982), and the "problem of reworking is overwhelming" (Raffi and Rio 1979, 143), making the last occurrences of nannofossil taxa very difficult to use (Schmidt 1973).

5.6.2 Mediterranean Neogene calcareous nannofossil zonations

It appears from the evidence provided by calcareous nannofossils, that the Mediterranean behaved as a "distinct planktonic biogeographic province" in the late Neogene (Rio *et al.* 1990, 513). As such, it has been necessary for biostratigraphers working in the Mediterranean to make adjustments to the standard zonations or propose Mediterranean-specific schemes for this time period, based upon species "which are normally neglected in extra-Mediterranean biostratigraphy" (Rio 1982, 326).

Early calcareous nannofossil zonations of the Mediterranean Neogene were "rough and partial" (Theodoridis 1984, 50). Cati and Borsetti (1970) constructed a broad scheme for the Miocene based upon discoasters from on-land sections in central Italy. However, many of these have since been found to be preservational morphotypes of long ranging species, and as such, are unsuitable as markers. A similar mistake was made by Schmidt (1973), who proposed a broad subdivision of the Late Miocene and Pliocene of the southern Aegean area, into six interval zones and one assemblage

zone. Schmidt's scheme was based solely upon land-based sections from Crete, Rhodes, Kassos, Karpathos and Gavdos, and as such it should be directly relevant to the present study. However, many of his zones are also defined by so-called "preservational species" (Theodoridis 1984, 51), or other forms which are extremely rare in the Mediterranean. As one of the earliest detailed studies on calcareous nannofossil biostratigraphy from Crete and the southern Aegean, Schmidt (1973) is still of some use to this report as well as for the Mediterranean as a whole (Müller 1978), and has experienced limited use by other biostratigraphers (e.g. Dermitzakis and Theodoridis 1978, eastern Crete).

In order to date and correlate the extensive Neogene sediments penetrated by the first phase of deep sea drilling (DSDP) in the Mediterranean, the standard calcareous nannofossil zonations, based on open-ocean sediments, were modified by Bukry (1973), Stradner (1973) and Müller (1978). Bukry (1973) applied his own open-ocean zonation, developed for the analysis of oceanic DSDP material, to the Miocene and Pliocene sediments of Site 132, Leg 13, with varying success. Some samples could be dated to one or another of Bukry's zones, but most of the material was given broader age assignments (e.g. "probably Late Miocene; Late Miocene to Early Pliocene; late Neogene", 823-827) due to "a provincial nannofossil suite with key species lacking" (Bukry 1973, 823). The standard zonation of Martini (1971) was preferred by Stradner (1973) on Leg 13, and Müller (1978) on Leg 42A. However, both workers encountered difficulties in applying this low-latitude, open-ocean zonation to the sediments of the Mediterranean. Müller (1978) found ten of Martini's marker species to be rare, missing or sporadic in the Early Miocene and Late Miocene to Early Pliocene of the Mediterranean, and proposed alternative taxa that could be used to

recognise the boundaries of his various NN zones. Müller (1978) also reported the atypical shape of some species as well as heavy overgrowth on discoasters, both of which complicated the recognition of Martini's zones in the Mediterranean. However, she commented that "with regard to the whole assemblages, the zones are recognisable" (Müller 1978, 727).

Ellis (1979) claimed to have successfully applied the zonation of Bukry (1973) to Miocene, Pliocene and Pleistocene sediments from the eastern Mediterranean sites of DSDP Leg 42A, as well as on-land Mediterranean stratotype sections. Contrary to the observations of many other biostratigraphers of the Mediterranean Neogene, Ellis stated that "open marine, warm water species of *Discoaster* are present in significant numbers, throughout the Miocene and Pliocene" and "numerous specimens of *Ceratolithus* and *Amaurolithus* occur in earliest Pliocene assemblages", indicating that "cool-water influences in the eastern Mediterranean ... have not appreciably diminished the value of low-latitiude nannoplankton zonation schemes" (1979, 401). By making several adjustments to the zonations of Bukry (1973; 1975), Ellis recognised all 13 zones and 19 of the 21 subzones for the Neogene period.

Raffi and Rio (1979) reconfirmed the difficulties involved in using the genus *Ceratolithus* for Mediterranean biostratigraphy in their reassessment of the Pliocene and Pleistocene nannofossil biostratigraphy of DSDP Site 132, Leg 13 (Tyrrhenian Sea), which was studied initially by Bukry (1973). By assessing the validity of the various markers used in Martini (1971) and Bukry (1973; 1975), Raffi and Rio (1979) proposed a scheme which was rather similar to the standard zonations for the Pliocene, but achieved a finer subdivision of the Pleistocene period by utilising

several new datum events. They quantified the changes in abundance of various species through the Plio-Pleistocene by counting as many as 3000 specimens per sample and used the following as markers: the last common occurrence of *Amaurolithus spp.*, the first common occurrence of *Discoaster asymmetricus* and the end of the dominance of small specimens in the population of *Gephyrocapsa*. This scheme represented the beginning of a more scientific approach to the study of Mediterranean Neogene biostratigraphy, which was continued by Driever (1984; 1988), Theodoridis (1984) and Raffi *et al.* (1990), and has resulted in a surprisingly fine subdivision of the Pliocene and Pleistocene.

Driever (1981) carried out a quantitative study of discoasters from Pliocene on-land sections on Crete and Sicily, and established changes in the relative abundance of the various species, in order to identify alternative events with which to subdivide the Pliocene period, due to the difficulties which were involved in determining the LO of *Reticulofenestra pseudoumbilica* and the absence of *Ceratolithus/Amaurolithus* in these areas. By counting 100 discoasters per sample and using a simple but effective key which is discussed in Section 5.6.2.2, Driever (1981) divided his sections into six non-mutually exclusive units based upon the acmes of *Discoaster asymmetricus* and *Discoaster tamalis,* and the 'paracme' of *Discoaster pentaradiatus* (syn. *Discoaster quintatus* Driever 1988; *Eu-discoaster misconceptus* Theodoridis 1984). On their own, the six subdivisions are of limited correlative value, however "within a framework of additional biostratigraphic data ... these intervals may greatly refine biostratigraphy" (Driever 1981, 447). This is demonstrated in Driever's monograph of 1988 (Section 5.6.2.2), in which he utilised several horizons based upon *Discoaster*

Standards			Mediterranean Specific Calcareous Nannofossil Zonations						
	M & W	O & B	C & B	Schmidt	Muller	Ellis	Raffi and Rio		Rio *et al.*
Holocene	NN 21	CN 15			E. huxleyi	E. huxleyi	Emiliania huxleyi		MNN 21 a + b
Pleisto-cene Late	NN 20	CN 14			G. oceanica	C. cristatus	Gephyrocapsa oceanica		MNN 20
	NN 19				P. lacunosa	E. ovata	Pseudoemiliania	S. pul + sm Geph	MNN 19 e + f
Early		CN 13				G. carib + E. ann	lacunosa	H. sel l + C. mac	MNN 18 b + c + d
Pliocene Late	NN 18	CN 12		D. brouweri	D. brouweri	D. pentaradiatus	C. pelagicus	C. doric + D. br	MNN 18 + MNN 19 a
	NN 17			Interval zone	D. pentaradiatus		Discoaster	D. pentaradiatus	MNN 16b / 17
	NN 16			D. surculus	D. surculus	D. surculus	surculus		
				Interval zone		D. tamalis		D. tamalis	MNN 16 a
Early	NN 15	CN 11		R. pseudoumbilica	R. pseudoumbilica	D. asym + S. neo	Reticulofenestra		MNN 14 / 15
				Interval zone			pseudoumbilca		
	NN 14	CN 10			D. asymmetricus	C. rugosus			
	NN 13			C. amplif. Int.	C. rugosus		Amaurolithus	C. rugosus	MNN 13
	NN 12			D. quinqueramus	C. tricorniculatus	C. acut + T. rug	delicatus	Amaurolithus spp.	MNN 12
				Interval zone					
Miocene Late	NN 11	CN 9	D. variabilis		D. quinqueramus	A. primus			
						D. berggrenii			
	NN 10	CN 8	D. pentaradiatus	D. pent. Int.	D. calcaris	D. neo + D. bell			
	NN 9	CN 7	D. phyllodus	D. aulakos-divaricatus Int.		D. hamatus			
Middle	NN 8	CN 6				C. coalitus			
	NN 7	CN 5	D. challengeri mediterraneus		D. kugleri	D. kugleri			
	NN 6				D. exilis	C. miopelagicus			
	NN 5	CN 4			S. heteromorphus	S. heteromorphus			

Figure 5.21 Comparison chart of Mediterranean Neogene calcareous nannofossil zonations and the 'standard' open-ocean Neogene calcareous nannofossil zonations of Martini (1971) and Okada and Bukry (1980).

counts, along with data from 'reticulofenestrid' coccoliths, to establish a very fine subdivision of the Mediterranean Pliocene.

In the Mediterranean Legs of the Ocean Drilling Project, which superseded the Deep Sea Drilling Project, further data has been presented on the calcareous nannofossil biostratigraphy of Mediterranean Neogene sediments, in particular through the work of Rio *et al.* (1990) on Plio-Pleistocene material from Sites 650-656 in the Tyrrhenian Sea. Using quantitative and biometric data, Rio *et al.* (1990) assessed the relevance of some 31 calcareous nannofossil marker events used by various authors to subdivide the Plio-Pleistocene period, and established those most applicable to the biostratigraphy of the western Mediterranean by using operational and quantitative definitions to produce an amended version of the Raffi and Rio (1979) zonation.

In terms of resolution, the zonation of Rio *et al.* (1990) is identical to Raffi and Rio (1979), except in the Early Pleistocene, which was represented by the *Helicosphaera sellii* subzone of Raffi and Rio (1979) but has been subdivided into two subzones (MNN 19c and d). However, the events which were used to divide the zones were better defined and easier to establish using the quantitative data. In the present report, the Pliocene scheme of Driever (1988) is preferred to that of Rio *et al.* (1990) on account of its superior resolution, and its emphasis on the genera and species of 'reticulofenestrid' coccoliths which dominate the calcareous nannofossil assemblages in the archaeological ceramics analysed in the present report (Appendix II). Nevertheless, a few of the markers featured in the zonation of Rio *et al.* (1990) are utilised here, in addition to some taxonomic definitions, such as that for the various species of *Gephyrocapsa* (Section AI.5).

5.6.2.1 Theodoridis (1984)

One of the most important calcareous nannofossil references for the Mediterranean Miocene time period is the work of Theodoridis (1984). This study is one of the few monographs on Miocene nannofossils (Young *et al.* 1994) and presents a thorough revision of the helicoliths and discoasters, as well as a 'Mediterranean Miocene Zonation' (Figure 5.22) and an 'Integrated Miocene Zonation', for extra-Mediterranean regions.

The Mediterranean Miocene zonation of Theodoridis (1984) is based upon material from DSDP cores as well as sections from Spain, Sicily, Malta and Gozo, Crete, Koufonisi, Zakynthos, Israel and Egypt. It contains 11 zones and some 21 subzones based on events in "several new, emended or hitherto neglected species" (Theodoridis 1984, 48), and as such, represents a vast improvement upon the standard zonations, and other Mediterranean zonations for the Miocene.

One of the genera which is utilised extensively in the zonation of Theodoridis (1984), is *Helicosphaera*, which is usually more abundant in the Mediterranean than in open ocean settings; the species of which are used to define seven biohorizons in the lower half of the Miocene. Theodoridis (1984) presented a comprehensive review of the structure, taxonomy and evolution of this genus as well as that of *Discoaster*, which he subdivided into '*Eu-discoaster*' and '*Helico-discoaster*' (not followed here). Both reviews have been used extensively for the identification of species in the present report, and the comprehensive Mediterranean Miocene range charts which Theodoridis (1984) presented, have been invaluable as a means of supplementary information where marker species are absent. One drawback of the zonation of

Standard Zonations		Mediterranean Miocene Zonation	
Martini and Worsley	Okada and Bukry	Zone	Subzone
PLIO Zancl. — NN 12	CN 10	C. leptoporus	C. leptoporus MC

	Martini and Worsley	Okada and Bukry	Zone	Subzone
PLIO Zancl.	NN 12	CN 10	*C. leptoporus*	*C. leptoporus* MC
Mess.	NN 11	b CN 9		*C. leptoporus* MB
				C. leptoporus MA
			R. rotaria	
		a	*A. primus*	
			C. pelagicus	
Tort.	NN 10	CN 8	*M. convallis*	*G. rotula*
				D. pentaradiatus
	NN 9	CN 7		*D. pseudovariabilis*
			D. calcaris	*D. hamatus*
	NN 8	CN 6		*D. bellus*
Serr.	NN 7	b	*D. exilis*	*D. bollii*
				D. kugleri
	NN 6	CN 5		*H. intermedia*
		a		*H. orientalis*
				H. walberstorfensis
	NN 5	CN 4	*S. heteromorphus*	*D. musicus*
				H. waltrans
Lang.	NN 4	CN 3		*H. perch-nielseniae*
				E. signus
				H. obliqua
			H. ampliaperta	

Figure 5.22 The Mediterranean Miocene calcareous nannofossil zonation of Theodoridis (1984).

Theodoridis (1984) is that it does not deal with the plexus of 'reticulofenestrid' coccoliths which are very dominant in late Neogene assemblages, especially those from archaeological ceramics. For this purpose, the extra-Mediterranean data of Backman (1980), Flores (1985), Young (1990), Gartner (1992), Mock and Bralower (1993), Takayama (1993) and others, must be considered for this taxonomic group (Section A1.8).

5.6.2.2 Driever (1988)

The zonation scheme of Driever (1988) represents the state-of-the-art in Mediterranean Pliocene biostratigraphy (Figure 5.23). Using quantitative data extracted from the record of *Discoaster* and 'reticulofenestrid' coccoliths in samples of on-land sections from Crete and Sicily, Driever defined 25 biohorizons, which in addition to the LOs of *Sphenolithus spp.* and *Calcidiscus macintyrei*, were used to produce a very detailed subzonal revision of Martini (1971).

Driever's (1988) scheme features 14 interval zones for the Pliocene Epoch, as opposed to the eight of Bukry (1973) and seven of Martini (1971), Raffi and Rio (1979) and Rio *et al.* (1990). In the Lower Pliocene, Zanclian stage, from which the raw material of many of the pottery samples in the present report are suspected to have come (Sections 11.2 and 11.3), the assignment of samples to one or more of the five subzones can be facilitated by establishing the relative abundance of the various 'reticulofenestrid' species (Section AI.8) and the comparison of this to Driever's (1988) charts. Two important datum events for this time period are the change in dominance of large reticulofenestrids (> 5 μm), from *Dictyococcities antarcticus* to

Standard Zonations		Mediterranean Pliocene Subzonal Scheme		
Martini and Worsley	Okada and Bukry	Zone	Subzone	Event and Date
Pleisto-cene **P. lacunosa** NN 19	*Crenalithus doronicoides* CN 13	*E. ovata* (NN 19)	NN 19B	
			NN 19A	m1 (1.4 MA)
P L I O C E N E				d13 (1.9 MA)
D. brouweri NN 18		*D. brouweri* (NN 18)	NN 18B	
			NN 18A	d12 (2.15 MA)
NN 17?				d11 (2.4 MA)
			NN 16-17F	
	D. brouweri CN 12		NN 16-17E	d10 (2.6 MA)
D. surculus NN 16		*D. surculus* (NN 16-17)	NN 16-17D	d7 (2.9 MA)
			NN 16-17C	n7 (3.2 MA)
				d6 (3.4 MA)
			NN 16-17B	
			NN 16-17A	s1 (3.5 MA)
				n5 (3.6 MA)
NN 15	CN 11	*Reticulofenestra pseudoumbilica* (NN 14-15)	NN 14-15B	
			NN 14-15A	d3 (3.7 MA)
NN 14				
			NN 12-13C	d2 (3.8 MA)
C. rugosus NN 13	*Amaurolithus triconiculatus* CN 10	*A. delicatus* (NN 12-13)	NN 12-13B	n2 (4.1 MA)
				n1 (4.5 MA)
NN 12			NN 12-13A	

Figure 5.23 Part 1. The Mediterranean Pliocene calcareous nannofossil zonation of Driever (1988) with the standard open-ocean zonations for comparison (n1-m1 = nannofossil datums).

n1	Change in the large-size coccoliths from predominantly *D. antarcticus* to *R. pseudoumbilica*.
n2	Size increase of *R. pseudoumbilica*, increase in the frequencies of *R. minutula* and *P. lacunosa*, decrease of *R. minuta* and appearance of rare small *Gephyrocapsa sp.*
n3	Increase in frequency of small *Gephyrocapsa sp.*
n4	Further increase in frequency of small *Gephyrocapsa sp.*
n5	Almost complete disappearance of *R. pseudoumbilica*, preceded by the appearance of subcircular *P. lacunosa.*
n6	First frequency increase of small *Gephyrocapsa sp.*
n7	Second frequency decrease of small *Gephyrocapsa sp.*, small *R. minuta* and large *R. minutula.*
n8	Second frequency decrease of small *Gephyrocapsa sp.*
n9	Low frequency of *R. minuta*, increase in *R. minutula.*
n10	Increase of *R. minuta*, decrease of *R. minutula* and third but gradual increase of small *Gephyrocapsa sp.*
n11	Further increase in frequency of small *Gephyrocapsa sp.*
n12	Appearance of larger *Gephyrocapsa sp.* (*G. caribbeanica*).
d1	Decrease in frequency of *D. variabilis* and increase of *D. surculus.*
d2	Increase in frequency of *D. asymmetricus* and increase in frequency of *D. brouweri.*
d3	Increase in frequency of *D. tamalis.*
d4	First decrease in frequency of *D. pentaradiatus.*
d5	Temporary decrease in frequency of *D. surculus, further* increase in frequency of *D. tamalis.*
d6	Reappearance of *D. pentaradiatus.*
d7	Top of the acmes of *D. asymmetricus* and *D. tamalis.*
d8	Base of the short-term reappearance interval of *D. asymmetricus* and *D. tamalis.*
d9	Virtual disappearance of *D. asymmetricus* and *D. tamalis.*
d10	Base of the stratigraphically highest acme of *D. pentaradiatus.*
d11	Decrease in the total abundance of *Discoaster* and virtual disappearance of *D. pentaradiatus* and *D. surculus.*
d12	Base of the acme of *D. triradiatus.*
d13	Top of the acme and LO of *D. triradiatus.*
s1	LO of *Sphenolithus.*
m1	LO of *Calcidiscus macintyrei.*

Figure 5.23 Part 2. Key to calcareous nannofossil datums of Driever's (1988) Pliocene zonation.

Reticulofenestra pseudoumbilica (or the last common occurrence of *D. antarcticus*), at the NN 12-13 A/B boundary, and the size increase of *R. pseudoumbilica* at the NN 12-13 B/C boundary. These can be established by counting 30 large specimens of *Reticulofenestra/Dictyococcities* and measuring 30 specimens of *R. pseudoumbilica*, respectively. This sort of approach represents a vast improvement to biostratigraphy in those assemblages where important marker species are rare and the nannoflora is dominated by 'background taxa' such as the reticulofenestrids, for example, the assemblages which are recorded in the smear-slides of archaeological ceramics in the present report (Appendix II).

Although specimens of the genus *Discoaster* are extremely rare in much of the archaeological and geological material which is analysed in this report, thorough searching has revealed small numbers of individuals in some samples. By making simple counts of the numbers of specimens belonging to the various species of the genus *Discoaster* in Pliocene assemblages (Section 5.10), it has been possible to apply some of the patterns of *Discoaster* relative abundance which were established by Driever (1988). The utilisation of these, and other patterns of relative abundance, to interpret calcareous nannofossil assemblages from archaeological ceramics, must always be undertaken in conjunction with other more conventional stratigraphic markers, as it is suspected, that several processes have the potential to alter the composition of calcareous nannofossil assemblages in ceramics (Chapter 3, Section 5.4. and 5.5).

5.7 Intra-Mediterranean variation of Neogene calcareous nannofossils

Despite the cosmopolitan distribution of calcareous nannofossils, some minor differences do exist between the late Neogene assemblages of the east and west Mediterranean. For example, in the Miocene, Müller (1978) has reported that *Sphenolithus belemnos* is missing from some parts of the western Mediterranean, yet it is common in the eastern Mediterranean. In addition, Rio *et al.* (1990) found *Ceratolithus/Amaurolithus* to be poorly represented in ODP cores from the western Mediterranean, whereas Ellis (1979) working on DSDP material from the eastern Mediterranean, claimed that numerous specimens of this group are present.

Müller (1978) suggested that different depositional conditions in various parts of the Mediterranean during the latest Miocene (Messinian) stage may have been responsible for variations in calcareous nannofossil assemblages, due to the isolation of some areas. However, these differences are not well established and are likely to be in the form of variations in the abundance and diversity of assemblages as well as the degree of reworking.

In the Upper Pliocene to Recent, differences exist between the calcareous nannofossil assemblages of the east and west Mediterranean, for example, in the terminal record of the genus *Discoaster*. Discoasters tend to be extremely rare or missing in the uppermost Pliocene (NN 16-18) of the western Mediterranean (Müller 1978; Bizon and Müller 1977), which has reduced the utility of their sequential extinction in this area (Rio *et al.* 1990). This appears to be the result of a greater influence of cooler North Atlantic water in the western Mediterranean since the onset of the glaciation in the northern hemisphere, at around 3 MA, which has also been responsible for the

larger and better evolved marine fossils in the eastern Mediterranean in general (Bizon and Müller 1977).

These slight biogeographical differences which exist between the equivalent-aged calcareous nannofossil assemblages of the east and west Mediterranean, although not insignificant, are too few in number and ill-defined, to be of any use for the provenancing of archaeological ceramics (Section 5.9.2). On the other hand, it is possible that the far greater dissimilarities which occur between the late Neogene Mediterranean and open-ocean calcareous nannofossil assemblages (Section 5.6.1; Figure 5.20), may be useful for identifying extra-Mediterranean material, although this is beyond the scope of the present report.

5.8 Analysis of other calcareous nannofossil taxa

Biostratigraphically-significant late Neogene calcareous nannofossil taxa, such as the species of *Discoaster*, *Amaurolithus/Ceratolithus* and *Triquetorhabdulus*, are generally very rare in the assemblages of Bronze Age archaeological ceramics which have been analysed in the present report. In fact, members of the latter two genera are almost entirely absent in the study material, having only been encountered on one or two occasions. Therefore, it has been necessary to utilise events in the late Cenozoic record of several other taxa, including the reticulofenestrids, sphenoliths, helicoliths, lopadoliths and members of the calcareous nannofossil family Coccolithaceae, in addition to the scanty information provided by the more conventional markers, to interpret some samples.

In order to establish the way in which these less important, but numerically more frequent, calcareous nannofossils can be used for biostratigraphy, a thorough review has been made of the range, biometry and variations in abundance of the various taxa, by utilising all available literature on Neogene Mediterranean calcareous nannofossils, in addition to important studies from extra-Mediterranean areas. This discussion is presented in Appendix I.

5.9 Approach to studying archaeological ceramics using calcareous nannofossils

5.9.1 Description

By analysing the calcareous nannofossil assemblages in smear slides of archaeological pottery, it is possible to describe ceramics according to the preservation and total abundance of nannofossils, the occurrence and relative abundance of particular taxa, and the geological age or nannofossil 'zone' of which the assemblage is indicative. The procedures adopted in this study for the routine analysis of calcareous nannofossil smear slides from archaeological pottery are outlined in Section 5.10, and descriptions of the calcareous nannofossil assemblages in all of the analysed pottery samples are presented in Appendix II.

5.9.3 Classification

Considering the range of processes which have the potential to remove calcareous nannofossil assemblages from archaeological ceramics, from the time of clay procurement until their analysis under the microscope (Chapter 3, Section 5.4 and

5.5), it is unwise to classify samples using the presence/absence of calcareous nannofossils. Likewise, it is inadvisable to group archaeological ceramics by the abundance, preservation or overall composition of their calcareous nannofossil assemblages; a problem encountered with the work of Troja *et al.* 1996 (Section 2.3.1.3). A more reliable approach, which may be used to infer similarity or dissimilarity between samples of nannofossiliferous pottery, is the interpretation and comparison of their assemblages in terms of the calcareous nannofossil zone/subzone, geological period or date of which they are indicative (Sections 11.2 and 11.3). As with all methods of grouping and classifying archaeological ceramics it is imperative to consider other compositional data, as well as any information pertaining to the technology of pottery production.

5.9.2 Provenance

The biostratigraphic interpretation of calcareous nannofossil assemblages from archaeological ceramics, outlined above, can be used to relate the geological age of the raw materials used in ceramic production, to deposits of equivalent aged sediments (Burnett and Young n.d., Section 2.3.2.5). Calcareous nannofossils are not well-suited to the interpretation of palaeoenvironment, and as discussed in Section 5.1, their distribution is very uniform over large areas, such as the Mediterranean (Section 5.7), which rules out a biogeographical approach to provenancing ceramics.

The success with which calcareous nannofossils can be used to provenance ceramics in this way is highly dependent on the precision of the biostratigraphic interpretations

based upon them, which in turn, is related to the state of preservation, abundance and diversity of the assemblage.

5.9.3 Technology

Calcareous nannofossils are not particularly applicable to the routine analysis of ceramic technology. However, they can be used to determine the geological date of calcareous material added as temper in pottery (Sections 11.2 and 11.3), and their occurrence has been utilised to confirm the admixture of marine and non-marine raw materials in Section 11.4. In the light of the experiments which are presented in Section 5.4, the presence/absence of calcareous nannofossils may only be used to make very crude interpretations of the degree of firing in ceramics (Section 5.4.8).

5.10 Procedures for logging and dating calcareous nannofossil smear slides of archaeological pottery

5.10.1 Introduction

The following section outlines the procedures which have been followed, during the logging of calcareous nannofossil smear slides of archaeological pottery in the present report and discusses the applicability of standard biostratigraphic techniques to ceramic assemblages.

5.10.2 The '100-counts'

In order to determine the relative abundance of the various calcareous nannofossil taxa in the assemblages from archaeological ceramics, a count of 100 specimens was made for each slide. It was decided to count 100 specimens, rather than the usual number of 200, because of the extremely low abundance of calcareous nannofossils in many of the samples. The binomial error in a count of 100 specimens is much greater than that for 200 (Dermitzakis and Theodoridis 1978), however, the results produced a reasonably accurate indication of the relative abundance of the various taxa in the assemblages when presented as semi-quantitative categories.

Due to the dominance of 'reticulofenestrid' coccoliths and the low proportion of other groups of calcareous nannofossils in the smear slides of archaeological ceramics, modifications were made to the semi-quantitative scheme of the above authors, in order to introduce more categories for taxa scoring < 20 % in the 100 counts (see below). Although these labels have a somewhat different meaning in terms of relative abundance to some other authors, comparisons can still be made between the relative abundance of two or more species in the same assemblage, such as *Reticulofenestra minuta* and *Reticulofenestra minutula* in the Pliocene, in order to utilise the results of Driever (1988) and others.

All of the specimens which were encountered during the 100 calcareous nannofossil counts were measured to the nearest 0.5 μm, using an eyepiece graticule. These measurements were necessary in order to classify specimens of *Reticulofenestra, Dictyococcities, Calcidiscus* and *Coccolithus* into their various species, as well as to

date samples in relation to size increases or decreases of certain taxa (e.g. the SRI, Section AI.8.2.1).

> **Extremely abundant (EX)**: > 40 % of total nannofossil assemblage
>
> **Very abundant (VA)**: 21-40 %
>
> **Abundant (A)**: 11-20 %
>
> **Common (C)**: 6-10 %
>
> **Few (F)**: 2-5 %
>
> **Rare (R)**: < 2 %
>
> **Present (P)**: species which did not score in relative abundance counts
> but which were observed during extemded searching

5.10.3 Apres-counts

Due to the scarcity of stratigraphically important taxa, such as *Discoaster*, *Ceratolithus* and *Amaurolithus*, in the archaeological samples which are analysed in the present report, a thorough search was carried out, after the completion of the 100-counts, for these and any other very rare calcareous nannofossils. All new taxa, as well as any significant specimens of those taxa which had already been encountered in the counts, were recorded and measured during closely-spaced traverses of the rest of the slide. In some low abundance samples, this procedure was not carried out, as the whole slide had to be covered in order to count 100 specimens. This searching revealed many important forms which were a minor component of the assemblage and would otherwise have been missed. These taxa are labelled as 'Present' (P) in the assemblage descriptions (Appendix II).

During the apres-counts, additional specimens of large reticulofenestrid coccoliths were counted and measured, in order to orient Early Pliocene samples with respect to

the change in relative abundance between *Reticulofenestra pseudoumbilica* and

Dictyococcites, as well as the size increase of the former, which are documented by

Driever (1988) and used as markers in his zonation scheme.

In a few Pliocene samples, in which well-preserved discoasters occurred in significant

numbers, a simple count was made of the various species using the method of Driever

(1981). This helped to orient these samples in relation to his six non-mutually

exclusive units, based upon the relative abundance of discoasters in the Pliocene

(Section 5.6.2).

5.10.4 Preservation and abundance estimates

Rough qualitative estimates of the overall abundance and preservation of the

calcareous nannofossil assemblages in the various samples were made in order to

highlight those samples which were particularly rich or poor in calcareous

nannofossils, as well as those in which the calcareous nannofossil assemblages were

very well preserved or badly preserved.

5.10.5 The reliability of FOs, LOs, and the relative abundance of calcareous nannofossils for biostratigraphically interpreting floras from archaeological ceramics

In attempting to interpret the calcareous nannofossil assemblages of archaeological

ceramics using conventional biostratigraphic techniques, it is necessary to be aware of

the many processes which may alter the assemblage at various stages in its history

(Chapter 3). Of particular importance is the potential of processes such as firing

(Section 5.4) and post-depositional alteration to remove delicate and rare taxa from the assemblage, as well as that of contamination during clay preparation and clay mixing, which may add anomalous specimens to the assemblage.

Considering these potential sources of bias, it is necessary to weigh up the reliability of the numerous lines of evidence provided by the calcareous nannofossil assemblage, when attempting to interpret a sample biostratigraphically. For example, it would be unwise to use the absence of a species to indicate a date prior to its FO or after its LO, when the presence of another more reliable species indicates an earlier or later date. It is also important to be aware of any signs of reworking and consider the overall preservation and abundance (Section 5.10.4), in order to determine whether a particular species may be absent due to etching, or may have an extended range as a result of reworking or contamination. When utilising the relative abundance of various taxa, such as in the scheme of Driever (1988) which is applied in this study, equal caution should be taken and consideration of any visible bias must be made. Quantitative calcareous nannofossil data preferably should be used to supplement the presence of other biostratigraphic markers, wherever possible, because of the potential alteration of the assemblage composition which may have taken place.

6 Foraminifera

6.1 Introduction

Foraminifera are minute, dominantly marine, protozoa with a mineralised, agglutinated or less commonly organic shell or 'test', enclosing an amoeboid body of continuously streaming, minutely granular, reticulose cytoplasm which has many delicate anastamosing pseudopodia (Figure 6.1). These single-celled marine animals can be subdivided into two broad groups based upon their life habit. These are, the planktonic (free floating) and benthic (bottom dwelling) foraminifera.

Planktonic foraminifera have a widespread latitudinal distribution within the world's oceans, which is strongly affected by water temperature, nutrients and ocean currents. Benthic foraminifera, on the other hand, have a less cosmopolitan distribution which is related to substrate parameters, depth and position on the ocean floor, as well as the availability of oxygen and the supply of nutrients. Benthic foraminifera can be subdivided into two groups, depending upon the structure of their test wall. Calcareous benthic foraminifera (sometimes just referred to simply as 'benthic foraminifera') have a simple, perforate, single or multi-layered calcite (or more rarely aragonitic) test, whereas 'agglutinated foraminifera' have a test constructed of mineral grains (collected by the organism), which are bound in a calcareous or proteinaceous cement (Figure 6.1).

Within the geological record, only the mineralised tests of foraminifera and their chitnous inner linings are preserved, as the soft cytoplasm quickly decays after death.

Foraminiferal tests are common in many types of argillaceous marine sediments, as well as biogenic limestones and chalks, from the Cambrian to Recent. The first foraminifera had a benthic life habit and it was not until the Middle Jurassic that planktonic foraminifera evolved.

Fossil foraminifera are subdivided into 14 orders on the basis of their test composition (calcite, aragonite, silica, proteinaceous and agglutinated) and ultrastructure (microcrystalline, hyaline or agglutinated), as well as the number and arrangement of layers, and the presence of pores (Loeblich and Tappan 1992). Of these, six represent the calcareous benthic foraminifera, four represent the agglutinated foraminifera, one contains all planktonic foraminifera (order Globigerinida), and the other three are minor orders which contain aragonitic and proteinaceous forms. Within these orders the foraminifera are classified by the morphology of their tests (e.g. overall shape of test, number and arrangement of chambers, type and degree of coiling), as well as the nature of specific features (e.g. the number and position of apertures, the nature of any surface ornament, the presence of keels, spines) Figure 6.1.

Foraminifera were one of the earliest groups of microfossils to be studied, and as a result some 4000 or so genera have been established since the description of *Ammonia* by Van Luenhock in 1772 (Loeblich and Tappan 1992). Fossil foraminifera are an extremely useful tool for biostratigraphy (especially planktonic forms), the interpretation of palaeoenvironment (particularly benthic foraminifera) and the reconstruction of oceanographic phenomena. The importance of foraminifera as a means of dating and correlating sediments has, like in other microfossil groups, fuelled intensive research since the 1950's, due to their widespread use in the oil
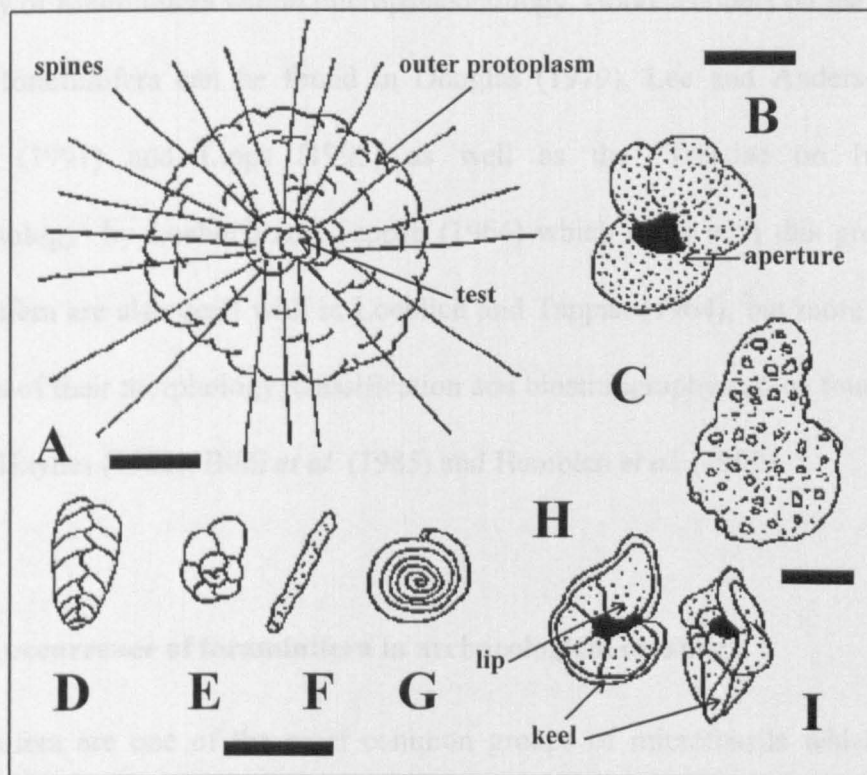
Figure 6.1 The biology and morphology of foraminifera. Living planktonic foraminifer (A), planktonic foraminiferal test (B), agglutinated foraminiferal test (C), elongate biserial test (D), globular trochospiral test (E), elongate tubular agglutinated test (F), tubular planispiral test (G), biconvex trochospiral planktonic foraminiferal test with apertural lip and keel (umbilical view H, peripheral view I). Scale bars = 1 mm (A and D-G), 0.5 mm (B, C and H). After Brasier (1980) and Haynes (1982).

industry, as well as a means of studying the deep ocean cores of the DSDP and ODP expeditions.

The study of living foraminifera is almost as old, and has developed in tandem with the study of foraminifera within micropalaeontology. Good accounts on the biology of Recent foraminifera can be found in Douglas (1979), Lee and Anderson (1991), Murray (1991) and Lipps (1993) as well as the 'Treatise on Invertebrate Palaeontology' by Loeblich and Tappan (1964) which deals with this group. Fossil foraminifera are also dealt with in Loeblich and Tappan (1964), but more up to date accounts of their morphology, classification and biostratigraphy can be found in Blow (1979), Haynes (1982), Bolli *et al.* (1985) and Hemblen *et al.* (1990).

6.2 The occurrence of foraminifera in archaeological ceramics

Foraminifera are one of the most common groups of microfossils which occur in archaeological ceramics (Davis 1951; Riley 1981; 1983; MacGillivray *et al.* 1988; Day 1991; Whitbread 1995; Vaughan *et al.* 1995; Troja *et al.* 1996; Stilborg 1997; Burnett and Young n.d.; Riley *et al.* n.d.), and are present in many of the thin sections which have been analysed in the present report. They occur mainly in those ceramics which contain a fair amount of fine calcium carbonate in the groundmass, i.e. 'calcareous fabrics', but can also be present in 'non-calcareous' ceramics (Section 11.2).

Foraminifera may appear in several components of a ceramic thin section. Most commonly, they occur as isolated inclusions within the clay matrix, however it is often possible to observe foraminiferal tests and their remains within clay mixing

(Stilborg 1997; Riley 1981), or other larger aplastic inclusions (Figure 6.2). In the present report foraminifera have been observed in calcite inclusions and, more rarely, within fragments of chert. In addition, isolated occurrences of foraminifera within clay pellets and grog (inclusions of crushed pottery), have been noted (Figure 6.2). It is possible for foraminifera to appear in more than one component within a single thin section, for example, where ceramics are tempered with a microfossiliferous material, and some tests have broken free to form separate inclusions in the matrix. One thin section which was analysed in the present report (Kn 92/53), contained foraminifera in three different components; within the clay groundmass, as part of several calcite inclusions, and within a piece of grog. It is very important to consider the context in which foraminifera appear within ceramics, as this has a direct influence on the type of information which can be attained through their study.

In thin sections of archaeological ceramics, foraminifera appear as complex, chambered, calcite structures (Figures 6.2 and 6.3). They are usually unmistakable, except when poorly-preserved, and have a great range of morphologies. A single foraminifer may appear very different, depending upon its orientation relative to the section, because of the effect of taking a two-dimensional slice of a complex three-dimensional structure (Figure 6.3). This effect, which also presents problems for other techniques of ceramic analysis (Riley 1984), makes the identification of foraminifera in thin section rather difficult.

Within the study of micropalaeontology, foraminifera and other larger calcareous microfossils are usually isolated from rock and sediment samples, in order to identify them as three-dimensional specimens. Whilst it is also possible to isolate well-

preserved foraminifera from vitrified archaeological ceramics in this way (Section 6.5), the quantity of material which must be destroyed in the process is far too great (Burnett and Young n.d.).

Nevertheless, it is sometimes possible to identify foraminifera to genus (MacGillivray *et al.* 1988; Troja *et al.* 1996; Alaimo *et al.* 1997) and even species level (Riley *et al.* n.d.) in thin sections of archaeological ceramics, if a specimen has been favourably sectioned so that particular, unmistakable features of its test are represented (Figure 6.3), or several different sections of one form occur in the same sample, so that an impression of its three-dimensional morphology can be attained. Thin section analysis is the only available method of studying foraminifera in certain other situations. For example, Sliter (1989) has established a detailed 31-part, Cretaceous planktonic foraminiferal zonation scheme from thin sections of indurated carbonate rocks of the circum-Pacific margin, which achieves a mean stratigraphic resolution of approximately two million years by using a series of instantly recognisable primary species as well as the size, diversity and morphology of the entire assemblage.

The late Neogene planktonic foraminifera which appear in ceramic thin sections of Bronze Age archaeological ceramics in the present report, are much less distinctive than the ornate Cretaceous forms which characterise Sliter's zonation. However, many of their morphological features can be identified (e.g. wall structure, proloculus, aperture) and a genus level identification can usually be made. Benthic foraminifera are equally common in many of the thin sections which are analysed in the present report, and can be more distinctive than planktonic foraminifera (Figure 6.3). Because of the greater range of morphologies in late Neogene benthic foraminifera, compared
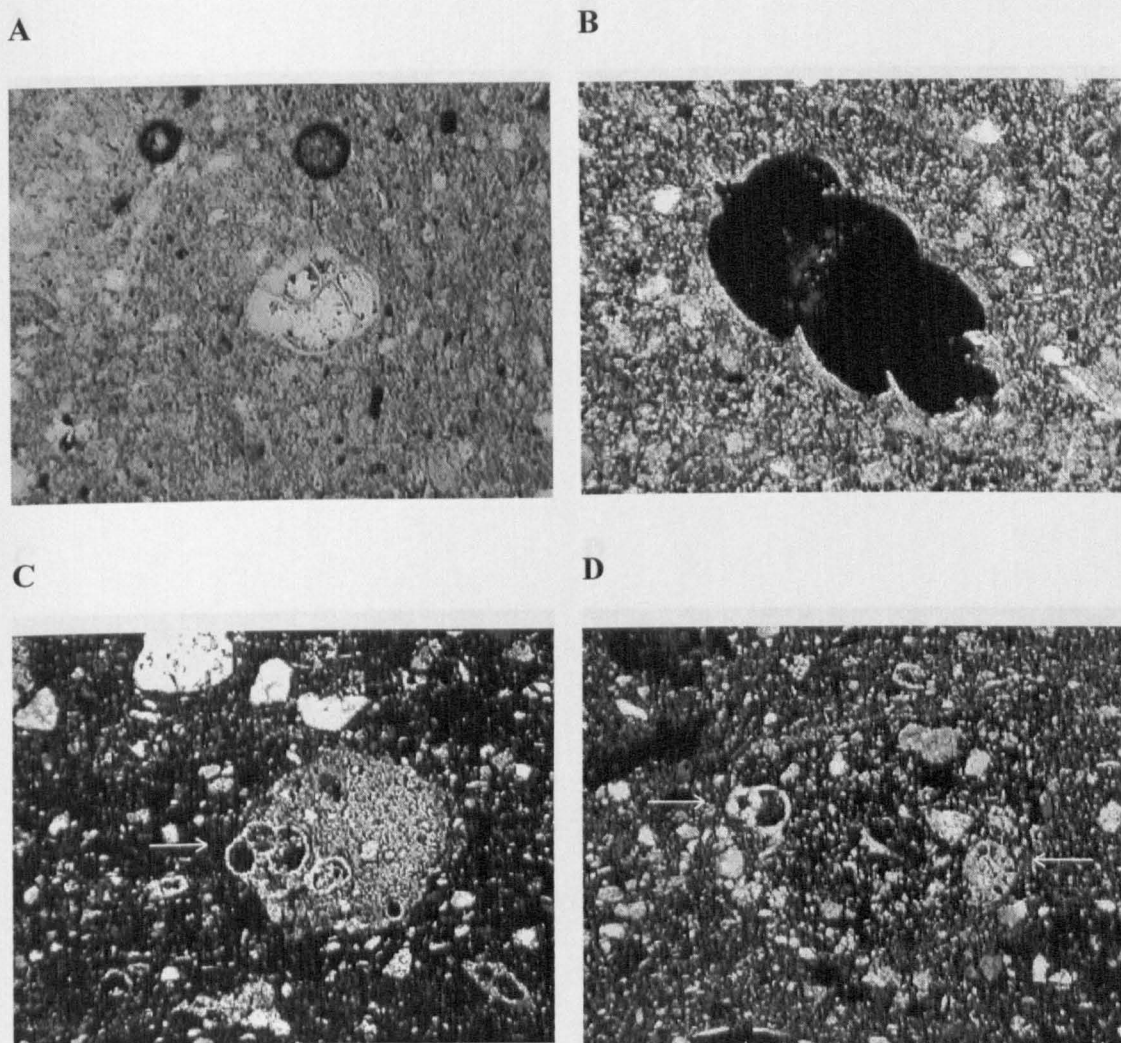
Figure 6.2 The occurrence of foraminifera in Bronze Age archaeological pottery from Crete. Within the clay matrix of Kn 95/187 (A and B), within a calcareous inclusion in sample Kn 95/382 (C) and within the clay matrix of a piece of 'grog' in Kn 92/53 (D). A = PPL, B, C and D = XP. Field of view = 0.5 mm (A and B), 1.5 mm (C and D).

Figure 6.3 The identification of foraminifera in thin sections of Bronze Age archaeological pottery from Crete. Differential representation of a single species of foraminifera in thin sections Kn 95/187 and Kn 92/13 (A and B), and well-preserved benthic foraminifera in Kn 95/238 and Kn 95/187 (C. *Stilostomella adolphina* and D. *Bolivina spathulata*). All pictures XP. Field of view = 0.5 mm.

to planktonics, it is much easier to identify specimens in thin section to genus level and beyond.

Despite the difficulties which are involved in identifying foraminifera in thin section, this is by far the most suitable method of analysing these microfossils from archaeological ceramics, as large quantities of original artefacts are not normally available for destruction, whereas ceramic thin sections often exist. The analysis of foraminifera in thin section has been applied extensively in the present report, in order to characterise, provenance and infer the technology of archaeological ceramics (Section 6.7).

6.3 The preservation of foraminifera in thin sections of Bronze Age archaeological ceramics from Crete

Foraminifera can be preserved in many ways within thin sections of archaeological ceramics. Simple observations of their preservation have been made by Riley (1981) and Troja *et al.* (1997). However, a more detailed study of the types and variation in the preservation of foraminifera within archaeological ceramics can reveal useful information pertaining to their origin, as well as the history of the artefact (e.g. Davis 1951).

Foraminiferal tests are commonly composed of calcite and, in the majority of cases, this original calcareous structure is retained in thin sections of archaeological ceramics. Thus, for example, it is possible to identify the cancellate, macroporous wall structure of planktonic foraminifera belonging to the genus *Globigerina*, the smooth, microperforate wall of the genus *Globorotalia*, as well as features such as the

gametogenic calcification of foraminiferal tests. However, in certain circumstances the original calcite wall of a foraminifer may be altered, replaced, or totally removed, so that its initial microstructure is destroyed.

Within the thin sections of Bronze Age archaeological ceramics which are analysed in the present study, foraminiferal tests can be replaced by micritic calcite, a single crystal of calcite, microcrystalline quartz, or may be represented by a characteristically shaped void where the mineralised test has been removed (Figure 6.4). The internal chambers of a foraminiferal test may also be infilled in various ways; by micrite or by sparry 'dog tooth' calcite, growing from the internal surface of the test (Figure 6.4), by interlocking equant crystals of calcite or quartz, by a single continuous crystal of calcite or quartz, or a combination of the above.

Dark red, brown and opaque black organic matter may infill the chambers of foraminifera, and can be found associated with their tests in many ways. This material represents the oxidised remains of organic structures which were attached to the test during the life of the foraminifera. The amorphous organic matter, which can surround the external surface of the test in a discrete layer, or may appear as a less distinct 'staining' within the clay matrix, is thought to represent the oxidised protoplasm of the dead foraminifer. Organic matter may also stain the calcareous walls of foraminiferal tests or calcite which infills the chambers, but is more commonly found as discrete bodies or membranes. As with the preservation of ostracods in thin sections of archaeological ceramics (Section 7.3), oxidised organic matter of this kind is often in the form of variable sized, black opaque spheres. These can occur singularly, but usually appear in number and may completely fill the chambers. This

A

B

C

D
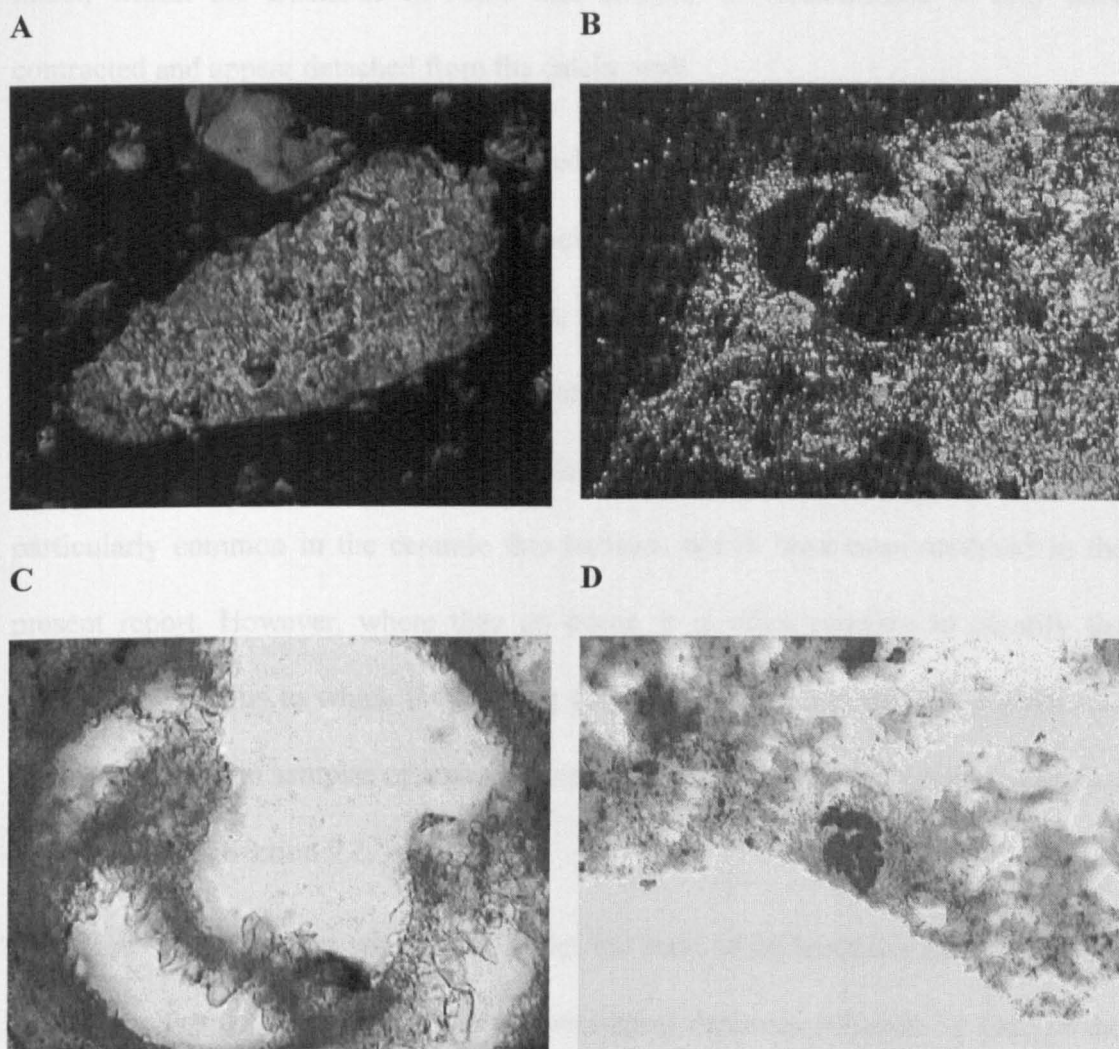
Figure 6.4 The preservation of foraminifera in thin sections of Bronze Age archaeological ceramics from Crete. Total calcite preservation in Kn 92/53 (A), total void in Kn 92/13 (B), 'dog tooth' sparry calcite growth in Kn 86/5 (C) and a biserial microforaminifera cf. *Bolivina sp.* in Kn 86/13 (D). A and B = XP, C and D = PPL. Field of view = 0.15 mm (A and C), 0.5 mm (B and D).

is likely to have come from the chitinous organic membranes which coat the internal surface of many foraminiferal tests. These tough, continuous linings can be observed intact, within the chambers of some thin sections of foraminifera, or may have contracted and appear detached from the calcite wall.

In some specimens in which the mineralised test wall has been removed, all that is left of the foraminifer is its internal organic lining (Figure 6.4). These membranes, which reflect the overall morphology of the test, are often found in acid digested residues, prepared for the study of organic microfossils (Section 9.1), and have been termed 'microforaminifera' by Wilson and Hoffmeister (1952). Microforaminifera are not particularly common in the ceramic thin sections which have been analysed in the present report. However, where they do occur, it is often possible to identify the foraminiferal genus to which they belong (Figure 6.4). Microforaminifera have also been liberated from samples of archaeological ceramics in the present report, and they are discussed in Section 9.2.

There are several factors which may affect the state of preservation of foraminifera and other calcareous microfossils in archaeological ceramics (Chapter 3). One of the most important processes in this respect is firing, as outlined by the following section.

6.4 The behaviour of larger calcareous microfossils during the firing ceramics

6.4.1 Introduction

In Section 3.6, we speculated that the process of firing may significantly affect microfossil assemblages in archaeological ceramics. This hypothesis was confirmed

by the experimental firing of Gault Clay briquettes, in which calcareous nannofossils were degraded and eventually removed by progressive heating (Section 5.4).

Foraminifera were not sufficiently abundant in the Gault Clay which was utilised in these experiments. Therefore, in order to determine the way in which these larger calcareous microfossils behave during firing, their state of preservation was recorded in several thin sections of Bronze Age archaeological ceramics from Crete, for which the approximate firing temperature was known. All of these samples come from research on Early Minoan pottery which was carried out at the Department of Archaeology and Prehistory, University of Sheffield and the Demokritos National Centre for Scientific Research, Athens. As part of this project, various samples of Cretan pottery were analysed with the SEM in order to observe their clay vitrification microstructures and determine approximate firing temperatures, using the method which was developed by Tite *et al.* (1982). As many of these samples contained foraminifera and ostracods, a direct comparison could be made between firing temperature and the preservation of these microfossils.

Forty microfossiliferous Early Minoan thin sections were chosen, covering the entire range of ancient firing temperatures which have been determined for this pottery (< 750 to > 1080 °C). Each thin section was scrutinised under the microscope and a qualitative estimation of the state of preservation of any foraminifera and ostracods was made, according to the scheme which is outlined below.

good pres.	well-preserved original calcite wall structure and microfossil morphology.
med. pres.	some of the original wall structure may be preserved; wall may be replaced by micritic calcite, but remains intact, as do the internal septa of foraminifera.
poor pres.	original wall structure is rarely preserved; wall and septa may be fragmented or missing entirely so that the fossil is represented by a void; microforaminifera may be present (organic lining without mineralised wall).
intermediate categories (med-poor pres.; med-good pres.)	
	attributed when the preservation is intermediate between the above categories or situations in which there is a range of preservation, in which case, these intermediate labels refer to the mean state of preservation.

6.4.2 Results

The firing temperatures, atmosphere, petrographic/ware groups and archaeological period of each of the 40 samples, as well as the preservation of their calcareous microfossils is presented in Figure 6.6 (Parts 1 and 2). In these tables, the samples are arranged with increasing firing temperature, so that any trends in the preservation of foraminifera and ostracods can be seen.

Sample	Firing temp.	Atmosphere	Foraminifera.	Ostracods
Kn 92/37	< 750 °C	O	good pres.	-
PET 94/63	< 750 °C	O	good pres.	-
PET 94/51	< 750 °C	R	good pres.	-
Kn 92/252	< 750 °C	O-R	med-good pres.	-
MFK 93/3	< 750 °C	O-R	good pres.	-
MFK 93/103	< 750 °C	O-R-O	good pres.	-
Kn 92/14	~ 750 °C	O	med pres.	med pres.
Kn 92/36	~ 750 °C	O-R-O	med pres.	-
Kn 92/64	750-800 °C	O	med pres.	-
Kn 92/91	750-800 °C	O	med-poor pres.	-
PET 94/35	750-800 °C	O	med-good pres.	-
PET 94/64	750-800 °C	O	med-good pres.	-
MFK 93/6	750-800 °C	O	med pres.	-
MFK 93/40	750-800 °C	O	good pres.	-
MFK 93/45	750-800 °C	O	med-good pres.	med pres.
MFK 93/65	750-800 °C	O	med-poor pres.	med-poor pres.
MFK 93/130	750-800 °C	R	med pres.	med pres.
Kn 92/4	750-800 °C	O-R	med pres.	med pres.
MFK 93/141	750-800 °C	O-R	med-poor pres.	med-poor pres.
MFK 93/87	750-800 °C	O-R-O	med-poor pres.	-
PET 94/26	750-850 °C	O	med pres.	med-good pres.

Figure 6.6 Part 1. The preservation of foraminifera and ostracods in Early Minoan Cretan ceramics fired from < 750 °C to 850 °C. Continued below. O = oxidising and R = reducing atmospheres.

Sample	Firing temp.	Atmosphere	Foraminifera	Ostracods
Kn 92/38	750-850 °C	O-R-O	med pres.	-
MFK 93/46	750-850 °C	O-R-O	-	med pres.
Kn 92/61	800-850 °C	O	med-poor pres.	-
PET 94/67	800-850 °C	O	med-poor pres.	-
MFK 93/42	800-850 °C	O	med-poor pres.	med-poor pres.
MFK 93/97	800-850 °C	O	good pres.	-
MFK 93/132	800-850 °C	O-R	-	good pres.
PET 94/37	800-850 °C	O-R-O	med-poor pres.	-
Kn 92/20	850-950 °C	O	poor pres.	-
Kn 92/221	850-950 °C	O-R-O	med pres.	med pres.
MFK 93/51	850-950 °C	O-R-O	poor pres.	-
MFK 93/115	850-950 °C	O-R-O	poor pres.	poor pres.
PET 94/22	850-1050 °C	O	med-good pres.	-
PET 94/57	850-1050 °C	O	med-good pres.	-
MFK 93/107	850-1050 °C	O-R-O	-	poor pres.
PET 94/65	850-1050 °C	O	poor pres.	poor pres.
Kn 92/230	1000-1080 °C	O-R-O	med-poor pres.	-
MFK 93/87	1050-1080 °C	O-R-O	med-poor pres.	-
Kn 92/212	> 1080 °C	O-R-O	v. poor pres.	-

Figure 6.6 Part 2. The preservation of foraminifera and ostracods in Early Minoan Cretan ceramics fired from 750 °C to > 1080 °C. O = oxidising and R = reducing atmospheres.

6.4.3 Discussion and implications for the study of archaeological ceramics

Both foraminifera and ostracods are present in highly fired ceramics (up to 1000 °C). By studying the overall state of preservation in Figure 6.6 (Parts 1 and 2), a general trend of decreasing preservation can be seen with increasing firing temperature. All samples which were fired to < 750 °C have well-preserved calcareous microfossils, and most samples which may have been fired > 850 °C have a rather poorly-preserved microfossil assemblage.

As speculated in Chapter 3, the preservation of microfossil assemblages in archaeological ceramics is likely to be the result of many processes, of which firing is but one. Other factors, which include the state of preservation of the original microfossil assemblage in the unfired clay body, the vessel function, post-depositional alteration and poor sample curation, may be responsible for the variations in preservation which are observed in samples that were fired to an equivalent temperature.

Nothing positive can be concluded about the response of calcareous microfossils to different firing atmospheres. Too few microfossiliferous, reduction-fired Early Minoan samples were available, and the sherds which were both reduced and oxidised (O-R and O-R-O) do not exhibit differences in preservation, compared to the oxidised samples that were fired to same temperature.

By comparing the preservation of foraminifera and ostracods in the various samples, it can be concluded that these two groups of calcareous microfossils appear to react in a similar way to progressive firing. Despite the absence of ostracods in the highest and lowest fired samples, it can be seen that both groups exhibit a general decrease in

preservation with firing. The more sporadic occurrence of ostracods as compared to foraminifera, is a feature of Cretan Bronze Age pottery in general, and not a result of firing.

The degradation of foraminifera and ostracods in fired ceramics is likely to be a result of the transformation of their $CaCO_3$ tests/valves into CaO and its subsequent re-hydration to $Ca(OH)_2$ after firing, as well as the breakdown of calcite and its reaction with the surrounding clay minerals at very high temperatures. It has not been possible, in the present report, to document the occurrence of these processes in detail, due to the poor precision of the SEM firing temperature estimates for the archaeological ceramics which have been analysed, as well as the possible occurrence of variations in other factors, such as the firing temperature and the length of firing. However, the general trend of a decrease in the state of preservation of larger calcareous microfossils in progressively fired archaeological ceramics, which has been highlighted, is very significant (see below) and requires further investigation.

The alteration and eventual destruction of foraminifera and ostracods during firing has several implications for their use in the study of archaeological ceramics (Sections 6.7 and 7.7). Firstly, the identifiability of these larger calcareous microfossils in thin section will be severely reduced as their calcite tests/valves are progressively degraded. This point is not as relevant to ostracods, which are extremely difficult to identify to even the broadest taxonomic level in thin sections regardless of their state of preservation. However, foraminifera, which can be identified to genus, or even species level in thin sections of archaeological pottery (MacGillivray *et al.* 1988; Troja *et al.* 1996; Riley *et al.* n.d.; Sections 11.2 and 11.3) by the number and

arrangement of their chambers, are likely to become less recognisable as their original wall structure and septa break down (Figure 6.6). This has major implications for the study of archaeological ceramics using foraminifera, as without accurate identification of the taxa which are present in thin section, it may not be possible to biostratigraphically or palaeoenvironmentally interpret the raw materials of ceramic manufacture (Section 6.7.4).

Whilst the destruction of foraminifera and ostracods in fired ceramics clearly affects the accuracy with which archaeological ceramics can be characterised and compared by the composition of their assemblages (Sections 6.7.2 and 6.7.3), the presence of these larger calcareous microfossils can still be noted. Unlike calcareous nannofossils, which are so small that they can be removed without a trace, by firing (Section 5.4) or etching (Section 5.5), foraminifera and ostracods leave characteristically-shaped voids in the groundmass. Although it is not possible to identify taxonomically the specimens of ostracods or foraminfera which once filled such voids; with a little experience, their former presence can be detected. This phenomenon is important for the utilisation of the presence/absence of larger calcareous microfossils as a means of classifying archaeological ceramics (Sections 6.7.3 and 7.7.3).

The preservation of larger calcareous microfossils in ceramics has been noted by various authors, including Riley (1981) and Troja et al. (1996), however, only Davis (1951) has used this to infer the degree of firing in ceramics. Davis, who identified a single specimen of the foraminiferal genus *Nubeculinella* in a sherd of Iron Age pottery from England, noted that "there are no signs of calcination or decay in the fossil-shell fragments" and considered this to indicate that "probably the pots were

only partly fired" (1951, 148). Whilst the interpretation of this author was probably correct, the application of the state of preservation of foraminifera, and ostracods, to the study of archaeothermometry in this way, is likely to be very limited.

6.4.4 Scope for further research

The investigations which are described above are very limited, and have only considered a limited number of medium to high fired archaeological ceramics. Nevertheless, a significant trend in the state of preservation of larger calcareous microfossils in progressively fired ceramics has been elucidated. In order to investigate this trend in detail, as well as to determine the effect of the length of firing and the firing atmosphere on the state of preservation of foraminifera and ostracods, controlled experiments are clearly required. The author has attempted to experimentally fire briquettes containing these two groups of larger calcareous microfossils, however the results in this case were very limited and further experiments are planned.

6.5 The isolation of foraminifera from archaeological ceramics

In order to determine whether or not three-dimensional specimens of foraminifera can be isolated from reasonably well-fired archaeological pottery, as well as to compare the identification of foraminifera in ceramic thin sections with that from isolated specimens, two samples of Cretan Bronze Age pottery were processed using the method which is standard to micropalaeontology (Section 6.5.3).

6.5.1 Samples

The two samples which were analysed (MFK 93/105 and Kn 95/187) came from the Cretan archaeological sites of Myrtos Fournou Korifi and Knossos respectively. These were chosen because they contained rich, well-preserved assemblages of foraminifera in thin section, and belong to significant micropalaeontological groups of pottery from the two sites. Due to the constraints posed by artefact conservation, only a small quantity of each sample (c. 2-3 cm^3) was available for these experiments. Although, by archaeological standards the destruction of such quantities of original artefacts may seem rather wasteful, much more material is usually required for a standard micropalaeontological preparation.

6.5.2 Equipment

Pestle and mortar; Enamel bowl or saucepan; Hot plate or bunsen burner and tripod; Soda crystals; Stirring rod: 63 μm sieve; Large flat-ended artists paint brush and a very fine version; Evaporating dish; Oven; 63 to 500 μm sieve tower; Envelopes; A4 paper and tissue; Binocular microscope; Small metal picking tray with grid-lines etched on it: Compartmentalised microscope slides; Paper glue stick; Access to a sink and fume cupboard.

6.5.3 Procedure

1. The sample of archaeological pottery was wrapped in tissue paper and gently crushed in the pestle and mortar. The resulting fragments were then transferred to the saucepan and covered with plenty of tap water.

2. The saucepan was placed on the hot plate, a couple of spoonfuls of soda crystals were added, the mixture was brought to the boil and then left to simmer.

3. Every few hours the saucepan was removed from the hot plate and its contents were sieved over the sink. If the residue which was left in the sieve contained disaggregated lumps of pottery then it was washed back into the saucepan, topped up with water and soda crystals, and placed back on the hot plate. This process was repeated until the pottery had broken down into a relatively fine sediment.

4. The sedimentary residue was then transferred to the evaporating dish, left to stand, decanted and transferred to the oven (preheated to approximately 30 °C), and left for 24 hours.

5. The dry sample was split into various fractions using the sieve tower, the A4 paper, large paint brush and the envelopes.

6. Each fraction of the sieved sediment was then analysed individually under the binocular microscope on the picking tray. The foraminifera, and anything else of interest, was picked out using the fine paint brush, wetted with tap water, then transferred to the compartmentalised slides where they were fixed in place using a small amount of paper glue.

<u>6.5.4 Results</u>

6.5.4.1 Introduction

The two small samples of pottery were scrutinised under the binocular microscope at magnifications of 15-35 x before processing, in order to determine whether the foraminifera which were visible in thin section could be seen on the surface of the sherds. A thorough search over all surfaces revealed many planktonic and benthic foraminifera embedded in the clay matrix of the sherds, as well as a mould of an ostracod shell, exposed on the surface of one sample.

In general, the two pottery samples were difficult to break down using the standard processing techniques which are described above. Both had to be boiled, washed and sieved many times until a suitable amount of residue was attained. Even after this, lengthy, process some relatively large pieces of disaggregated pottery remained. These particles, which appeared in the > 250 μm fractions, often contained foraminifera embedded in their surface. Nevertheless, a reasonable quantity of fine residue (63-250 μm), containing foraminifera, was recovered from each sample. This also contained small pieces of pottery which had failed to break down.

6.5.4.2 Sample MFK 93/105

This sample contained approximately 100 specimens of foraminifera, with roughly equal proportions of benthic and planktonic forms. Despite the occurrence of a few broken specimens, most of the foraminifera were well-preserved. These mainly occurred in the 250-125 μm and 125-63 μm fraction, however, rare lumps of pottery

with foraminifera were found in the larger fractions. The assemblage of foraminifera which was isolated from this sample of pottery contained adult and juvenile tests of many species, and was dominated by the benthic genera *Bolivina*, *Bulimina* and *Cibicides*, as well as the planktonic foraminifer *Sphaeroidinellopsis*.

Assemblage: *Asterigerina mamilla, Bolivina dilatata, Bolivina spathulata, Bolivina subexcavata, Bolivina sp., Bulimina costata, Bulimina cf. exilis, Bulimina subulata, Cibicides ungerianus, Cibicides sp., ?Dentalina sp., Elphidium cf. fichtellianum, Globigerina acostaensis* (dwarfed)*, Globigerina sp., Globorotalia cf. subscitula, Sphaeroidinellopsis sp.*

6.5.4.3 Sample Kn 95/187

This sample contained less foraminifera than MFK 93/105. These were, on the whole, less well-preserved than the previous slide, and the coarse fractions of Kn 95/187 yielded a greater number of disaggregated lumps of pottery with foraminifera. The assemblage of foraminifera which was isolated from this sample contains both benthic and planktonic forms, juveniles and adults, and is dominated by species of the genera *Bolivina* and *Cibicides*.

Assemblage: *Bolivina spathulata, Bolivina subexcavata, Cibicides cf. dutemplei, Cibicides ungerianus, Elphidium fichtellianum, Globigerina acostaensis* (dwarfed)*, Globigerina sp.,* non-keeled *Globorotalia cf. scitula, Sphaeroidinellopsis sp., Stilostomella advena, Stilostomella adolphina, Uvigerina bononiensis cf. compressa.*

6.5.5 Discussion

The results of this pilot study indicate that it is possible to liberate foraminifera from samples of reasonably well-fired archaeological pottery. It was feared that the alteration of their $CaCO_3$ tests to CaO during firing, and its re-hydration after firing, would cause them to disintegrate during the mechanical crushing of the pottery sherds. However, the foraminifera on the whole remained structurally intact. The number of specimens which were isolated from the samples of archaeological pottery were too few for a detailed micropalaeontological analysis, but could be interpreted in terms of the broad geological date and palaeoenvironment in which the raw materials of ceramic manufacture were deposited (Section 6.6.4).

By comparing the assemblages of foraminifera which were isolated from archaeological pottery samples MFK 93/105 and Kn 95/187 (Sections 6.5.4.2 and 6.5.4.3), with those which were determined from ceramic thin sections of the same artefacts (Figures 11.3 and 11.8), it is possible to assess the reliability of the latter approach, which has been used extensively in the present report as well as by other authors (Riley 1983; MacGillivray *et al.* 1988; Troja *et al.* 1996; Alaimo *et al.* 1997; Riley *et al.* n.d.). The associations of benthic foraminifera which are present in the thin sections of samples MFK 93/105 and Kn 95/187, have a lower species diversity than the corresponding assemblages which were isolated from the original samples. This is to be expected, and is the consequence of the small numbers of foraminifera which appear in thin sections of archaeological pottery, the random sample which is captured by this method, and the fact that not all specimens can be identified. The

difficulties which are involved in identifying foraminifera from thin sections of archaeological pottery, comprise a major drawback of this method (Section 6.2).

The benthic foraminifera which occur in many of the thin sections of archaeological pottery which have been analysed in the present report, can sometimes be speciated (Figures 11.3 and 11.8), whereas, most planktonic foraminifera can only be identified to genus level. Benthic foraminifera are much less valuable for biostratigraphy than planktonics, and therefore, the poor identifiability of the latter in thin section poses problems for the biostratigraphic interpretation of the raw materials used in ceramic manufacture (Section 6.7.4).

6.6 Foraminifera and biostratigraphy in the Mediterranean late Neogene

6.6.1 Introduction

The value of foraminifera within the study of archaeological pottery lies in their biostratigraphic and palaeoenvironmental potential, which can be used to characterise and indicate the provenance of the raw materials which were used in ceramic manufacture. In order to interpret the assemblages of late Neogene foraminifera which have been recorded from thin sections (Figures 11.3 and 11.8) and digested residues (Section 6.5.4) of Cretan archaeological ceramics in this way, it is necessary to review the numerous biostratigraphic and palaeoenvironmental schemes which have been proposed for the late Neogene of the Mediterranean basin.

Both planktonic and benthic foraminifera have been reasonably well studied in the Mediterranean late Neogene. This is due, in part, to the isolated nature of

Mediterranean foraminiferal assemblages in the Late Miocene and Pliocene, which has necessitated the erection of specific biostratigraphic schemes for this area. But also, because many late Neogene stratotype sections occur in the central and western Mediterranean (particularly southern Italy and Sicily).

The late Neogene sediments of the Mediterranean sea floor have been drilled extensively by several Deep Sea Drilling Project and Ocean Drilling Project expeditions (DSDP Legs 13 and 42A and ODP Legs 107, 160 and 161). The analyses of foraminifera which have been published in the reports of these expeditions have provided a basis for several of the Mediterranean Cenozoic biozonations as well as the geological, oceanographic and climatic interpretation of this area during the last c.15 MA.

In the southern Aegean (eastern Mediterranean), the extensive efforts of micropalaeontologists and stratigraphers from the University of Utrecht has resulted in the erection of several foraminiferal zonation schemes which deal specifically with this area, as well as many isolated biostratigraphic and palaeoenvironmental studies dealing with specific geological formations at one or more localities (Sections 6.6.2 and 6.6.3).

6.6.2 Planktonic foraminiferal biostratigraphy of the Mediterranean late Neogene

6.6.2.1 Introduction

As mentioned above, the Mediterranean behaved as a distinct bioprovince, separate from the open-ocean, during the late Neogene (Iccarino 1985). This provincialism,

which is particularly well expressed in the record of planktonic marine organisms including calcareous nannofossils (Section 5.6.1) and planktonic foraminifera, has caused difficulties when attempting to apply the 'standard' biozonations for the late Neogene (Iccarino and Salvatorini 1982). It is possible to subdivide the Neogene record of planktonic foraminifera in the Mediterranean at a level close to the FO of the species *Orbulina universa* in the Middle Miocene (Cita 1976), in terms of its similarities with the equivalent assemblages of the open-ocean.

At this point in the Middle Miocene, the Mediterranean section of the shrinking Tethys Ocean became separated from the Indian Ocean to the east and communications with the open-ocean were restricted to the narrow Gibraltar Sill which existed between Spain and North Africa in the west (Berggren and Philips 1971). This had a strong effect on the Mediterranean circulation patterns and resulted in a climatic control of the marine flora and fauna of this area (Cita 1976). Prior to the '*Orbulina* datum', the planktonic foraminiferal assemblages of the Mediterranean exhibited strong similarities to those of the open-ocean and as a result, the standard biostratigraphic zonations which have been established in the tropics, can be successfully applied to the sediments of this period. The assemblages of foraminifera after this crucial point, however, have progressively less affinities with those of the open-ocean and developed strong provinciality, due to the isolation of the Mediterranean, the climatic deterioration which started in the Middle Miocene and pronounced eustatic movements (Iccarino 1985).

In the latest Miocene, the Mediterranean was affected by the Messinian salinity crisis, during which the connections with the Atlantic Ocean to the west, were severely

restricted, sea level fell and extensive evaporite sedimentation took place (Chapter 4). Foraminifera can be found within the Messinian stage of the Late Miocene, in marine incursions which punctuate the evaporite deposition (Bizon and Müller 1977), but are characteristically impoverished and consist of dwarfed specimens of a few of taxa, including *Orbulina universa* and *Globigerina multiloba* (Cita 1976).

In the earliest Pliocene, a sudden re-establishment of connections across the Gibraltar Sill and the flooding of the desiccated Messinian basins, resulted in a rejuvenation of the marine faunas. An acme of the genus *Sphaeroidinellopsis*, is a characteristic feature of the sediments from this time period throughout the Mediterranean. The Pliocene foraminifera which were introduced from the Atlantic during the early Zanclian, evolved separately from those of the open-ocean. Therefore, provincialism developed once more in the Mediterranean.

6.6.2.2 Zonation schemes

Due to the distinct differences between the assemblages of late Neogene planktonic foraminifera in the Mediterranean and the open-ocean, and the corresponding difficulties which are involved in applying the 'standard' zonations, such as Blow (1969), various Mediterranean-specific schemes have been established (Figure 6.7). An early attempt at this, was the zonation scheme of Bizon (1967). This was established on land sections from western Greece and formed the basis for several schemes which succeeded it. All of Bizon's zones were based upon events in the late Neogene record of the genus *Globorotalia*, with the exception of the highly distinctive

Early Pliocene '*Sphaeroidinellopsis* zone' which is a feature of all the Mediterranean planktonic foraminiferal zonations (Figure 6.7).

Cita's (1973-1976) zonations which followed (Figure 6.7), were based upon the cores which were recovered by the first Mediterranean DSDP expedition (Leg 13, Tyrrhenian Sea), and differed from that of Bizon (1967) in its species concepts and the adoption of lineage and interval zones instead of assemblage zones. Cita (1976) estimated the ranges of the most common planktonic foraminifera in the Mediterranean Neogene and used the last occurrences of taxa, calibrated with palaeomagnetic data, to establish her zones. As well as presenting a detailed planktonic foraminiferal biozonation for the Late Miocene and Pliocene of the Mediterranean, which could be applied to ocean cores and land sections, Cita (1976) interpreted the faunal changes which she observed, in terms of the geological and oceanographic evolution of the Mediterranean during this period.

The earliest attempt at a planktonic foraminiferal biostratigraphy of the southern Aegean, was the work of Gradstein (1974). Working on land sections from Crete, Gradstein (1974) conducted a statistical study of Pliocene *Globorotalia* and discovered that the late Neogene history of this genus was characterised by the succession of three assemblages. These are the *Globorotalia margaritae/Globorotalia punctictulata, Globorotalia bononiensis* and *Globorotalia inflata* groups. Using these successive populations of *Globorotalia*, Gradstein (1974) proposed a tentative three-fold subdivision of the Cretan Pliocene (Figure 6.7).

The subdivision of Gradstein (1974) was subsequently incorporated into a more detailed Cretan zonation by Zachariasse (1975). Working on nine closely-sampled sections from western Crete, Zachariasse (1975) proposed a scheme of seven interval

Epoch	Stage	Blow '69	Bizon '67	Cita '73-'76	Z'hriasse '75	Spaak '83	Iccarino '85
Pleisto.	Early	N 22					*G. truncatulinoides*
							G. cariacoensis
Plio.	Piacenzian	N 21	*G. inflata*	*G. inflata*	*G. inflata* Assem.	*G. inflata*	*G. inflata*
			G. crassaformis	*G. elongatus*	*G. bononiensis* Int.	*G. crassaformis*	*G. aemailiana*
		N 20		*S. subdehiscenns*		*(G. punct)*	*G. puncticulata*
	Zanclian	N 19	*G. puncticulata*	*G. marg. / G. punct.*	*G. punt* Int	*G. m / G. p* Con Range	*G. punct. - G. marg.*
			G. margaritae	*G. marg. marg.*	*G. marg* Int. /	*G. margaritae*	*G. margaritae*
		N 18	*Sphaeroidinellopsis*	*Sphaeroidinellopsis*	*Sphaeroidinellopsis* ACME	*Sphaeroidinellopsis* ACME	*Sph. seminulina* s.l.
M i o c e n e	Messinian	N 17	*G. menardii mioceanica*		*G. conomiozea* Interval Zone		Non-distinctive Zone
							Globorotalia conomoizea
	Tortonian	N 16	*G. menardii menardii*		*G. acostaensis* Interval Zone		*G. suterae*
							G. obl. ex./ G. bull
							G. acostaensis
		N 15	*G. mayeri*		*G. continuosa* Assem.		*G. menardii* s. l.
	Serravalian	N 14					*G. siak / G. obl. obl*
		N 13					*G. subquadratus*
		N 12					*G. altispira*
		N 11					*G. preamen / G. peri*
		N 10					*O. universa*
	Langhian	N 9					*O. suturalis*
							Po. glomerosa s. l.
	Burdigal'n	N 8					*G. altiap / Cat dissi,*
		N 7					*G. dehiscens*
	Aquitanian	N 4 + 5 + 6					*G. d. dehiscens*

Figure 6.7 The various planktonic foraminiferal zonation schemes proposed for the Mediterranean Neogene, with the standard open-ocean zonation of Blow (1969) for comparison.

and assemblage zones and one acme zone, for the Middle Miocene to Late Pliocene period of the southern Aegean (Figure 6.7), which he calibrated with the benthic foraminiferal zonations of Freudenthal (1969) and Meulenkamp (1969), Section 6.6.3. The zonation of Zachariasse (1975) also illustrated the importance of the species of *Globorotalia* in the Mediterranean late Neogene, with this genus accounting for six of the eight events which defined the zones; the other two being the FO of *Globigerina acostaensis* and the acme of *Sphaeroidinellopsis*. The zonation scheme of Zachariasse (1975) has been applied by several authors, studying the stratigraphy or micropalaeontology of various late Neogene sections on Crete, such as Fortuin (1977), Dermitzakis (1980), Dermitzakis *et al.* (1979). As such, this zonation scheme is directly relevant to the present report.

The most recent biostratigraphic zonation for planktonic foraminifera in the Cretan late Neogene is that of Spaak (1983). Spaak made a quantitative study of the planktonic foraminifera in some 1400 Pliocene samples from Cretan, Sicilian and Calabrian sections, in order to evaluate the previous Mediterranean zonation schemes for this period. He also analysed material from the Atlantic Ocean and related the Mediterranean planktonic foraminiferal datum levels to climatically-induced migrations of the bioprovinces from this region, as well as studying water temperature through the analysis of *Orbulina* diameter, and proposing a hypothesis for the formation of the Pliocene laminites.

On Crete, Spaak (1983) re-evaluated many classic sections, such as Prassas, Kalithea, Agios Vlassios, Finikia and Myrtos, which had been studied previously by other Utrecht micropalaeontologists. As such, his work represents the most detailed study of

the Pliocene planktonic foraminifera from this area. The result of Spaak's quantitative analysis, was the proposition of a detailed nine-part subdivision for the whole of the Pliocene, determined by distinct datum levels in the genera *Globorotalia* and *Neogloboquadrina*. Spaak (1983) calculated the recogniseability of these nine intervals from single samples, and subsequently proposed six readily identifiable zones (Figure 6.7), of which five are present on Crete.

In the Lower Pliocene, Spaak's zonation does not differ greatly from the earlier schemes of Bizon (1967), Cita (1975) and Zachriasse (1975). Here, he recognised four zones; a *Sphaeroidinellopsis* acme zone, followed by three zones based upon the appearance, disappearance and combined ranges of *Globorotalia margaritae* and *Globorotalia puncticulata*. In the middle part of the Pliocene, due to "less distinct faunal changes" (1983, 51), the zonations of Spaak and other authors differ, and the recogniseability of his intervals five to eight, is poor. Spaak (1983) therefore grouped these intervals together in his larger *Globorotalia crassaformis* zone, which is succeeded by the uppermost Pliocene *Globorotalia inflata* (not present on Crete). Although Spaak's (1983) zonation is not a great improvement on the work of Zachriasse (1975) and others, in terms of the biostratigraphic assignment of single samples, a finer subdivision can be made when his nine intervals are applied to sedimentary sections.

Outside of the Aegean, Iccarino and Salvatorini (1982) proposed a refined planktonic foraminiferal zonation based upon their knowledge of Neogene assemblages from land sections in countries bordering the Mediterranean and several deep sea cores from ODP sites. Their zonation, which covers the entire Neogene and Quaternary,

achieves a high level of biostratigraphic subdivision by the use of events which were particularly widespread in the Mediterranean, but previously disregarded. Within the late Neogene interval, which is of interest to the present report, the zonation scheme proposed by these authors achieves a five-fold subdivision of the Upper Pliocene, by the introduction of the *Globigerina apertura, Globorotalia bononiensis, Globorotalia aemiliana/Sphaeroidinellopsis seminula* s.l. and the *Globorotalia crassaformis crassaformis* subzones (Figure 6.7). However, these four subzones were subsequently disregarded by Iccarino (1985) in her updated version of the Iccarino and Salvatorini (1982) zonation, because of doubt surrounding the FO of *Globorotalia bononiensis,* the range of *Globorotalia crassaformis crassaformis* and the LO of *Sphaeroidinellopsis.*

6.6.3 Studies on benthic foraminifera from the Cretan Neogene

There have been several palaeoenvironmental and biostratigraphic studies on late Neogene benthic foraminifera from the southern Aegean area, since the 1960s. The majority of these were conducted by micropalaeontologists from the University of Utrecht, and focus on numerous well-studied sections in central and western Crete.

Freudenthal in his extensive report on the stratigraphy of the Khania province of Crete (1969), paid special attention to the evolution of the large benthic foraminifera *Planorbulinella, Heterostegina* and their related genera. By analysing the length of the early spiral and the size of the initial chambers in specimens of *Planorbulinella,* Freudenthal (1969) established three species of this genus (*P. rokae, P. astriki* and *P. caneae*), which appear in succession during the Tortonian stage (Figure 6.8). Using

	Freudenthal (1969) *Planorbulinella* range-zones	Meulenkamp (1969) *Uvigerina* range-zones

PLIOCENE — PIA / ZAN

MIOCENE — MESS / TORT / SERR

Freudenthal zones: *caneae*, *astriki*, *rokae*

Meulenkamp zones: *arquatensis*, *lucasi*, *cretensis*, *felixi*, *gaulensis*, *selliana*, *praeselliana*, *melitensis*
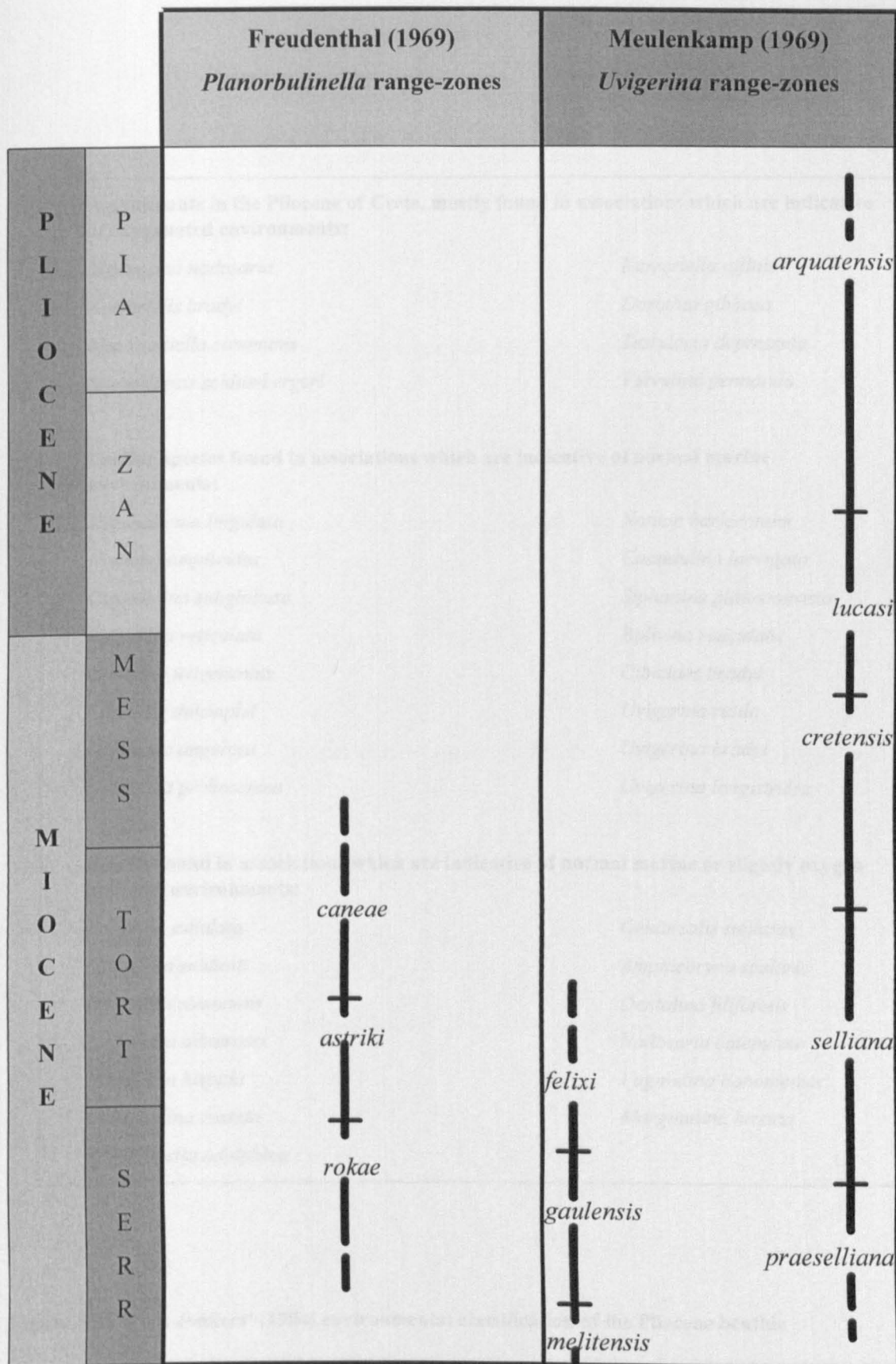
Figure 6.8 Benthic foraminiferal zonations proposed for the Cretan late Neogene.

Agglutinants in the Pliocene of Crete, mostly found in associations which are indicative of oxygenated environments:

Bigenerina nodosaria   *Karreriella affinis*

Karreriells bradyi   *Dorothia gibbosa*

Martinottiella communis   *Textularia depressula*

Sigmoilopsis schlumbergeri   *Vulvulina pennatula*

Benthic species found in associations which are indicative of normal marine environments:

Heronallenia lingulata   *Nonion barleeanum*

Nonion pompiloides   *Cassidulina laevigata*

Cassidulina subglobosa   *Siphonina planoconvexa*

Siphonina reticulata   *Bolivina reticulata*

Cibicides uvigeranius   *Cibicides bradyi*

Cibicides dutemplei   *Uvigerina rutila*

Uvigerina angulosa   *Uvigerina bradyi*

Uvigerina proboscidea   *Uvigerina longistriata*

Species found in associations which are indicative of normal marine or slightly oxygen deficient environments:

Bulimina subulata   *Oridorsalis stellatus*

Gyroidina soldanii   *Amphicoryna scalaris*

Dentalina communis   *Dentalina filiformis*

Nodosaria albatrossi   *Nodosaria catenulata*

Nodosaria hispida   *Vaginulina bononiensis*

Marginulina costata   *Marginulina hirsuta*

Stilostomella adolphina

Figure 6.9 Part 1. Jonkers' (1984) environmental classification of the Pliocene benthic foraminifera of Crete. Continued below.

Species found in slightly oxygen deficient environments:

Bulimina barbata   Uvigerina pygmea

Uvigerina carinata   Bolivina alata

Bolivina antiqua   Bolivina dilatata

Species found in associations which are indicative of a moderate degree of oxygen deficiency:

Valvulineria complanata   Baggina totomiensis

Cancris auricula   Bulimina costata

Species found in associations which are indicative of a high degree of stagnation:

Uvigerina cylindrica   Uvigerina c. gaudryinoides

Stilostomella advena   Fursenkoina sp.

Bulimina elongata   Bulimina exilis

Uvigerina bononiensis   Bolivina spathulata

Epiphytes, which are relatively abundant as allochthonous elements in oxygen depleted environments:

Bolivina subexcavata   Asterigerina mamilla

Asterigerina planorbis   Cibicides lobatulus

Elphidium fichtellianum   Elphidium aculeatum

Elphidium crispum   Hanzawaia boueana

Planorbulina mediterranensis   Rosalina globularis

Reusella spinulosa

Figure 6.9 Part 2. Jonkers' (1984) environmental classification of the Pliocene benthic foraminifera of Crete.

this simple, lineage-based zonation of the Tortonian, Freudenthal attempted to correlate between the various formations which he established in the west of Crete, however, he stated that "the possibilities for correlating Neogene formations by means of *Planorbulinella* are rather restricted" (1969, 159).

In the same year, Meulenkamp (1969) published his report on the Neogene geology of the Rethymnon province of Crete, in which he also utilised benthic foraminifera as a means of biostratigraphy and correlation. He analysed the phylogeny of two groups of the benthic genus *Uvigerina*; the *U. meltensis* and the *U. cretensis* groups. The evolutionary trend within these two groups is that of an increase in the proportion of fully developed and primitive uniserial chambers, the development of a more regular series of uniserial chambers and the development of more slender tests. In addition, Meulenkamp studied the relationship between the number of primitive and fully developed uniserial chambers in individuals at the same growth stage from different stratigraphic positions within the late Neogene record of both groups, and used this to subdivide the lineages of *Uvigerina* into eight biometrically-defined species (Figure 6.8). The combined range of the two lineages covers the interval from the base of the Serravalian to the Late Pliocene, which permitted Meulenkamp to make a detailed subdivision of the late Neogene into seven range zones (Figure 6.8).

This scheme was a distinct improvement on Freudenthal's (1969) benthic foraminiferal zonation and has been used, often in conjunction with the planktonic foraminiferal zonation of Zachariasse (1975), by other stratigraphers working with the Cretan Neogene. Meulenkamp's (1969) zonation, however, does suffer from two drawbacks. First, the evolution between the biometric 'species' is gradual, so that the

boundaries separating the successive zones are not well defined. Secondly, whilst the Upper Miocene part of the zonation is finely subdivided, the Pliocene is represented by only two zones; the fairly large *Uvigerina lucasi* zone, which covers the late Messinian to early Zanclian time period, and the huge *Uvigerina arquatensis* zone, which represents the rest of the Pliocene. More recently, in attempting to apply the uvigerinid zonation scheme of Meulenkamp (1969) to correlate between the various Neogene formations of the Ierapetra region of Crete, Fortuin (1974) described the species *Uvigerina praeselliana* which "precedes the first member of Meulenkamp's cretensis lineage" (Zachariasse 1975, 13) and overlaps with *Uvigerina gaulensis* of the melitensis group.

In an attempt to prove or disprove the various models which have been proposed for the formation of the highly distinctive repetition of laminated and non-laminated marls in the Pliocene of Crete, Jonkers (1984) studied the interrelation between the changes in environmental variables (sediment compound analysis, oxygen and carbon isotope analysis) and the benthic foraminiferal faunas of eight Cretan sections. He classified the Pliocene benthic foraminifera of Crete into those species which are indicative of normal marine environments, and water which is progressively oxygen deficient, or 'stagnated' (Figure 6.9). Jonkers (1984) then interpreted the changes in the proportions of these taxa through the sections, in terms of the marine environment at the time of deposition. His quantitative results are not directly relevant to the present study, however the descriptions of the Pliocene benthic foraminiferal faunas from the Finikia Formation of the Iraklion province of Crete and the Myrtos Formation of south-central Crete have been indispensable to the present study (Sections 11.2 and 11.3). Jonkers (1984) discussed the taxonomy of many species of

benthic foraminifera, and identified a few events in the Early Pliocene history of *Uvigerina* and *Bulimina*, which may be useful for biostratigraphy.

6.6.4 The biostratigraphic and palaeoenvironmental interpretation of planktonic and benthic foraminifera isolated from samples of archaeological ceramics

By utilising the studies outlined above, the assemblages of planktonic and benthic foraminifera which were isolated from two small samples of Bronze Age archaeological pottery in Section 6.5.4., can be interpreted in terms of the biostratigraphic zone, geological period, and palaeoenvironment in which the raw materials of ceramic production were deposited.

On the basis of the common occurrence of the planktonic foraminiferal genus *Sphaeroidinellopsis*, dwarfed specimens of *Globorotalia acostaensis* and a possible specimen of the species *Globorotalia subscitula*, the assemblage of foraminifera in sample MFK 93/105 (Section 6.5.4.2) is indicative of the Early Pliocene (Zanclian). In the earliest Pliocene, the planktonic foraminiferal assemblages of the Mediterranean are dominated by *Sphaeroidinellopsis*, and many authors have used this acme to propose a *Sphaeroidinellopsis* zone (Figure 6.7). Zachariasse, in his study of planktonic foraminifera from the Neogene of Crete, noted that the species *Globorotalia subscitula* first occurs in the earliest Pliocene, near to the base of this zone, and stated that its presence "may be used as an additional marker to recognise the Mediterranean limit between Upper Miocene and Pliocene" (1975, 37). The same author also reported that dwarfed specimens of *Globigerina acostaensis* are characteristic of the Pliocene part of the Cretan Neogene succession.

The assemblage of benthic foraminifera in sample MFK 93/105 (Section 6.5.4.2) is similar to the rich associations which are reported by Jonkers (1984) from the homogeneous and laminated Pliocene marls of Crete. By comparing the abundance of the various benthic species which occur in this sample with the environmental classification of Jonkers (1984; Figure 6.9), it can tentatively be suggested that the assemblage which was liberated from MFK 93/105 is characteristic of relatively normal marine environments, due to the high proportion of the species *Cibicides ungerianus* (Figure 6.10). There is also a small proportion of species, such as *Bolivina spathulata, Bulimina exilis, Astigerina mamilla* and *Elphidium fichtellianum*, which are indicative of highly stagnated marine environments or allochthonous epiphytic species which are usually found in oxygen depleted environments (Figure 6.10). This could be interpreted in terms of bioturbation and burrowing by organisms living in the sediment after deposition, or may suggest that the potter deliberately or unintentionally utilised raw material from more than one horizon for the production of the vessel from which sample MFK 93/105 originated. However, it is difficult to distinguish between these and other factors such as the reworking of foraminifera at the time of deposition.

Jonkers (1984) analysed several hundred specimens from each sample, in order to interpret the environmental trends in the Pliocene laminites of Crete. Therefore, the above interpretation, which is based very small numbers of specimens is likely to be less reliable.

Planktonic foraminifera are not very well represented in the assemblage which was liberated from sample Kn 95/187 (Section 6.5.4.3). However, the presence of
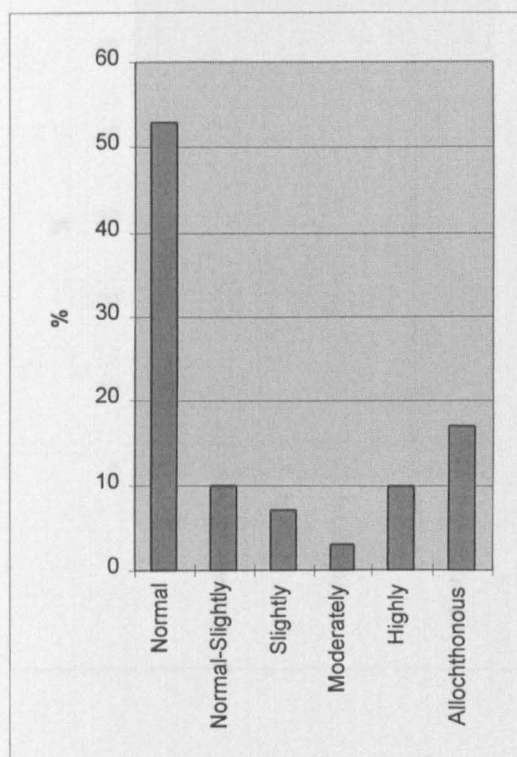
Figure 6.10 Palaeoenvironmental analysis of the assemblages of Pliocene benthic foraminifera isolated from sample MFK 93/105, according to the classification scheme of Jonkers (1984: Figure 6.9). The labels 'slightly', 'moderately' and 'highly' refer to the degree of stagnation, and 'normal' refers to normal oxygenated marine conditions. The allochthonous group is composed of epiphytes and commonly occurs in oxygen depleted (stagnated) environments.

Sphaeroidinellopsis (though not in abundance) but desired specimens of the species *Globigerina oceanensis*, may suggest a Pliocene date for this assemblage.

The relatively rich assemblage of benthic foraminifera which was isolated from this sample (Section 6.5.4.3) is also similar to the Pliocene Pliocene assemblages which are presented by Jenkers (1984). This assemblage is dominated by species which are indicative of highly stagnated marine conditions (i.e. the appearance) and also contains several epiphytic species. These species are allochthonous components in oxygen depleted environments. The other characteristic components of the assemblage are species such as *Bolivina robusta* and *G. chinensis*, which are usually found in associations indicative of stagnated/stressed conditions (Figure 6.11). Notwithstanding the presence of reworked foraminifera, which may be a consequence of several factors, the exceptionally good preservation which has been liberated from sample Kn 95/187 is indicative of Pliocene sediments, which were deposited in a poorly oxygenated environment.

The significance of the foraminiferal assemblages which have been isolated from samples MPK 93/101 and Kn 95/187 in terms of the inferences and timing of organic production is discussed in Sections 7.1.2 and 11.3.

The chart shows:
- Normal: ~17
- Normal-Slightly: ~6
- Slightly: 0
- Moderately: 0
- Highly: ~53
- Allochthonous: ~24

Y-axis: % (0 to 60)

Figure 6.11 Palaeoenvironmental analysis of the assemblages of Pliocene benthic foraminifera isolated from sample Kn 95/187, according to the classification scheme of Jonkers (1984: Figure 6.9). The labels 'slightly', 'moderately' and 'highly' refer to the degree of stagnation, and 'normal' refers to normal oxygenated marine conditions. The allochthonous group is composed of epiphytes and commonly occurs in oxygen depleted (stagnated) environments.

Sphaeroidinellopsis (though not in abundance) and dwarfed specimens of the species *Globigerina acostaensis*, may suggest a Pliocene date for this assemblage.

The relatively rich assemblage of benthic foraminifera which was isolated from this sample (Section 6.5.4.3) is also similar to the Cretan Pliocene associations which are presented by Jonkers (1984). This assemblage is dominated by species which are indicative of highly stagnated marine environments (e.g. *Bolivina spathulata*) and also contains several epipyhtic species which are often found as allochthonous components in oxygen depleted environments (Figure 6.11). Less frequent components of the assemblage are species such as *Cibicides uvigeranius* and *C. dutemplei*, which are usually found in associations indicative of normal marine environments (Figure 6.11). Notwithstanding the presence of these normal marine species, which may be a consequence of several factors, the association of benthic foraminifera which has been liberated from sample Kn 95/187 is indicative of Pliocene sediments which were deposited in a poorly oxygenated environment.

The significance of the foraminiferal assemblages which have been isolated from samples MFK 93/105 and Kn 95/187 in terms of the provenance and technology of ceramic production is discussed in Sections 11.2 and 11.3.

6.7 Approach to studying archaeological ceramics using foraminifera

6.7.1 Procedures for analysing assemblages of foraminifera in ceramic thin sections

In the present report, thin sections of archaeological ceramics were systematically scrutinised under the light microscope at a magnification of 100 x in order to study all

specimens of foraminifera which were present. A magnification of 400 x was used for the detailed analysis of small specimens, as well as the observation of fine details such as wall structure. All of the identifiable, partly identifiable, and distinctive, but unidentifiable specimens of foraminifera were illustrated, so that a visual comparison could be made between the foraminifera in different thin sections. This also aided the identification of unidentifiable specimens at a later date. Whilst studying the foraminifera which were present within in a particular thin section, observations were made of any particularly significant styles of preservation, the association of foraminifera with any other microfossils, as well as the component(s) of the ceramic in which the specimens appeared.

6.7.2 Description

By studying the associations of foraminifera which are present in thin sections of archaeological pottery using the method which is described above, it is possible to describe ceramics in several ways. In most thin sections which contain foraminifera, some specimens can be identified to genus or species level. From these identifications, a description of the foraminiferal assemblage can be compiled for each sample of archaeological pottery (e.g. Figures 11.3 and 11.8). These descriptions cannot be considered to be representative of the whole assemblage of foraminifera in the sherd, due to the low level of identifiability of some specimens, but may however, be used to characterise individual samples of archaeological pottery. It is also important to accompany this information with observations on the state of preservation of

foraminifera in each sample, as well as the component(s) of the ceramic in which the specimens occur (i.e. in the groundmass, inclusions or clay mixing).

Another means of describing samples of archaeological pottery by their assemblages of foraminifera is through an interpretation of the biostratigraphic zone, geological period or palaeoenvironment in which the raw materials of ceramic manufacture were deposited. If it is possible to identify numerous specimens of foraminifera in a particular thin section, then biostratigraphic and palaeoenvironmental schemes, such as those described in Section 6.6, can be applied to the assemblage. In the present report, very few assemblages could be positively dated with foraminifera alone, as it was not often possible to identify the specimens of planktonic foraminifera to species level in thin section. However, these assemblages were compared to the more precise biostratigraphic assignments that were achieved by studying calcareous nannofossils (Appendix II).

6.7.3 Classification

It is not wise to classify thin sections of archaeological ceramics by the presence/absence of foraminifera alone. Riley (1983, 287) believed that this group of microfossils "provide independent and unequivocal criteria for grouping fine wares ... into fossiliferous and non-fossiliferous" in his analysis of Late Minoan ceramics from Knossos. However, as ceramic thin sections represent a very small part of the complete sherd or vessel from which they are prepared, it is possible that some sections will not cut any specimens of foraminifera, if they were an infrequent component of the whole sample (Section 11.2). Therefore, it is inadvisable to consider

the presence/absence of foraminifera in ceramic thin sections independently of the other components of the fabric. By treating foraminifera as but another type of aplastic inclusion, and considering their occurrence along with the rest of the thin section, it is possible to identify those samples which may contain foraminifera but suffer from poor representation. In this way the resulting ceramic classifications will have more meaning.

In all cases, the utility of foraminifera, and indeed all microfossil assemblages, as a means of grouping and classifying samples of archaeological ceramics relies heavily upon the success with which they have been described (Section 6.7.2). In this respect it is very important to note the component(s) of the ceramic in which foraminifera occur within thin sections of ceramics (Section 6.2), as well as their preservation (Section 6.3). If it has been possible to identify the specimens of foraminifera in thin sections of archaeological pottery, then comparisons can be made between the corresponding assemblage descriptions (Figures 11.3 and 11.8) in order to confirm similarities between samples. If identification has not been possible, then it may be possible to infer relationships between samples which petrographically are very similar, on the basis of distinctively-shaped individuals. In this case, illustrations are indispensable, although some experience is needed in order to determine those shapes which are significant and those which are not.

Where it has been possible to compile assemblage descriptions of foraminifera from individual thin sections of archaeological pottery, these can interpreted in terms of geological age or palaeoenvironment, as outlined above. This information represents the final criterion which can be used to classify sherds, and is highly contextual, in

that it pertains to the nature of the raw materials of ceramic manufacture, which is one of the main purposes of classification.

6.7.4 Provenance

Foraminifera may be used to provenance ceramics in several ways. On a very broad scale, their mere presence, as seen in ceramic thin sections, may well be used, in combination with the rest of the fabric, to infer the possible location of the raw materials used in ceramic manufacture, where sediments containing foraminifera have a very restricted distribution (e.g. Stilborg 1997). However, where this is not the case (e.g. Crete and the southern Aegean), the biostratigraphic and palaeoenvironmental interpretation of foraminiferal assemblages can be used to ascertain the provenance of archaeological ceramics (Riley 1983; MacGillivray *et al.* 1988; Troja *et al.* 1996; Riley *et al.* n.d.). In this case, the precision of the biostratigraphic, or palaeoenvironmental information will determine the stratigraphic and geographic precision with which the specific sources of raw materials which were used for ceramic manufacture can be located (compare the provenance interpretations of pottery from Knossos by Riley 1983 and Riley *et al.* n.d.).

Because of the poor identifiability of planktonic foraminifera in the thin sections of archaeological ceramics which are analysed in the present report (Figure 11.3 and 11.8), the accuracy of biostratigraphic interpretations based upon them is equally as low. Therefore, the more detailed geological assignments which were achieved by the analysis of calcareous nannofossils (Appendix AII), have been used. Nevertheless, these assemblages of foraminifera have been very useful where calcareous

nannofossils were absent, as well as to supplement the information provided by this latter group of microfossils.

6.7.5 Technology

The nature of foraminifera in thin sections of archaeological ceramics is not well suited to the study of pottery technology. Crude inferences about the degree of firing in ceramics may be based upon observations of the preservation of foraminifera in thin section (Davis 1951; Section 6.4.3), however, this has no routine application. The presence of foraminifera in calcareous temper or clay mixing, can be used to determine the nature and geological age of such materials, where they occur in ceramics (Sections 11.2 and 11.3).

7 Ostracods

7.1 Introduction

Ostracods are microscopic crustaceans with a bivalved calcite shell, which inhabit many types of aqueous environments, from the deep sea to rivers and lakes. They have a very long geological history which begins in the Cambrian and despite being complex metazoans, they are commonly classified as microfossils, due to their small size (c. 0.7-5 mm). The calcareous, bivalved carapaces of ostracods frequently occur in sieved sedimentary residues, prepared for the study of foraminifera, and these two groups may be informally referred to as the 'larger calcareous microfossils', to distinguish them from calcareous nannofossils which are an order of magnitude smaller. The ostracod carapace contains many body parts, including appendages, eyes and complex sexual organs (Figure 7.1), however, only the valves are preserved after death (either singularly or less commonly, as a pair), as the soft organs quickly decay. Consequently, fossil ostracods are classified by the internal morphology of their shells, which reflect some details of the soft parts which they once contained (e.g. muscle scars, and eye spots, stalks and sinuses), as well as the external 'morphology' and 'ornament' (Figure 7.1). Ostracod valves also exhibit strong sexual dimorphism, and given that they grow by a stepwise series of moultings ('ecdysis'), it is possible to identify juveniles ('instars'), as well as male and female adult carapaces, even in fossil assemblages.

Ostracods may have a benthic or planktonic life habit. Benthic ostracods can be epifaunal (crawling and grazing on the sediment surface), infaunal (burrowing
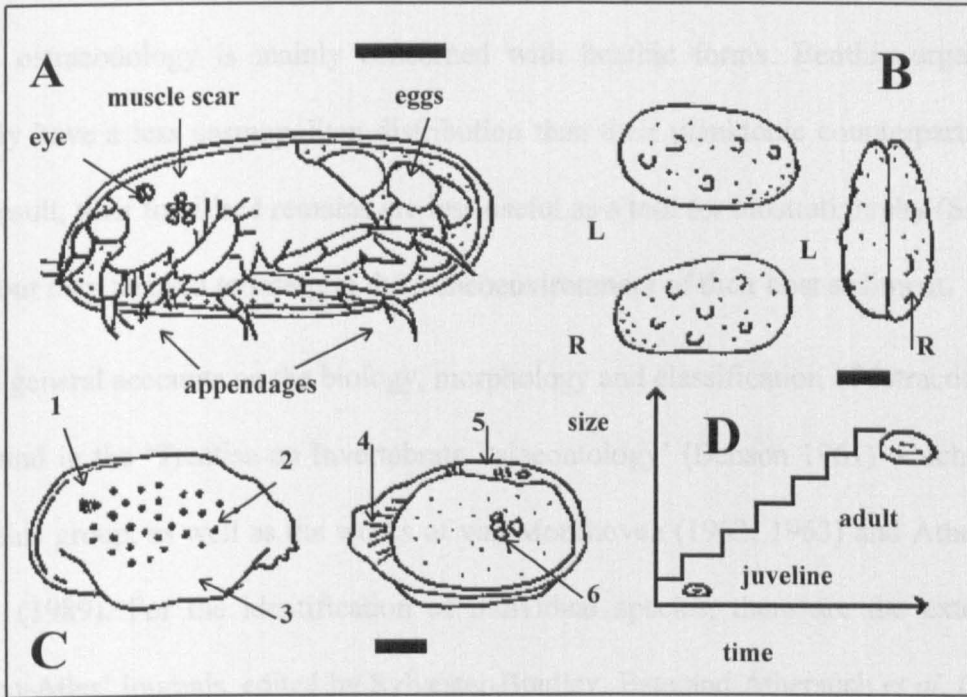
Figure 7.1 The morphology of ostracod shells and soft parts. Living ostracod with one valve removed to reveal soft parts (A), orientation of ostracod valves (B), morphology and ornament on exterior and interior of ostracod valves (C: 1 = eye spot, 2 = pits, 3 = alar wing, 4 = inner lamella with marginal pore canals, 5 = hinge and 6 = muscle scar), and step wise growth of ostracods. Scale bars = 0.1 mm. After Brasier (1980) and Sissingh (1972).

through the uppermost layers of sediment), or epiphytic (living on algae and other plants). Planktonic (free swimming) ostracods evolved in the Devonian and Carboniferous Periods, but have a poor geological record due to the dissolution of their thin calcareous shells by undersaturated sea water after death. For this reason, fossil ostracodology is mainly concerned with benthic forms. Benthic organisms usually have a less cosmopolitan distribution than their planktonic counterparts, and as a result, their fossilised remains are less useful as a tool for biostratigraphy (Section 7.4), but may be used to interpret the palaeoenvironment of their host sediment.

Good general accounts on the biology, morphology and classification of ostracoda can be found in the 'Treatise on Invertebrate Palaeontology' (Benson 1961) which deals with this group, as well as the works of van Morkhoven (1962; 1963) and Athersuch *et al.* (1989). For the identification of individual species, there are the extensive 'Stereo-Atlas' journals, edited by Sylvester-Bradley, Bate and Athersuch *et al.* (1973-1991), as well as Bate and Robinson (1978) and Bate *et. al.* (1982).

7.2 The occurrence of ostracods in archaeological ceramics

Ostracod shells are very conspicuous in ceramic thin sections, where present, and have been observed by Whitbread (1995, Lesbos), Alaimo *et al.* (1997, Sicily), Whitelaw *et al.* (1997, Crete), Day *et al.* (1999, Israel) and Riley *et al.* (n.d., Peloponese). They can occur in calcareous inclusions, clay pellets, or more commonly, within the groundmass (Figure 7.2). In the thin sections of archaeological ceramics which have been analysed in the present report, there were no occurrences of ostracods within 'grog', however, this is a definite possibility, if crushed pottery

containing ostracods is used as temper. Ostracod valves mainly occur in those ceramics which contain a reasonably high percentage of calcite in the matrix. Within the thin sections of such 'calcareous fabrics', ostracods can appear in great numbers (Riley *et al.* n.d.) or may be very scarce (Whitbread 1995). There are several factors which determine the abundance of ostracods valves within a particular thin section, such as the density of shells in the original artefact, the orientation of the section relative to the original artefact, as well as the size of the thin section itself. In a particular fabric group, some variation in the numbers of ostracods per section can be expected, and within those fabric groups in which they represent a low-frequency inclusion, it is not unusual for some small thin sections to contain no ostracod valves (Section 11.2).

In ceramic thin sections, ostracods appear as thin crescent-shaped calcite inclusions when the two valves are dislocated, or calcite ellipses when both valves are intact (Section 7.3). The size and curvature of ostracods in thin section is strongly related to the orientation of the section relative to the carapace, so that a range of shapes may arise from the differential sectioning of a single specimen. This is the first difficulty associated with the study of ostracods in thin section, the second is that very few of the important morphological features which are used to classify ostracod shells are visible (Figure 7.1).

Thin sections of ostracod shells often feature inflated ends which is a consequence of the characteristic thickening of the valve margin. In some rare cases, it may be possible to identify other features of the valve margin, such as hinges (Figure 7.2), inner lamella structures and fused zones. However, little additional information for the

identification of ostracod specimens can be gleaned by the occurrence of these features in thin section. Genotypic ridges, tubercles, reticulae and alar projections or wings appear as two-dimensional bumps and depressions in the outline of the valve, and as such are not much help in classifying ostracods in thin section.

In fact, the study of ostracods in thin section is so difficult that it is often impossible to distinguish between male and female, juvenile and adult valves, or orientate specimens (i.e. determine between anterior and posterior, dorsal and ventral). As a result, very little work has been carried out on ostracods in thin section. Ostracods are usually studied as three-dimensional specimens, mechanically and chemically isolated from rock or sediment samples.

In some cases it can be difficult to distinguish between ostracod valves and other types of shells occurring in thin sections of archaeological pottery, especially when the ostracod valves are fragmented. The two main types of shell with which ostracod fragments can be confused are small pieces of macrofossil (bivalve, brachiopod or cephalopod) shell and broken foraminiferal tests. Characteristic features which may appear at the margin of ostracod shells usually assist their identification, these include the hinge and inner lamellar structures which are mentioned above. Where such features are absent, it is necessary instead to study the structure of the calcite wall. Foraminifera can be identified by the presence of pores, cancellate ridges and spines (Section 6.1) and macrofossil fragments should be distinguishable on the basis of their complex multi-layered wall structure, whereas ostracod valves usually have a homogeneous single-layered structure (although they may have a thin outer layer; Section 7.3). If it is still difficult to determine whether a particular shell fragment, seen in thin section, is that of an ostracod, it may be necessary to make a decision

A

B

C

D

Figure 7.2 The occurrence of ostracods in thin sections of Bronze Age archaeological ceramics from the Mediterranean: (A) within the clay matrix (MAK 96/9), (B) within a calcareous inclusion (MAK 96/166) and (C) within a clay pellet (MAK 96/229). (D) the preservation of an ostracod hinge structure in thin section (MAK 96/119). A, B and C = XP, D = PPL. Field of view = 0.5 mm (A and D), 2 mm (B and C).

based upon the overall nature of the particular sample, (i.e. whether or not any definite ostracod valves are present, as well as the nature of their wall structure and preservation). In such cases, previous experience is very important.

7.3 The preservation of ostracods in archaeological ceramics

There is an interesting variation in the state of preservation of ostracods in the ceramic thin sections which have been analysed in the present report, in terms of the carapace itself as well as the clay matrix surrounding it. This can been interpreted in terms of the taphonomy of the original sediment as well as the history of the ceramic artefact. Examples of the main types of ostracod preservation which have been encountered in ceramic thin sections in the present report are illustrated and interpreted below.
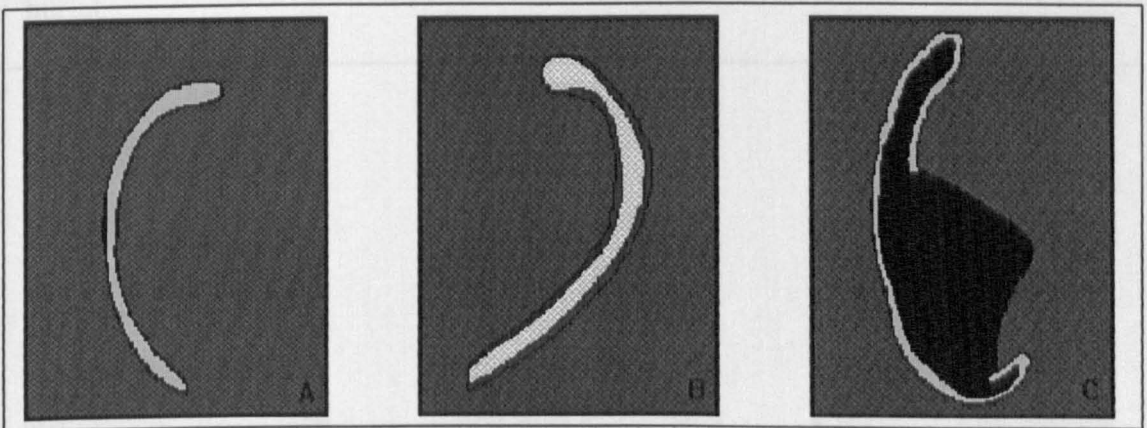
A. Homogeneous micrite wall

A well preserved, unaltered ostracod valve.

B. Three-layered calcite wall

A three-layered structure consisting of a thick central layer surrounded between two thinner, outer layers. This can be preserved in micrite, as a single crystal of calcite, or

as micrite sandwiched between two layers of perpendicular fibrous calcite growth. All of these situations are likely to represent the preservation of the main calcite wall of the shell and its chitinous outer layer by replacement at some stage in the taphonomy of the ostracod valve. The preservation of an outer layer by fibrous (acicular) crystals is likely to be the result of calcite deposition in a void, left by the decomposition of the chitinous layer.

C. Double micrite layered shell with a single central void

Although this could be interpreted as the preservation of the chitinous outer layer of the shell without the main wall, it is unlikely. More realistically, this type of preservation arises from the incomplete infilling of an ostracod-shaped void by calcite, precipitated from a solution which penetrated the artefact during usage or burial.
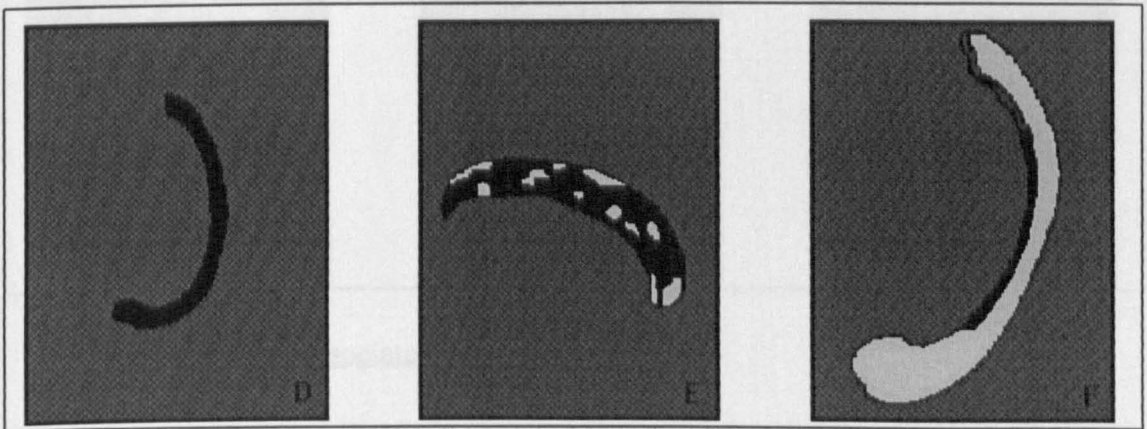
D. Ostracod-shaped void

In this situation, the calcareous ostracod valve has been removed at some stage in the history of the artefact by acidic conditions or high firing (Section 7.4). It may also be due to excessive abrasion, during thin section preparation.

E. Ostracod-shaped void with fragmentary calcite remains

This scenario may arise by the incomplete removal of the calcite wall by the processes which are described above, or alternatively, the partial infilling of an ostracod-shaped void by calcite precipitation. The former is more likely, as the precipitation of calcite into voids usually takes place in an ordered fashion from the void margin inwards, and not randomly.

F. Linear void(s) along the inner margin of valve

This may arise by the loss of the outer part of the three-layered structure due to dissolution or the exploitation of weaknesses during thin section preparation. The outer layer can be missing entirely or represented by fragments of calcite.
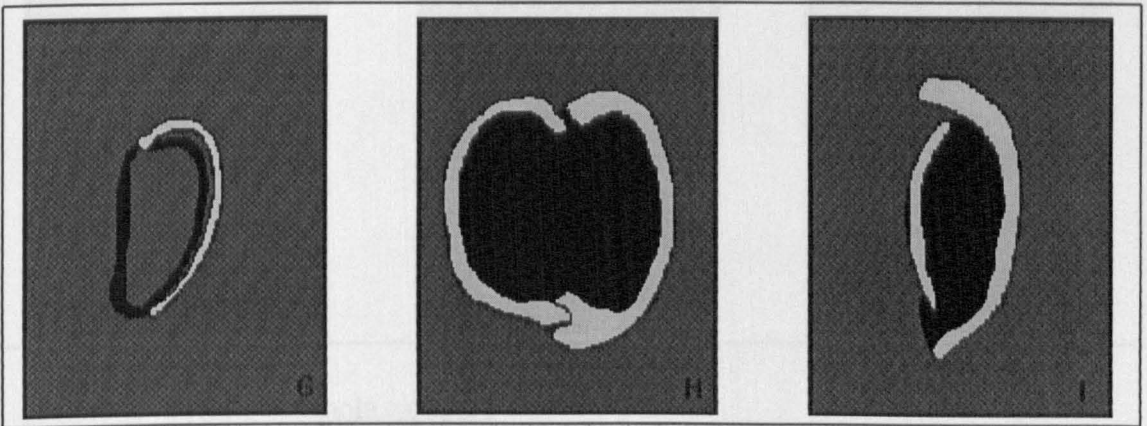
G. Ring-shaped void associated with a single valve

The clay pellet inside the ring-shaped void has detached itself from the surrounding matrix and its association with the curved ostracod valve suggests that this process may be due to some sort of expansion or contraction of the shell. However, no definite explanation can be given.

H. Two valves joined together enclosing a void

Preservation of the whole carapace with a void representing the former position of the organisms soft-parts. Note the hinge structure which is present in this example.

I. Two associated valves with one apparently larger than the other

Notwithstanding the fact that many ostracod taxa posses a slight size difference between their two valves, this type of preservation in thin section is likely to arise from the dislocation of the two valves and their movement relative to each other in an anterior-posterior direction. The two valves can be in contact with one another or apart, and the area between the two can be occupied by a void or various types of clay.
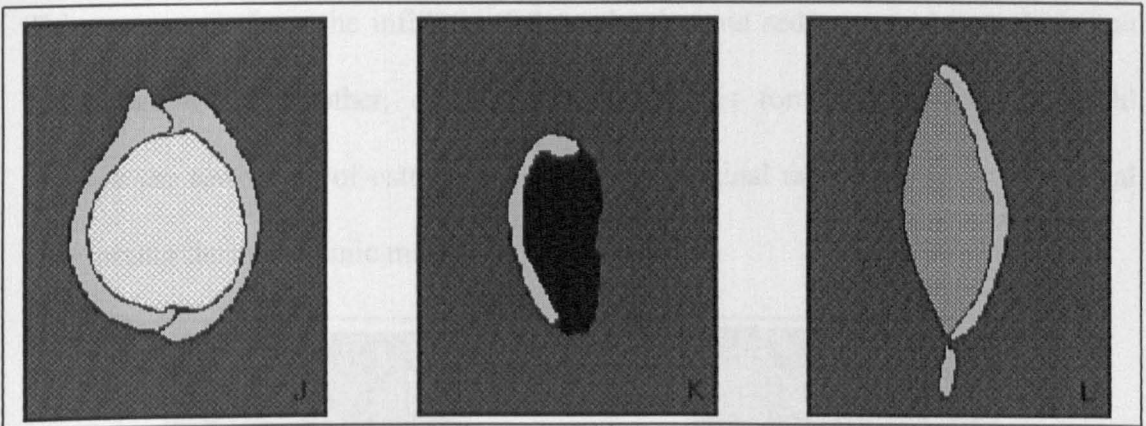
J. Calcite infilling of whole carapace

The void between the two valves of a complete carapace can be infilled by micrite, sparry calcite, a single large calcite crystal or a combination of these. This is likely to be the result of secondary calcite precipitation taking place in the original sedimentary environment during diagenesis or by groundwater passing through the ceramic artefact during burial in the archaeological record.

K. Single valve with a large associated void

This situation may arise from the removal of one of the valves by dissolution after burial, or during thin section preparation. However, ostracods found in this way are often well-preserved and it is therefore unlikely that the other shell could have been selectively removed. It is feasible that one valve was lost prior to burial and the void then represents the space left by the decomposition of the soft parts which has not been invaded by the clay matrix. These voids may also be infilled by calcite in a number of ways.

L. Different coloured/textured clay inside a single valve

This may result from the infilling of the valve by one sediment and its subsequent incorporation into another, different sediment. This form of preservation could indicate the reworking of ostracod valves in the original raw material or intentional clay mixing during ceramic manufacture.
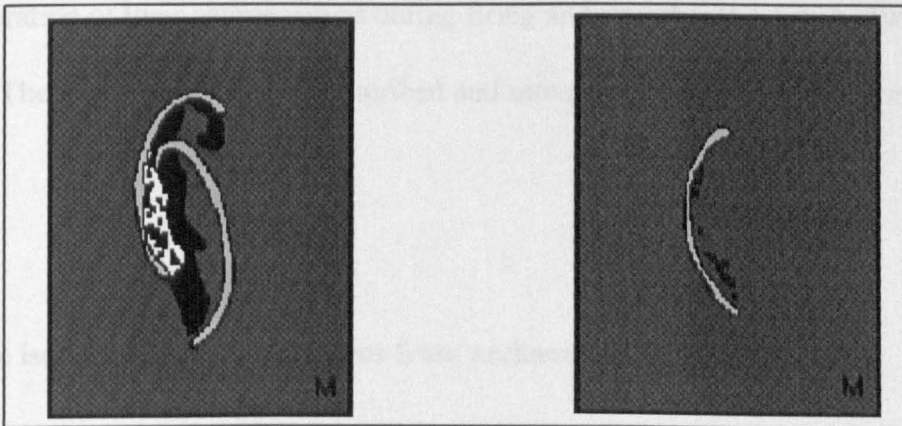
M. Oxidised organic matter

Organic matter can be associated with ostracod valves in various ways and is likely to have originated from the oxidation of chitinous soft matter during the process of firing. The organic deposits can be divided into amorphous organic 'staining' of the

clay micromass and discrete round to sub-round bodies of various sizes. The colour of this organic matter seen in plane-polarised light, ranges from translucent red-brown to opaque black.

7.4 The behaviour of ostracods during the firing of ceramics

In order to assess the way in which ostracod valves behave during the firing of pottery, numerous thin sections of differentially-fired Early Minoan archaeological ceramics were scrutinised under the microscope (Section 6.4). These samples, for which firing temperature and atmosphere estimations were obtained by the observation of clay vitrification structures in the SEM, revealed that ostracods and foraminifera exhibit a decrease in their state of preservation with increasing temperature above c. 750 °C (Figure 6.6). This process of degradation, which can result in the total destruction of ostracods in ceramics, is likely to be a consequence of the alteration of their calcite valves during firing and its subsequent re-hydration after firing. These investigations are described and interpreted in detail elsewhere (Section 6.4)

7.5 The isolation of ostracod valves from archaeological ceramics

7.5.1 Introduction

Due to the severe difficulties involved in identifying ostracod specimens from thin sections of archaeological ceramics, attempts were made to isolate complete valves from small samples of original artefacts. The mechanical and chemical isolation of

ostracods and foraminifera from rock samples is the standard method of preparation in micropalaeontology and from suitable samples produces large numbers of complete specimens which can be studied easily using a binocular microscope. In order to determine whether three-dimensional ostracod specimens can be isolated from reasonably well-fired archaeological ceramics, five samples of Late Neolithic pottery were processed in this way.

7.5.2 Samples

Five Late Neolithic sherds from the site of Makrygialos near Thessaloniki, Greece, were chosen for this pilot study. All of the samples contained ostracods in thin section and were reasonably well-fired, although clearly not so highly fired that their calcite had been destroyed. Only a small piece of each sample (c. 2-3 cm^3) was available for this purpose. Although, by archaeological standards the destruction of such quantities of material may seem rather wasteful, much more is usually required for a standard micropalaeontological preparation.

7.5.3 Equipment and procedure

The equipment and procedure used to isolate ostracods from the Neolithic pottery sherds in this experiment, were identical to those described for the isolation of foraminifera from archaeological ceramics (Section 6.5).

<u>7.5.4 Results</u>

The five samples from Makrygialos were scrutinised under the binocular microscope at magnifications of 15-35 x before processing, in order to determine whether the ostracods which were visible in thin section, could be seen on the surface of the sherds. A thorough search over all surfaces revealed several ostracod valves embedded in the clay matrix of the sherds, as well as a section of an ostracod shell exposed on a flat surface of one sample, which had been produced by thin sectioning.

As in the experiments which are outlined in Section 6.5, the Neolithic pottery sherds were difficult to break down using the standard processing techniques. Each sample had to be boiled, washed and sieved many times until a suitable amount of residue was attained. Even after this lengthy process some relatively large pieces of disaggregated pottery remained. These particles, which appeared in the > 250 μm fractions, often contained ostracods embedded in their surface. Nevertheless, a reasonable quantity of fine residue (63-250 μm) was recovered from each sample. This also contained small pieces of pottery which had failed to break down, in addition to a variable number of adult and juvenile ostracod valves, as well as a larger quantity of small ostracod fragments, rare complete carapaces, internal moulds of ostracod shells and a few foraminifera.

7.5.4.1 Sample MAK 96/3

The residue which was recovered from the breakdown of this sherd contained unidentifiable adult and juvenile ostracod fragments and whole valves of the species *Cyprideis torosa torosa*, all of which had smooth or finely pitted external surfaces. In

addition, one foraminifer belonging to the genus *Nonion* and a possible specimen of the genus *Rotaliatina*, were found.

7.5.4.2 Sample MAK 96/9

This sherd contained adult and juvenile fragments and whole valves of the ostracod species *Cyprideis torosa torosa*, all with smooth or finely pitted external surfaces.

7.5.4.3 Sample MAK 96/21

A single adult carapace and several fragments of the ostracod species *Cyprideis torosa torosa*, with smooth or finely pitted external surfaces, were recovered from MAK 96/21, in addition to a half-broken valve and an anterior fragment of the ostracod species *Bythocypris bosquetiana* and one specimen of the foraminiferal genus *Nonion*. This sample also contained a couple of internal ostracod moulds and a single bivalve macrofossil shell fragment.

7.5.4.4 Sample MAK 96/136

The residue from this sample contained a single adult carapace and several fragments of *Cyprideis torosa torosa*, all of which had smooth or finely pitted external surfaces, plus a posterior fragment of the ostracod species *Bythocypris bosquetiana* and a single valve of the ostracod genus *Bardia*. This sherd also contained one bivalve shell fragment.

7.5.4.5 Sample MAK 96/137

MAK 96/137 only contained two ostracod fragments, one from an adult and one from a juvenile carapace. The well-calcified adult fragment may have originated from a valve of the ostracod species *Cyprideis torosa torosa*, and has a finely pitted surface.

7.5.5 Discussion

The results of this pilot study indicate that it is possible to liberate fossil ostracods from samples of fired archaeological ceramics. It was feared that the alteration of their $CaCO_3$ tests to CaO during firing, and its re-hydration after firing (Section 6.4), would cause them to disintegrate during the mechanical crushing of the pottery sherds. The large proportion of broken valves which were encountered in the final, processed residues, are likely to be a result of the maceration process.

The number of ostracod specimens which were isolated from the pottery sherds were too few for detailed micropalaeontological analysis, but could be interpreted in terms of the broad geological date and palaeoenvironment in which the raw materials of ceramic manufacture were deposited (Section 7.6.4), using the ostracod zonations of Sissingh (1972; 1973; 1976a) which are outlined in Section 7.6 below.

7.6 Mediterranean Neogene ostracod biostratigraphy

7.6.1 Introduction

In order to interpret the ostracod assemblages which were isolated from individual samples of archaeological ceramics, in the above experiments (Sections 7.5.4.1 to

7.5.4.5), it is necessary to review the biostratigraphic and palaeoenvironmental schemes which have been proposed for this group of microfossils in the Mediterranean.

7.6.2 Pre-1970's

Prior to the work of Sissingh in the 1970's, a substantial number of articles had already been published on Mediterranean Neogene ostracods from as far back as the eighteenth century. However, most of these dealt with the faunas of small numbers of samples from single sections, and were taxonomically confused. Previous studies on late Cenozoic ostracods from the eastern Mediterranean were few (Terquem 1878; Bonarelli 1901; Christodoulou and Haralambos 1960; Bignot *et al.* 1963; Grekoff, *et al.* 1967; Gramann 1969; Gramann and Kockel 1969; Becker-Platen 1970), and equally inadequate (for a review see Sissingh 1972).

7.6.3 Sissingh (1972; 1973; 1976)

Sissingh's (1972) monograph on southern Aegean ostracoda represented the first detailed study of Mediterranean ostracods from several geographically separated sections covering a large stratigraphic interval and several different environments. As such it is perhaps the definitive study on ostracods for this region. Sissingh's sections from Crete, Karpathos, Gavdos and Rhodes had been studied previously by Freudenthal (1969) and Meulenkamp (1969), who determined their relative stratigraphic position by means of evolutionary stages in the foraminiferal genera

Planorbulinella and *Uvigerina* respectively (Section 6.6.3, Figure 6.8). Using these detailed age assignments as well as those afforded by the study of planktonic foraminifera, Sissingh (1972) documented the approximate stratigraphic ranges of many of the ostracod species which occurred in his material. By combining this with a rough palaeoenvironmental interpretation of brackish, shallow marine and deep marine (as determined by the ecology of the modern relatives of his fossil taxa), he was able to construct a tentative ostracod zonation for the late Cenozoic of the southern Aegean (Figure 7.3).

Although Sissingh's first zonation scheme was incomplete and featured very broad 'assemblage' zones, it represented a step forward in the study of Mediterranean ostracods, who's "contribution to the solution of stratigraphic problems was limited" (Sissingh 1976a, 276), and formed the basis for more detailed zonations by the same author. This three-part zonation (Figure 7.3) relied upon several major changes which took place in the eastern Mediterranean ostracod faunas during the late Cenozoic, which he related to the geological development of this area. These major breaks in the faunal succession were studied in detail by Sissingh (1976b), and are outlined below:

Tortonian: Increase in the diversity of ostracod faunas associated with a marine transgression.

Messinian: Significant reduction of the fauna as a result of a marine regression and the subsequent 'salinity crisis'.

Zanclian: New diversified fauna introduced into the region by a marine transgression.

Piacenzian: Less pronounced faunal turnover caused by tectonic movements between the African and Aegean plates as well as an influx of cold Atlantic bottom water.

Pleistocene: Extinction of psychospheric, deep water ostracods and appearance of new immigrants, associated with the relaxation of the cold Atlantic current and a move towards interglacial conditions.

A detailed consideration of the evolution of late Neogene Mediterranean ostracod faunas in relation to tectonics, climate and water conditions has been presented by Benson (1976) and Sissingh (1976b).

Sissingh (1972) tested his ostracod zonation by attempting to apply it to previous studies from several late Cenozoic and Pleistocene stratotype sections in Sicily, Italy and southern Spain, as well as classic sections in northern Algeria, Cephalonia, the Rhone Basin, north-west Bulgaria and Greek Macedonia. He found that most of his assemblage zones could be recognised outside the Aegean and particularly in Italy. This led to the proposal of a more detailed ostracod zonation for the Middle Miocene to Holocene of the entire central and eastern Mediterranean (Sissingh 1976a), which differs from the equivalent fauna in the western Mediterranean and was restricted in its migration by extensive Messinian evaporite deposits occurring between the Balaeric Islands, Corsica and Sardinia. In this late Cenozoic Mediterranean scheme, Sissingh (1976a) also incorporated his tentative ostracod zonation for the Mediterranean Quaternary (Sissingh 1973), which was established upon samples from the type Sicilian and Calabrian sections in Sicily, as well as his work on the southern Aegean area (Figure 7.3). The result was a four-part, multiple zonation, covering the

	Southern Aegean (1972)			Mediterannean Quaternary (1973)				Mediterannean Late Cenozoic (1976)			
	Brackish	Relatively Shallow Marine	Relatively Deep Marine	Brackish	Infralittoral	Circalittoral-Upper Bathyal	Lower Bathyal-Abyssal	Brackish	Infralittoral	Circalittoral-Upper Bathyal	Lower Bathyal-Abyssal
Recent-Holocene					*Heterocythereis albomaculata*	*Eucythere declivis*	*Argilloecia-Polycope* Zone		*Heterocythereis albomaculata*	*Eucythere declivis*	*Argilloecia-Polycope* Zone
PLEISTO Late	*Cyprideis torosa torosa* Zone	*Aurila speyeri speyeri* Zone		*Cytheromorpha fuscata* Zone	*Sclerochilus contortus* Zone	*Macrocypris succinea* Zone		*Cytheromorpha fuscata* Zone	*Sclerochilus contortus* Zone	*Macrocypris succinea* Zone	
PLEISTO Early					*Aurila speyeri speyeri* Zone	*Cytheropteron testudo* Zone	*Agrenocythere-Bythoceratina* Zone		*Aurila speyeri speyeri* Zone	*Cytheropteron testudo* Zone	
PLIO Piac.		*Urocythereis margaritifera margaritifera* Zone						*Cyprideis torosa torosa* Zone	*Urocythereis margaritifera margaritifera*		*Agrenocythere* Zone
PLIO Zanc.			*Aurila convexa emathiae*						*Aurila convexa emathiae*	*Bythocypris lucida* Zone	
MIOCENE Mess	*Cyprideis cf. sarmatica*	*Loxoconcha hodonica*						*Cyprideis p. agrigentina*	*Loxocorniculina djaffarovi*		
MIOCENE Tort.		*Cytheridea acuminata acuminata*	*Cytheretta vandenboldi* Zone					*Cyprideis cf. sarmatica* Zone	*Cytheridea acuminata acuminata*	*Cytherella vandenboldi* Zone	*Bythoceratina* Zone
MIOCENE Serr.		*C. p. verrucosa*	*Cytheretta semiornata*						*Cytheridea paracuminata v.*		

Figure 7.3 The Mediterranean Neogene ostracod zonation schemes of Sissingh (1972; 1973; 1976a).

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interval from the Serravalian to Recent, and featuring a total of 19 assemblage zones, separated by several major events in the development of the Mediterranean ostracod fauna (Figure 7.3).

Sissingh (1976a) divided the ostracod faunas into infralittoral, circalittoral-upper bathyal and lower bathyal-abyssal marine categories as well as those preferring brackish environments using more refined ecological assignments of the fossil taxa than in his 1972 zonation. The scheme was again considered to be tentative and very accurate correlations were not expected, however, many of the zones were considered to be recognisable "in relatively wide areas at corresponding stratigraphic intervals" (Sissingh 1976a, 282).

Of particular interest to the present study (Section 7.6.2), is Sissingh's refined biostratigraphic subdivision of the late Neogene brackish facies. By the incorporation of the *Cytheromorpha fuscata* Zone from his Quaternary zonation, (characterised in the latter version by the occurrence of the nominate species in addition to *Cyprideis torosa torosa* and *Loxoconcha elliptica*), Sissingh (1976) restricted the *Cyprideis torosa torosa* Zone (defined as a rather large range-zone in his earliest scheme), to the Upper Pliocene interval only, where the named species occurs in monospecific assemblages from brackish environments.

7.6.4 The biostratigraphic and palaeoenvironmental interpretation of ostracod faunas isolated from samples of archaeological ceramics

The ostracod assemblages which were isolated from five late Neolithic archaeological pottery sherds in Section 7.5, can be interpreted in terms of the biostratigraphic zone,

geological date or period, and palaeoenvironment in which the raw materials, used in their production, were deposited, by utilising the studies which are outlined above. This information may then be used to ascertain the provenance of the ceramics in question, by considering the geographical distribution of geologically compatible sedimentary deposits.

The dominant ostracod species in the sieved residues from all five Makrygialos pottery samples, is *Cyprideis torosa torosa*. Adult and juvenile carapaces, complete valves and fragments of valves belonging of this species occur in all samples except perhaps MAK 96/137, which contains but a single poorly-identified ostracod fragment. In all samples, the valves of *Cyprideis torosa torosa* have a smooth or finely pitted external surface, and many of the specimens feature a small spine on their postero-dorsal angle.

Cyprideis torosa torosa is an extant species which appeared in the Mediterranean during the Upper Pliocene (Sissingh 1972, southern Aegean) and inhabits brackish to shallow marine environments. The occurrence of monospecific assemblages of this species was used by Sissingh (1972) in his multiple ostracod zonation of the southern Aegean (Section 7.6.2, Figure 7.4) to denote the brackish '*Cyprideis torosa torosa* Zone' which covers the Late Pliocene to Recent time period. This zone is restricted to the Late Pliocene only in Sissingh's (1976a) refined Mediterranean Neogene ostracod zonation, with the succeeding Pleistocene to Holocene '*Cytheromorpha fuscata* Zone' defined by the concurrence of *Cyprideis torosa torosa* with the nominate species and *Loxoconcha elliptica*.

The occurrence of almost monospecific assemblages of *Cyprideis torosa torosa*, without *Cytheromorpha fuscata* or *Loxoconcha elliptica*, in many of the Makrygialos pottery samples, indicates that they may be associated with Sissingh's (1976a) refined brackish water *Cyprideis torosa torosa* Zone. However, this does not account for the occurrence of foraminifera and rare specimens of the true marine ostracod *Bythocypris bosquetiana*, in two samples. This latter species is also extant and has been reported by Sissingh (1972) from the Early and middle Pliocene of Crete, in assemblages belonging to his shallow marine '*Aurila convexa emathiae*' and '*Urocythereis margaritfera margaritfera*' zones.

The carapace of *Cyprideis torosa torosa* can exhibit a smooth, finely-pitted external surface or may posses a variable number of pronounced nodules. The occurrence of nodules in this species is suspected to be related to salinity. In general, the *Cyprideis torosa torosa* specimens which inhabit brackish environments tend to be more nodular than those living in normal marine water, which are usually smooth or pitted. In Sissingh's discussion of the species *Cyprideis torosa torosa*, he mentions that "only larval valves may show nodes on the surface" (1972, 87), in his Mediterranean samples. However, in the Makrygialos pottery samples, neither the adult or juvenile specimens of *Cyprideis torosa torosa* were found to possess any nodules, instead all were smooth and finely pitted. This may well indicate that the ostracod faunas which were isolated from the pottery sherds are less brackish/more marine than those described by Sissingh in his zonations of 1972 and 1976a. This assumption is in agreement with the rare occurrence of the shallow marine ostracod species *Bythocypris bosquetiana* and benthic foraminifera.

The various environments indicated in Sissingh's three zonation schemes (1972; 1973; 1976a) are based primarily on salinity (e.g. brackish and true marine) and water depth (e.g. infralittoral, circalittoral, bathyal, abyssal), and as such represent highly transitional categories. It is therefore highly feasible to record assemblages which exhibit characteristics of two adjacent zones. With this in mind, it may be possible to assign the ostracod assemblages from pottery samples MAK 96/3, 96/9, 96/21 and 93/136 to the Late Pliocene, brackish '*Cyprideis torosa torosa* Zone' of Sissingh (1976a). It is not possible to interpret the poor fauna of sample MAK 96/137 in terms of any of Sissingh's zones.

By comparing these palaeoenvironmental and biostratigraphic interpretations of the ostracod faunas isolated from the Makrygialos ceramics with the Neogene geology of this region, it has been possible to identify specific sedimentary deposits which may have been the source of the raw materials used for Late Neolithic pottery production at this site.

7.7 Approach to studying archaeological ceramics using ostracods

7.7.1 Procedures for analysing ostracod faunas in ceramic thin sections

Due to the large sample size which it is necessary to destroy in order to liberate ostracods from archaeological ceramics, the analysis of these larger calcareous microfossils was carried out using thin sections, in the present report.

The procedure which was employed in the study of ostracods from ceramic thin sections was rather simple, but did reveal some useful information. Thin sections were

scrutinised under the light microscope at a magnification of 100 x in order to determine whether or not they contained ostracods. The component(s) of the ceramic in which any ostracod valves occurred (within the micromass, inclusions, clay pellets or clay mixing) was noted, as well as their abundance, and any distinctive features of the overall assemblage (e.g. preservation, size, association with other microfossils). Distinctive individual specimens were studied in detail (at a magnification of 400 x), in order to observe any features which may be used for identification, as well as to determine the range of ostracod preservation which was present in each thin section (Section 7.3).

As the majority of ostracod valves which were observed in the present report appeared rather featureless and could not be identified with any taxonomic precision, it was necessary to make visual comparisons between the overall appearance of the ostracod valves in particular thin sections, in order to note any striking similarities and differences. The process of visually comparing numerous thin sections is extremely arduous, therefore the individual ostracod specimens were scanned into a computer and hard copies were printed out for comparison (Section 11.5). The digital images were captured on the *Aequitas 1.01* image database and image archive management system for *Windows 95* using a *Moritex μ-Scopeman MS-500* camera attached to the eyepiece of a *Leitz Laborlux 12 pols* light microscope. The images were saved as *Windows* bitmap files and could then be annotated in *Paintbrush* and imported into *Word 6* for *Windows*.

7.7.2 Description

By studying the ostracod faunas which are present in thin sections of archaeological ceramics, using the methods outlined in Section 7.7.1 above, it is possible to characterise individual samples in several ways. These are; the context in which ostracods appear, their abundance, their overall shape and size, and their state of preservation. If sufficient quantities of the original sherds are available for destruction, and three-dimensional ostracod specimens can be isolated from them, then it may be possible to identify the genera and species which constitute the faunas of individual samples (Section 7.5.4), as well as the geological period or palaeoenvironment of which they are indicative (Section 7.6.4). However, in the present report this method was impractical for the routine analysis of ostracod assemblages, and it was necessary to rely upon the study of ceramic thin sections.

7.7.3 Classification

As a result of the often low abundance of ostracod specimens in ceramics and their potential non-representation in thin section (observations in the present report), it is unwise to group and separate samples on the basis of the presence/absence of ostracods alone. However, it is possible to form meaningful classifications by considering the presence of ostracods in combination with the other characteristics of the fabric, as seen in thin section. Those thin sections which contain ostracods, can be compared in several ways, including the preservation and overall appearance of their faunas, as well as the component(s) of the ceramic in which they occur (Section 11.5). The abundance of ostracods in individual ceramic thin sections is not a valid criterion

for comparison (Section 7.2). If complete ostracod valves can be isolated from sufficient quantities of the original artefacts, as in the pilot study which is outlined in Section 7.5, then the individual faunas can be compared on the basis of the genera and species which they contain, as well as the geological date or palaeoenvironment of which they are indicative (Section 7.6.4).

7.7.4 Provenance

In thin section, ostracods may only be used to indicate the provenance of ceramics in combination with other methods of characterisation (Day *et al.* 1999; Section 11.5), due to the low level of specific geological information which can be attained by their study. If the faunas which are contained within archaeological ceramics can be studied in detail and identified taxonomically (Section 7.6.4), then more precise provenance interpretations can be made by a consideration of local and regional geology. As it has been necessary to rely upon thin sections for the routine analysis of ostracod assemblages from most ceramics in the present report, the information provided by the study of calcareous nannofossils and foraminifera has been used to provenance archaeological ceramics wherever possible (Sections 5.9.2 and 6.7.4).

7.7.5 Technology

The nature of ostracods in thin sections of archaeological ceramics is not well suited to the study of pottery technology. Crude inferences about the degree of firing in ceramics may be based upon observations of the preservation of ostracods in thin

section (Section 6.4.3), however, this has no routine application. Nevertheless, the presence of ostracods in calcareous temper or clay mixing, can be used to determine the nature of such materials, where they occur in ceramics.

8 Diatoms and other siliceous microfossils

8.1 Introduction

There exist several groups of aquatic micro-organisms, including diatoms, radiolaria and silicoflagellates, which are characterised by having a siliceous skeleton. Their remains occur in Palaeozoic, Mesozoic and Cenozoic sediments deposited in marine and non-marine environments, as well as archaeological ceramics from different parts of the world (Section 8.2). The biology and morphology of these siliceous microfossils as well as their occurrence, preservation and utility in archaeological ceramics is outlined below.

8.1.1 Diatoms

Diatoms are 'golden-brown' algae belonging to the division Bacillariophyta. These unicellular plants range from approximately 10-100 μm in size and have an external siliceous skeleton or 'frustule' (Figure 8.1). The diatom frustule is composed of two equal sized 'thecae' which fit together rather like a pill-box, and protect the soft cell inside. Diatoms are extant and appear to have evolved in the Jurassic period, although earlier reports have been made. In the geological record, all that remains of these algae are their siliceous frustules, which may or may not occur intact. The earliest diatoms were marine, but they appear to have colonised fresh water environments in the Eocene, and today diatoms can live wherever there is moisture, (e.g. in soils). Diatoms can be planktonic; free floating in oceans and lakes, or benthic; attached to various
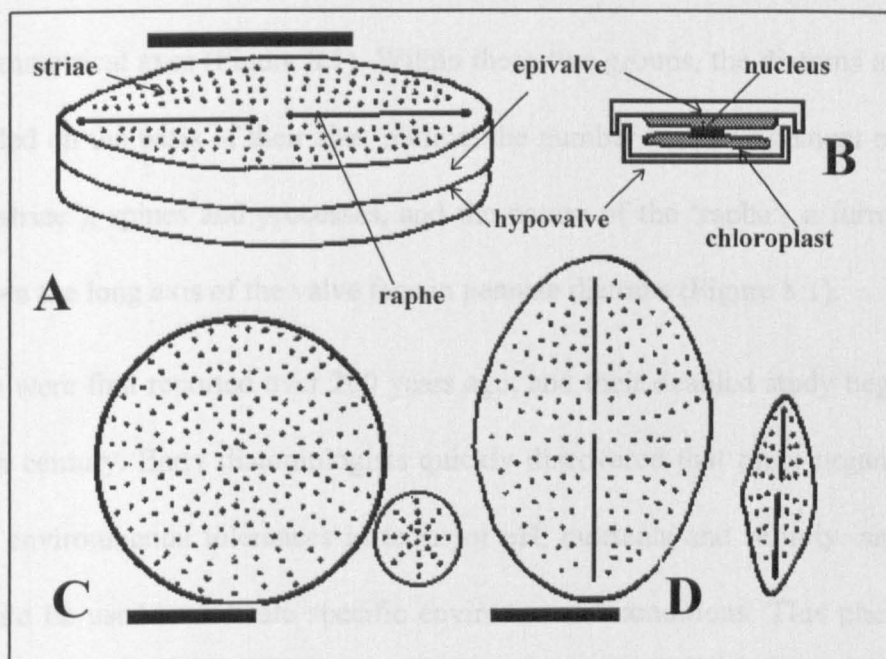
Figure 8.1 The biology and morphology of the diatom frustule. Oblique view of a complete pennate diatom frustule (A), schematic cross-section with soft parts (B), valve view of central diatoms (C) and valve view of pennate diatoms (D). Scale bars = 100 μm (A) and 10 μm (C and D). After Brasier (1985) and Barber and Haworth (1981).

organic and mineral substrates, migrating within the uppermost layers of sediment, or growing in thread-like colonies from the sea bed.

Fossil diatoms are classified on the basis of their frustule morphology. There are two main groups of diatoms in this respect; the 'centrales' which are usually circular in plan view and have radial symmetry, and the 'pennates' which are elongate and have three symmetrical axes (Figure 8.1). Within these two groups, the diatoms are further subdivided on the basis of their size, outline, the number and arrangement of lines of pores ('striae'), spines and processes, and the nature of the 'raphe'; a furrow which runs down the long axis of the valve face in pennate diatoms (Figure 8.1).

Diatoms were first reported over 200 years ago, and their detailed study began in the late 19th century. Early diatomologists quickly discovered that these organisms had specific environmental tolerances in terms of pH, nutrients and salinity, and certain taxa could be used to indicate specific environmental conditions. This phenomenon has been used extensively in the latter part of this century to reconstruct fresh water, estuarine and coastal environments, and detect human-induced environmental changes, such as the acidification and eutrophication of fresh water lakes.

Planktonic marine diatoms are a less reliable tool for biostratigraphy than other microfossils such as planktonic foraminifera and calcareous nannofossils. This is a consequence of their 'evolutionary conservatism' (Burckle 1978), and the problems which are associated with the geological record of diatoms in regions of upwelling. However, in high latitude areas, where calcareous microfossils are less abundant, diatoms and other siliceous microfossils are being used more for biostratigraphy (e.g. Ocean Drilling Project cruises).

Fossil diatoms can be isolated from sedimentary rocks by using the floatation method (Section 8.4), in which the disaggregated sediment is mixed with water, stirred and allowed to settle, or centrifuged. The minerogenic matter sinks and the diatoms float to the surface, where they can be extracted and mounted on a strew slide (Section 8.4). It is often necessary to treat sediments with hydrogen peroxide, in order to oxidise any organic matter, and hydrochloric acid, if they are calcareous. The strew slides are observed with transmitted light at a magnification of 400-1000 x.

Good accounts of the biology, morphology and ecology of diatoms can be found in Chandra (1992) and Tappan (1980). The modern classification of diatoms is dealt with by Crawford *et al.* (1990), and some key floras can be found in the publications of Cholnoky (1968), Cleve-Euler (1951-1955), Foged (1980, 1982), Germain (1981), Hudstedt (1927-1966), Krammer and Lange-Bertalot (1986; 1988; 1990; 1991), Patrick and Reimer (1966; 1975), Salden (1978), Schmidt (1874-1959), Van Heurck (1880-1885) and van der Werf and Huls (1957-1974). General summaries of the application of fossil diatoms to the reconstruction of Holocene environments and the biostratigraphy of marine diatoms, can be found in Battarbee (1986), Burckle (1978) and Lipps (1993).

8.1.2 Radiolaria

Radiolaria are unicellular marine zooplankton of the class Actinopoda, which possess an internal siliceous skeleton. There are several orders within the subclass Radiolaria, however, only one of these, order Polycystina, has a significant geological record, as the siliceous skeletons of the other two are not well-preserved.
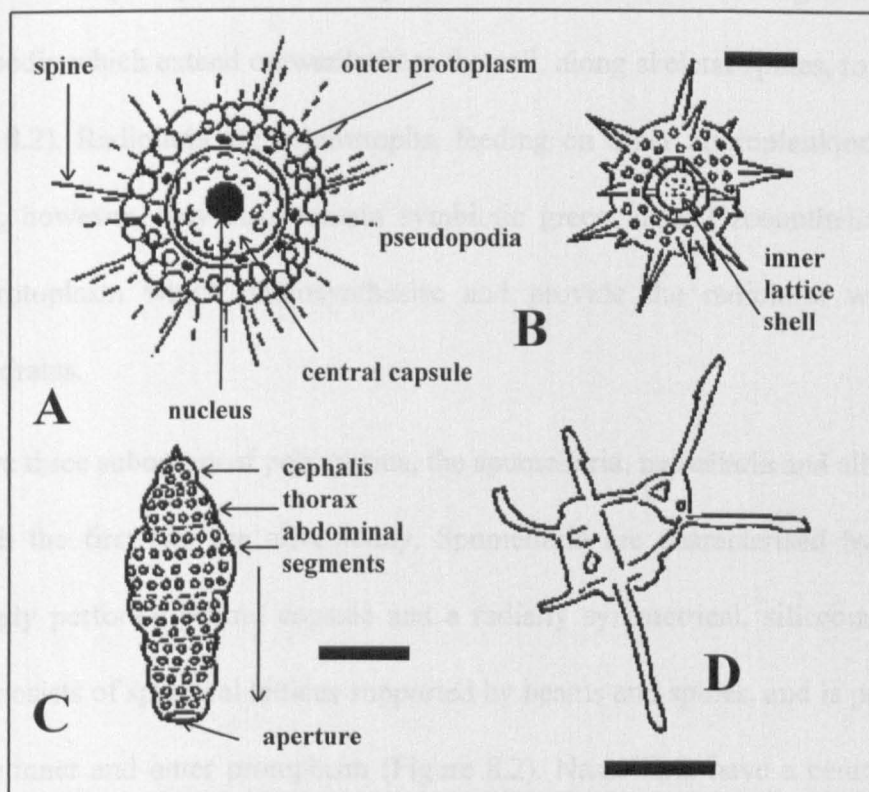
Figure 8.2 The biology and morphology of polycystine radiolaria. Living polycistine radiolarian (A), spumellarian skeleton (B), nassellarian skeleton (C) and albaillellarian skelton (D). Scale bars = 50 μm. After Bignot (1980), Brasier (1985), Campbell (1954) and Holdsworth (1969).

All polycystine radiolaria possess a two part protoplasm consisting of a perforate 'central capsule' surrounded by an outer protoplasm. The inner protoplasm or 'endoplasm' contains one or more nuclei which control the vital functions of the cell, and the outer protoplasm or 'ectoplasm' is constantly streaming and produces pseudopodia which extend outwards from the cell, along skeletal spines, to catch food (Figure 8.2). Radiolaria are heterotrophs, feeding on other microplankton including diatoms, however, they also contain symbiotic green algae ('zooanthellae') in the outer protoplasm which photosynthesise and provide the radiolaria with simple carbohydrates.

There are three suborders of polycystina, the spumellaria, nassellaria and albaillellaria, of which the first two are alive today. Spumellaria are characterised by having a completely perforate central capsule and a radially symmetrical, siliceous skeleton, which consists of spherical lattices supported by beams and spines, and is produced in both the inner and outer protoplasm (Figure 8.2). Nassellaria have a central capsule which is perforated at only one end, and a bell-shaped skeleton consisting of several segments, with an aperture (Figure 8.2). The segments of the nassellarian skeleton can be divided into a cephalis, a thorax and one or more abdominal segments (Figure 8.2). In all Mesozoic and Cenozoic nassellarians, the cephalis contains a siliceous 'spicule' which presses against the central capsule, in order to alter its surface area for important physiological reactions. Albaillelleria are an extinct suborder of polycystine radiolaria which have a bilaterally symmetrical skeleton, constructed of three rods (Figure 8.2). The interesting Silurian to Permian evolution of albaillelleria can be followed through the progressive modification of this basic structure.

All modern radiolaria have an open-ocean distribution, although they were near-shore dwellers in the Palaeozoic. The distribution of individual taxa can be related to water depth and temperature, and often corresponds to the distribution of particular water masses. Spumellaria are more common in the photic zone (< 200 m) and migrate up and down by way of contractile vacuoles, in order to satisfy the light requirements of their zooanthellae. Nassellaria on the other hand do not possess these symbiotic algae and can follow deep water masses (> 2000 m).

In the geological record, the skeletons of radiolaria occur from the Late Cambrian to Recent and are commonly preserved in cherts and limestones. They can be isolated from these types of rocks by using acids, and are studied in strew slides, or picked by hand like foraminifera (Section 6.2).

Radiolaria are a useful tool for precise biostratigraphy, due to their rapid evolution. They can also be used to model Quaternary palaeoceanography and palaeoclimatology, as a result of their relationship with specific water masses.

The biology, morphology and ecology of fossil and living radiolaria is discussed in detail by Campbell (1954), Kling (1978) and Casey (1993), and good accounts of the geological evolution of this group can be found in Nazarov and Ormiston (1985), Pessagno (1977) and Riedel and Sanfilippo (1977).

8.1.3 Silicoflagellates

Silicoflagellates are unicellular, flagellate marine algae which posses an internal, tubular siliceous skeleton, ranging in size from 30-100 μm (Figure 8.3). Because of

their small size and free swimming life habit, they are often classified as nannoplankton, along with calcareous nannofossils. The skeletons of silicoflagellates first appear in the geological record in the Late Cretaceous, Campanian stage, and are present in significant numbers, along with other siliceous microfossils, in areas of ancient or modern upwelling, as well as marine sediments which have been deposited as a result of volcanic activity. Fossil silicoflagellates are classified by the gross morphology of their delicate siliceous skeletons as well as the details of any surface ornamentation, although the latter may be related to water temperature (Martini and Müller 1976).

The study of silicoflagellates is more recent than that of other branches of micropalaeontology. A great deal of information has been gleaned by the study of high latitude oceanic cores from the Deep Sea Drilling Project (1968-1986) and Ocean Drilling Project (1986-present). Here, silicoflagellates have been used to date marine sediments, which contain very few foraminifera and calcareous nannofossils. Silicoflagellates have also been used to interpret palaeotemperatures, by referring to the known preferences of extant species. Important genera in this respect are *Dictyocha* and *Distephanus* (Figure 8.3); the relative proportion of which, Mandra (1969) and Ciesielski and Weaver (1974) have used to indicate surface water temperatures in the Southern Ocean.

Silicoflagellates can be liberated from sedimentary rocks in the manner which is described for diatoms and radiolaria (Sections 8.1.1 and 8.1.2). However, they are more commonly studied by the preparation of smear slides. Silicoflagellate smear slides are prepared in a similar fashion to those for the study of calcareous
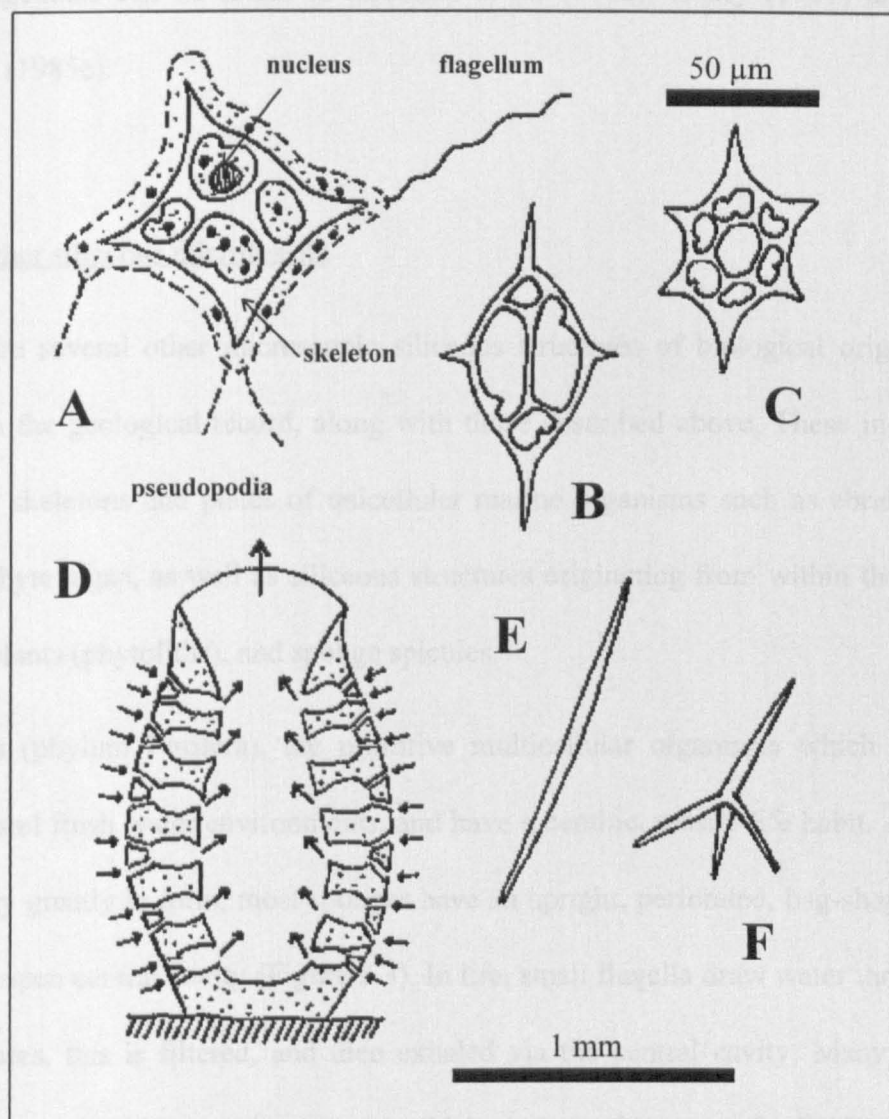
Figure 8.3 The biology and morphology of silicoflagellates and sponges. A. Living silicoflagellate;

B. *Dictyocha*: C. *Distephanus*; D. Living sponge showing water currents; E; Monaxon and D.

Tetraxon sponge spicules. Scale bars = 50 μm (B and C) and 1 mm (E and F). After Bignot 1982,

Perch-Nielsen 1985c and Moore (1955).

nannofossils (Section 5.3), and must be viewed with plane polarised transmitted light at magnifications of 250-400 x.

Good accounts of the morphology, classification and biostratigraphy of silicoflagellates can be found in Loeblich *et al.* (1968), Bukry (1981) and Perch-Nielsen (1985c).

8.1.4 Other siliceous microfossils

There are several other microscopic siliceous structures of biological origin which occur in the geological record, along with those described above. These include the spicular skeletons and plates of unicellular marine organisms such as ebridians and chrysophyte algae, as well as siliceous structures originating from within the cells of higher plants (phytoliths), and sponge spicules.

Sponges (phylum Porifera), are primitive multicellular organisms which occur in marine and fresh water environments, and have a benthic, sessile life habit. Although they vary greatly in form, most sponges have an upright, perforated, bag-shaped body with an open central cavity (Figure 8.3). In life, small flagella draw water through the outer pores, this is filtered, and then exhaled via the central cavity. Many sponges have an internal skeleton of some sort, which supports the mass of cells. In some, for example, the demosponges (Class Demospongea) this is in the form of numerous siliceous spicules, which are < 1 mm in length and consist of simple rods (monaxon spicules) or four diverging rays (tetraxon spicules, Figure 8.3). In the geological record, these spicules are often all that is preserved of siliceous sponges, which range from the Cambrian to the present day.

Despite occurring in large numbers in some sediments, sponge spicules are of very little stratigraphic value. The biology and morphology of sponges and their spicules is outlined in the *Treatise on Invertebrate Paleontology (Part E Archaeocyatha and Porifera)*, edited by Moore (1955), although the taxonomy in this book is somewhat out of date. Other references include Bergquist (1978) for modern sponges, and Reid (1964) on classification, as well as Clarkson (1986), upon which the above account is based.

8.2 The occurrence and preservation of diatoms and other siliceous microfossils in archaeological ceramics

Several groups of siliceous microfossils have been encountered in archaeological ceramics from various parts of the world. Diatoms are perhaps the most commonly reported group of siliceous microfossils in this respect. They are a relatively consistent component in Neolithic to Medieval ceramics from northern Europe (Foged 1968, Norway; Edgren 1970, Finland; Jansma 1977; 1981; 1984; 1990, Netherlands and England; Alhonen and Matiskainen 1980; Alhonen *et al.* 1980; Alhonen and Väkeväinen 1981; Matiskainen and Alhonen 1984, Finland; Gibson 1983a and b, England; Håkansson and Hulthén 1986; 1988, Germany and Sweden; Stilborg 1997, Denmark), as well as further afield (Troja *et al.* 1996, Neolithic to Bronze Age Sicily; De La Fuente and Martinez Macchiavello 1997, Inka pottery of Argentina).

As with other groups of microfossils which occur in archaeological ceramics (Sections 5.2, 6.2, and 7.2), diatoms can occur within the clay micromass (Håkansson and Hulthén 1986), associated with shell or grog temper (Jansma 1984; 1990), within
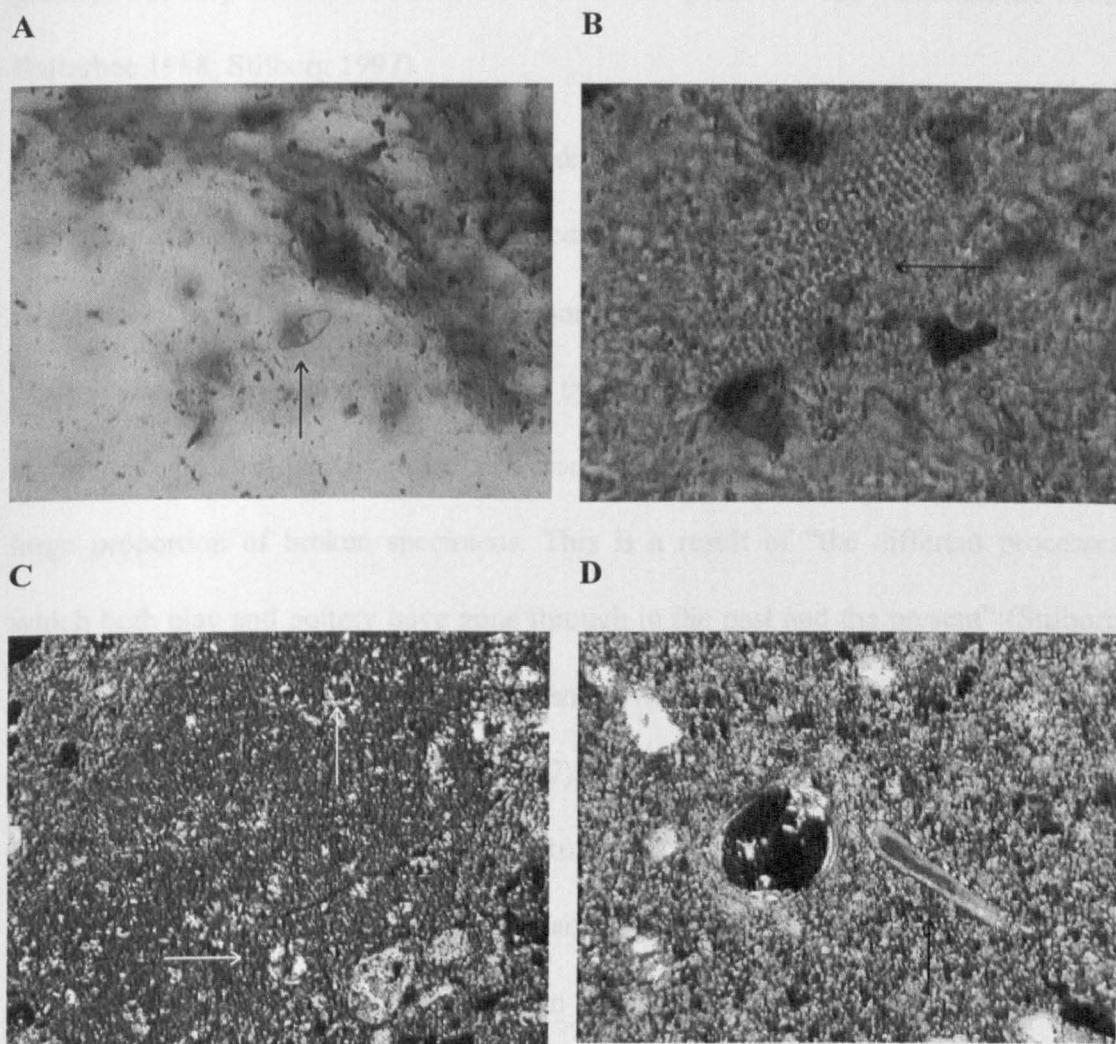
Figure 8.4 The occurrence of siliceous microfossils in thin sections of Bronze Age archaeological ceramics from Crete. A. a pennate diatom within the calcareous slip of Kn 95/400, B. a fragment of radiolarian skeleton within a calcareous inclusion in Kn 95/407, C. chalcedony radiolaria within a chert inclusion in MFK 93/7, D. a rod-shaped sponge spicule within the clay matrix of Kn 95/187. A and B = PPL, C and D = PPL. Field of view = 0.15 mm (A), 50 μm (B), 1.5 mm (C) and 0.5 mm (D).

clay mixing (Stilborg 1997), or as part of a slip or paint applied to the exterior of the vessel (Figure 8.4). The abundance of diatoms in archaeological ceramics varies greatly, but they usually occur in small numbers (Alhonen and Matiskainen 1980; Battarbee 1988; Stilborg 1997).

In ceramic thin sections (c. 30 μm thick), diatom frustules are usually obscured by the fine clay matrix, which makes them difficult to recognise and identify taxonomically (Håkansson and Hulthén 1986; Håkansson 1997). For this reason it necessary to analyse diatoms by isolating them from the rest of the ceramic (Section 8.4). The diatom assemblages which are isolated from archaeological ceramics often contain a large proportion of broken specimens. This is a result of "the different processes which both clay and pottery have gone through in the past and the present" (Stilborg 1997, 109), including the transportation and reworking of specimens in the original sedimentary environment (Jansma 1977), the maceration and working of raw materials during ceramic manufacture (Gibson 1983a), firing (Gibson 1983b), and the process of isolation itself (Håkansson and Hulthén 1986; Battarbee 1988). The fragmentation of diatom specimens in archaeological ceramics hinders their identification (Stilborg 1997) and the production of quantitative assemblage descriptions, or 'diatom profiles' (Gibson 1983a). Those diatoms which inhabited brackish water usually have sturdier thecae than those from fresh or purely marine environments (Jansma 1977; Gibson 1983a), and the degree of damage in ceramics appears to be related to the shape of individual species (Jansma 1981), with the longer pennate forms being particularly fragile. This may have a serious affect on the determination of allochthonous and autochthonous species in archaeological ceramics, by the proportion of broken and unbroken individuals of diatoms from different

environments (Section 8.4). The effect of firing on diatom thecae has been studied by several authors, and is discussed separately in Section 8.3.

Other groups of siliceous microfossils which have been observed in archaeological ceramics, include radiolaria (Farnsworth 1964; Whitbread 1995, Section 2.2), sponge spicules (Linné 1957; Brissaud and Houdayer 1986; Håkansson and Hulthén 1988; Keech McIntosh and Macdonald 1989; Riley *et al.* n.d.), silicoflagellates and chrysophyte algae (Håkansson 1997).

Sponge spicules have been found to occur in very high abundance (17 %) in some Iron Age pottery from Mali, by Keech McIntosh and Macdonald (1989). These conspicuous inclusions which can be added as temper during ceramic manufacture (Krausse 1911; Linné 1957), appear as glassy, isotropic 'rods' (longitudinal profile) or 'bulls eyes' (transverse profile) in thin sections (Keech McIntosh and Macdonald 1989), and can be identified in terms of the types of sponges from which they originated, by scanning electron microscopy (Brissaud and Houdayer 1986). Linné (1957) has commented upon the distribution and orientation of sponge spicules in thin sections of archaeological ceramics from Brazil and Bolivia and, from this, interpreted technological aspects of ceramic manufacture.

Very few siliceous microfossils were encountered in the present report, despite the detailed analysis of many thin sections and smear slides of archaeological ceramics. Rare diatoms and fragments of the perforate siliceous skeletons of radiolaria were observed within the clay matrix and calcareous slips or paints, of some thin sections (Figure 8.4). In addition, rare sponge spicules, as well as chalcedony radiolaria within chert inclusions, were encountered (Figure 8.4).

8.3 The behaviour of diatoms and other siliceous microfossils during the firing of ceramics

As with other groups of microfossils (Sections 5.4, 6.5, 7.4, 9.4), firing is one of the most important factors which may alter the nature of diatom assemblages in archaeological ceramics. Various authors have commented on the effect of this aspect of pottery manufacture (Jansma 1977; 1981; 1984; Gibson 1983a and b; Håkansson and Hulthén 1986; Battarbee 1988), however further research is clearly needed (Gibson 1983a).

It appears that the silica of diatom thecae, when heated, breaks down at a critical temperature. This temperature has been variously quoted as 800 °C (Gibson 1983b), 1000 °C (Jansma 1977), or 1400 °C (Matiskainen and Alhonen 1984), although none of these authors appear to have determined this for themselves, or indicated the source of their information. A similar process also takes place at a critical point during the firing of ceramics, and this too is poorly defined.

The minimum temperature which has been quoted most often for the destruction of diatom valves in archaeological ceramics during firing is 800 °C (Jansma 1981; 1984; Battarbee 1988). This seems to be based upon firing tests carried out by Jansma (1981), in which "at temperatures not exceeding 800 °C, the frustules ... remained intact", and "at temperatures of 800 °C or more, most of the frustules disappeared" (Jansma 1984, 529). However, the same author, according to Gibson (1983b, 21) found diatoms "in some Medieval Dutch ceramics which have been fired at 1000 °C". Gibson (1983b) did not state the method by which the firing temperature of these archaeological ceramics was determined, nor did Jansma report the findings himself.

Nevertheless, the results of a set of experiments carried out by Håkansson and Hulthén (1986), may support such findings.

In order to determine how heating affects diatom frustules, Håkansson and Hulthén fired three samples of recent diatomaceous clay at 125 °C for approximately twelve hours, 550 °C for three to four hours, and 925 °C for approximately two hours, respectively. It was not stated why these particular temperatures or firing durations were chosen, neither were any other details of their experimental firings outlined. However, by analysing the fired samples, it was discovered that the diatom frustules were still well-preserved, even after firing at 925 °C. This contradicts the experimental work of Jansma (1981; 1984), and may explain the presence of diatoms in archaeological ceramics fired to a temperature of 1000 °C (Jansma *in* Gibson 1983a and b).

On the other hand, Jansma (1990) has also indicated that diatom specimens can be destroyed during firing, at much lower temperatures. In his analysis of variously fired Neolithic pottery from the former island of Schokland in the Netherlands, he noted that whilst the very low fired pottery (400 °C or less) of the Funnel Beaker Culture contained relatively rich diatom floras (average 120 specimens), those of the Vlaardingen Culture which were fired at slightly higher temperatures (over 400 °C) contained a less abundant diatom flora (average 70 specimens). In support of this, Jansma reported that one sample of pottery from the Vlaardingen Culture contained more than 400 specimens, which he believed to be due to its lower firing temperature and consequently, "a better conservation of the thecae" (1990, 305).

The various estimates which have been quoted for the temperature at which diatoms are destroyed during the firing of ceramics, highlights the complexity of this process. It is highly probable that several other factors, in addition to temperature, determine the threshold at which the silica of diatom thecae 'melts' (Gibson 1983b), or reacts with other components of the clay, such as the exact chemical composition of the clay matrix and the silica itself. It is suspected that different diatom taxa will be destroyed at different levels during the firing of ceramics, as Jansma (1977, 77) stated that "diatoms from brackish water have sturdier thecae than those from marine or freshwater as they have to tolerate a greater fluctuation in environment".

One way in which the degree of firing in ceramics indirectly affects the diatom assemblages in strew slides of archaeological ceramics, is by the vitrification of the clay micromass. In order to liberate diatoms from archaeological pottery sherds using the floatation method (Section 8.4 and Figure 8.5), it is first necessary to break the samples into small pieces. This can be done by hand where the sherds are low-fired, however those samples which have undergone substantial vitrification (> 700 °C, Håkansson and Hulthén 1986) must be crushed with greater force (e.g. in a pestle and mortar). This latter process has been demonstrated to cause very extensive fragmentation of the diatom frustules in the final residue (Håkansson and Hulthén 1986). The physical strain which is caused by the process vitrification of clay micromass during the firing of ceramics itself may also be responsible for the fragmentary nature of the diatom assemblages which are isolated from highly fired ceramics. In addition, Brissaud and Houdayer (1986) have postulated that this process, rather than melting, is responsible for the destruction of sponge spicules at 800 °C during the firing of ceramics from Mali.

8.4 The preparation and analysis of diatoms from archaeological ceramics

8.4.1 Preparation

8.4.1.1 Introduction

The presence of diatoms in archaeological ceramics can be determined by viewing thin sections with the light microscope. However, individual specimens are often be obscured by the clay minerals, and characteristic features of a valve may be cut off, hindering their identification (Håkansson and Hulthén 1986; Håkansson 1997). It is therefore necessary to liberate three-dimensional diatom specimens from archaeological ceramics in order to analyse the floras in detail. Several authors have successfully isolated diatoms from pottery sherds using an adaptation of the 'floatation method', which is the standard procedure for the liberation of diatoms from sedimentary rocks (e.g. Gibson 1983a; Håkansson and Hulthén 1986). This procedure is summarised below, and illustrated in Figure 8.5.

8.4.1.2 Procedure

1. In order to liberate diatom specimens from archaeological ceramics using the floatation method, it necessary to destroy pieces of original artefacts. The amount of material which is destroyed by this method, relies heavily upon the quantity which is available. This can range from as much as ten grams (Alhonen and Matiskainen 1980), to as little as one gram, and it may even be possible to isolate diatoms from a scraping of the base or interior of a vessel (Gibson 1983a). Theoretically, a larger sample will give a better representation of the diatom flora of the whole artefact and

will suffer less from the effect of contamination during preparation (Battarbee 1988), however, in reality only very small quantities are available.

Pottery which is plastic impregnated (a method sometimes used to preserve delicate artefacts), cannot be treated in the manner which is described below as it fails to break down (Håkansson and Hulthén 1986).

2. Before a chip or sherd of pottery can be processed, it must be cleaned thoroughly (Jansma 1981; 1984). This involves brushing the sample in order to remove any secondary deposits which have adhered to it during burial (Section 3.8), and may contain diatoms.

3. The clean pottery sample must now be disaggregated in order to increase the surface area available for the chemical treatment in step four. Very low fired, or heavily weathered ceramics may be easily separated by the solution itself (Gibson 1983b), though it is usually necessary to crush or crumble sherds by hand (Jansma 1990). Heavily fired, well-vitrified ceramics are often too hard to break down in this way and may require more force. This can be achieved by splintering the sample with pliers (Jansma 1984; Gibson 1983b), or crushing it to fine-medium gravel sized pieces with a pestle and mortar (Matiskainen and Alhonen 1984; Stilborg 1997). Håkansson and Hulthén (1986) claim that the mechanical destruction of highly fired ceramics damages the diatom specimens within, and in his description of this process, Håkansson (1997, 109) states that the samples must be "carefully crushed - not ground".

An alternative method of breaking down well-vitrified pottery for the isolation of diatoms suggested by Gibson (1983b), is by the use of an ultrasonic bath.

4. The disaggregated sample must now be placed in a beaker and treated with hydrogen peroxide (H_2O_2) in order to oxidise any organic matter. This can be done with a concentration of 20 % (Alhonen and Matiskainen 1980; Matiskainen and Alhonen 1984) to 30 % H_2O_2 (Alhonen *et al.* 1980; Gibson 1983a; Jansma 1984; Jansma 1990), and the reaction can be facilitated by warming the solution at a constant temperature of 50-60 °C for 12 hours (Alhonen and Matiskainen 1980) or adding some crystals of potassium permanganate (Gibson 1983a; Jansma 1984). If the pottery sample contains significant amounts of calcareous matter, then one or two drops of concentrated hydrochloric acid (c. 30 %) may be added (Jansma 1990).

In what was perhaps the earliest attempt at isolating diatoms from archaeological ceramics, Foged (1968) 'cooked' his samples in concentrated HCl only, without the use of H_2O_2 (Alhonen and Matiskainen 1984). Furthermore, it may be possible to dissolve pottery samples by warming them in 10 % phosphoric acid (H_3PO_4) at a temperature of 50 °C, for a period of a few days to several weeks, as outlined by Håkansson and Hulthén (1986) and others. During the chemical treatment of pottery samples, it is necessary to stir the solution at regular intervals.

5. After the various forms of chemical treatment which are described above, the sample should have separated into a coarse, sand to silt-sized residue and a cloudy suspension containing clay minerals and diatoms. The finer particles, which are in suspension can now be decanted with care from the coarser material. Whilst it is possible to mount and analyse diatoms from this fraction, the clay minerals tend to coat the diatom specimens (Gibson 1983b), and can interfere with their identification

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┌─────────────────────────────────┐
│      Original vessel or sherd     │
└─────────────────────────────────┘
                 │
        ┌──────────────────┐
        │  1-10 g subsample  │
        └──────────────────┘
                 │
┌─────────────────────────────────┐
│       Brushing or cleaning        │
└─────────────────────────────────┘

   Medium-fired    Poorly -fired    Well-fired

  ┌──────────────┐              ┌──────────────┐
  │ Break by hand │              │ Crush in mortar│
  └──────────────┘              └──────────────┘

┌─────────────────────────────────┐
│ Treat with 20-30 % H₂O₂ and KMnO₄ │
│        at 50°C for 12 hours.       │
└─────────────────────────────────┘

     Non-calcareous        Calcareous

                      ┌──────────────┐
                      │ Treat with HCl │
                      └──────────────┘

┌─────────────────────────────────┐
│  Decant and discard coarse fraction│
└─────────────────────────────────┘

┌─────────────────────────────────┐
│ Centrifuge at 2000 rpm for 2-3 mins│
│    and discard upper two-thirds   │
└─────────────────────────────────┘

  ┌──────────────┐    ┌──────────────┐
  │  Strew slides  │    │    SEM stub    │
  └──────────────┘    └──────────────┘

  ┌──────────────┐    ┌──────────────┐
  │ Study with LM  │    │ Study with SEM │
  └──────────────┘    └──────────────┘
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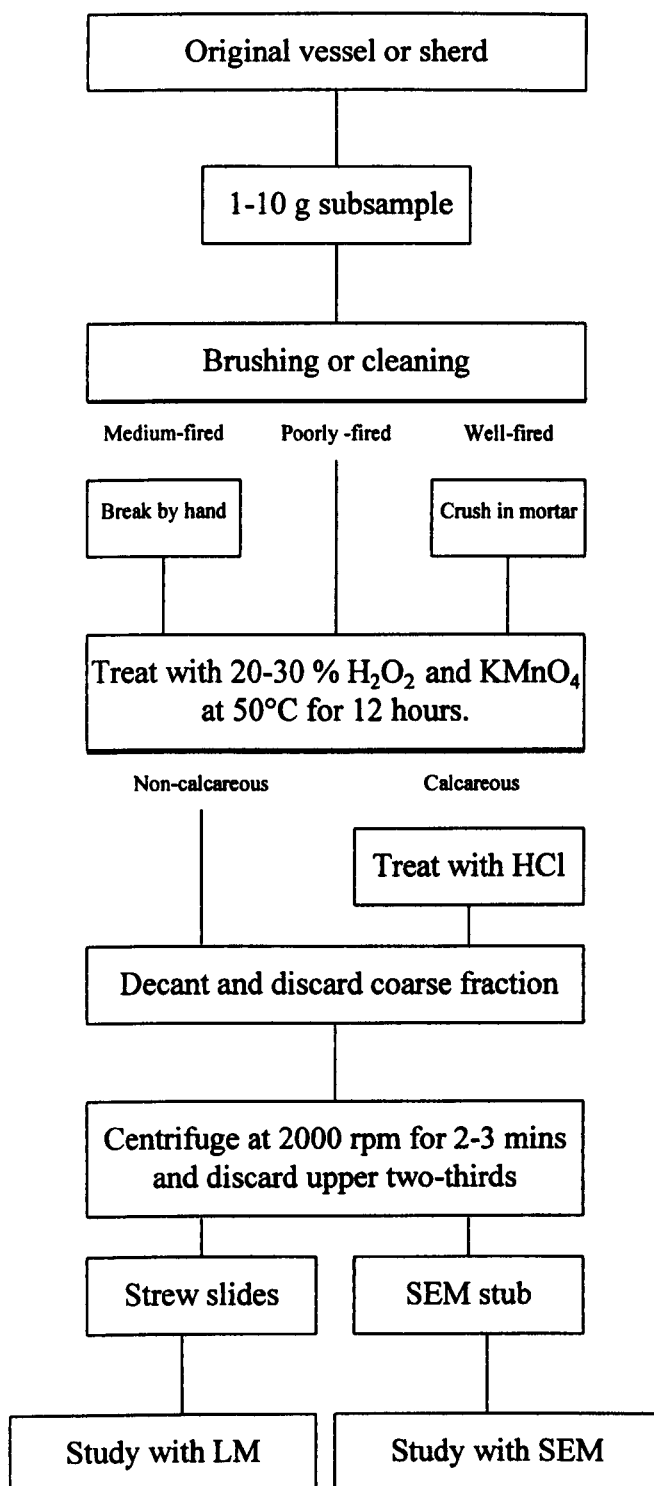
Figure 8.5 Diagrammatic representation of the floatation technique of diatom preparation, after

Gibson (1983a) and others.

(Matiskainen and Alhonen 1984). In order to remove these fine clay particles (< 2 μm) it is necessary to dilute the solution with distilled water and centrifuge it at 2000 revolutions per minute, for two to three minutes (Gibson 1983b; Jansma 1984). After this process, the clay minerals remain in suspension and may be decanted, leaving the diatoms and other siliceous microfossils at the bottom of the centrifuge tube. The clay particles can also be separated from the diatoms by allowing the solution to settle for two hours at a time, decanting the upper two-thirds and diluting the remaining fraction with more water (Håkansson and Hulthén 1986), however this is extremely time consuming.

6. The product of steps two to six is a clear solution which will hopefully contain diatoms, as well as a small proportion of clay minerals > 2 μm. Strew slides can now be prepared from this, by pipetting 2-3 ml of the solution onto a microscope coverslip, placed on a hotplate. The dried coverslip will contain a fine residue, and must be adhered face-down onto a standard microscope slide immediately. For this purpose, it is necessary to use a mounting medium with a high refractive index, such as *Naphrax* or *Caedax*, which will enhance the structure of the diatom valves when viewed under the microscope (Jansma 1981). Alternatively, the digested diatom solution can be dried onto a stub and studied with the SEM (Håkansson and Hulthén 1988).

8.4.2 Analysis

In strew slides prepared from samples of archaeological pottery in the manner which is described above, diatoms can be viewed in isolation, unobscured by the clay minerals which are present in ceramic thin sections. These slides are usually studied

with the transmitted light microscope using a 1000 x oil-immersion lens and a mechanical stage.

There are two methods of recording the diatoms in a strew slide; a qualitative and a quantitative method. In the qualitative method, all species which are present in the flora are recorded, and a note is made of any dominant species. The quantitative method however, involves counting and identifying a set number of individuals in each strew slide, as well as the number of broken and unbroken specimens of each species. A count of 400 specimens is usually made (Jansma 1981; 1984; 1990; Gibson 1983a), however some strew slides will not contain this many specimens; in which case as many as possible should be counted.

The identification of quaternary diatom specimens in strew slides of archaeological ceramics can be facilitated by referring to published 'floras', such as Cholmoky (1968), Cleve-Euler (1951-1955), Foged (1980, 1982), Germain (1981), Hudstedt (1927-1966), Krammer and Lange-Bertalot (1986; 1988; 1990; 1991), Patrick and Reimer (1966; 1975), Salden (1978), Schmidt (1874-1959), Van Heurck (1880-1885) and van der Werf and Huls (1957-1974). These floras can also be used to determine the salinity preferences of the various diatom species, in terms of the aqueous environment which they inhabited during life. It therefore is possible to calculate the 'MBF ratio' (Marine-Brackish-Freshwater) of the diatom flora, by recording the number of species (qualitative method), or the number of individuals (quantitative method), belonging to each environment.

The MBF ratio gives an indication of the type of environment in which the original clay source, used for the manufacture of the pottery, was deposited. However, as

diatom valves can be transported from their original habitat and re-sedimented, the assemblage in a particular layer of sediment may contain allochthonous diatom specimens which are indicative of different depositional environments, in addition to the *in situ* flora which was living in the water column above (Jansma 1977). The presence of many allochthonous diatom specimens in the strew slides of archaeological pottery, may therefore hinder the determination of the original depositional environment of the raw materials of ceramic manufacture, using the qualitative method. Nevertheless, as the action of re-sedimentation very often damages the diatom valves (especially long pennate species), a comparison between the number of broken and unbroken individuals of diatom species from the different environments (as determined by the quantitative technique), may be useful in identifying the allochthonous component of the flora, and therefore the true nature of the original sedimentary environment.

If marine fossil diatoms occur in the strew slides which have been prepared from samples of archaeological ceramics, then it may be possible to interpret floras in terms of the geological date in which they were deposited, by the identification the fossil taxa and the application of suitable biostratigraphic schemes.

8.5 Approach to studying archaeological ceramics using diatoms

8.5.1 Description and classification

It is possible to characterise and classify archaeological ceramics in many ways on the basis of their diatom floras (Section 2.3.1). On the simplest level, the presence/absence of diatoms in ceramics can be used classify samples (Jansma 1977),

as can the component of the ceramic in which they occur (Stilborg 1997). A more sophisticated form of description and classification can be made on the basis of the taxonomic composition of the diatom assemblage (Alhonen *et al.* 1980; Alhonen and Matiskainen 1980; Matiskainen and Alhonen 1984; De La Fuente and Martinez Macchiavello 1997). However, it is preferable to characterise and classify diatomaceous archaeological ceramics in terms of the depositional environment of which their flora are indicative (Jansma 1984), as this is highly contextual and assists the determination of provenance (Section 8.5.2). It is very important, when grouping archaeological ceramics on the basis of their diatom assemblages in this way, to consider other forms of characterisation and classification, such as thin section petrography, typology and chemistry.

8.5.3 Provenance

The detailed description of diatom assemblages from archaeological ceramics and the interpretation of palaeoenvironment from the salinity tolerances of the various taxa (Section 8.4.2), facilitates the determination of provenance. Diatoms can be used to indicate the provenance of ceramics on many scales (Section 2.3.2), by comparing this environmental interpretation with the proximity of the site of excavation relative to the coast (Jansma 1984; Stilborg 1997), pre-existing geological knowledge (Alhonen and Matiskainen 1980; Alhonen and Väkeväinen 1981; Matiskainen and Alhonen 1984), or the diatom analysis of representative clay samples (Jansma 1977; 1981; 1990; De La Fuente and Martinez Macchiavello 1997). Where fossil diatom floras occur in the residues which have been isolated from archaeological ceramics, it may

be possible to use biostratigraphy to indicate provenance, by considering the occurrence of contemporaneous sediments of a suitable lithology.

8.5.3 Technology

Diatoms are not well suited to the direct interpretation of ceramic technology. The occurrence of diatoms with conflicting salinity tolerances in strew slides of archaeological pottery samples must not be used to infer tempering or clay mixing without the analysis of ceramic thin sections, as was the case in Jansma (1977; 1982; 1990) and Matiskainen and Alhonen (1984, Section 2.3.3.2). However, the presence of diatoms in clay mixing or temper, as seen in thin section, may be used to infer the nature, or even the origin of this material (Stilborg 1997). In addition, their presence/absence may possibly be used, indirectly, to make crude inferences about the degree of firing, if other aspects of the ceramic fabric are considered (Jansma 1977; 1990).

9 Organic microfossils

9.1 Introduction

Several types of organic-walled microfossils, or 'palynomorphs' occur in sediments dating from the Cambrian to the present day, as well as samples of archaeological ceramics (Section 9.2). Most palynomorphs are constructed of an extremely durable organic polymer called sporopollenin and can be extracted from lithified sediments by the use of strong acids (Section 9.3). The most common groups of palynomorphs are pollen, spores, dinoflagellate cysts and acritarchs, however many other types occur, e.g. chitinozoans, scolecodonts, fungal bodies and colonial algae, but these are very rare. The study of organic microfossils forms a specialised branch of micropalaeonotology called 'palynology'.

9.1.1 Pollen and spores

Pollen and spores are the minute (usually < 200 μm) reproductive organs of higher plants, which are produced in vast numbers, dispersed by wind, water or other organisms, and are often incorporated into marine and non-marine sediments. Pollen and spores have a very long geological range (Figure 9.1) and the appearance of different forms of these two palynomorphs reflects the evolution of land plants. Spores have a roughly spherical form and often occur in clusters or tetrads (Figure 9.1). Different types of plants usually have their own morphologically distinct pollen
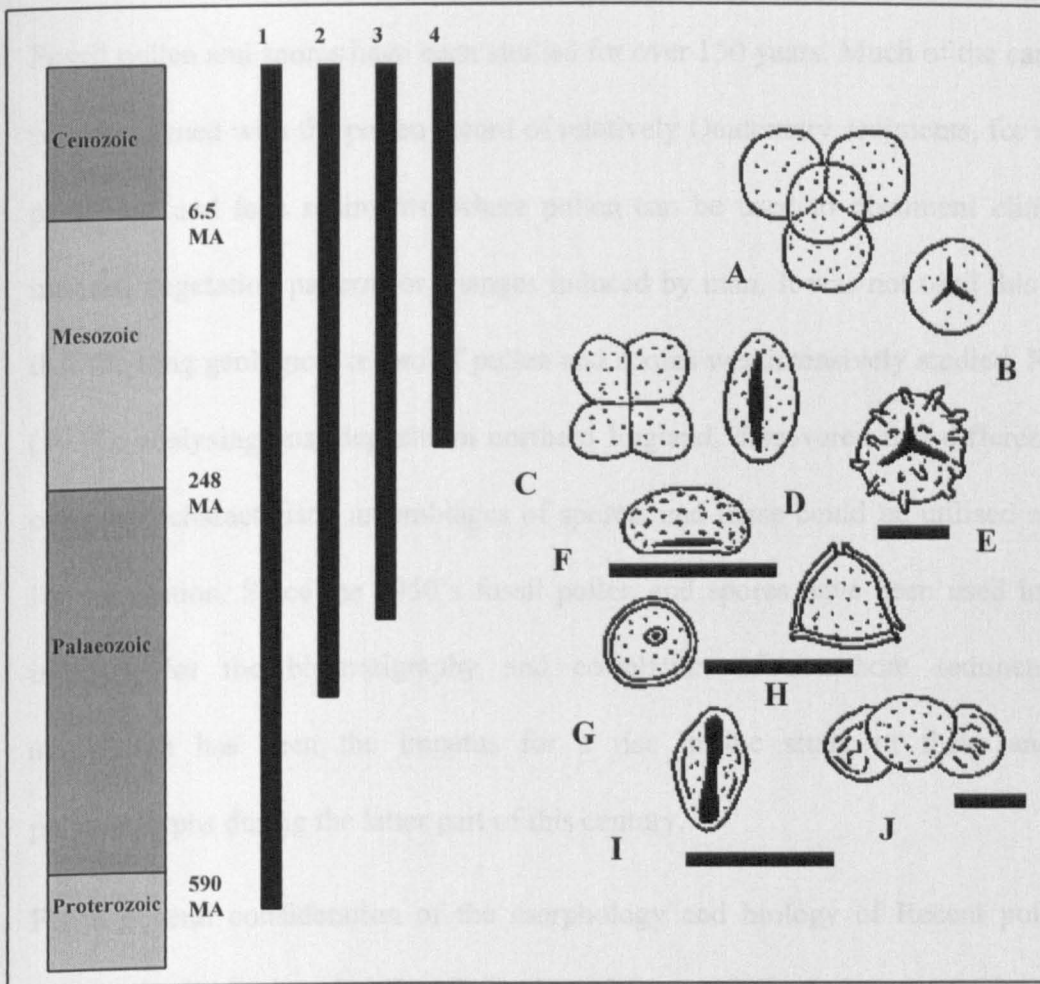
Figure 9.1 The geological range of the main groups of organic microfossils, and the morphological features of fossil pollen and spores. 1 = Acritarchs, 2 = Spores, 3 = Pollen, 4 = Dinoflagellate cysts. (MA = million years). A = tetrahedral tetrad of trilete spores and B = isolated trilete spore, C = tetragonal tetrad of monolete spores and D = isolated monolete spore, E = ornate trilete spore with bristles, F = ornate monolete spore with granules, G = monoporate pollen grain, H = triporate pollen grain, I = monosulcate pollen grain and H = bisaccate pollen grain. Scale bars = 50 μm (F to J), 200 μm (E). After Traverse (1988) and Bignot (1985).

or spores, characterised by size, overall shape, wall structure, surface ornamentation, the number and position of pores, colpae (furrows), sulcae (folds), the presence of markings from the tetrad, as well as air-sacs (Figure 9.1).

Fossil pollen and spores have been studied for over 150 years. Much of the early work was concerned with the pollen record of relatively Quaternary sediments, for example peat bogs and lake sediments, where pollen can be used to document climatically induced vegetation patterns or changes induced by man. It was not until this century that the long geological record of pollen and spores was intensively studied. Raistrick (1934), analysing coal deposits in northern England, discovered that different seams contained characteristic assemblages of spores, and these could be utilised as a tool for correlation. Since the 1950's fossil pollen and spores have been used in the oil industry for the biostratigraphy and correlation of nearshore sediments, this application has been the impetus for a rise in the study of these and other palynomorphs during the latter part of this century.

For a general consideration of the morphology and biology of Recent pollen and spores, the reader is referred to Tschudy and Scott (1969), Erdtman (1969) and Pons (1970), as well as more general works such as Brasier (1980) and Bignot (1985), upon which the above account is based. The application of plant microfossils to geological problems, including biostratigraphy, is dealt with in Muir and Sarjeant (1977); Traverse *et al.* (1957) and more recently, Jansonius and McGregor (1996).

9.1.2 Acritarchs

The group acritarcha was erected by Evitt in 1963 to include all hollow, organic-walled unicellular vesicles with unknown affinities. This group contains a great range of forms which are classified into 13 subgroups based upon their morphology (Figure 9.2). However, certain features are common to many acritarchs; a single-layered wall enclosing a central cavity, conspicuous processes, an opening or 'pylome' and surface ornamentation or 'sculpture'. Acritarchs are approximately 20-150 μm in size and therefore it is necessary to sieve organic residues using a very fine mesh (7 or 5 μm) to recover these palynomorphs.

Although the exact affinity of acritarchs is unknown, it is clear that they are the remains of some kind of planktonic marine organism, due to their cosmopolitan distribution and increase in abundance in offshore sediments. They commonly occur in many types of marine strata (especially argillaceous sediments), from the Pre-Cambrian to Devonian, and have a much less conspicuous geological record, consisting of several long-ranging forms, in the late Palaeozoic, Mesozoic and Cenozoic. The superabundance and rapid evolution of acritarchs in Pre-Cambrian and lower Palaeozoic marine sediments makes them a very useful tool for dating and correlating rocks of this period. In addition, the abundance, diversity and preservation of acritarchs can be used to distinguish near and offshore sediments, and certain forms can be characteristic of particular oceanic environments.

Good accounts on the morphology, classification and stratigraphic application of acritarchs can be found in Muir and Sarjeant (1977), Deunff et al. (1971), Tappan (1980) and more recently, Stover et al. (1996).
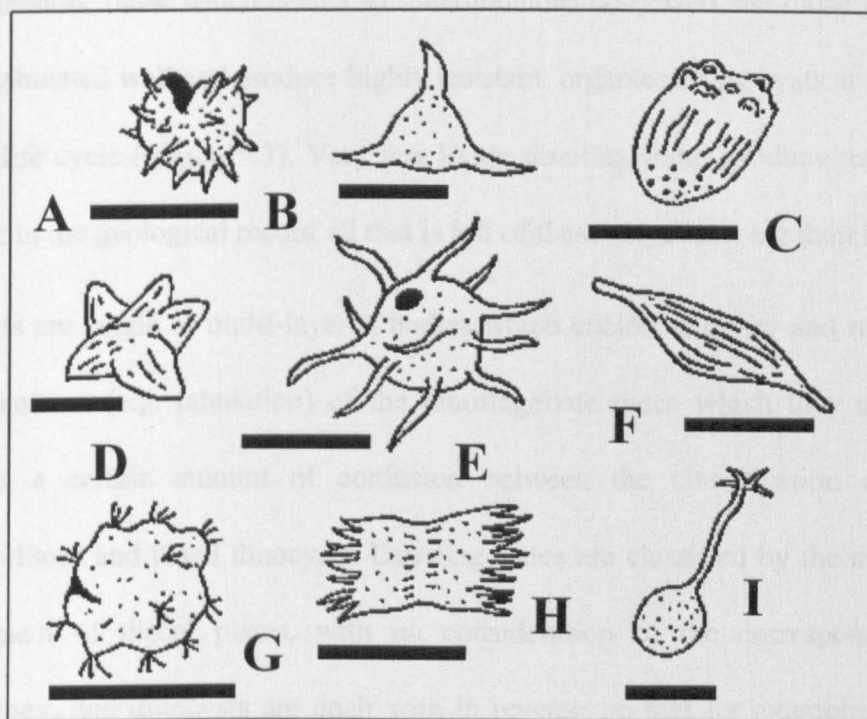
Figure 9.2 The morphological features of acritarchs. A = *Micrhystridium* **spherical body with processes and slit-like opening, B =** *Veryachium* **polygonal body shaped by number and position of processes, C =** *Ooidium* **ornamented spherical body, D =** *Estiastra* **body formed of several broad open processes, E =** *Baltisphaeridium* **spherical body with processes and circular pylome, F =** *Leiofusa* **elongate fusiform body with polar processes, G =** *Vulcanisphaera* **sub-spherical body with short branching processes, H =** *Acanthodiacrodium* **rectangular body with smooth equator and thread-like polar processes, and I =** *Deunffia* **spherical body with single elongate branching process. Scale bars = 50 μm. After Bignot (1985) and Brazier (1980).**

9.1.3 Dinoflagellate cysts

Dinoflagellates are small (usually < 100 μm), single-celled, marine phytoplankton which have a prominent nucleus, chloroplasts and two flagella. There are two kinds of dinoflagellates; those which have a soft membranous body wall and those which have a platy tabulated wall and produce highly resistant, organic resting cysts at some point in their life cycle (Figure 9.3). Very few living dinoflagellates produce resting cysts, however in the geological record all that is left of these organisms are their cysts.

Dinocysts are single or multi-layered bodies which enclose a cavity and reflect some of the features (e.g. tabulation) of the dinoflagellate theca which they once filled. There is a certain amount of confusion between the classification of modern dinoflagellates and fossil dinocysts. Dinoflagellates are classified by the number and arrangement of thecal plates, with no consideration of the corresponding cysts morphology, and dinocysts are dealt with in reverse, so that for example, the fossil genus *Spiniferites* is the cyst of the living genus *Gonyaulax* (Figure 9.3).

There are three main types of dinocysts, based upon the arrangement of the one or more wall layers and the way in which the cyst filled its dinoflagellate theca (Figure 9.3). Proximate cysts develop with their outer wall in direct contact with the inner surface of the dinoflagellate theca. They are a similar size and shape to the theca and exhibit strong 'paratabulation'. Chorate cysts are much smaller than the dinoflagellate theca from which they originate, and have two wall layers; an endophragm surrounded by a periphragm moulded into conspicuous processes which were in contact with the inner wall of the dinoflagellate. The processes of chorate cysts can be randomly arranged (non-tabulate) or may reflect the tabulation of the thecae in which they were
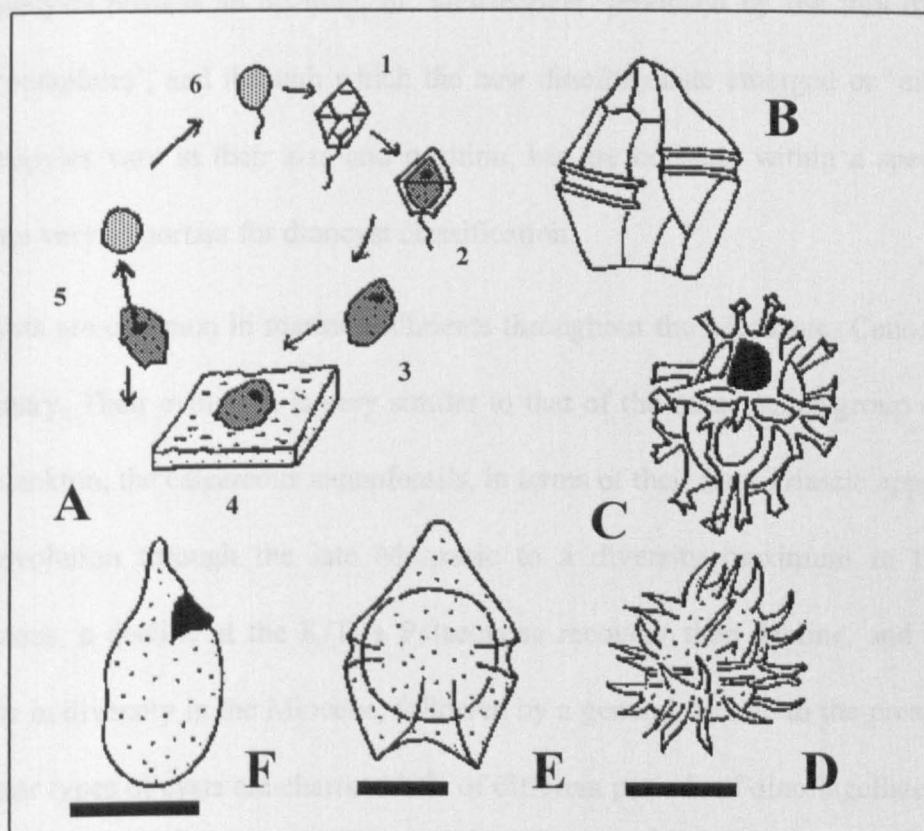
Figure 9.3 The life cycle of a tabulated cyst-producing dinoflagellate (A), the living dinoflagellate genus *Gonyaulax* (B) and its fossil dinocyst *Spiniferites* (C), the three main types of dinocyst: proximate (F), cavate (E) and chorate (D) cysts. 1 = planktonic thecate stage, 2 = encystment within the theca, 3 = loss of theca, 4 = benthic resting stage, 5 = excystment and deposition of cyst, 6 = naked planktonic gymnodinoid. Scale bars = 50 µm. After Evitt (1985) and Bignot (1985).

enclosed (tabulate). The other group of dinocysts (cavate cysts), have two wall layers which are partially separated, to produce large cavities. These cavities are usually situated at the poles of the cyst and may be elongated into 'horns' (Figure 9.3).

All dinocysts possess an opening, or 'archaeopyle' produced by the loss of one or more 'paraplates', and through which the new dinoflagellate emerged or 'excysted'. Archaeopyles vary in their size and position, but are constant within a species and therefore very important for dinocyst classification.

Dinocysts are common in marine sediments throughout the Mesozoic, Cenozoic and Quaternary. Their evolution is very similar to that of the other major group of fossil phytoplankton, the calcareous nannofossils, in terms of their Late Triassic appearance, rapid evolution through the late Mesozoic to a diversity maximum in the Late Cretaceous, a decline at the K/T, a Palaeogene recovery then decline, and a slight increase in diversity in the Miocene, followed by a general decline to the present day. Particular types of cysts are characteristic of different periods of dinoflagellate history and their rapid evolution in the Mesozoic and early Cenozoic makes them a very useful biostratigraphic tool for this time period. The biostratigraphic utility of dinoflagellate cysts has been utilised extensively in the oil industry and is the main reason for an increase study of dinoflagellates during the latter part of this century. Late Cenozoic and Quaternary dinocyst assemblages are characterised by a few, long-ranging species and are therefore not very useful for detailed biostratigraphy and correlation. However, by studying the environmental tolerances and distribution of these extant forms it is possible to make palaeoecological and palaeoenvironmental interpretations.

The biology and morphology of living dinoflagellates is dealt with in Sarjeant (1974) and Fensome *et al.* (1993; 1996). For the classification and biostratigraphy of fossil dinoflagellate cysts, the reader is referred to Williams (1977); Williams and Bujak (1985), Fensome *et al.* (1993) and Stover *et al.* (1996), as well as more general references such as Brasier (1980) and Bignot (1985), upon which the above account is based.
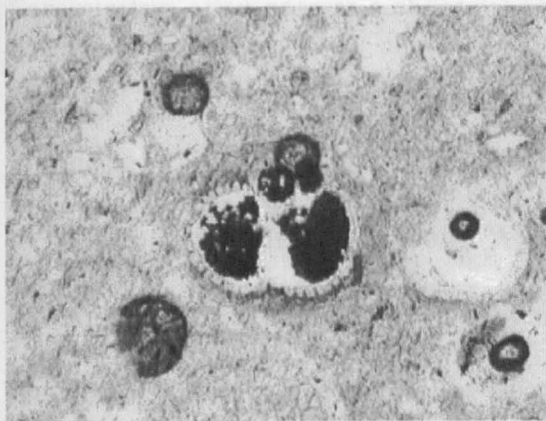
9.2 The occurrence of organic microfossils in archaeological ceramics

Organic microfossils have been observed in archaeological ceramics by Hunt (1996) and Tsaila (n.d), as well as mudbricks (Ayyad *et al.* 1991). Hunt (1996, 69), who isolated pollen, spores and dinocysts from reduction-fired sherds of British Iron Age pottery (Section 2.3.2.5), noted that "considerable organic matter and possible palynomorph fragments" were visible in thin sections of the same samples. The presence of palynomorphs in ceramic thin sections has also been noted by Tsaila (n.d.), who identified a fern spore (Polypodiaceae) in her analysis of the Middle Minoan Age Dark Faced Incised Ware pyxides from Knossos (Section 11.3). Ayyad *et al.* (1991) did not analyse thin sections, but instead, isolated recent and fossil pollen and spores, as well as macrofossil plant fragments, from three unfired Egyptian mudbricks.
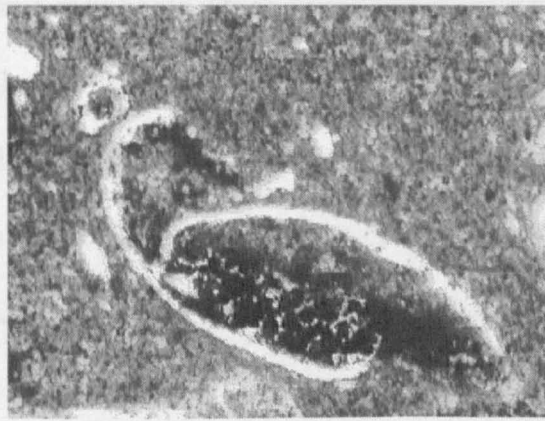
Although much of the archaeological pottery which has been analysed in the present study contains organic matter in thin section, very few identifiable palynomorphs have been observed. The oxidised organic matter appears reddy brown in thin section, and forms amorphous structures as well as dark spherical bodies within the clay matrix,

Figure 9.4 The occurrence of organic matter in thin sections of Bronze Age archaeological ceramics from Crete. Organic matter associated with a foraminifer in Kn 95/211 (A), and with an ostracod in MFK 93/57 (B). The occurrence of 'microforaminifera' in Kn 86/13 and Kn 95/237 (C and D). All pictures PPL. Field of view = 0.5 mm (A and B), 0.15 mm (C and D).

Amorphous organic matter may be found associated with foraminifera and ostracods (Figure 9.4), or can form distinct linings which are directly related to their mineralised remains.
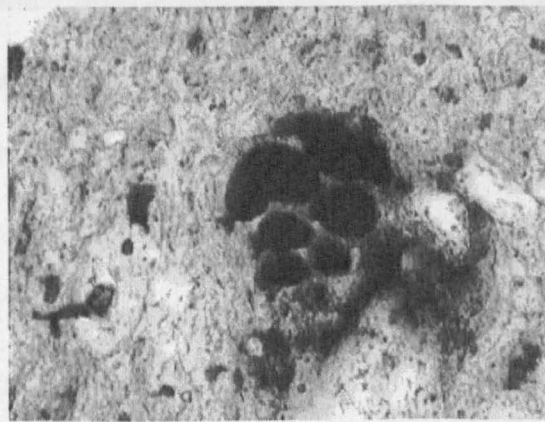
Foraminifera often possess a chitinous organic membrane which lines their chambers and can be seen in thin sections of archaeological pottery. These linings often appear in digested palynological residues, where they have been termed 'microforaminifera' by Wilson and Hoffmeister (1952). Within thin sections of archaeological ceramics, the linings of foraminiferal tests are often highly oxidised and have contracted within the chambers, but can sometimes be left *in situ*. In rare cases, foraminifera have been dissolved and all that is left is a dark red-brown, opaque organic structure which reflects the form of the test which it once lined (Figure 9.4). Strictly speaking, these organic remains of foraminiferal tests are also 'microforaminifera', especially where they occur without a calcite shell. As such, they are the only type of palynomorph which has been identified in the thin sections of archaeological ceramics in the present report. Microforaminifera were not present in the digested residues of Cretan pottery which are analysed in Section 9.3. This may be due to the brittle, thermally altered condition of these organic structures, which is evidenced by their dark, opaque appearance under the microscope (Figure 9.4).

Palynomorphs have flexible, translucent sporopollenin walls and are therefore difficult to observe and study in ceramic thin sections (Hunt 1996). However, by treating sherds with strong acids, it may be possible to isolate these organic microfossils and study them separate from the rest of the ceramic (Section 9.3).

9.3 The isolation of organic microfossils from archaeological ceramics

9.3.1 Introduction

Whilst Bryant and Holloway (1996, 914) reported that "there is little chance that archaeologists will find pollen trapped in most ceramic pottery", Hunt (1996) has successfully isolated pollen, spores and other types of palynomorphs from reduction-fired archaeological ceramics by dissolving sherds with hydrofluoric (HF) and hydrochloric acid (HCl). In the present report, several Bronze Age archaeological pottery sherds from Crete have been treated in a similar manner.

9.3.2 Samples

Six Bronze Age cooking pot samples were chosen for the purpose of this investigation. The sherds, which date from the MM II and LM III periods were excavated on Crete and are low calcareous.

9.3.3 Procedure

The various archaeological pottery samples were processed at the Industrial Palynology Unit of the University of Sheffield, in the manner described below.

1. Each sherd was individually crushed and ground to a powder in a pestle and mortar.

2. The fine powdered samples were then transferred to a disposable plastic container and wetted with ordinary tap water.

3. A small amount of dilute Hcl was then added to each container in order to dissolve any calcareous matter. The samples were left for 30 minutes, after which the reaction was completed.

4. The reactant was decanted and 30 ml of HF, plus 10 ml of Hcl, were added to the samples. This mixture was stirred with a glass rod, a labelled lid was placed on each container, and the reaction was left to proceed.

5. The contents in each container were stirred every day until all argillaceous particles were dissolved. Some of the samples took longer to digest than others, particularly those which were fired to a higher temperature.

6. The digested samples were decanted and diluted with tap water until they had a neutral pH.

7. Each sample was then washed and sieved at 7 μm..

8. Standard palynological strew slides were prepared from each sample by transferring four drops of the organic residue into another phial, diluting them with 3 ml of distilled water then pipetting 1 ml of this onto a coverslip, placed on a hotplate in a fume cupboard. The dried coverslip was then mounted face down on a labelled microscope slide using 'Entellan' optical adhesive, and left to set.

All slides were viewed under plane polarised light at magnifications of 25, 40, 100 and 400 x in order to determine the nature of any organic material present.

<u>9.3.4 Results</u>

No identifiable pollen, spores or marine palynomorphs were present in the residues of the six samples in this pilot study. Some organic matter was liberated from most samples, although this was oxidised and amorphous. It is not possible to determine the exact reasons for the absence of palynomorphs in these sherds. The raw materials from which they were manufactured may not have contained any organic microfossils to begin with, or palynomorphs may have been present but were destroyed during firing. In order to investigate this latter alternative it is necessary to document the behaviour of various organic microfossils during the firing of ceramics (Section 9.4).

9.4 Investigation into the behaviour of palynomorphs during the firing of ceramics

<u>9.4.1 Introduction</u>

In the present report, experiments were devised in order to investigate the behaviour of organic microfossils during the firing of ceramics. It was hoped that the results of these experiments would reveal useful information pertaining to the thermal alteration of palynomorphs, as well as the temperature(s) at which they are destroyed during firing.

<u>9.4.2 The thermal alteration of organic matter in geological contexts</u>

Organic microfossils are known to undergo a distinct transformation, when subjected to increasing temperatures as a result of geological processes (burial, overthrusting,
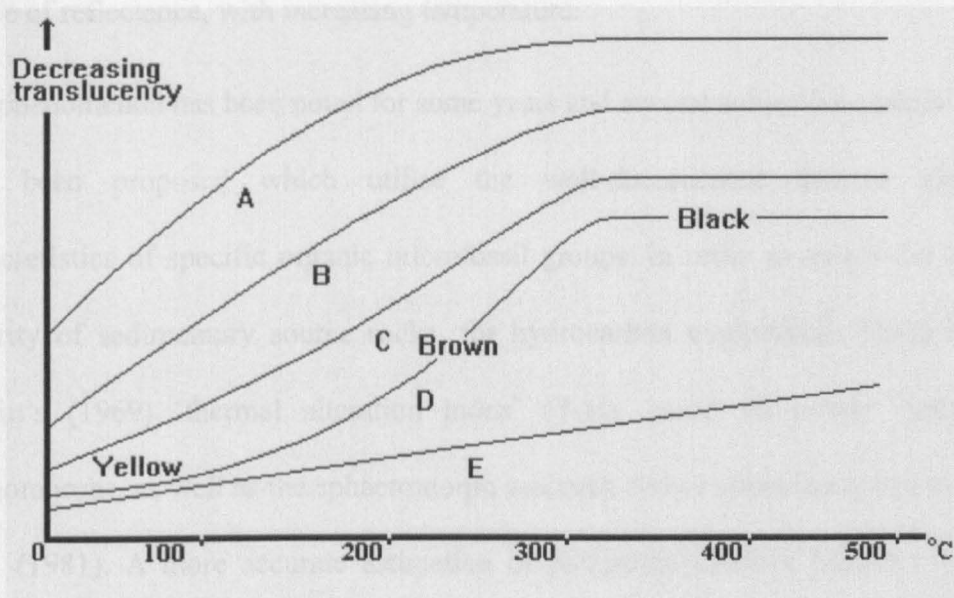
Figure 9.5 The behaviour of different palynomorphs with increasing temperature from 0-500 °C, in terms of their translucency and colour, as well as the abundance of inertinite in assemblages heated to different temperatures (after Brooks and Dorning 1997, 186-187: Figs. 1 and 2). A = Pollen and spores, B = Amorphous organic matter, C = Acritarchs, D = Dinoflagellate cysts, E = abundance of inertinite.

igneous intrusion) or intentional heating (heat-treatment of flints, firing of pottery). The specific rates of thermal alteration exhibited by different groups of organic microfossils vary considerably, in relation to their composition and thickness (Dorning 1986). However, the general trend is that of a colour change (usually yellow to brown to black), a decrease in the degree of translucency and an increase in the degree of reflectance, with increasing temperature.

This phenomenon has been noted for some years and several subjective optical indices have been proposed which utilise the well-documented thermal alteration characteristics of specific organic microfossil groups, in order to assess the thermal maturity of sedimentary source rocks, for hydrocarbon exploration. These include Staplin's (1969) 'thermal alteration index' (TAI), based on colour changes in palynomorphs, as well as the sphaeromorph acritarch colour alteration index of Legall *et al.* (1981). A more accurate estimation of palaeotemperatures however, may be obtained by assessing the degree of thermal alteration of more than one group of organic microfossils occurring in a sample. There is often some variation in the degree of colouration, translucency or reflectance within a single sample, and a comparison between the different stages reached by the various organic microfossils provides a means of cross-checking the palaeotemperature estimates obtained. In general, non-marine organic fossils such as pollen and spores tend to exhibit greater alteration at lower temperatures than marine organic microfossils, such as dinoflagellate cysts and acritarchs. However, the latter provide a more reliable means of estimating palaeotemperature, due to the high variation in the composition of non-marine sediments and the more extreme thermal alteration and variable weathering of organic-walled microfossils from this realm (Dorning 1986). By utilising the thermal

alteration characteristics of the various types of palynomorphs, it is possible to measure palaeotemperatures from as low as 50°C to as high as 400 °C.

9.4.3 Previous archaeothermometric studies

The thermal alteration of organic microfossils is a potentially useful tool for the determination of firing temperatures in archaeological materials ('archaeothermometry'), as exemplified by the studies of Brooks and Dorning (1997) and Hunt (1996).

Brooks and Dorning (1997) have used the thermal alteration of palynomorphs to investigate the deliberate heat treatment of siliceous raw materials for the production of stone tools. Heating flint and chert to a temperature of c. 250 °C (Purdy and Brooks 1971), appears to improve their 'knapping' quality and initiates fracturing in previously intractable materials. Chert and flint deposits often contain various quantities of marine and non-marine palynomorphs, which can be affected by natural thermal alteration, as well as intentional heating by man. Brooks and Dorning (1997) have investigated the degree of intentional heat treatment in flint samples from the Late Mesolithic to Neolithic archaeological site of Lismore Fields near Buxton, and the Late Bronze Age to Early Iron Age site of New Buildings in Hampshire, England, by extracting organic microfossils with hydrofluoric acid. In both investigations, the organic microfossils which were liberated from the fragments of flint were compared to a set of geological reference samples, produced by the controlled heat treatment of Late Cretaceous flints.

At Lismore Fields, Brooks and Dorning (1997) discovered that the majority of the samples which were analysed, had been heated to a temperature of between 200 and 250 °C. This was in agreement with the optimum temperature for heat pre-treatment, established by Purdy and Brooks (1971). As none of the samples exhibited any characteristics in hand specimen which suggested that they had been heat treated, the results of these studies question the reliability of the macroscopic flint analysis.

Brooks and Dorning (1997) analysed ten samples of burnt flint from the site of New Buildings, Hampshire in order to determine the origin of cracking and crazing seen in hand specimen. The thermally altered organic matter which was liberated from these samples indicated that they were heated to a temperature of 300-400 °C. This in itself, would not have been sufficient to induce the fracturing which was seen, and the authors therefore inferred that the burnt flint was used as a material for heat retention. Here, gradual heating followed by rapid cooling, probably due to the dumping of water, would have produced the cracking and crazing.

Hunt (1996) has utilised the thermal alteration of palynomorphs in Iron Age pottery from North Furzton, near Milton Keynes, England, to infer approximate firing temperatures. He treated ten oxidised and ten reduction-fired sherds with hydrofluoric and hydrochloric acid in the manner which is described in Section 9.3. However, only the latter contained any significant organic matter. By comparing the colour of the palynomorphs in the reduction-fired sherds with the 'Thermal Alteration Index' of Staplin (1969), he determined that the pottery was relatively low-fired (approximately 400 °C or '2 +' to '3' in Staplin's 'TAI').

9.4.4 Material

Several kilograms of raw material were collected from the Eocene London Clay Formation at Walton-on-the-Naze in the county of Essex, England. This clay which contained a rich assemblage of pollen, spores and dinoflagellate cysts, was ideal as a means of studying the differential effect of firing on the various groups of organic microfossils. Standard palynological preparations were made from the raw London Clay, in order to compare its assemblage with that of the fired samples.

9.4.5 Clay preparation

The raw material was broken into small pieces and allowed to dry in a bucket for a period of one week. The dry pieces of clay were then crushed into a fine powder (<1mm) with a pestle and mortar, and sieved using a 1 mm mesh. This powder was mixed with tap water until a reasonably stiff clay paste was achieved. The clay was kneaded by hand and transferred to ice cube containers, which were left to dry. The small cubes of clay were ideal for these experiments as they were easy to produce and of roughly equal dimensions. After one week, the briquettes were removed from their container and placed in a warm oven (30 °C) for several days, in order to drive out any excess water in preparation for the firing process.

9.4.6 Details of the firing process

The firing of the London Clay briquettes was carried out in tandem with the experiments into the behaviour of calcareous nannofossils during firing (Section 5.4,

Figure 5.5). This produced twelve samples, fired to a maximum temperature of 700, 800, 900, 1000 and 1100 °C in an oxidising and a reducing atmosphere respectively.

9.4.7 Processing

The fired London Clay briquettes were processed at the Centre for Palynology of the University of Sheffield, in order to liberate any organic matter which had survived the firing process. This procedure was almost identical to that applied to the archaeological ceramics in Section 9.3. The reduction-fired briquettes contained a far greater quantity of organic matter than the oxidised briquettes, and it was therefore necessary to divide the residues from these samples into two fractions. One fraction was washed and sieved through a 7 μm nylon mesh, and the other fraction was treated with 'Schulze's solution' (an acidic oxidant), in order to reduce level of amorphous organic matter and then sieved.

9.4.8 Results

9.4.8.1 Oxidised briquettes

None of the oxidised London Clay briquettes contained any recognisable palynomorphs after processing. The strew slides from these briquettes were dominated by undigested mineral grains, black inertinite, brown to black amorphous organic matter and secondary minerals, which were precipitated during or after the digestion process. There was a progressive decrease in the absolute abundance of organic matter in the oxidised briquettes with increasing firing temperature.

Despite the high degree of thermal alteration exhibited in all samples, a definite colour change can be seen in the amorphous organic matter, by comparing the lowest fired (600 °C) and the highest fired (1100 °C) samples. The sample which was fired to a maximum temperature of 600 °C, contained dark brown to black organic matter, whereas that of the sample fired to 1100 °C was dominantly black or more rarely a very dark brown colour. It was not possible to discern any colour change between those samples which were separated by a smaller temperature range (e.g. 700-800 °C), due to the high thermal alteration of all samples.

9.4.8.2 Reduction-fired briquettes

The overall quantity of organic matter which was liberated from the reduction-fired samples, was far greater than that obtained from the briquettes which were fired in an oxidising atmosphere. The abundance of organic matter in the residues of these samples was so high that they had to be diluted before strew slides could be made (Section 9.4.7) It appears that the process of reduction firing had less affect on the organic matter in the London Clay, as many recognisable palynomorphs could be seen in all samples, even those fired to a temperature of 1100 °C. The organic matter in most of the reduction-fired samples was grey or black in colour, which is indicative of the high degree of thermal alteration that can be expected at these temperatures. There is however, a slight difference between the overall colour of the organic matter in the lowest and that of the highest fired samples. At this level of thermal alteration, the colour of organic matter appears to be related to the origin of the material in question, as well as its thickness and translucency. For example, in the
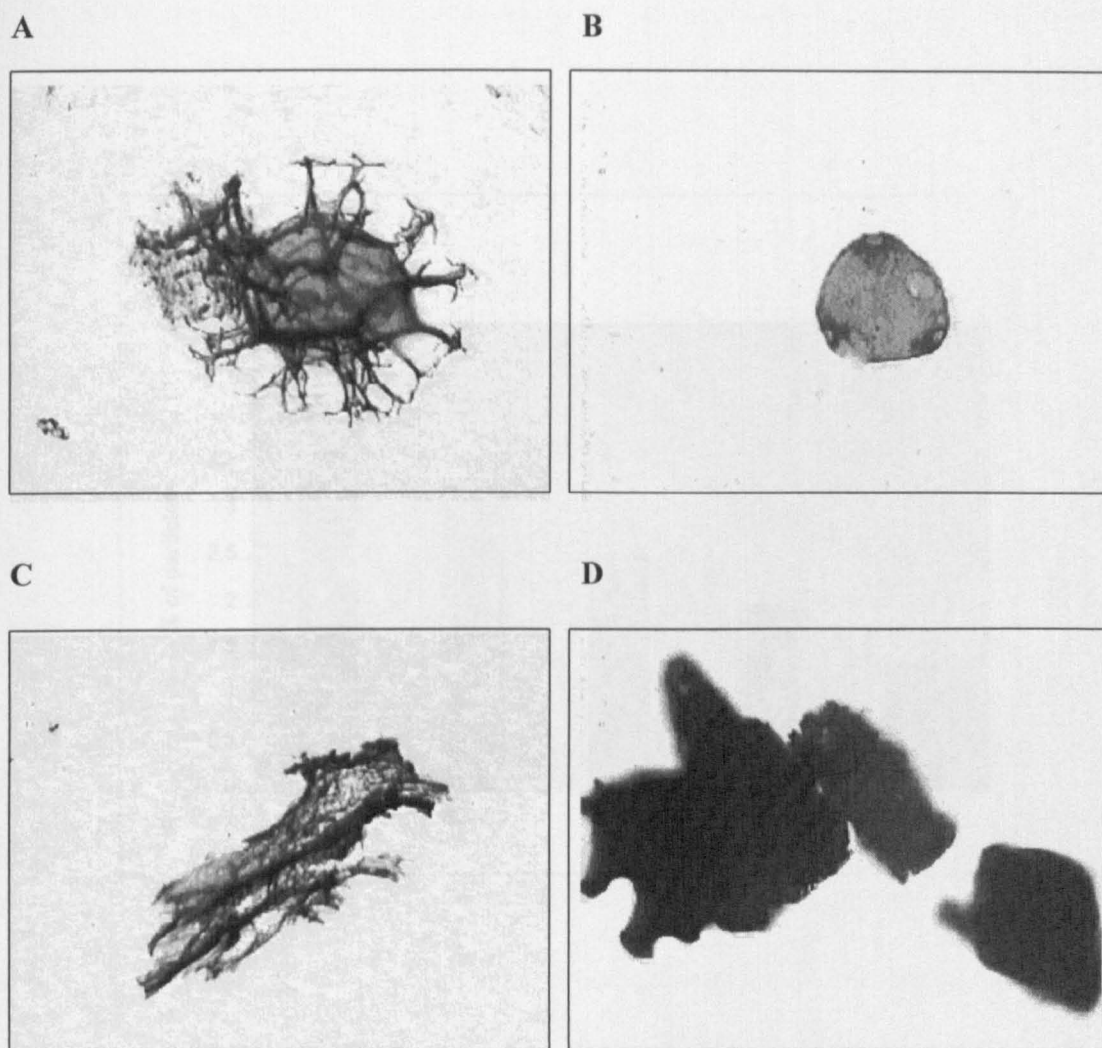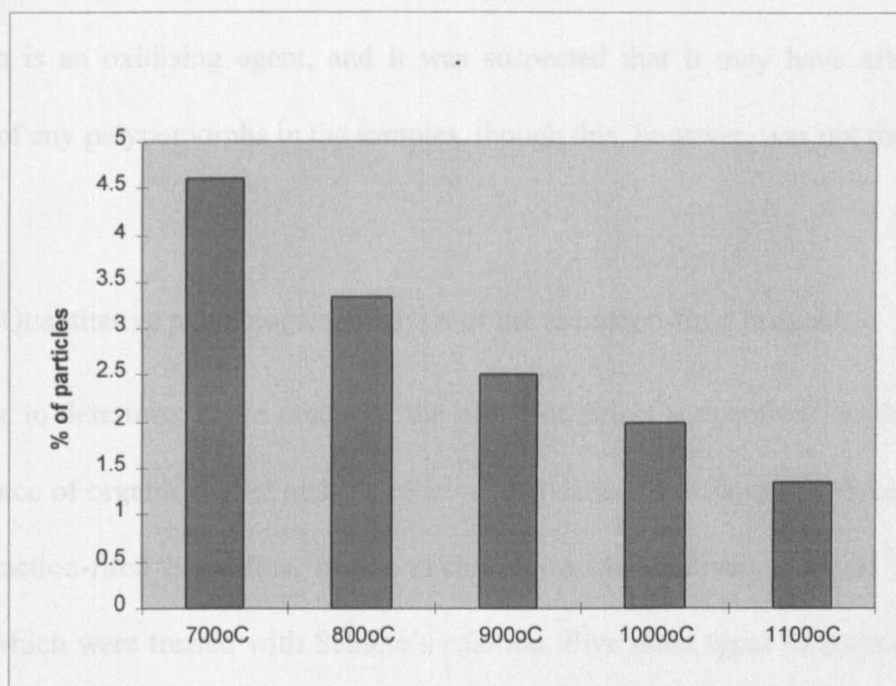
Figure 9.6 The different types of organic particles which were counted during the quantitative analysis of reduction-fired London Clay briquettes. A. dinocysts, B. pollen and spores, C. woody matter, and D. inertinite macerals. Amorphous organic matter is not illustrated. All pictures PPL. Field of view = 0.15 mm.

Figure 9.7 The overall abundance of palynomorphs in samples of London Clay fired to different maximum temperatures in a reducing atmosphere, as determined by the % field of view represented by all organic particles.

highest fired samples, the thick graphitic woody fragments have an opaque black colour, whereas the thin transparent palynomorphs are grey.

The strew slides which were prepared from the reduction-fired subsamples treated with Schulze's solution, indicated that this process reduced the amount of secondary precipitation as well as hindering the clumping of the organic matter. Schulze's solution is an oxidising agent, and it was suspected that it may have affected the colour of any palynomorphs in the samples, though this, however, was not the case.

9.4.8.3 Quantitative palynological analysis of the reduction-fired briquettes

In order to determine more precisely the effect of firing temperature on the overall abundance of organic matter and the relative abundance of different palynomorphs in the reduction-fired briquettes, it was necessary to quantitatively analyse the strew slides which were treated with Schulte's solution. Five main types of palynomorphs; dinoflagellate cysts, pollen and spores, woody matter, inertinite macerals and amorphous organic particles were counted during a single traverse of each strew slide, at a magnification of 400 x (Figure 9.6). In addition, an impression of the total abundance of palynomorphs was determined for each slide by calculating their percentage in 10 fields of view, using the *Aequitas 1.01* image database and archive management system for *Windows* 95. The results of this quantitative analysis are presented in Figures 9.7, and discussed in Section 9.4.9 below.
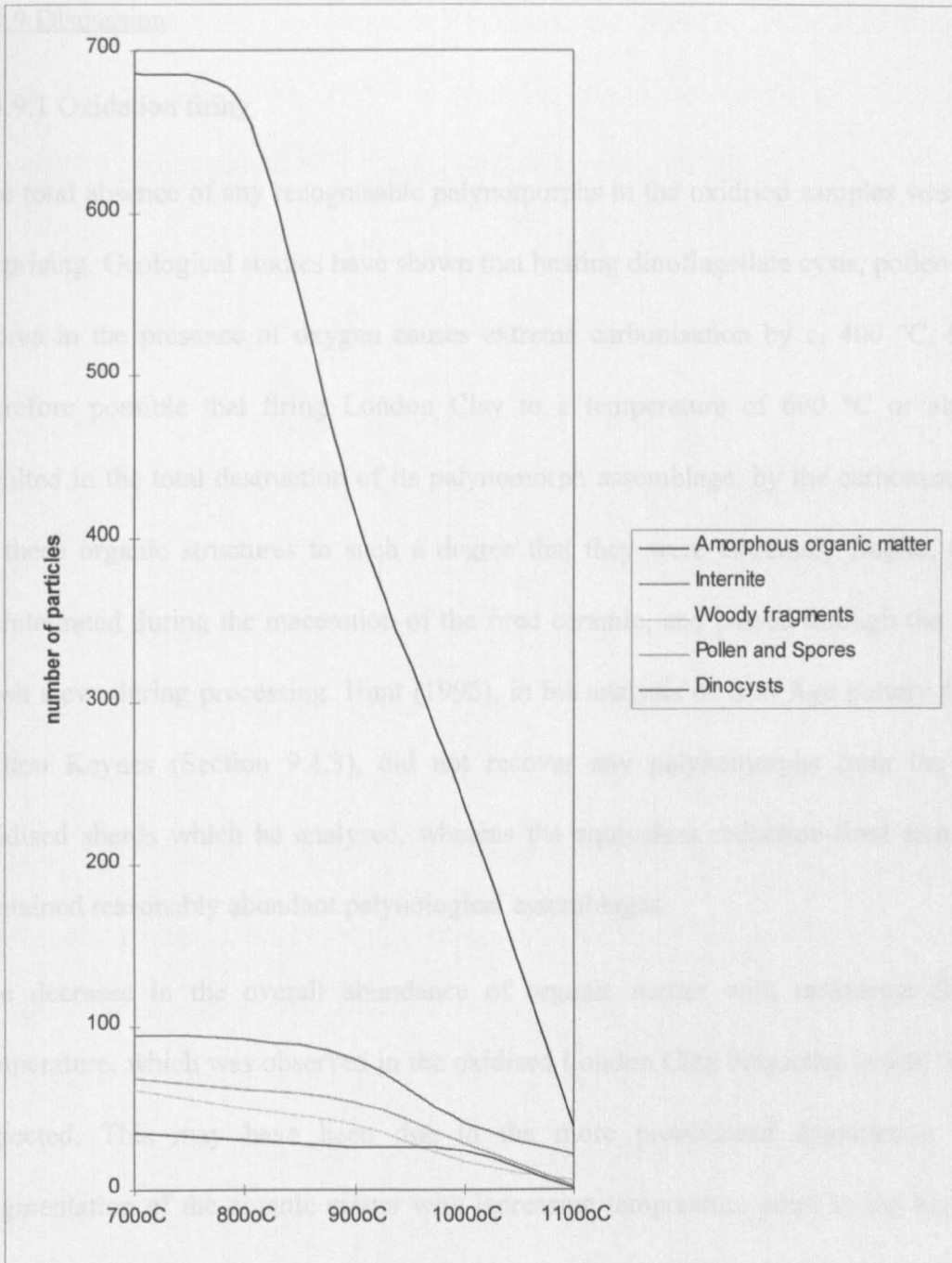
Figure 9.8 The overall abundance of the five principle groups of palynomorphs in London Clay fired to different maximum temperatures in a reducing atmosphere, as determined by the total number of particles belonging to each group which were encountered during a single traverse across a strew slide at a magnification of 400 x.

9.4.9 Discussion

9.4.9.1 Oxidation firing

The total absence of any recognisable palynomorphs in the oxidised samples was not surprising. Geological studies have shown that heating dinoflagellate cysts, pollen and spores in the presence of oxygen causes extreme carbonisation by c. 400 °C. It is therefore possible that firing London Clay to a temperature of 600 °C or above resulted in the total destruction of its palynomorph assemblage, by the carbonisation of these organic structures to such a degree that they were extremely fragile, then disintegrated during the maceration of the fired ceramic, and passed through the fine mesh sieve during processing. Hunt (1996), in his analysis of Iron Age pottery from Milton Keynes (Section 9.4.3), did not recover any palynomorphs from the ten oxidised sherds which he analysed, whereas the equivalent reduction-fired samples contained reasonably abundant palynological assemblages.

The decrease in the overall abundance of organic matter with maximum firing temperature, which was observed in the oxidised London Clay briquettes is also to be expected. This may have been due to the more pronounced degradation and fragmentation of the organic matter with increasing temperature until, in the highest fired sample (1100 °C), the proportion of black amorphous organic matter and inertinite was greatly reduced.

It was possible, by comparing the highest and lowest fired samples, to observe a slight colour change in the organic matter with increased firing (Section 9.4.8.1). This was very subtle and very difficult to detect between successive samples, due to the high degree of thermal alteration which was achieved in even the lowest fired samples. The

work of Doming (1986) and others has indicated that once organic matter has been heated to a temperature of c. 400 °C, there is very little significant change in its colour and opacity/translucency with increasing temperature (Figure 9.5).

9.4.9.2 Reduction firing

The presence of identifiable palynomorphs in the reduction-fired London Clay briquettes which were heated to temperatures of between 700 and 1100 °C, clearly illustrates the importance of oxygen in the carbonisation process. Sporopollenin is extremely resistant to decay, for example, by the corrosive action of hydrofluoric acid, which dissolves minerogenic matter. However, when heated in the presence of oxygen, these tough polymers begin to decay. It is therefore not surprising that, in the absence of oxygen, the process of carbonisation is greatly reduced.

The organic matter in the reduction-fired samples, was however not totally unaffected. It appears to have undergone a distinct colour change as a result of reduction firing, from yellow to dark brown and grey. Although there was a slight difference in colour between the lowest (dark brown and grey) and highest fired samples (grey), the appearance of pollen, spores and dinocysts in all samples is indicative of a high degree of thermal alteration; equivalent to that produced by a temperature of 400-500 °C in the presence of oxygen. This indicates that, despite the lack of oxygen, the process of thermal alteration still took place, but at a slower rate.

Figure 9.8 indicates that the progressive heating of London Clay, from 700 to 1100 °C under reducing conditions results in a steady decrease in the overall abundance of organic matter from 700-1100 °C. This is likely to be the result of the progressive

thermal alteration of different types of organic matter throughout the course of the experiments, as seen in Figure 9.8 and discussed below.

Figure 9.8 indicates that the abundance of all groups of London Clay palynomorphs decreased with increasing firing temperature. This is a result of the progressive thermal alteration of all organic matter with increasing temperature. The graph in Figure 9.8 however, highlights the differential response of the five groups of organic particles in terms of the rate and timing of their thermal alteration and progressive destruction. The abundance of amorphous organic matter experienced a sudden decrease between 800 and 900°C and fell steadily between 900 °C and 1100 °C. The proportion of woody matter decreased from 700 to 900 °C, though not nearly as rapidly as the amorphous organic matter, and all other groups of palynomorphs exhibited little or only slight change in comparison. Between 900 and 1000 °C a significant threshold in the thermal alteration of inertinite macerals and pollen and spores may have been reached, this resulted in a rapid decrease in the abundance of these particles. The abundance of dinoflagellates however remained unchanged until a level somewhere between 1000 and 1100 °C, after which it too exhibited a rapid decrease. The relationship between the abundance of pollen and spores and dinoflagellate cysts in Figure 9.8 is interesting, in that it indicates that pollen and spores are more susceptible to thermal alteration at lower temperatures than dinoflagellate cysts. This agrees with the work of Dorning (1986) who suggests that non-marine organic microfossils exhibit greater thermal alteration than those from marine environments at equivalent temperatures.

These familiar patterns indicate that the thermal alteration of organic matter in reduction-fired ceramics proceeds in a similar fashion to that which has been documented in the presence of oxygen. However, the process appears to take place at significantly higher temperatures.

9.4.10 Conclusions

Several conclusions can be drawn from the results of the above experiments. Firstly, it appears that palynomorphs may be destroyed during the firing of ceramics, at temperatures of 600 °C or more, in the presence of oxygen. This is likely to be a result of the extreme carbonisation of organic matter which takes place at this temperature, and has serious implications for the characterisation and classification of archaeological ceramics with palynomorphs (Section 9.5.1).

On the other hand, reasonably well-preserved pollen, spores, dinoflagellate cysts, inertinite and woody fragments may be isolated from reduction-fired ceramics heated to a temperature of up to 1100 °C. The survival of palynomorphs at such high temperatures is likely to be directly related to the absence of oxygen, which is crucial to process of carbonisation. In the present report, the palynomorphs which were isolated from the reduction-fired samples, were dark-brown to grey in colour, which indicates that some thermal alteration had taken place, (approximately equivalent to that achieved at 400°C in the presence of oxygen). The thermal alteration of the London Clay assemblage between the temperatures of 700 to 1100 °C in a reducing atmosphere, resulted in a steady decrease in the total abundance of all organic matter, as well as predictable trends in the overall and relative abundance of the different

types of palynomorphs (Section 9.4.9.2 and Figure 9.8). This clearly indicates that the process of thermal alteration proceeds in a similar fashion in the presence and absence of oxygen, but takes place at significantly higher temperatures in the latter case.

In the present report, there was a slight difference between the overall colour of the palynomorphs and amorphous organic matter in the London Clay fired at 600 and 1100 °C in reducing and oxidising conditions respectively. However, this was very indistinct, and more research is clearly required before the thermal alteration of organic-walled microfossils may be successfully used to determine the firing temperatures of ancient ceramics (Quinn and Dorning, work in progress). In the light of the experiments which are outlined in the present report, Hunt's (1996) interpretation of ancient firing temperatures in reduction-fired Iron Age ceramics (Sections 2.3.3.3 and 9.4.3), is likely to be an underestimation.

9.5 Approach to studying archaeological ceramics using organic microfossils

9.5.1 Description and classification

As with all other groups of microfossils which occur in ceramics (Sections 5.6, 6.7, 7.7 and 8.5), organic microfossils can be used to characterise and classify samples of archaeological pottery in terms of their presence/absence, their preservation and abundance, the context in which they occur and the taxonomic composition of the assemblage. In terms of the nature and origin of the raw materials of ceramic manufacture, a classification based upon the geological period or palaeoenvironment of which the palynomorphs are indicative, is preferred. Presence/absence and preservational groupings of archaeological pottery samples can be misleading, and in

the case of organic microfossils, are as likely to be related as much to firing technology, as to the presence/absence of organic microfossils in the unfired ceramics.

9.5.2 Provenance

By identifying the palynomorphs which are present in digested residues of archaeological ceramics, it may be possible to determine the geological age or depositional environment of the raw materials of ceramic manufacture. Such information may then be used to indicate the possible origin of these raw materials, by consulting published geological reports, or analysing representative clay samples (Hunt 1996).

9.5.3 Technology

Of all the groups of microfossils which occur in archaeological pottery, palynomorphs are perhaps the best suited to the interpretation of ceramic technology. The presence of Recent non-marine palynomorphs such as pollen and spores in samples of archaeological pottery, can be used to indicate the addition of organic temper during ceramic manufacture (Hunt 1996), especially when combined with plant macrofossil evidence (Ayyad *et al.* 1991). A potential technological application of organic microfossils in archaeological ceramics, is the determination of ancient firing temperatures (archaeothermometry), by their degree of thermal alteration (Hunt 1996). However, this technique requires further investigation, as experiments in the present

report (Section 9.4), indicate that the thermal alteration of palynomorphs during the firing of ceramics is highly dependent upon the atmosphere in the kiln.

9.5.4 Seasonality of pottery production and ancient agricultural practices

It may be possible, by identifying the palynomorphs contained within samples of archaeological ceramics, to determine the approximate time of the year during which pottery manufacture took place. As indicated in Section 3.4, the raw materials of ceramic manufacture can receive large quantities of allochthonous wind borne pollen and spores during their procurement, transportation, storage and preparation, as well as the pottery forming process itself. The composition of these contaminant palynological assemblages will be determined by the types of plants which ocurred in the landscape surrounding the potters workshop, which is in turn heavily dependent upon the time of year.

One potential source of contaminant pollen and spores in archaeological ceramics is agriculture. Pollen, spores and plant macrofossil material such as seeds can be incorporated in the raw materials of ceramic manufacture both intentionally, for example through the practice of chaff and straw tempering, and unintentionally by airborne contamination. By analysing the plant macro and microfossils contained with pottery sherds it may therefore be possible to gain an insight into the types of crops which were cultivated in ancient societies. This subject has been approached by Ayyad *et al.* (1991) in their analysis of unfired mubricks from the Giza pyramid area, Egypt.

Both of these potential applications of palynomorphs in archaeological ceramics are of course severely limited by the fact that organic microfossils can be destroyed at relatively low temperatures during the firing of ceramics, as well as the difficulties in distinguishing between the fossil, sub-fossil and Recent pollen already present in the raw materials of ceramic manufacture and those grains which were subsequently incorporated after its procurement. A further problem, which may hinder the determination of seasonality of manufacture, is the storage of raw materials for long periods (Rice 1987, 115), during which they may receive allochthonous pollen and spores from different times of the year. Nevertheless, both approaches may be feasible where reduction-fired ceramics occur with stored quantities of their original raw materials, so that it is possible to compare the palynological assemblages of the two.