EARLY IRON SMELTING IN NSUKKA-NIGERIA: INFORMATION FROM SLAGS AND RESIDUES

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A thesis submitted for the degree of Doctor of Philosophy
in the Department of Archaeology and Prehistory,
University of Sheffield.
January, 1992

SUMMARY

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The presence of large and diverse quantities of iron smelting residues indicates that iron smelting was practised in Nsukka. Unfortunately most of the present inhabitants of Nsukka do not know how the residues originated. Although some of the residues are indestructible, there is urgent need to study them now as they are either being quarried away for constructions or bulldozed to give way for development projects.

Hence an attempt was made in this research to study iron smelting in Nsukka through the analyses of the residues. Three investigation techniques were used during this research. These are ethnographic investigation, archaeological and analytical techniques. The analyses were conducted at two levels, morphological and microscopic. Carbon samples were processed to give carbon-14 dates and to identify species. The dates show that iron was smelted in Nsukka from about 765 cal. BC to about 1950 cal. AD. During this period, the industry passed through three distinct technological phases.

The study of the results of the slag, iron ore and the slag-inclusion analyses show that Nsukka iron smelters smelted local goethite and haematite in shaft furnaces. Initially the high presence of Al_2O_3 in the iron ores enforced the use of high smelting temperatures. During the late phase, the temperature was lowered by the use of sand flux. The presence and the location of iron ores determined the site of the industry. The sites were relocated when the ores were exhausted.

Available evidence suggests that Nsukka iron smelting was conducted by a closed-caste of specialised craftsmen, who moved from one location to another as they exhausted available raw materials on each site. These craftsmen limited themselves to the production of blooms. They never refined or forged the blooms. The blooms were sold to blacksmiths who refined and forged them into tools.

ACKNOWLEDGEMENTS

This research has come to light through the contributions of many people and organisations. It is difficult to attempt mentioning all who have been involved in this project by name, since such an attempt will produce an even bigger volume than the present thesis. I assure all who contributed in various ways towards the success of this project, but whose names are not mentioned here, of my sincere gratitude.

I am very grateful to the British Foreign and Commonwealth Office for their scholarship, which enabled me to undertake this research. My thanks are also due to the British Council who administered the scholarship. I am grateful to the University of Nigeria, Nsukka, for the three year study leave I needed for this research. The Accelerator dating laboratory, University of Oxford, processed twelve radiocarbon samples for which I am grateful. I thank the staff of the Royal Botanical Garden Laboratory, Kew for their study of my charcoal samples for species identification. The Robert Kiln Research Award financed part of my field work in Nigeria, for this I am very grateful. I would like to thank the Department of Prehistory and Archaeology, University of Sheffield for financing part of my field work in Nigeria.

Due to the interdisciplinary nature of this research, I had a number of supervisors. I thank God for blessing me with such wonderful, dedicated and sincere scholars. Their inspiration and encouragement were a source of strength to me throughout the research period. To Professor K. Branigan and Dr. B. S. Ottaway, my supervisors in Archaeology Department, I owe a lot of thanks for their devotion, patience and encouragement throughout my research. To Dr. J. A. Whiteman, from the Department of Materials, I can only say, "you are great and wonderful". His interest and commitment to this research have been extremely helpful and encouraging. Dr. Pat Phillips was a co-supervisor with Professor K. Branigan before she took early retirement. Within the limited period she supervised the research, she was able to chart its course and made the necessary connections that facilitated the execution of this research. Pat, please accept my sincere appreciation of your personal contributions, (moral, financial and material) "towards the advancement of African Archaeology".

My appreciation and gratitude go to other staff of the Archaeology Department who in various ways have contributed to the success of this research. I owe a lot of thanks to my fellow research students in Archaeology Department for their co-operation and help while I was attempting to use computer programs for this research. I am very grateful to Professor P. O. Nggody and Mr. B. A. C. Onwurah, both staff of the University of Nigeria, Nsukka, for their sustained encouragement and advice during the decisive period when this research was about to begin. Mr. Bernard Coffey, a former Regional Director, British Council Office Enugu, Nigeria saw the need for this research and made efforts to find sponsors for it. I owe a lot of thanks to him. Without his directions and advice I would not have gained the FCO Award.

Finally, to my wife Egoyibo, who took over the full responsibilities of our family and four children during the three years, I owe a lot of gratitude. Her encouragement and daily letters to me reduced my lonely moments.

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CHAPTER 1

Background To The Research

1:1 Aims And Scope of The Study:

The purpose of this research is to study the developments of bloomery iron smelting in the north western Igboland, Nigeria. The area covers Nsukka Division. Nsukka Division occupies the extreme north western part of the former Eastern Region of Nigeria (see fig. 1:1). With the evolution of the local government system and state structure in Nigeria in 1967, Nsukka Division is presently in Enugu state and has been carved into six local government areas. Detailed discussion of this is in section 2:1 below.

In Igboland in general, and in Nsukka in particular, a great deal of evidence of early iron working exists. This evidence comprises mostly slags and refractory clay. To date not much is known about the technological process involved in the early iron working of this area. The little that is known about it derives from few and scattered archaeological excavations and ethnographic, historical and oral traditions collected by people of various disparate interests.

Despite this lack of systematic study and understanding of iron working processes in Igboland, many assumptions, speculations and hypotheses have been put forward by various people concerning early iron working in Nsukka. Njoku (1986:120) for instance, from reconstruction of historical and oral traditions concluded that Nsukka Division is the oldest iron working community in Igboland from where this technology spread to other neighbouring iron working communities like Nkwere, Udi, Okigwe, Awka and Abiriba. Stating the above assumption in another form Afigbo (1973a, 1977) and Okpoko (1987:217) assert that Nsukka was a major iron producing centre in the precolonial Igboland. Developing this view further, Anozie (1979) and Anozie and Ray (1983a:25-27) put forward many assertions and hypotheses about early iron working in Nsukka and Igboland. For them Leja is the oldest iron working centre in Nsukka. This assertion was made without the support of any data. They went further to assume similarity in technological techniques between Nsukka iron working and north west Tanzania in practising preheating of air in the tuyere before the air enters the furnace chamber. They stated further that the cylindrical blocks of slags discovered at Leja are rich in iron and have been a bloom bank (Clark 1983:1), from where blacksmiths collect material for forging tools. Isichei (1976:9-16) from oral and historical studies recorded that the furnace types used in iron working in Nsukka were similar to those discovered at Taruga and assumed that iron working in Nsukka diffused from the Nok culture area, presently the generally accepted earliest centre of iron working in sub-saharan Africa.

Then Ekechukwu (1988), Ezeugwu (1986), Ezike (1989), Okafor (1984a), Onyeke (1986) and Njoku (1986) assumed without corroborative analysis that haematite was the main ore used in Nsukka iron working. Finally Van der Merwe (1980:492-493) and Anozie and Ray (op cit) claimed that Nsukka iron workers produced high carbon steel direct from the furnace. This claim was made without any analysis of iron samples from the area. In making this claim Van der Merwe recorded.

'In 1976 the author visited an Ibo village near Nsukka in the eastern region of Nigeria. Most of the men in the village are blacksmiths who produce iron objects of traditional design... Previously iron was smelted in the natural draft furnace in a neighbouring village but it is no longer produced. One blacksmith exhibited a small anvil which had been forged from the locally smelted iron and likewise claimed that it was 'much stronger' than the imported metal currently available but I assume that it is high carbon steel'.

I investigated and found out that the Igbo village visited by Van der Merwe was Umundu (see fig. 1:2) and the visit was made in the company of F. N. Anozie of the department of archaeology, University of Nigeria, Nsukka. Iron working tools forged from locally smelted iron, anvil and hammer (see plate 1:1) have been collected from this village and samples taken from them for analysis. Descriptions of these tools and the results of the analyses are in section 5:8.

In the face of these unsupported assumptions and speculations, this research is undertaken to make a study of early iron working in Nsukka through the analysis of iron working residues. The study aims at discovering the technological processes of this extinct technology in the area and to clarify these assumptions and speculations on Nsukka early iron working. On a wider sphere, the research aims at increasing the data base for a general understanding of African Iron Age.

Although iron working remains have been found in other parts of Nigeria outside Nsukka Division, (Bellamy and Harbord 1904, Effah-Gyamfe 1981, Fagg 1969, Nicklin 1981, Okafor 1983, Sassoon 1964, Sutton 1976 and Tylecote 1975), none can be compared with the remains in Nsukka both in volume and variety of techniques employed in the industry. In Nsukka division mounds, aggregates and cylindrical blocks of slags (see plate 1:2) of early iron working have been discovered at Owerre-Elu, Opi, Orba, Umundu and Leja (see fig. 1:2). Ethnographic, ethno-archaeological and archaeological investigations by Njoku (op cit) Okafor (op cit) and Onyeke (op cit) show that there were many differing systems of iron smelting with different apparatus and techniques within the same ethnic group. Unfortunately harsh weather had led to disintegration of most of the remains of this technology so that what has survived are tuyere and furnace fragments and slags which are almost indestructible.

Similar surviving residues, slags and refractory clay have been used with success by many people both within and outside Africa in the reconstruction of technological processes (Allen 1988; Bachmann 1982; Fells 1983; Friede 1977; Friede and Steel 1977, 1988; Friede *et al*.1982; Goucher 1983; McDonnell 1986; Morton and Wingrove 1969a, 1972; Hallett 1973; May 1905; Sperl 1980; Straker 1931; Todd 1976, 1979; Todd and Charles 1978; Tylecote 1975a; Tylecote *et al*. 1971; Tylecote *et al*. 1989; Van der Merwe and Killick 1979; Zacharia and Bachmann 1983). The study of such residues has been recommended as a reliable approach towards the reconstruction of extinct metallurgical processes (Bachmann 1982; Chami 1988; Coghlan 1977; Northover 1985; Pleiner 1980; Tylecote 1987). Pleiner (op cit), while recommending the use of slag study in technological process reconstruction, described the achievements of O. Voss in this field. Voss discovered that slag studies produce very valuable information leading to technological reconstruction. He not only discovered the various constituents of the analysed slags but was able to determine the melting points of the slag samples, the sources of the charcoal used in the smelting and finally discovered that the smelting was done without the aid of any flux while the furnace was adjusted to slag tapping.

This recommendation is not only useful but it is very appropriate for most iron smelting sites in Africa where most of the early iron working communities have no record of their technology. Iron working residue study is the only direct means of knowing what went on during these processes of iron smelting since in most of these communities, the account of these processes are from informants most of whose generations never witnessed the process in action while the majority of them, like in Nsukka will innocently tell you, *madu emeghi afuru, afuru si n' ala epuputa* (slags germinate from the ground and were never made by man). In situations such as this, the only way to establish the process of iron smelting is the study of the surviving residues.

Although these residues are said to be indestructible (Tylecote 1987:291; Van der Merwe 1969:100), the wind of rural development that is blowing across Nigeria now is felt at Nsukka where it is threatening the continued survival of these residues in such condition that useful information can be derived from them. In Nsukka, rural industries, schools, markets, roads, health centres and community squares are being constructed at an alarming rate. These constructions though beneficial to the communities, are destructive of archaeological sites. The worst hit archaeological sites in Nsukka are iron smelting sites that lose many tons of slags to road and house builders every year. Where these slags are not quarried away, they are bulldozed to form foundations for these developmental projects. For example a new timber shed in Nsukka, parts of Opi boys secondary school, Opi motor park, Opi primary school, Odinanso Idi Opi village square, Afor Opi market, Opi customary court, Opi health centre, Umundu secondary school, and Dunoka village square at Leja are all built on top of these smelting residues. The new Nsukka to Isiakpu road and Ndiagu Ndiuno Ama-Orba to Ohom Orba road all cut through Owerre-Elu and Ama-Orba early iron smelting sites respectively.

As the majority of Nsukka people are farmers and there is always land scarcity in the area, their constant farming on and around these sites threatens the survival of these residues in their primary context. Three factors have therefore made this study both necessary and urgent. The volume and variety of available residues in Nsukka communities, the lack of detailed study of these residues which had led to variety of views and assumption, and the kind of threats that face these residues mean that if reliable information is to be derived from them that it should be done without further delay.

Slags, cinder, fuel ash slag, furnace and furnace lining are common smelting residues found in iron smelting sites, (McDonnell 1983; Morton and Wingrove 1969a; Tylecote 1987:291-298). These residues are described and discussed in chapter four. Among them furnace and furnace lining, cinder and fuel ash slag survive or disappear from smelting sites according to weather conditions (Pole 1975:11-39), age of the site and the intensity of human activities in the area. The older the site, the less chances archaeologists have of recovering these particular residues from iron smelting sites. Though they result from the iron smelting process, when analysed they usually give biased information especially on the mineralogical and elemental constituents involved in the bloomery process of smelting (Friede and Steel 1977:226-227; Friede *et al.* 1982:41). Bearing these facts in mind, smelting residues used in this study are well formed slags that can give as unbiased and comprehensive information as possible on this extinct bloomery process, refractory clays and ores.

Analysis of these residues, and slags in particular, have led to the discovery of their constituent elements (major and minor elements). These element grades are indicators of the character of the slag and changes in them reflect technological changes and development in the operating system. Some of the elements at major and minor levels have been useful in provenance evaluation and some people have used them in locating the sources of raw material used in the manufacture of certain tools (Haldane 1970; Hedges and Salter 1979; Tylecote 1970a; Van der Merwe and Killick op cit). This study also reveals that the mineralogical composition of slags reflect ore composition and the operating process used and has led to the discovery of the free running temperature of the slags in Nsukka and the furnace operating temperature, and the slag density. And most importantly, it has been a source of chronological reconstruction for many of these sites. None of these was known about early iron working in Nsukka before this research was done. Details of these discoveries are in chapters five, six and seven.

This present study has been limited to the study of iron smelting slags and residues and so does not include iron smithing residues. This is necessary in order to limit the number of processes that are to be considered, bearing in mind the pressing constraint of time and resources. Again iron smithing operations are still a very buoyant industry in Nsukka area and have been studied in some depth by many people (Anozie 1979; Ekechukwu 1988; Ezike 1989; Njoku 1986; Okafor 1989; Onyeke 1986). Further more, discoveries by people that have done residue analysis and classification show that, apart from morphological features, there are not many differences that distinguish iron smelting slags from smithing ones (McDonnell 1986:224). Physical analytical investigations also show that the only difference that may distinguish smithing slags from smelting slags is the relatively higher percentage of manganese in the smelting slags; often by more than 0.5% (McDonnell ibid).

1:2 State Of Early Iron Smelting Study In Africa:

African early iron-working studies, like other branches of African archaeology, has been bedevilled by too much unco-ordinated research. Most importantly the approach to the subject has not been well ordered. This has resulted in a situation where most of the written works on the topic lack basic information and details and this has caused much concern to many people. Andah (1979:135-136) lamented that not much is known about the Early Iron Age of West Africa. Njoku (1986:45) observed that impressive number of iron working sites have been identified in Igboland but only a couple have been excavated and only one radiocarbon dated. Schmidt and Childs (1985:54) also lamented that one of the paradoxes in the archaeology of Iron Age Africa is the absence of well dated and well documented evidence of Early Iron Age technology.

The major obstacle to a clear understanding and study of African early iron working is that most of the pioneer works on this topic were exotic in approach rather than contextual. Initial attempts to study African iron working were wrongly geared towards finding the origin of African iron working instead of studying iron working *per se*. Because of this faulty foundation, nearly all the major works on African iron working were devoted to the debate on the origins of the technology in Africa (Andah 1979:135-142; Arkell 1968; Diop 1968; Huard 1966; Kense 1985; Lhote 1952; Mauny 1952; Keteku 1987; Van Noten 1979; Okafor 1977; Phillipson 1985:148-186; Rustard 1980; Schmidt and Avery 1983:432-434; Shaw 1969; Shinnie 1966; Todd 1976:5-25; Trigger 1969; Tylecote 1970b, 1975a; Van der Merwe and Avery 1982:150-152). It is not my intention to get into these debates here for I have properly treated it elsewhere and concluded that it is like putting the cart before the horse (Okafor 1977).

Again it has been my view (Okafor 1984a:1-4) that the study of African iron working should concern itself first with studying the nature and processes of iron working as practised in the various communities of Africa that had that technology in all its ramifications to acquire a data base for comparative studies. Without such a data base no study of origins will lead to objective conclusions. It is only when the technological processes of most African communities that did bloomery iron smelting have been established that one can have raw data for comparing the African type(s) of iron smelting with any other. It is the result of such studies that can point out if African early iron working diffused from the projected donor areas or was an independent development or an amalgam of both factors.

Presently African iron working studies lack volumes of comparative data. As far as published works are concerned the data base is still very low and is very dispersed in time and space. A realistic study of the origin of African iron working should include a comparative study using amassed data from most parts of Africa and the supposed donor zones. The present study is an attempt to provide such a data base from Nsukka early iron working communities. We are at a stage where data accumulation is very essential, some form of foundation has to be laid before synthesis and comparisons can emanate.

As hinted above pioneer studies of early African iron working were concerned more with finding the origin of iron technology in Africa. Presently there are three schools of thought as regards the origin of African iron working, the diffusionist school, the indigenous school and the cautious school (Okafor 1984a:2).

In the diffusionist school are people like Arkell (1968) and Shinnie (1966) who hold that African iron working came from Egypt through Meroe and the Sudan. This view was modified by Huard (1966) who proposed that African iron working diffused from Meroe and the Gulf of Syrte in Libya. The view that African iron working diffused from Meroe has been put to rest by work done by Tylecote at Taruga (Tylecote 1975a-b) and Meroe itself (Tylecote 1970b) which show that Meroic iron working was much later than the earliest Taruga dates. Trigger (1969) postulates that African iron working originated from East Asia through Madagascar or the East coast of Africa, while Williams (1969, 1974) believes that the African iron working came from Meroe and Carthage. The view that Carthage was the source of African iron working is presently the most favoured by the diffusionists. This idea was originated by Mauny (1952) and since then been held and developed by other people like Shaw (1969, 1975), Tylecote (1970b, 1975a) and Van der Merwe and Avery (op cit). Mauny (1952) speculates that the Berbers from the Phoenician settlements of north Africa were the agents through which iron working diffused into Africa and possibly through trade voyages the Phoenicians organised along the coast of West Africa. Presently not much is known of the nature of iron working in the Punic North Africa and the Sahara, and yet this idea is held to. Further more, the inscription found in the temple of Baal-Hamon, which recorded the voyage of Hanno king of Carthage around the Atlantic coast of Africa in 425 BC on which the voyage tradition was based has been discovered to be a forgery (Mauny 1970:78-80).

Tylecote (1975a:4-5) and Van der Merwe and Avery (1982:150-151) hold that it is difficult to support the idea that iron was independently worked in Africa because Africa had no pyro-metallurgical traditions before iron. For them the most likely source of African iron is Carthage where little or nothing is known about early bloomery iron working. The view that Africa had no pyrotechnology before iron is presently being challenged by discoveries of some archaeologists working in Niger and Mauritania. In Niger at a site in Sekkiret area, copper furnaces were dated to 1662 ± ? BP. and 1175 ± 117 BP. (Calvocoressi and David 1979:10) and in Agades many copper smelting furnaces were dated to the second millennium BC (McIntosh, S. R. and McIntosh, R. J. 1983:241). Then at Azelick close to Agades, dates ranging from early to mid first millennium BC were obtained for copper working (ibid). From Akjoujt in Mauritania dates of ninth and sixth centuries BC were obtained for copper working (Posnansky and McIntosh 1976). These discoveries have led Avery and Schmidt (1979) and Schmidt (1983) to propose a hypothesis that iron smelting might have developed independently from a copper smelting technique which might have used iron oxide as flux.

Opposed to this diffusionist model are people in the indigenous school. The protagonists of this school include Lhote (1952) who observed a total lack of evidence for early iron working among the Saharan Berbers and on that fact suggests that iron working was independently developed in Africa. Diop (1968) and Keteku (1987) hold that iron was independently worked in Africa. Diop (op cit) after considering the early dates of 3500 BC obtained for the iron working sites at Nok (see page 10) concluded that he did not see why the negroes should not have discovered how to work iron by themselves. Rustard (1980:237) supporting the independent development view concluded that, 'the emergence of iron technology in the Nok culture cannot be explained by diffusion from Meroe. Further it is highly unlikely that Carthage was a source of knowledge about iron technology for the participants in the Nok culture. Therefore in the absence of the evidence to the contrary, iron technology may be considered an autochthonous development in the Nok area or culture'. Schmidt and Avery (1983: 433) supporting independent development of iron in Africa argue that the knowledge of iron smelting might have been gained by Africans through the use of iron oxide as flux in copper smelting which has been demonstrated to have great antiquity in West Africa. This phenomenon, they suggest occurred at the turn of the second and first millennium BC. They further suggested that the earliest evidence of this independent development of iron in Africa are at Taruga in Nigeria and Kagera north west Tanzania which developed different technological process of preheating of air in a tuyere for higher temperatures in their smelting operations.

The third group comprise the people which I have labelled the cautious group (Okafor 1984:4-3) and they are represented by Andah (1979) and Phillipson (op cit:170-171). These people who, to a certain extent, entertained the idea of independent development of African iron working, observe that more work needs to be done before a firm stand can be taken on the issue. For this reason Andah recorded that not enough is yet known about when, with whom and how iron working began in West Africa for any one to assert positively that the knowledge of the process was transmitted from outside. Phillipson (op cit) on his part notes that, 'the first point is that knowledge of iron working can no longer be assumed to have been brought to West Africa from the North... The most plausible explanations for the initial development of West African iron working are either that it is a local invention based upon earlier copper smelting technology or that the relevant knowledge was transmitted south of the Sahara from north Africa. Elaboration of these hypotheses... will have to await the recovery of detailed evidence as to the smelting methods that were practised in these two areas'.

Meanwhile, while the debate about the origins was raging, several early dates were accepted for iron working in parts of Africa. For many years dates from Taruga in the Nok culture area have been held as the earliest for iron working in Sub-Saharan Africa. These dates published by Fagg (1969) and Shaw (1969) include dates of 440, 300 and 280 BC. Other dates from Nok culture area include 591 ± 75 BC, 538 ± 84 BC, 341 ± 133 BC from furnaces at Taruga (Calvocoressi and David (1979:10). Dates of 555 ± 210 BC (Calvocoressi and David op cit) and 620 ± 230 BC. (Shaw 1981:630) were obtained on the basis of the thermoluminescence dating from Nok itself. Outside the Nok figurine area, early dates for iron working in Nigeria are a 4th century BC date at Ise Dura rock shelter (Calvocoressi and David 1979:11), a second century BC date at Baha mound (Shaw 1978:97), a fifth /sixth century AD date at Daima (Connah 1968:317; 1981:146) and 685 ± 80 AD and 750 ± 155 AD dates from furnaces at Samaru West site I Zaria (Sutton 1976:18).

Recently Calvocoressi and David (op cit) published a date in the mid ninth century BC obtained from dating a piece of wood covered by iron slag in a smelting furnace at Do Dimmi in Niger, while at Meroe, the widely suggested source of African iron, dates of third to sixth century BC were obtained from materials associated with slag (Green 1975). From Buhaya in north western Tanzania, dates of the ninth and fifth centuries BC were accepted for early iron working (Avery and Schmidt 1979; Schmidt and Avery 1978). Kense (1985:16) published and accepted mid first millennium BC to mid first millennium AD dates for Daboya iron working site in Ghana. And from the Congo basin de Maret *et al*·(1977:495) accepted fourth to third century BC dates for the earliest iron working there.

It is to be observed that associated with some of these accepted dates are older dates which were rejected as, 'unacceptedly earlier' or 'impossibly old' or 'earlier than what is assumed' to be the date for the beginning of iron working in Africa. Such rejected dates include a 3265 ± 65 BC date from Nok Nigeria published by Barendsen *et al* (1957:916-918), 3190 ± 129 and 3580 ± 130 BC (Davies 1966:471) and 1630 ± 130 to 1240 ± 120 BC dates from Ntereso Ghana (Shaw 1969:236) and a second millennium BC date from Buhaya in Tanzania (Schmidt and Avery 1978).

Many people have associated the beginning and spread of iron working in most parts of Africa with the Bantu and their spread (Fagan 1965; Greenberg 1963; Mason 1974:211-216; Davies 1966). In fact, many anthropologists and archaeologists have looked on the Bantu spread as being synonymous with the spread of iron in Africa (Hiernaux 1968:506). Recently one of the experts in Bantu studies, Ehret (1982:57-65), has discovered that the beginning of the Bantu spread from their ancestral homeland, the Nigerian-Cameroon border area, was around 2000 to 3000 BC. This discovery has thrown into confusion those who see no relation between Ehret's 2000 to 3000 BC dates for Bantu spread and the accepted dates for iron beginning in Sub-Saharan Africa. But a closer study reveals that Ehret's dates bracket most of those rejected dates obtained from iron working sites in various parts of Africa. Probably a fresh review and reconsideration of opinions are needed for an objective assessment of this problem.

1:3 Archaeological And Experimental Studies Of Early Iron Smelting In Africa

Despite the fact that much energy and time were wasted debating the origin of African iron working, some advances and positive discoveries have been made on certain aspects of African Iron Age studies. The most positive achievements are the number of Iron Age sites that have been recognised, mapped and described in various parts of Africa. In South Africa, Van der Merwe and Killick (op cit) and Van der Merwe and Scully (1971) have extensively studied Iron Age sites around Phalaborwa, while Friede and Steel (op cit) have studied and reconstructed an Iron Age smelting pit furnace at Bultfontein in central Transvaal as well as Iron Age sites at Broederstroom, Utkomist cave and Olifantspoot which were mapped and described by Friede (1977).

In north western Tanzania, the efforts of Schmidt and Avery are producing what is turning to be a new insight into the African Iron Age. Schmidt (1977, 1983), Schmidt and Avery (1978) and Schmidt and Childs (1985) described extensive Iron Age site near Kemondo Bay in Kagera region, the KM2 and KM3 sites, where techniques used in the smelting include the preheating of air in the tuyere before the air enters the furnace chamber and the production of high carbon steel from the furnace before decarburization in the forge. Details of this are in section 1:4 of this chapter where peculiarities and explanations of the African Iron Age are treated. Among the Maa speaking pastoralists of north central Kenya, Roy Larick (1986) studied Iron Age sites scattered within the hills and valleys there and established the settlement pattern of the communities in that region.

In the Congo Basin, studies undertaken by Van Noten and his group (Van Noten 1985) have not only revealed numerous Iron Age sites in Zaire, Rwanda and Burundi but the furnace types, smelting techniques and the chronology of the Iron Age in that region is now one of the best known in Africa. In northern Cameroon, around Bamenda and Ndop plain, work done by Warnier (1975), Warnier and Fowler (1979) and Rowlands (1989) have brought to light extensive bloomery iron working in the 'Iron Belt' of the grassfields of Cameroon. It is now possible to trace the development of this industry, its decline, revival and trade exchanges connected with it through the discoveries of these people. Furthermore, the present research programme mounted by the University of Calgary under Nicholas David (David *et al* 1989) on the early iron working in the Mafa region of Cameroon is revealing the technological processes involved in this extinct technology.

In the Sudan, Shinnie (1985) Shinnie and Kense (1982) and Tylecote(1970b, 1975b) have through their studies established the nature of Meroic iron working and its chronology. Discoveries by Tylecote (1975a) in particular helped in redirecting the minds of people interested in the beginning of iron technology in Africa away from Meroe. In Ethiopia recent work by Todd

(1976, 1979) and Todd and Charles (1978) among the Dimi iron workers is the most detailed Iron Age study in Africa that involved physical analysis of iron smelting residues. Todd not only discovered the furnace type used by the Dimi iron smelters but analysed the produce of these furnaces as well as ores recovered from the sites.

From iron smelting sites in western and central Sudan, and the Nile valley Haaland (1985) discovered the techniques of iron smelting in Mema, Darfur and Meroe and from the study inferred the various consequences of iron working which include deforestation, environmental deterioration and shift in settlements. Work done by Fagg (1969) and Tylecote (1975a) at Taruga and Nok produced the earliest accepted dates for iron working site in sub-saharan Africa whose furnace type and the associated cultural materials are well known. It is now known that the Nok figurine area with over 30 sites covers some 300 miles long and 100 miles across to the west and south of Jos plateau (Aiyedun and Shaw 1989:6).

From other parts of Nigeria, results of investigations conducted by Anozie (op cit), Effah-Gyamfi (1981), Nicklin (1981) Okafor (1983, 1984, 1988), Sassoon (1964) and Sutton (1976) have revealed that extensive and complex iron working was practised in various parts of Nigeria. Goucher (1983) and de Barros (1988) have discovered, mapped and studied iron working sites in Bassar, Togo. Goucher applied physical analytical techniques in the study of the produce of the technology to determine their mineralogical and elemental composition while de Barros delved into the consequences of this technology on Bassar community. Finally from northern Ghana, works by Pole (1974, 1975, 1985) have helped in the understanding of the various furnace types and smelting techniques that were involved in the Early Iron Age of northern Ghana.

Besides archaeological investigations of Early Iron Age smelting sites, some people have undertaken what Childs and Schmidt (1985:122) termed ‘imitative experiments’ based on ethnographic models aimed at reconstructing techniques of African iron smelting. In South Africa, Friede and Steel (1977,1988) conducted two smelting experiments using shaft and bowl furnaces. In north west Tanzania ethnographic information gathered by Schmidt (1977) among the Haya formed the base for his experimental iron smelting at Brown University in 1975 and subsequent observation of a series of six replicated Haya smelts in 1976 (Childs and Schmidt 1985). Except for the experiments based on Haya tradition, which had the specific objective of testing the preheating hypothesis, the rest, like the ethnographic observation of local smelters replicating the process such as Todd did at Dimi (Todd op cit) Haaland in Darfur, Pole in Ghana, Sassoon in Surkur and Van Noten in Madi and Gisagara (Van Noten 1985), aimed at providing clues for better understanding of archaeological iron smelting remains. They also sought to describe iron smelting techniques as still known by the people that remember the processes involved in the operation.

One fundamental observation common to these experiments and replications, is that most of them produced little or no iron. However, an exception to this remains the replication smelt at Mafa in northern Cameroon. This low yield of iron according to Friede and Steel (1977:239) is probably due to the inherent complexity of Iron Age smelting processes, a complexity partly due to large numbers of operational factors, partly due to accidental changes such as fluctuations in temperature and air flow, and partly due to environmental influences such as rain and strong wind. Todd and Charles (1978:68) attribute the low yield obtained from the Dimi smelt they observed, probably to their failure to observe certain norms of the technology. They recalled that they attempted to take flash photographs when the smelting was on and the flash was deemed to be lightning which was a bad omen during smelting. It should be borne in mind, that except the smelt at Sukur observed by Sassoon, which yielded much bloom, and that observed at Dimi by Todd, both of which were conducted by people still practising the trade, that the rest of the replications and experiments were either conducted or directed by people acting from hindsight. Most of these had broken with the trade for over sixty to eighty years past. Some only witnessed the smelting as small children. In such a situation it is impossible to expect good yield from those smelts.

1:4 Peculiarities And Explanations In African Iron Smelting:

An intriguing aspect of African iron working is the observation of many peculiarities which are not paralleled any where else. Such peculiar aspects and techniques have put African iron working into a category apart from all others. Three recurrent aspects of African iron working reported from many parts of Africa are the production of high carbon steel direct from the furnace which was later decarburizied in the forge, the preheating of air in the tuyere before it enters the furnace chamber and the development of the natural draft tall furnace (McIntosh, S. R and McIntosh, R. J. op cit :244).

The production of steel is a phenomenon that was achieved through different techniques in many parts of the world in recent time. This was achieved in the Eastern Mediterranean by carburizing during forging of low carbon blooms produced from smelting. In China wrought iron of very high carbon content produced from the smelt was decarburized in the forge to obtain steel. But in Africa high carbon steel was produced direct from the furnace and this has been documented by archaeologists in their ethno-archaeological and archaeological investigations (Van der Merwe 1980:497).

In Africa among the ethnographic records of this phenomenon are those of Bellamy and Harbord (1904:123) in Ola Igbi near Oyo in Nigeria where bloomery smelting using roasted ore produced blooms of 1.6% carbon. An analysis of blooms produced by the Shona smelters from Zimbabwe during the 1930 agricultural show in Johannesburg revealed that the bloom had areas of high carbon steel interspersed with low carbon regions (Stanley 1931). In north west Tanzania the Haya iron smelters reported by Schmidt and Avery (op cit) and Schmidt (op cit) produced high carbon steel from the furnace.

As described by Schmidt (1977:337) preroasting of ore is essential in this technique for the introduction of carbon into the ore. This is enhanced by the carbon produced by charred swamp grass at the base of the furnace. Because of the high temperature involved, the ore melts and forms a fayalite-wustite slag which mixes with the fibrous carbonaceous swamp grass that provides a high carbon contact area. The entrapped carbon reacts with the slag to produce carbon monoxide bubbles which remove oxygen from the slag now saturated with iron whose crystals precipitate and grow. Metallurgical analysis of these precipitated irons show that the crystal perfection was good with straight Neuman bands and well formed crystals. The carbon from the grass carburizes the iron surrounding it and this results in a steel with variable local carbon content.

Discoveries from archaeological contexts confirm that the production of high carbon steel was an integral aspect of African iron smelting. Pieces of iron excavated by Caton-Thompson (1931) from below the 14th century conical tower in Great Zimbabwe were found to consist of medium carbon steel in the centre with low carbon material surrounding it. This does not show a carburized structure and has been found to be medium carbon steel decarburized on the surface during forging (Van der Merwe op cit :497). From Tana in Ethiopia iron specimens excavated by Dombrowski (1971) dated to the 13th and 14th centuries AD yielded a carbon content of 0.62, 0.79 and 1.62 percent. From Jenne Jeno in

Mali artefacts of medium carbon steel with a homogeneously carburized structure were recovered in second century B.C context (McIntosh, S. R. and McIntosh, R. J. op cit). Finally iron specimens dated to 14th century AD recovered from archaeological sites near Phalaborwa Transvaal and Orange Free state provence of South Africa consist of uniform medium carbon steel (Van der Merwe and Scully 1971).

A study of oral tradition of the Haya in north western Tanzania during 1969 and 1970 led to the discovery of another outstanding aspect of African iron working, the preheating of air in the tuyere before it enters the furnace chamber (Schmidt 1977). Archaeological investigations at the site of Rugomora Mahe confirm the accuracy of oral traditions concerning early iron smelting in the kingdom 2400 to 2550 years ago (Schmidt and Avery 1978). Analysis of archaeological slags from the site indicate a free flow temperature of 1350 to 1400°C. From results of bloomery smelting experiments conducted by some people, this is within the highest temperature limits that can be obtained in European bloomery technology (Todd and Charles 1978; Tylecote *et al.* 1971). Analysis of tuyere fragments recovered from the site suggest that they have come from inside of the furnace.

Van der Merwe and Avery (1982:153) outlining the hypothesis of the preheating technique, noted that in Buhaya, tuyeres are inserted up to 60cm into the furnace, and bellows placed about 5cm from the end of the tuyere. Air is then blown into a bell-shaped opening. The gap between the bellows and the tuyere and the bell-shaped opening work together to suck in considerable more air than emitted by the bellows. Some air is pulled back when the bellows are pulled up, so that the air oscillates while passing through the tuyere. Combustion gas flows back along the tuyeres inside the furnace, and the surface is wet with slag, indicating the temperature on the outside of the tuyere, to be in excess of 1250°C. At this range slag penetrates about one-third of the way through the tuyere wall. This causes the air passing through the tuyere to be heated and enter the furnace at 500 to 600°C. Calculations from furnace types without preheating techniques indicate that the highest temperature with reducing atmosphere is 1250°C to 1300°C. Then in the preheated furnace, where a start is made with air blast of 500 to 600°C the same conditions will produce temperatures from 1800°C and above.

In iron working employing the preheating technique the process is quite different from ordinary bloomery techniques where ore is reduced and slagged leaving a spongy bloom on top in the furnace. Rather in the preheating process the ore melts and the slag saturated with iron undergoes a carbon boil. Iron crystals precipitate and grow as temperature increases (Schmidt op cit :337).

From investigations of Schmidt and Childs (op cit) at KM2 and KM3 sites near Kemondo Bay in north west Tanzania, it has become possible to recognise smelting sites where preheating of air was practised. This is readily observed by analysing tuyere fragments recovered at smelting sites. Tuyeres used in the preheating technique display evidence of slagging, vitrification and reduction and these are held as diagnostic indications of preheating. Apart from Rugomora Mahe and KM2 and KM3, sites with evidence of preheating have been suggested at Phalaborwa in South Africa (Van der Merwe and Scully op cit), and among the Dimi in Ethiopia (Todd 1976). This technique has also been reported at Ola Igbi near Oyo in Nigeria (Bellamy and Harbord op cit) and at Sukur in northern Nigeria (Sassoon 1964:175). Anozie and Ray (op cit) have suggested that this technique was also practised by Leja iron smelters in Nsukka, Nigeria.

Explaining situations that led to the invention of the preheating technique, Schmidt (1983), Schmidt and Avery (1978) postulate that apart from the need to achieve high temperatures for the smelting that the technique developed in response to dwindling supplies of the necessary fuel for smelting, since the technique achieves high fuel economy. Outside Africa the only place the preheating of air in the tuyere during the Early Iron Age has been recorded is in China (Tylecote 1965). It is to be noted that the preheating hypothesis has not won the acceptance of some archaeometallurgists especially Professor Tylecote (personal communication 1989). He holds that the furnace working temperature is not affected in any degree by any length of tuyere inserted into the furnace, and has thus rejected the hypothesis.

An aspect of African iron working where large volumes of literature has been produced is on furnace description and typology. Outstanding works on this topic include those of Cline (1937), Bellamy and Harbord (op cit), Forbes (1933), Francis-Boeuf (1937), Friede and Steel (1988), David *et al.* (1989), Pole (1985), Sassoon (1964), Sutton (1976, 1985) and Tylecote (1970, 1975). Evidence from these works reveals the multiplicity of furnace types that were in use in Africa and most of these were widely distributed. Typologically they are grouped into four, (1) the simple bowl furnace, regarded as the earliest, (2) low shaft furnace with no provision for slag tapping, (3) low shaft furnace with a hole for removing bloom and tapping slag and (4) shaft furnace of about two to seven metres tall operated without any bellows (Van der Merwe 1980:490-491).

This fourth type has been observed to be peculiar to Africa. Except in the Upper Burma and India, reported by Tylecote (1965), where similar self-induced furnace was operated, no other evidence is available from other parts of the world since Tylecote (1980:216) doubts a suggestion that furnaces of the Roman period in Austria and the British Isles were self-induced. The technique capitalizes on the low density of hot air inside the furnace. Hot air inside the furnace has a lower density than the air outside and thus the hot air rises resulting in the low pressure in the furnace. The low pressure causes more air to be drawn in through the tuyere below (Van der Merwe and Avery op cit). Parts of Africa where this type of furnace has been documented include Nigeria, Ghana, Togo, Cameroon, the Congo Basin, Zambia, Southern Tanzania, Malawi and the Horn of Africa (Van der Merwe op cit).

In the borderlands of northern Nigeria and Cameroon are found a special unique type of forced draft furnace (David *et al* 1989:189-191; Sassoon 1964:215; Tylecote 1965:345). The furnace is cylindrical in shape and is built of local clay on a natural bank. The forced draft is supplied by a bellowsman seated on top of the bank behind the furnace. A thin wall guard about a metre high is built from the top of the bank to shield the bellowsman from the heat from the furnace. The draft from the bellow is forced into the furnace through a large long tuyere that occupies the centre of the furnace. This particular tuyere device has the effect of delivering air to the centre of the fuel bed and ensures even combustion. It further saves labour since only one bellowsman was needed at a time to operate the bellows from the top. Tylecote and Sassoon feel that some level of preheating of air in the tuyere is possible with this device.

With these distinctive achievements and sophistications, it is puzzling that iron smelting did not survive and develop to modern blast furnace technique or into any other form in Africa. Hypotheses and explanations have been developed to explain this anomaly. The earliest explanation on the reason for the decline of African iron smelting centred around trade models whereby imported European metal goods were seen as the sole cause of the decline of African iron working (Flint 1974:387; Williams 1974:69, 72, 86). This hypothesis is deficient in explaining this phenomenon since dates from iron smelting sites in parts of Africa show that iron smelting continued even till the middle of this century. For example, in Babungo and Bamenga area of the Grassfields of Cameroon, large scale iron smelting continued into the late 1940s (Warnier *et al.* 1979:338). In Mafa northern Cameroon, bloomery iron smelting continued into the late 1950s (David *et al.* 1989:86). The Bandjeli of Bassar in northern Togo continued bloomery iron smelting till 1951 (de Barros 1988:96). Six carbon-14 dates processed for the present research show that the late phase of Nsukka iron smelting continued up to 1950 cal. AD. Details of this are in Chapters five and six. There are instances too where the imported metal goods were rejected as inferior (Sassoon 1964:174) and some smelters after many years of abandoning the trade reverted to it despite the availability of imported European metal goods (Sassoon ibid).

In a bid to overcome this deficiency in the trade model, a model centred on ecological and environmental implications of iron smelting was developed. Goucher (1981) and Haaland (1985) attribute the demise of the industry more to the deforestation effects of the industry which made huge demands on hardwoods, which are difficult to regenerate. This as Haaland noted aided environmental deterioration through erosion and eventual shift in settlements.

Pole (1982) argues that iron smelting, particularly the way it was organised in Africa was labour intensive. The ultimate result was that eventually the cost of local produced metal was far higher than the imported one. Okafor (1984a:66-67) considers the ecological model an adequate explanation for the demise of iron smelting if it is combined with the long term effects on the industry of its high labour costs. It is known that by the early decades of this century internal slave trade, such as the raids for slaves on Bassar iron smelters by the Tykossi and Dagomba (de Barros 1988), and the insecurity created by inter-ethnic wars, such as the Igbo-Igala wars of the 18th-19th centuries AD (Oguagha 1983:58, Oguagha and Okpoko 1984) made the procurement of the labour necessary for iron smelting difficult. The shortage of the vital fuel and the uncertainty of the supply of labour to help in the smelting, and the ready alternative provided by European imported iron caused the decline of iron smelting in parts of Africa.

1:5 Past Works On Iron Smelting Slags.

The least explored aspect of African iron working is iron smelting residues. The apparent lack of interest in this aspect of the African Iron Age result from a general ignorance of types of the information that can be obtained from such materials and the dearth of technical resources needed for this type of investigation in many parts of Africa. Some people however, have tried, imperfections in their works not withstanding, to incorporate residue analysis in their reconstruction of African iron working.

In West Africa, the earliest published attempt on residue analysis was that by Tylecote (1975a) which gave bulk analysis of a slag each of Taruga and Meroe iron working residues plus four other analyses of ores from both sites. This publication however, gave only the oxide constituents of the samples and nothing is known of the mineralogical phases of these samples. Again the number of slags used in this study was very small and so the results are inadequate for comparative studies. From sites in Igboland, Okafor (1984) analysed four slags. But he sought only the major elements in the samples and lumped minor and trace elements together as 'others'. Nothing is known about their mineralogical phases, their free flowing temperature, density and viscosity. These deficiencies make the work less useful for any comparative studies.

From Togo, Goucher (1983) published the summary of analysis of 15*th* century Bassar smelting slags. Although details of the oxide constituents were not published he was able to identify major phases in these slags as wustite, fayalite and glass. From the Ivory Coast Zacharia and Bachmann (1983) made an analysis of slags from the Senufo. Various mineralogical phases in the slags as well as their oxide constituents were identified. Major phases were identified as fayalite, wustite, spinel, hercynite and magnetite. By calculating the basic/acidic oxides ratios in the samples they were able to discover their viscosity which were found to be very low and their free flowing temperature which ranged from 1160°C to 1190°C.

Works by Todd (op cit) and Todd and Charles (op cit) among the iron working people of Dimi in Ethiopia remain to date the most detailed residue study on Afrcan iron working. Todd analysed ores, slags and metal specimens from Dimi and compared the results with results of analysis of some smelting residues received from Hani, Brong Ahafo in Ghana and Central and Western Sudan. From these studies they discovered that it is hard to do provenance studies from slag analysis using trace elements since trace elements are affected by operating furnace conditions. Intead they recommended the use of major and minor non metallic inclusions in the artefacts for provenance studies. From the comparative studies of analysed specimens from various parts of Africa, they discovered further that most of the smelting slags have a high flowing temperature around 1400°C.

In South Africa, Van der Merwe and Killick (1979) analysed smelting residues from Square near Phalaborwa and from comparative studies of titanium/calcium and titanium/iron ratios in the slags and ores, discovered the sources of ores used during early iron smelting at Square. Friede *et al.* (1982) made analyses of iron smelting slags from South Africa, Swaziland and Botswana. This is the most detailed work from Southern Africa which provided not only the oxide constituents of the slags but also determined their free flowing temperature and mineralogical phases. Other residue analysis conducted on slags from iron smelting sites in Magaliesberg area (Friede 1977) and Melville Koppies nature Reserve Johannesburg are of very limited value, since they only gave oxide constituents of the specimens.

In Europe, unlike in Africa, a great deal of work has been done on residues from early iron working sites. Fells (1983) gave a good review of these published residue studies up to 1983. Among these works are those of Bachmann (1982), Tylecote (1962:252-255, 1987:291-324), Coghlan (1977:9-10) McDonnell (1983, 1986), Morton and Wingrove (1969a, 1969b, 1972), Reimann and Sperl (1979) and Sperl (1980) which described various types of slags and various approaches and techniques that can be used in their study as well as different types of information one can derive from them. Among the studies conducted on slags from specific iron working sites, the ones by Morton and Wingrove (1969b,1972) on slags from Sharpleypool and Worcester, Muncaster, West Runton, Harthope Mill, Rushall, Bourne Pool and Nun's Well have become reference works for most people that delve into this aspect of iron working. Morton and Wingrove (op cit) laid bare the wealth of information that can be derived from slag analysis which include the reconstruction of technological changes within any metallurgical technocomplex and the operating temperature of the furnace.

However, their adoption and recommendation of anorthite-SiO_2-FeO ternary phase diagram as the appropriate phase diagram for bloomery slags has been found to be inapplicable to slags used in the present study. Details of this are in Chapter Six. Other people (Fells op cit :80; Kresten 1986:43; Todd and Charles op cit:72) have found out that anorthite-SiO_2-FeO ternary is not of universal application to bloomery slags. Results of analysis of slags from Wealden iron smelting site and Wilderspool and Stockton Heath near Warrington published by Strakey(1931) and May (1904) respectively are of limited value since they analysed only the major and minor oxides and nothing is known about their mineralogical phases. However, Allen's (1988) analysis of Romano British bloomery slags from the Wetlands of the Severn estuary not only identified compositional variations within the slag samples but used statistical methods to confirm his findings.

In recent years slags from Early Iron Age sites have formed research topics in some universities. In 1976 Todd (op cit) obtained a doctoral degree from Cambridge University after research which included analysis and study of bloomery slags. Her work highlighted the usefulness of major and minor non-metallic inclusions found in artefacts in provenance studies. Fells (op cit) obtained a doctoral degree in 1983 after her research on the structure and constitution of archaeological ferrous process slag. This work showed that the mineralogy of iron smelting slags is not dependent on furnace types, or age or intensity of industrial operations but on operating complex conditions in the furnace. Since smelting affects the concentration of the trace and minor elements in the artefacts, she recommends the use of non partitioning oxides Al_2O_3 , MnO, V_2O_5, TiO_2 and MgO for provenance studies. McDonnell (op cit) in 1986 obtained a doctoral degree from Aston University after completing a study on slag classification. He discovered that little distinction exists between smelting and smithing slags and that any distinction that does exist can be observed morphologically since from physical analysis the only difference observed between the two types of slags is the relatively higher percentage in smelting slags of manganese of more than O.5%.

1:6 Methods Used In This Study:

Four iron smelting sites in Nsukka Division Opi, Orba, Owerre-Elu and Umundu were isolated for this study (see Fig. 1:2). Descriptions of these sites are in Chapter three. They were chosen (1) due to the volume and extent of residues found in them (2) due to the variety of these residues and (3) because the four sites embody various features observed in other iron smelting sites in Igboland. For example, four furnace types were thought to have been used for iron smelting within the zone; pit furnace, shaft furnace without slag tapping facilities with internal wooden frame, shaft furnace without slag tapping facilities made of only clay and shaft furnace with slag tapping facilities. Opi iron smelting was thought to have been conducted in a pit furnace, while Owerre-Elu iron smelting was in a shaft furnace with slag tapping facilities. Orba smelters conducted their smelting in a shaft furnace with internal wooden frame and the slag was not tapped; while Umundu smelting was conducted in self-draft shaft furnace without slag tapping facilities.

Pit furnace technique was also thought to have been used at the remaining iron smelting site in a neighbouring town, Leja (Anozie 1979:127). The process of iron smelting at Leja is presently being studied by Anozie. However, five slag samples from that site were analysed during the present research for comparative reasons. The results of the analyses of these Leja slags are in ~~a~~Appendix 1.

Each of the four chosen sites was surveyed in February 1990 and their dimensions determined. For sample collection each of the sites was divided into residue cluster areas according to the spread of the residues. The number of samples collected from each cluster area depends on the diversity or homogeneity and volume of residues in each area and their location within the sampled area. Sampling sections opened in some cluster areas were geared towards collecting samples for radiometric dating and refractory clays for mineralogical analysis.

Because ores found in smelting sites are often treated or rejected ores (Friede *et al*· 1982:42; Fells 1983:71,162; McDonnell 1986:56; Tylecote 1975a:7), and do not give correct information about the actual ores smelted, ore samples used in this research were collected from various ancient ore pits and locations discovered through ethnographic and oral interviews. Descriptions of the samples analysed in this study and the results obtained from them are in chapter five.

Slag samples were analysed morphologically and physically. Presently the widely used analytical technique in slag study is scanning electron microscopy. But because the amount of slag from each sample analysed with scanning electron microscope, the major technique used in this study, is so small, $15mm^2$ and 3mm thick compared to the volume of slag discovered, attention was paid to the morphological study of the entire slag to determine their coverage, colour, texture, inclusions, magnetic levels, rust and porosity. Many people like Argent (personal communication 1989), Bachmann (1982), Claugher (1988), Freestone (1985), Olsen (1988), Parker (1986), Knudson (1978) and Tite (1972) have studied the usefulness and the reliability of the results obtained from slag study using S.E.M and found that the results were highly reliable and that the technique has many other points to its credit. They have thus recommended it to people interested in slag analysis.

This recommendation has been taken by many people involved in slag study including Goucher (1983), McDonnell (1986), Todd (1976) and Todd and Charles (op cit). They found that S.E.M with attached energy dispersive x-ray system (EDS) is a very useful tool for rapid analysis for composition greater than O.1 wt %. The technique not only analyses the elemental constituents of the slag samples but elucidates individual mineral phases of the sample which are also analysed. The use of S.E.M in slag analysis also reduces the discrepancies that exist between bulk and phase analyses since these two analyses are executed under the same conditions and on the same specimen. The bulk analysis results were obtained by making raster scan of the electron beam over the sample at magnification of X2OO covering approximately an area of about O.25mm. The phase analyses were obtained at the same magnification using the spot mode of the S.E.M.

Todd (1976:73) observed that the S.E.M energy dispersive x-ray system has the following advantages (1) simultaneous detection and recording of x-ray spectrum (2) display of both peaks and back ground intensities for most major elements (3) rapid visual display of the spectrum (4) high collection efficiency of the results (5) less sensitive to x-ray source position than wavelength spectrometers (6) sensitive to a larger x-ray energy range (1 to 50 kev) (7) variable shape and size of the detector and (8) no moving part or alignment of the sample.

Another advantage of the S.E.M technique is that samples analysed by it require minimum preparation. The samples are polished scratch-free and made conductive by carbon and silver dag coating to eliminate any error due to charging or heating of the sample surface by electron beam (Claugher 1988; Olsen 1988). Details of the various techniques used in preparing the samples are in section 5:3. Furthermore the S.E.M technique is non destructive of the samples. It does not require much grinding or much chemical treatment of the sample which means that one sample can be analysed with some other technique if desired after it had been analysed with S.E.M. Again S.E.M has the advantage of giving two types of images in its photomicrograph. In a study where the aim is to discover the surface and the morphological features of the sample, S.E.M secondary electron images are produced and these result from electrons knocked off the surface of the sample by an incident electron beam. These secondary electrons have low energies and are used to show the relief or morphology of samples in shades of grey grading into one another without sharp boundaries (Brothwell 1969; Freestone 1985).

Secondary electron images have been mostly employed in the study of artefact morphology and surface features such as microwear on stone tools (d'Errico 1988) and tooth wear (Hillson 1988). In studies such as the present one, where the interest is to discover the composition of the samples, the backscatter images of the S.E.M are produced. The backscatter images show the compositional character of the samples in terms of different phases, (composite chemical compounds in the samples). On the whole, whether for secondary images or backscatter image, the images obtained by S.E.M are much easier to interpret than those from transmission microscope, so that it is often possible to obtain the desired results from an examination of an image as it is formed on the viewing screen (Brandes 1983:10-68).

A raster scan with the electron beam across the sample surface gives the elemental constituents of the sample and where desired each of the phases are analysed to determine their elemental composition. Backscatter images of S.E.M are usually identified by sharp changes in grey level. Heavy elements such as iron and lead appear brighter in S.E.M backscatter images (Freestone op cit).

Besides images produced by S.E.M it produces x-ray spectra of various constituent elements in the sample. Chemical elements in a sample produce x-ray spectra of a unique series of energies. Samples composed of many elements produce x-rays of each in proportion to the relative amount of the elements present. These X-ray spectra are displayed on a TV screen and they give qualitative indication of the composition of the sample, that is, the elements present in high or low concentrations.

In the operation of the S.E.M, a narrow beam of electrons is generated by heating a wire filament to high temperature under a strong electrical potential. This beam passes through a cylindrical 'column' around which is a series of electromagnetic 'lenses' which allow the beam to be focused and moved. At the base of the column, the electrons hit the sample and generate a number of different types of signals. This signal from the sample is converted electronically to provide an image of the sample on a TV screen (Brothwell 1969).

The S.E.M housed at the department of Material Science of the University of Sheffield was used in the analysis of the samples used in this study.

This Camscan, model 2, has a computer Link System which makes it possible for x-rays and the results of the analysis to be displayed on a TV screen and to produce a print out of the results. This 'Link' Energy Dispersive x-ray microanalyser enables qualitative analysis (analysis using only the number of x-ray counts) and quantitative analysis (with ZAF. 4 atomic number absorptions and fluorescence correction) to be carried out. The ZAF. 4 analytical package strips the background before peaks fitting. Cobalt was used as reference to define a count rate for all the analysis and oxides as standards for the quantitative analysis. *

Because of the inhomogeneity and variability that characterise slags and the unrepresentativeness of a single bulk raster analysis (McDonnell op cit:224), at least three raster analyses were made on each sample (see tables 1-56 in Appendix 1) and the mean of each of the detected oxides calculated (see tables 57- 61 in Appendix 1). From the mean values, the free flowing temperature of each slag sample was obtained by normalising the highest analysed oxides to 100% after Morton and Wingrove (1969a) and Bachmann (1982). The results were then plotted on the appropriate phase diagram. The viscosity of each slag sample was estimated using the formula developed by Bachmann (1982) which involves the determination of the viscosity coefficient of the slag through calculation of basic/acidic oxide ratios using the mean values of the raster results (Zacharia and Bachmann 1983).

Various mineral phases observed in each sample were analysed and their proportions calculated using the Digipad program of the Link-System of the Camscan. For a check on the results obtained from the S.E.M various mineral textures observed by the S.E.M were verified using a metallurgical microscope and the findings confirm the results obtained from the S.E.M. Another check on the technique is that most of the samples after analysing in the S.E.M were analysed using x-ray diffraction technique especially for comfirmation of the mineral phases observed in the samples. In each case similar results as obtained with the S.E.M were arrived at. Details of these are in Chapters five and six.

* The level of O_2 in the samples was obtained by stoichiometry and then normalised to 100%. The counting time was 30 seconds with a count rate of about 2500 to 3000 counts per second. The standards used were pure alumina, pure silica and a synthetic wustite. The ZAF. 4 program produced normalised data to 3 decimal places and these were reported to 2 decimal places in the tables. The error on any elemental analysis will be of the order of 5% of element and no arguments are made that assume an accuracy greater than this. The micrographs presented in this thesis were all obtained by the use of a back scattered electron detector.

CHAPTER 2

NSUKKA AND ITS ENVIRONMENT

2:1 LOCATION:

Since an understanding of man's environment provides one of the keys to his history (Shaw 1963:4), and realising that for a better understanding of technological and cultural achievements of a people a knowledge of the people's environment is vital, in this chapter the survey of geology, climate and geography of Nsukka Division is set out.

Nsukka Division, located on the Anambra River Basin of the former Eastern Nigeria is on the Nigerian survey map sheet 287, located to the northeast between latitude 6°52" E and longitude 7°32" N. It is bounded northwards by both Adoru and Orokam respectively both in Benue state, southwards by Umulumgbe, westwards by the River Niger and eastwards by Nkalagu (see fig. 2:1). With the inception of a local government system in Nigeria in 1967, the Division was carved into six different local government areas namely Igbo-Eze, Igbo-Eze south, Igbo-etiti, Isi-uzu, Nsukka and Uzo-uwani local government areas. The Division has common geomorphological features, climate and vegetation (Ofomata 1972).

2:2 Geology of Nsukka Division

Nsukka Division is on Nsukka-Udi-Okigwe Cuesta and so stands on Similar geological formation. Geologically the Division forms part of the lower Benue trough filled with cretaceous sediments, (Hoque 1976:516; Ofomata 1975:8; Umeji 1980:110). This older sequence of Albian to Turonian age was folded in the Santonian times (Nwachukwu 1972) and was later overlain by seawards dipping shallow marine paralic and fluviatile sediments. Eze (1985), Ofomata (1967), Simpson (1954) and Umeji (op. cit) have recognised four major stratigraphic formations in the area that are resistant to erosion and which make impressive landforms. These formations are Nsukka Formation also known as

Upper Coal Measures, Ajali Formation or Ajali Sandstone, Mamu Formation or Lower Coal Measures and Nkporo Shale also known as Asata Shale (Umeji 198O:111). All these formations dip gently at 2 to 8° west and southwest. Figure 2:2 shows the general geology of Nsukka Division.

The Nsukka Formation, which is the youngest, is a paralic sequence of mudstone and shale. It contains also lenses of coal often up to 3.5m thick. This formation outcrops over dip slopes and also as relict Mesas and outliers underlain by Ajali Sandstone (ibid). de Swardt and Casey (1963:7) observed that in Nsukka Division, the Nsukka Formation is nearly horizontal or dips at a low angle to the west and south.

The Ajali Sandstone which underlies much of the Division is a coarse friable cross-bedded sandstone, which is characterised by absence of feldspar and contains predominantly monocrystalline quartz grains. This, according to Hoque (1976:519), demonstrates the maturity and long transportation of the marine sediments. The sandstone is composed of 9O% subangular quartz fragments and less than 1O% of clay (Hoque and Ezepue 1977). Most of this formation is unconsolidated and poorly sorted sandstone, white to pale grey in colour, but sometimes stained red yellow or brown by iron oxides (de Swardt and Casey op. cit.). Beds of white shale occur at intervals, and become more numerous towards the base, where there is a gradual transition into the underlying Mamu Formation (ibid). This formation has a thick lateritic capping (Ofomata 1967) and is cut by numerous gullies.

Underlying the Ajali Formation is another coal bearing paralic sequence, mostly carbonaceous, the Mamu Formation (Umeji op. cit), which Ofomata (op.cit.) called the Lower Coal Measures. Simpson (1954) observed that most of Nigeria's coal seams are found within the Mamu Formation. In Nsukka Division, at Ezimo, several coal seams are observed within this formation along Iyi-nzu stream valley (Ezeugwu 1986). Nwachukwu (1972) in his study of tectonic evolution of the southern part of the Benue trough, which includes Nsukka Division, noted that a slight tectonic movement probably took place in the area during the Cenomanian. This might have been responsible for the submergence of the forest whose vegetation yielded the hydrocarbon.

Below the Mamu Formation is the Asata shale or Nkporo shale. This is the oldest unit and forms the base of the sequence. This Formation has been subdivided into three units-the carbonaceous, the grey and sandy shale (Ezeugwu op.cit.). These are greyish dark, soft, bluish and laminated alternating with mudstone and silt.

The main rock types in Nsukka Division are sandstones, sandy shales and carbonaceous shales. These are observed as one moves from the core Nsukka area through Eha-ndi-Agu to Ezimo, Imilike-Agu and Ogbodu-Aba (Eze 1985). de Swardt and Casey (op.cit:7) observed that at Nsukka, "there is a persistent basal sandstone horizon 3O to 7Oft (9.14 to 21.33m) thick and this is succeeded by a great thickness of dark shales with many sandstone beds". Gritty and pebbly bands are locally present in the sandstone, and the shales contain many concretions, bands and stringers of marcasite and siderite.

Other rock types in the Division are precambrian, which are mostly metamorphic in origin. However, a few old volcanic rocks like magnetite and quartzite are also present. But the majority of rock outcrops in the area are haematite rocks and this probably was one of the attractions for the development of early iron smelting in the area (Onyeke 1986). Large lumps of haematite are exposed at gravel ground surfaces, valley slopes and hills (see plate 2:1) and presently they are mined for other purposes such as building and road construction projects.

2:3. Landforms and drainages in Nsukka Division:

2:3:1 Landforms In Nsukka Division:

Nsukka Division consists of heterogeneous land forms with varying elevations. Ofomata (1972:3) observed that 4O% of its' land area of 3,4O6sq km is low land of less than 122m above sea level, 25% consisting of land lying between 122m and 244m above sea level while 2O% of the land area lies between 244m and 366m and the remaining 15% of the area is above 366m above sea level.

However the dominant land form in the area is north south trending Enugu escarpment which runs from Enugu through Ukehe, Opi, Ehalumona to Orokam in Benue state. From Opi the escarpment branched off through Edeoballa and Nsukka to Adoru and Ogboyega also in Benue state. This escarpment which is characterised by steep troughs in these places rises from about 1OO to 3OOm above sea level. In some places in Nsukka area the escarpment is capped by ferruginized Ajali sandstone paticularly at Ugwu

Ikwube in Ehandiagu and lateritic materials as in Obimo (Eze 1985; Ofomata,1967).

Three planation surfaces, assumed to be products of three erosion cycles (Umeji op.cit) have been recognised in Nsukka area and these are characterised by lateritic capping on the Nsukka-Udi-Arochukwu cuesta (ibid). The local names and ranges of these surfaces are (1) the Ukehe surface, 55O to 60Om (2) the Nsukka plateau surface, 43O to 46Om and (3) the Okigwe surface 27O to 30Om (see fig. 2:3).

The Ukehe surface occurs as hills and ridges characterised by flat tops and lateritized to a great depth over 15m. The surface is formed on the Nsukka Formation and are eroded remnants of the oldest surface on the escarpment. The Ukehe peak with an elevation of over 59Om rising about 14Om above the plateau below it is the highest and the best developed land form in Nsukka area (Umeji op.cit).

Nsukka plateau surface is the best documented land form in the area (Ofomata 1967). The southern extension of this plateau to Udi has been known as Udi plateau (Grove 1951). This plateau wherever it has been recognised is flat at 43 to 36Om or gently slopping west and south west (Umeji op. cit) and is bounded by steep slopes below. The greater part of this plateau is formed over Ajali sand stone but the Mesas are formed on Nsukka Formation. There is a great similarity in the degree of lateritization between Nsukka plateau surface and Ukehe surface. Lateritic crust and ferruginous soil reach depths of of 1O to 15m. And deposits of massive and rubbly or oolitic iron stone, 3O to 5O% iron occur at a few places about 3m below this surface (ibid:113).

The lowermost planation surface in Nsukka area, the Okigwe surface, has an altitude of 27O to 30Om. Few isolated hills within this range of elevation have preserved this surface. A thin 3 to 5m lateritic cover, mainly pisolitic is characteristic of this surface. Canyons and gullies with vertical walls 10Om high and flat floors have developed during the erosion of this surface.

This Nsukka-Udi-Arochukwu cuesta, (see fig. 2:4) on which these surfaces stand has an east facing escarpment rising between 2OO to 35Om above the Cross River plain. Its dip is south-wards about 44Om long, extending from the Benue valley to Arochukwu in Cross River plain and 8Okm across from Idah to Okaba and narrowing to about 4Okm in the vicinity of Nsukka (Umeji 198O:11O). This cuesta as we shall see below is the principal determinant of the drainage system in Nsukka area where it serves as water shed to rivers and streams.

Other prominent land forms in Nsukka Division are flat-bottomed dry valleys (Ofomata 1967) and U-shaped dry valleys (Ofomata 198O). The presence of these dry valleys had attracted the attention of some workers in the area. They were studied and commented upon by Ezeugwu (1986), Grove (1951), Jungerius (1964), Ofomata (1965a, 1965b, 1972, 198O) and Umeji (198O). It is generally thought that the dry valleys were formerly normal river valleys, developed on the upper coal measures but as these formations become eliminated, the rivers came incontact with the underlying highly porous Ajali sandstone and got infiltrated into the the latter, thereby leaving their valleys dry. Even as recent as 198O, it was observed that Nwokpa stream at Ibegwa dried up leaving a dry channel (Eze op. cit).

It is possible that the valleys evolved during climatic variations that prevailed during inter pluvial periods in Africa (Jungerius op cit). These dry phases of alternating wet and dry conditions must have persisted, as in fact it is felt, that the present holocene climatic conditions are relatively drier than in the past as to perpetuate the dry valleys (Ofomata 1967:8). Again the great width of these dry valleys, such as the one that runs from Ibuku Ekwegbe to Agu-Ekwegbe, would point to their formation under wetter conditions than is the case at present (Jungerius op ct:17O). The formation of these dry valleys would therefore, indicate that the water-table has continued to fall, which implies also that Nsukka is becoming drier.

It is also possible that the dry valleys resulted from local or regional uplifts, a situation that characterise the latter part of the pleistocene period in some parts of West Africa (Orajaka 1975:5). For instance, from Senegal to Mauritania, four such uplifts have been identified. However most of these valleys show no evidence of rejuvenation, as would be the case if such uplift were important. This however, does not deny the possibility of such local or regional uplifts only that there is not much in the form of evidence from Nsukka Division to support this hypothesis.

Ofomata (1967:8) suggests a possibility whereby the dry valleys resulted from a general denudation and progressive migration of the water-table following gulleying and ravination in areas beyond the immediate confines of Nsukka Division. The dry valleys nevertheless still function but periodically, mostly during the rains when flooded by rain water. Some of these valleys become marshy during the rains as in the case of the dry valley to the north of Ibagwa-Aka (Ofomata 1965b)

2:3:2 Drainage Systems In Nsukka Division:

The Ajali sandstones which underly most of the Division are highly permeable and hence facilitate the infiltration of rain water to great depths (Ofomata 1967:5). Most of the central areas of the Division are served by springs from perched aquifers on the slopes of the water-bearing Nsukka Formation at between 336 and 397m (Ofomata 1972:3). The water table is not very far below the surface, a situation which favours the sinking of wells in many areas of Nsukka Division.

As noted above the principal dominant drainage system in Nsukka Division is the Nsukka-Udi-Arochukwu cuesta (see fig. 2:4). This central highland of Nsukka Division forms the water shed for some upper course tributaries of the Niger and the Cross Rivers. These tributaries emanate from the scarp and some feed the Cross River in the east while others flow-west and southwards on the dip slopes as tributaries of both the Niger and Imo Rivers. Generally rivers and streams found in the Division are in the peripheral zones like Obollo, IKem and Eha-Ndi-Agu, where such streams like the Igbogbo, Ebonyi, Iyi-nzu, Amanyi-Orba and Uhere are found. And to the west of Nsukka only the Adada River at Nkpologu and Obina, a tributary of the Anambra, and itself a tributary of the Niger can be found.

The east flowing streams take their rise at about 366m and 274.5m, while most of the western section of the area lying above 244m carries no substantial surface drainage. This according to Ofomata (1972:3) is because the 366m contour line coincides reasonably well with the contact between the porous Ajali sandstones and the more impermeable Mamu Formation and thus constitutes a spring line, while the 274.5m contour line coincides with the contact between the Mamu Formation and the highly impermeable Nkporo shales, thereby forming another spring line at the foot of the escarpment. This accounts for the presence of more rivers and springs in the Isi-Uzu area of Nsukka, stretching from Amalla through Ikem, Eha-Ndi-Agu to Opi Agu. On the other hand, the western section of the Division is underlain by the Ajali sandstones which are highly permeable and thus facilitate infiltration of rain water.

2:4 Soils In Nsukka Division:

Soils in Nsukka Division derive from weathered materials from the underlying geological formations which have weathered to great depths to produce a thick mantle of weathered materials over which soils have developed. The presence of rain water which infiltrates the soils together with high temperature are responsible for weathering to great depths of these formations. Weathering in the zone also leads to precipitation of iron oxides, the accumulation of which leads to the formation of highly resistant iron stone concretions found throughout the Division (Ofomata 1967; Umeji 198O). These iron stones, mainly haematite (Njoku 1986) are found eroding from soils on slopes of dry valleys (Ofomata 1967, 198O) on the surface of residual hills and outcrops (Ofomata 1967; Umeji op cit). The presence of these haematites at easily accessible levels according to Njoku (1986) and Anozie (1979) was the major reason for the development of iron smelting early in the area.

Jungerius (1964) observed that the most important soils in the area consist of a deep red structureless and porous non gravelly coarse sand loam, with a loose reddish brown sandy top soil. Lower slope profiles show eluviation of clay into the subsoil, giving a reddish brown sandy surface soil over a red sandy clayey subsoil. Other prominent soils in the area are black or grey clays, which may be calcareous or non-calcareous. These black soils, sticky when wet and cracking deeply when dry are hydromorphic in character.

The minerals of the parent rocks have almost completely changed so that the soils consist of quartz fragments of sand, hydrous aluminium silicates and aluminium hydroxides of clay or clay-like substances and hydrated iron oxides giving red or red-brown colours to the soil (Hoque and Ezepue 1977; Onyeke 1986).

Soil characteristics of Nsukka Division are generally similar to those defined for red ferralitic soil (Ofomata 1972). However sectional variations exist. For example, areas on the Cross River plain, mostly parts of Isi-Uzo influenced by seasonal waterlogging have hydromorphic soils, and the impervious Imo-shale of the Anambra lowlands, occupied by the Uzo-Uwani people, give rise to brown hydromorphic mottled clay soils (Ofomata 1972:4). Whereas, in the central Nsukka plateau underlain by porous unconsolidated Ajali sandstone, the soil is sandy and coarse-grained and thus favours the easy infiltration of water to great depths.

As will be seen below, because the vegetation cover on these soils is very light and the seasonal rainfall very torrential, most soils in the Division are very much prone to erosion. Apart from the adverse effects of rainfall on these soils, farming methods in vogue in the area add to fast leaching of these sandy soils. Thus Inyang (1972:5) observed that soil deterioration has become "the cancer" of Nsukka Division.

2:5 Vegetation Of Nsukka Division

Although Menakaya and Floyd's (1972) map of West Africa included Nsukka Division within the Guinea Savannah; Nsukka was part of the tropical ever green forest that lies intermediate between the humid tropical rain forest and the slightly arid sudan savannah vegetation of the north. Before the area was inhabited by man, it was covered by high rain forest (Okigbo 198O) and the former northern limit of this type of vegetation in Nigeria is marked by a line joining all stations with "at least 5Oinches (1270mm) mean annual rainfall and lowest mean monthly humidity at 9 a.m of 7O%" (Uzoezie 1972:1). Nsukka Division with over 6Oinches (1524mm) and 75% of these climatic parameters lies south of this line (Inyang 1972).

Nsukka has a humid tropical climate best described as a tropical wet and dry climate (Ofomata 1972). From a moisture and temperature stand point, Nsukka Division is on the fringe of a monsoon climate and the wettest part of a savannah environment. This shows that it can support and has indeed supported a tropical rain forest which now is extant (Okigbo 198O). Man through his many sided activities destroyed this tropical forest and thus Nsukka Division today is regarded as being in "derived savannah" (Inyang op cit:2; Ofomata 1972:3; Udo 198O).

Ezeugwu (1986:14,16) in an attempt to explain this change in vegetation observed that Nsukka communities have been practising agriculture from the beginning using the slash and burn system and that the change in the vegetation was a direct consequence of the system and the intensity of agriculture practised by Nsukka communities.

A hypothesis of how human activities transformed this forest environment to savannah was formulated by Uzoezie (1972). He suggests that the forests on the Nsukka plateau were probably more open than their counter parts on the lowlands to the east and west. This factor combined with the relative ease of tilling the sandy soils made them very attractive to Igbo settlers. As their economy was based on yam cultivation (Okigbo 1980; Shaw 197O), which as a rule must be staked (Oguagha and Okpoko 1984), the forest provided the stakes

Initially the settlers practised forest fallow cultivation in which part of the forest was cleared, cropped for a few years then abandoned for sometime, probably ten to fifteen years, as yields declined. Since the area experience frequent drought (Inyang op cit:2), secondary forest were slow to grow and could not attain a climax stage before they were cleared again. More frequent cultivation following increases in population and the use of fire for clearing and hunting (Onyeke 1986; Reeves 1936) resulted in the replacement of the secondary forest by bush in which some economic trees were spared.

The system of land use thus changed from secondary forest fallowing to bush fallowing with a shorter priod of rest of five to ten years. With further increases in population and more frequent cropping, bush fallowing gave way to grass fallowing or short fallow cultivation in which the land rests for one or two years after use and grasses rather than shrubs and small trees form the vegetal cover.

Apart from agriculture, it is known that many Nsukka communities were deeply involved in iron working (Anozie 1979; Njoku 1986; Okafor 1984) and most iron producing areas of West Africa are today in regions considered by some people to be "derived savannah" (Goucher 1981; Ofomata 1965; Udo 198O), that is, a type of savannah created by human interaction. Of most ecological interest here is that the type of trees suitable for the production of fuel used in iron working are the ones that are very slow in regenerating. For example, the replacement of *ukpaka* **(pentecleth̲ea macrophylla)** and *okpenye* **(Ricinus Communis)**, used mostly for fuel production by Nsukka iron workers requires more than twenty years of growth (Njoku op cit).

Okafor (1984a:67) noted that the shortage of these two tree species in the Nsukka area was a major factor in the decline and eventual demise of the industry in the area. There is no way thousands of trees harvested by a smelter could be regenerated in his life time (Richards 1973:65). Added to the fuel needs of iron workers, is the wanton felling of trees for fire wood and other purposes which together make it impossible for true forest to regenerate.

Since the evolution of this savannah type of vegetation, a combination of climatic and human factors has prevented the regeneration of true forest vegetation, thus Nsukka Division today lies within the forest savannah mosaic portion of the former Eastern Nigeria (see fig. 2:5). Only fire resistant trees and shrubs survive the annual bush burning (Reeves op cit) which characterise agricultural activities in the area. The Division is thus predominantly covered by grass with patches of woodlands. Common grasses present in the area include *Ikpo* **(Loudetia Arundinacea)**, *Owa* **(Andropogon Tectorum)**, *Uma* **(Maranthacloa Cuspidata)**, and *Ata* **(Imperata Cylibndrica)**. These mix with tall trees and shrubs. The former include *Orji* **(Chlorophora Excelsia)** *Ofo* **(Deutarium Macrocarpa)** and hard woods *Ukpaka* **(Penteclethea Macrophylla)** and *Okpenye* **(Ricinus Comunis)**. These two latter ones as noted above provide iron workers in the Division with fuel for their industry. Other common trees in the area are *Ahaba* **(Acioa Barteri)**, used mostly for yam staking, *Ube* **(Diacryodes Edulis)**, *Ugiri* **(Irvignia Gabonensis)**, *Ukwa* **(Treculia Africana)**, *Oji* **(Cola nitida** and **cola accuminata)** and a gamut of other edible economic trees like oil palm trees **(Elaeis Guineensis)**.

There are however, some remnants of the pristine forest vegetation in the area, particularly in the area lying along the river valleys. Typical areas are Nimbo in Uzo-Uwani and Agamede in Isi- Uzo. In some other places they survive as groves used during ritual and cultural activities and are known locally as *Uham* or *Ikpogu.*

2:6 Climate Of Nsukka Division:

Nsukka Division located at the northern end of the rain forest zone experiences two major seasons annually, the wet and the dry seasons (Inyang 1972; Ofomata 1972). The wet season starts about the month of March when the south is under the influence of the rain bearing southwest monsoon winds and this lasts till October. About 89% of the annual rainfall of about 165Omm falls within this period (Inyang op cit). There is however, a brief interruption of a dry spell in August, locally called "August break". Monanu (1975:25) suggests that the "August break" is associated with an inversion in the tropical maritime air mass. Humidity, however, remains very high during this break. Rains in this Division, as noted before, usually fall in the form of torrential down-pours and this makes it possible for a large amount of water to be retained on the ground surface for a period of time, often throughout the duration of the rain (Ofomata 1972).

The pattern of rain fall, controlled by the movement of the sun, has not been consistent through the years (Oguagha and Okpoko op cit). In some years the rains or even the "August break" may be prolonged while in others the rains do not start when they are supposed to. These variations affect most economic activities in the Division especially farming. Inyang (OP cit:2) observed that in Nsukka Division rainfall "assumes significance in every phase of agricultural pursuit. It determines the timing of cultivation, planting and harvesting of crops of various sorts, application of fertilisers, selection of crop varieties and transplanting. It controls the operation of a number of occupations. For example, some people are migrant farmers in one season and casual labourers later".

The dry season sets in in October as a result of north-east trade winds originating from the Sahara and ends in March (Ofomata 1972). The hottest months of the year are February, March and April with the temperature dropping in December and January due to harmattan winds blowing from across the Sahara. Ofomata (1972:3) observes that the temperature in Nsukka Division is high. The Division has average mean daily minimum temperature of about 69.8°F (21°C) while its' average mean monthly maximum is about 75.5°F (24°C). Its average daily maximum is about 85°F (29°C) while the mean daily range varies from about 10 to 19°F (-12°C to -7°C) (Inyang op cit:1).

This wet and hot tropical climate is anti-archaeology. Most material remains, particularly organic remains, disappear at a fast rate in this climatic zone. Rarely do Archaeologists recover meaningful organic materials from their surveys. Only such highly weather resistant objects like stone, well fired clay and slags survive well within this area. Iron objects rust and disintegrate leaving brown or red stains on the soil.

2:7 Major Economic Activites In Nsukka Division:

Although the people of Nsukka Division are involved in many economic activities such as trading, wine tapping, iron working and hunting, their predominant occupation is farming which as noted above uses a fallowing system with slash and burn technique. According to the 1963 Nigeria population census, over 94% of the people of the Division were farmers. The nature of the soil, which is mostly sandy, clayey and loamy is encouraging to agriculture and the people avail themselves of the benefits of this resources. A very wide range of staple foods are produced in the Division but the most important are *Ji abana*

(Dioscorea alata), *Ji ocha* **(Dioscorea rotundata)**, *Ji oyogbe* **(Dioscorea cayenesis)**, *Ona* **(Dioscorea dumetorum)**, *Ede* **(Colocasis esculenta)**, *Akpu* **(Manihot esculenta)** *Oka* **(Zea mays)**, and *Ose* **(Capsicum annum)**. Rice is extensively cultivated only in the lowland areas of Isi-Uzo and Uzo-Uwani which are on hydromorphic soils. Palm products like palm oil and palm kernel are produced in great quantity and Nsukka communities are famous for their palm wine.

One outstanding feature of agricultural pursuits in the Division is the use of terracing on the residual hills that spread across the Division. This technique is widely practised around Aku, Leja, and Nkpologu and is generally thought to have been introduced by about 18th century by Igala conquerors who for strategic reasons settled on the hill tops and cultivated the immediate surroundings (Uzoezie 1972:8). But the fact that this technique is held to and practised up till now long after the Igala had withdrawn and peace restored is a pointer that the people realise that the hill wash sands of the lowlands are not as productive as the hill sides covered with materials of recent weathering. This is probably why the villagers have tended to grow the more important crops like yams, maize and cassava on the hill side using terraces in spite of the higher labour cost (ibid.)

Animal husbandry and poultry keeping are not neglected. Animals that are resistant to insect borne diseases are kept at home. These include dwarf horned cows kept mostly by Orba, Opi, Ibeagwa and Igbo-Etiti communities. Sheep and goats are generally reared by most families. Some of these are left to roam about while some are permanently tied at home and properly fed with fodder and peels from cassava, yams, banana, plantain and other food stuffs. Fowls are the main birds kept at home. They are left to fend for themselves during the day but are provided with shelter at night. Dungs from these animals provide part of the manure the farmers use for their cultivation

Besides farming, hunting also occurs especially during the dry season when there is less work to do in their farms. Being in the savannah, herbivorous animals such as grass-cutters, and antelopes abound, and leopards, squirrels, wild cats, rodents like rabbits and mice are available and are hunted during the dry season. Hunting is usually organised with locally produced traps, matchets, spears and guns. At times group hunting is organised at village levels.

Nsukka Division in precolonial period was the nerve-centre of the intra-and inter-ethnic trade and exchange of southeastern Nigeria (Afigbo 1973a, 1977). There were at least two main trade routes from central Igboland that passed through Nsukka to Igala and Idoma (Afigbo 1977:126). One of these routes passed through important Igbo markets in Okigwe, Awgu, and Udi into Nsukka. From Bende another route skirting eastern Igboland ran through Afikpo, Uburu, Ezza and Nkalagu into Nsukka. Then from Nsukka these routes branched out into many directions and entered Igala and Idoma at several points. From Aku in Nsukka, a town of astute traders, one trade route went through Obimo and Okpuje to Adoru in Igala and from there moved up to Idah. From Obimo, a branch went through Nkpologu and Nimbo to Ogurugu where it joined the main Anambra route (Afigbo 1977)

According to tradition, the most important traders on these routes were the Nupe and Igala from the north; the Aro, Awka, Nri and Nsukka from the south. Among the numerous trade goods involved in this trade were slaves, ivory, cola nuts, livestock, textiles, metal tools, salt and imported European goods (Afigbo 1973a:78; Shaw 1970:285). The bronze horseman's hilt excavated by Shaw (ibid) at Igbo Richard suggests that horse was familiar to northern Igbo people by the 9th century AD. Major market centres in Nsukka where these goods were traded include Oye-Orba, Aho-Ezi (Opi), Nkwo-Ike (Ozalla), Orie-Uwellu (Aku) and Nkwo-Ibeagwa.

Metal working is another occupation in which many Nsukka people excel now and in antiquity (Afigbo 1973:84; Anozie 1979; Eze 1972; Njoku 1986; Okafor 1984a). Many iron smelting sites covered by heaps and aggregates of slags such as those at Opi, Leja, Orba, Umundu and Owerre-Elu are concrete evidence of their involvement with this industry (Anozie and Ray 1983, Okafor 1983, 1984a, 1988, 1989). Iron smelting is no longer practised in the area but many villages in Nsukka Division are still engaged in blacksmithing (see plate 2:2). Amalla Amube Orba (Okafor 1984a, 1989), Ehalumona, Eha-Ndi-Agu, Ede-Oballa (Njoku 1986; Onyeke 1986) and Umundu (Anozie 1979) are the communities where blacksmithing is still a buoyant industry. Products of this industry are mostly utility tools, farming and ritual implements (see plate 2:3). These include the *Aga* needle, *Okpupke Isi* razor, *Anyike* axe, *Aha* spear, *Mkpa* trap, *Mma* knives, *mgwu* digger, *Mkpu* weeding blade, *Nkugoro* sickle, *Ogu* hoe, *Aro* ceremonial staff, *Aba* ritual pin, *Ogene* gong, and *Odenigbo* giant gong.

CHAPTER 3

BLOOMERY IRON SMELTING IN NSUKKA

3:1 Introduction:

One of the main purposes of this research is to discover the process of bloomery iron smelting in Nsukka Division through the analysis of smelting residues, especially slags. Before commencing the slag study and analysis general information on the bloomery iron smelting which leads to the production of slags and bloom is deemed necessary. It is also considered necessary to give information on the sites- Orba, Opi, Umundu and Owerre-Elu which provided the samples for this study.

3:2 Bloomery Iron Smelting Process:

Although the earliest iron tools used by man may have been made from meteoritic iron (Trigger 1969:23; Tylecote 1980:209), the Iron Age proper did not start until man was able to extract iron from smelted iron ores. The use of meteorites as the source of iron for tools was very much limited in antiquity due to a number of factors which made it an unimportant item in early man's tool kit. It was difficult to find this type of iron in sufficient and suitable pieces for making tools. When found there were difficulties of forging the pieces into the desired tools (Tylecote 1986:8). In archaeological investigations, meteoritic iron is generally distinguished from the smelted iron by its relatively high (over 5%) nickel content (Tylecote 1987:99) and a total absence in it of the slag inclusions which are very common in smelted iron (Haldane 1970; Todd 1976).

Iron next to silica and alumina is the most abundant mineral in the earth's crust where it occurs in various forms. The most common oxides of iron are haematite (Fe_2O_3), limonite ($Fe_2O_3.H_2O$), magnetite (Fe_3O_4) and siderite ($FeCO_3$) (Hodges 1964:81). These oxides of iron are widely distributed all over the world.

Haematite, the red oxide of iron, is found in large quantities near the ground surface and it is easily reduced; while limonite which is the hydrated form of haematite known as red ochre is used as pigment. It is also easily reduced during smelting. Magnetite is the black magnetic oxide of iron which is at times called lodestone. This particular oxide occurs in beach sands and in particles weathering from rocks. As will be seen below, this particular oxide, although discredited as source of iron in bloomery smelting by some people (Richardson 1934), has been found to provide high iron bloom during early iron smelting in parts of Africa (Tylecote 1965). Siderite is a carbonate ore also referred to as chalybite or spathic ore. It generally occurs as nodules and has a relatively low iron content, but this poses no problem in smelting (Van der Merwe 1969:15). Where the deposit is exposed, it easily weathers to haematite and limonite.

Early iron workers have smelted these various forms of iron ore where they are available (Andah 1979:142; Tylecote 1987:48). It was assumed that the early smelters preferred haematite/limonite to other forms of iron ore. However, there is no reason why other forms of iron ore, if available, could not have been worked without some form of crucible process as suggested by Richardson (1934:566). For example David Livingstone (1974) in Nyasaland and Sassoon (1964), in northern Nigeria, described iron smelting operations in which black iron oxides (magnetic sand) were smelted. David *et al*· (1989:191) in Mafa northern Cameroon recorded successful smelting of magnetite which yielded over 61% of iron from the ore charged into the furnace.

There are suggestions that iron ore good for smelting should have an iron content of between 60 and 40% in order to be easily smelted for a good yield of iron (Friede *et al*·1977:234). But it is known that some early iron workers in South Africa smelted ores which had about 21% iron (Friede 1980). Although it is surprising that such low grade ores were smelted with profit, evidence from other iron working communities shows that such lean ores were treated and concentrated by panning, pounding and washing to reduce the siliceous matter (Bellamy and Harbord 1904; Sassoon 1964; Tylecote 1965). The low or high level of iron in some iron ores was therefore no hindrance to their being used in early iron smelting. Where the level of iron was too low, the iron level was upgraded as just discussed and where it was very high sand or clay was added to increase the siliceous matter (Zacharia and Bachmann 1983:1; Tylecote 1987:313).

All these various forms of iron ore have different characteristics conferred on them by the presence or absence of other minerals. Various grades of siliceous matter known as gangue are present in iron ores. The commonest of these minerals include silica, alumina, manganese, magnesia, titanium, potassium, calcium, phosphorus, sulphur and vanadium. The ultimate aim of iron smelting is to separate the gangue minerals from the iron.

In most early iron producing communities of West Africa, iron ore was obtained through surface collection on the out crops, valley sides or by open cast mining (Anozie 1979; Tylecote 1965). In some places the ores, particularly magnetite, were collected from alluvial deposits and weathered rocks along river courses (Sassoon 1964). These ores which were collected as nodules were pounded and broken up into smaller bits for easy penetration of heat and carbon monoxide. This was sometimes, in some communities, followed by roasting (Bellamy and Harbord op cit). Roasting has the effect of driving off carbon dioxide, water and sulphur that may have been in the ore. It also produces a much more friable charge which is more readily broken up to desired sizes. From the present study, it is known that the Nsukka iron smelters broke up the ores into palm nut kernel sizes (about 150mm thick) (Okafor in preparation a). The roasting also increases the porosity of the ore and allows better penetration of the reducing gas during smelting.

After the ore has been prepared the next important step was the provison of fuel for the smelting. In the bloomery process, the main fuel used in iron smelting was charcoal which was derived by burning sap-wood, although Tylecote (1986:21) and McDonnell (1986), observed that peat was used by some early iron smelters in England. Wood was also directly used in smelting as was the case at the site near Kankara in Kano Nigeria (Darling 1963) and in Orba Nsukka (Okafor 1984a). This will be discussed fully in section 3:3 of this Chapter. But whether the fuel used was wood or charcoal produced from burnt sap-wood, most early iron workers preferred dense hard woods which burns slowly and produces less ash. In Nsukka Division *Ukpaka*, *okpenye* and *ahaba* were the main hard woods used by the iron workers while lead wood (***Combretum inberbe***) was the main hard wood used by the iron workers of Central and Southern Africa (Van der Merwe 1980: 488).

The next vital preparation was the provision of the reducing chamber. Reduction of iron ore to bloom requiries an enclosure with a limited supply of oxygen. In the bloomery smelting, this was achieved by the construction of a bloomery furnace. In the study of early iron smelting, different forms of furnaces have been discovered to have been used in iron smelting processes (Martens 1978; Pole 1975; Sutton 1985; Tylecote 1965; Van der Merwe 1980). These include bowl furnace, otherwise known as pit furnace, dome furnace and shaft furnace. These differ in form and operative techniques. Some of these have their slags tapped in the course of the smelting while some retained their slag within their chambers.

In Nsukka area and other parts of Africa these furnaces were made of local clay which was puddled and built in layers as for some shaft furnaces. Some of these shaft furnaces have internal wooden frames and their walls were constructed as wattle and daub (Okafor 1984a). The internal surface of these furnaces were lined with puddled clay. Evidence from some parts of the world indicates that some early iron smelting furnaces were made of stone (Freestone 1989:155), and bricks lined with clay; while turf is speculated to have been used for furnace construction in Iceland (McDonnell 1986:21).

Closely related to furnaces in some bloomery iron smelting are tuyeres and bellows which were means by which draught was blasted into the furnace chamber to achieve and maintain a high temperature. Tuyere shaped like pipes or tubes were mostly made of refractory clay. Tylecote (1965) however observed that some of them were made of stone but this was not common.

The study of early bloomery iron smelting in many parts of the world revealed that the location of the smelting sites was done with deep consideration. Generally the presence of raw materials, ores and fuel, was the dominant factor that decided the siting of the smelting sites (Friede and Steel 1977; Njoku 1986). However, where the two were not available in one location, the smelting site was located closer to the ore source. What appears to be an exception to this general pattern was recorded in West Yorkshire, England by Moorhouse (1982). In West Yorkshire, during the Medieval period, iron ore was carried over 20 miles to the smelting site located nearer to the fuel source. This occurred because of the difficulties encountered at the time in the transportation of either timber or charcoal over a long distance. In Nsukka Division, smelting sites were located in places away from the settlement area, nearer to the raw materials. Apart from the need to be near the raw materials, there was the need for secrecy and prevention of possible pollution of the rituals and purity of the site by people with wicked designs against the smelters. Also the furnace heat was considered very intense and dangerous to the community.

When the necessary preparations were completed, the next step was the actual smelting. Our understanding of bloomery process and conditions influencing and affecting the production of blooms and slags have increased through the experimental smelting conducted by Childs and Schmidt (1985), Cleere (1971), Friede and Steel (1977), Pleiner (1968) Schmidt and Avery (1978) Tylecote *et al.* (1971) and Wynne and Tylecote (1958). These demonstrate conditions under which slags and blooms are formed, which in the long run have aided our interpretation of bloomery products and residues. The experiment done by Wynne and Tylecote using a pit furnace and by Tylecote *et al.* using a shaft furnace clearly demonstrate how complex bloomery technology is and how variations in temperature, rate of air flow, sizes of ore and charcoal, sizes of tuyere bore and level of charge into the furnace can affect the smelting.

For a good reduction of iron ore to iron to occur, a sufficient temperature in the region of 1100°C to 1200°C is necessary for separation of gangue materials from the spongy bloom. In the bloomery process iron is reduced in the solid state below its melting point of 1535°C and above the melting temperature of slag which is between 1200°C and 1300°C (Morton and Wingrove 1969; Tylecote 1986). This is possible in a reducing atmosphere that affords the expulsion of oxygen from the ore. It is possible for reduction of iron to occur in temperatures of about 700°C to 800°C. But when iron is reduced at the temperature below 900°C the result is an unforgeable dark grey porous substance (Andah 1979:145). When it is reduced at a temperature of about 1000°C to 1050°C the result of the smelt will be a loosely coherent mass which would also be very difficult to forge (Richardson 1934:377).

The reduction of iron ore by carbon fuel depends very much on both the temperature and the presence of a sufficient amount of carbon monoxide. The combustion of carbon in a furnace chamber poor in oxygen produces two end products, carbon monoxide and carbon dioxide. Part of the carbon is fully oxidised to form carbon dioxide; the rest is partially oxidised to form carbon monoxide. With sufficient carbon monoxide present iron ore is reduced to iron. The following equations describe the reactions that take place during reduction.

$$C + O_2 \rightarrow CO_2$$

$$2C + O_2 \rightarrow 2CO$$

$$Fe_2O_3 + 3CO \rightarrow 2Fe + 3CO_2.$$

If the carbon monoxide supply falls below a critical level, the ore simply forms slag and no bloom can be produced. The separation of growing iron crystals from the slag is important for the production of the bloom. For slag to attain low viscosity which will enable it to separate from the iron, the slag requires a limited range of composition. For this reason, much iron is lost to the slag in order to flux the gangue minerals (Coghlan 1977). Some of these minerals especially silica and alumina have high melting points in excess of 1700°C, which is above the bloomery furnace temperature (Bachmann 1982). The addition of iron oxide and or any other oxides as flux into the smelt lowers the melting point of the gangue minerals i.e, the slag to below 1200°C (Tylecote 1986) and also helps to free more iron crystals from the slag.

In the bloomery process, part of the iron oxide being smelted acts as flux during smelting and this accounts for a high loss of iron to the slag. At times up to 40 to 50% of the iron charged is lost (Morton and Wingrove 1972). This high iron loss is therefore one of the characteristics of bloomery process and bloomery slags are characterised by their high iron content to the extent that some of them have been resmelted with profit (Bellamy and Harbord 1904; Rowlands 1989; Tylecote 1960: 455; Warnier *et al*·1979; Yarranton 1681).

Apart from iron oxide other materials used as flux in bloomery smelting include sea shells (Tylecote 1965), ground shells of land snails ***achatina***, bones and sand (Van der Merwe 1980:488). Calcium oxide which is commonly used as flux in modern blast furnace smelting was said to have been used in some bloomery smelting (Agricola 1945:420; Bachmann 1982; Tylecote 1965). But calcium oxide is useful as flux in iron smelting in quantities between 10% and 15%. Above that level it raises the free running temperature of the slag beyond the capacity of bloomery furnace (Bachmann 1982; Morton and Wingrove 1969; Van der Merwe 1980).

At the end of the smelting two main products are present at the site. These are (a) the bloom and (b) the smelting residues. The smelting residues comprise slags which are mostly fayalitic in composition with some levels of silicon dioxide, aluminium oxide, magnesium oxide, titanium dioxide, calcium oxide and potassium oxide. Other residues from iron smelting are refractory clay from furnaces and tuyere, cinder, fuel ash slag and furnace lining. Descriptions and consideration of these smelting residues are in Chapter four. The bloom consists of a heterogeneous mass of spongy iron encased in slag. This is refined in the forge to expel much of the slag before the iron is forged into the desired tools.

Studies conducted on early bloomery iron show the bloom is not always wrought iron with little or no carbon as some people believe (Butt 1963). Analysis of three bloomery iron samples from Umundu which form part of the present research show that Umundu smelters produced low carbon steel with carbon content of O.O5% to O.2% and not high carbon steel as assumed by Van der Merwe (1980:493).Details of this are in Chapter five. We have already seen in section 1:4 that high carbon steels were produced direct from the bloomery furnace which were later decarburized in the forge. In the Mafa, northern Cameroon, David *et al.*(1989:196-197) witnessed the production by Dokwaza of cast iron with a carbon content of between 3% and 4% which was later decarburized to steel.

Bloomery iron smelting was a tedious and complex venture that was both resource and time intensive. Because of the difficulties connected with the trade, any smelt failure or unsuccessful smelt was a sufficient reason for a smelter to mourn "as if one has lost a child" (Okafor 1984). In Nsukka area in particular, any smelt failure was seen from more than a financial perspective. The failure forebodes some disaster to the entire community due to the close association of smelting with the fertility of women (Njoku 1986) and the dependence of other crafts and trades (that derive their tools from the products of the smelting) on the smelting.

3:3 Orba Bloomery Iron Smelting:

Among the few places in Igboland where people remember that their ancestors did iron smelting in the past is Orba. Orba was in the old Nsukka Division (see fig. 1:1). But with the inception of local government system in Nigeria in 1967, it was in the resucitated Isi-Uzu local government, being once in the former Udenu local government area of Anambra State. On the Nigeria survey map sheet 287 Nsukka N. W, Orba town lies between latitude 6° 52'N and longitude 7° 32'E. It is bounded northwards by Imilike-Uno, eastwards by Ehalumona and westwards by Nsukka town. According to 1963 Nigeria population census figures, Orba had 16,421 people with land area of about 160 sq km.

Ngwu (1983) claims that Orba is the only community in Igboland where there seem to be a continuity between the ancient iron smelting and the present blacksmithing in the town. Orba therefore appears to be vital in the understanding of the ancient iron smelting in Igboland. Three dates derived from charcoal samples collected from smelting furnaces at Orba gave dates of 300 ± 90 BP, 215 ± 100 BP, and 295 ± 85 BP. When these dates were calibrated to two sigma levels they show that iron . . . . was probably smelted in Orba between 1430 and 1950 cal. AD. Details of this are in section 5:9. The terminal date for this smelting process in Orba appears to be corrrect since there are people alive today in the town who have knowledge of how the bloomery iron smelting was conducted there.

What follow are the results of oral interviews, ethno-archaeological and archaeological investigations carried out in Ama-Orba in the months of March, May and June 1984 (Okafor 1984a) and in the month of February 1990 on the system of bloomery iron smelting in Orba. Oral interviews were conducted at Ama-Orba in 1984. While adopting this approach, I recognise that oral interview is a complex subject and differs with particular objectives. Thus the design for eliciting information regarding genealogy or kinship is not normally the same as that for eliciting information about ancient or abandoned settlements or workshops of various kinds (Okafor 1984a:10). I recognise further that the first is a complicated business in which if properly done, the anthropologist has to take proper cognizance of the social structure of the group in question. The archaeologist interested in locating ancient settlements and workshops, while not necessarily ignoring the social set up in the area, need not be constrained by this. The design for the oral interview was done with full recognition of this fact.

This oral interview was conducted in Ama-Orba on Sunday 25th March, 1984 in the house of Eze Peter Attama. The primary aims of the interview were to get clues towards the discovery of the smelting sites, and a description of the methods and materials used in the smelting. Eze Peter Attama leading his elders informed me that their people in the past smelted *nne igwe* (iron ore), and sold the product to the blacksmiths of Amube Amalla Orba, another clan in Orba that specialise in blacksmithing. They disclosed that the smelting was done in two different locations at Agu Ama-Orba and Ndiagu- Ndiuno Ama-Orba. These locations were away from the settlement area but very close to the sources of raw materials iron ore and fuel (hard woods) needed for the smelting. Other facts relating to iron smelting in Ama-Orba disclosed during the interview were recorded. The interview yielded maximum benefits for all aspects of iron smelting in Ama-Orba were described. This information led to the discovery of the smelting sites used by Ama-Orba people in ancient time.

Armed with the information, three weeks reconnaissance survey of Ama-Orba aimed at locating and mapping the smelting sites was carried out in 1984. During the survey, the two sites mentioned by the Ama-Orba elders during the interview were discovered. The site at Agu Ama-Orba covered an area of 2187 sq metres while the one at Ndiagu Ndiuno covered an area of 2243. sq metres (see fig. 3:1).

Further investigations on the early iron smelting in Orba revealed that the early iron smelting was done only by the Ama-Orba clan in the ancient time. They produced bloom which was sold to Amube Amalla blacksmiths. It was some form of division of labour. Ama-Orba produced the bloom and it was the function of the Amube Amalla blacksmiths to refine and fashion the bloom into tools.

As noted above, the smelting was done in two locations and from descriptions and observations, the two areas were at the source of raw materials for iron smelting. The Agu Ama-Orba site was within the wooded part of Orba. This Agu is within what can be described as an ancient dry valley that runs in an east-west direction with gentle slopes. This valley falls within the group of valleys Ofomata (1977) described as flat bottomed dried valleys. Lumps of haematite are seen on the lateritic ground as one walks through the valley. The main tree types in the area where the smelting was done were mostly hard woods like *okpenyi*, *ukpaka* and *ahaba*. The fuel for smelting came from these hard woods.

Presently the *Agu* is made up of shrubs and few scattered big trees. Patches of cassava farms were seen within and around the site, while the site at Ndiagu Ndiuno was covered by yam plantation. On the ground surface were lumps of red baked clay in the slag heaps. There were no visible traces of furnace or any other apparatus used for the smelting. I was informed that it had been a long time since smelting was last done at that site, and constant farming by people in and around the site had led to the disintegration of the *Itoro* (furnace).

To collect reasonable diagnostic slag samples and charcoal for dating the site, a metre square test pit was excavated at the smelting debris mound at Agu Ama-Orba on 17th February, 1990 (Okafor in preparation b). The top soil of this pit was mostly loose and it contained aggregates of slags and lumps of baked clay. Below this layer was a dark grey soil that contained mostly slags and furnace fragments. At the western section of the pit, at a point 28cm below the surface, parts of a broken furnace wall were discovered. This wall was traced and it extended into layer three where a complete furnace base was discovered at a level 32cm below the surface. This furnace base was surrounded by dark-bluish soil. Within the base of this furnace were amalgams of slags and cinder encasing occluded charcoal (see Plate 3:1). This furnace base has a diameter of 46cm (ibid). There were no other cultural materials found in this pit except slags and charcoal, which afforded good samples for analysis. Results of the bulk analyses of slag samples from Orba are in tables 13-24 in appendix 1. These results are discussed in Chapters five and six.

It was difficult to categorize this base of the furnace as part of a bowl or shaft furnace, but from ethnograhpic data collected from the community, Ama-Orba people conducted their smelting in an *Itoro* which from description was a self draught shaft furnace. An account of the smelting techniques and description of the apparatus used by iron smelters in Orba by Okafor (1984a) is given below. In Ama-Orba the whole process of iron smelting is known as *Ise Itoro*; which means construction of oven or furnace. The *Itoro* which was estimated to be about 1.3m tall was constructed by first fixing medium sized timbers closely in circular form as if one was building a fence. Twining plants such as ***Landolphia spp.*** and ***Combretum spp.*** were used to tie the wood to make them firm. After the tying both the inside and outside of this frame were plastered and lined with thick layers of puddled clay in the same way that is done when building wattle and daub walls. After the construction, the *Itoro* was provided with an opening at the top and two or three openings at the base.

Iron smelters in Orba did not use charcoal in their smelting. Rather they used hard wood from *okpeyi ukpaka* or *ahaba*. It is significant to note that a similar furnace structure and fuel system have been described by another iron smelting community at Ugwu Ogu in Uturu Okigwe in Imo state, a community about 120km S.E of Orba (personal communication, Okpoko 1983). Orba furnace also resembles those of Umundu, a town 3km N.E of Orba, which is described in section 3:4 of this chapter as being a self draught shaft furnace.

In Orba iron ores are locally known as *nne igwe* (Okafor in preparation a). When these ores were analysed by x-ray diffraction technique it was discovered that they consist mainly of goethite. Details of this are in section 5:7. Ores from Orba have an average iron content of more than 51% and have relatively few gangue minerals (see tables 62 and 127 in Appendix 1). The ores are particularly remarkable in having significant levels of titanium. The clay minerals in them are mostly alumina, silica and little iron oxide. The ores were collected on the valley sides and erosion gullies as nodules and lumps which were broken up into small bits of about palm nut kernel sizes (about 150mm thick) (Okafor in preparation a) before they were fed into the furnace. No roasting or any other further treatment of the ores was reported.

When the furnace and all the necessary materials-ore, and fuel were ready, the smelting began with the loading of the furnace through the top opening. First the floor of the furnace was lined with dry grass and small dry woods that can catch fire easily. The layers immediately above comprised larger wood and then iron ore. From this layer wood and iron ore were loaded in alternate fashion until the furnace was three-quarters full. Fire was set to the entire load with dry grass through the openings at the base.

As the smelting progressed, the opening at the top was closed with thick material. It was learnt that some frightening sounds kru-kru-kru-kru were normally heard from the inside of the furnace, a sign that the smelting was progressing well (Okafor in preparation a). When this sound becomes continuous, the furnace was opened and through the top opening, some sand ~~were~~ was charged into the furnace. It was not possible to ascertain the role of the sand in the smelting. But the most probable explanation is that the sand acted as a flux in the smelting to help in the slag formation and flow at a low temperature and to free more iron crystals. Whatever effect the sand was desired to achieve, the analyses of slags from Orba, show that they contain more silica than could have been produced from ores derived from that town or any part of Nsukka. Details of this are in tables 13 to 24 and 58 in Appendix 1 and in Chapters five and six. This strongly suggests that the gangue materials from the ore reacted with the sand to produce slag of very high silica level. Iron smelting in Orba once begun lasts for about 36hrs.

At the end of the smelt, the contents of the furnace, bloom, slags, charcoal and other residues- were raked out with a wooden rake through the openings at the base of the furnace. Labourers, (male or female) were employed to sort out blooms from the other smelting residues off-loaded from the furnace. This explains why slags from Orba are mostly in aggregate form (see plate 3:2) as they were usually broken up in order to collect the bloom. They weigh between 12gm and 19gm and have mean specific gravity of 3.98gm\cm^3.

The recovered blooms were carried in baskets to Orie Orba market where they were sold to blacksmiths from Amube and those from other parts of Nsukka Division. Ama-Orba iron smelters never went beyond the stage of bloom production in their iron working. The blacksmiths that bought the blooms refined them by heating and hammering--off any adhering slags before forging them into tools.

3:4 Bloomery Iron Smelting In Umundu:

Umundu, a town located 10km north east of Nsukka metropolis, (see fig. 1:2) is one of the communities in Igboland where bloomery iron smelting took place in ancient time (Van der Merwe 1980). Iron production is still going on in the town but not with bloomery smelted iron. The present group of iron workers are blacksmiths who use metal scraps of motor chassis and iron bars in forging locally needed tools (Anozie 1979; Van der Merwe op cit).

Archaeological and ethno-archaeological investigations in the town have helped in the understanding of the early iron smelting in the community. This was possible because people who assisted their parents in iron smelting when they were young are still alive and were persuaded to re-enact the technique of the smelting for recording at the museum of Archaeology department University of Nigeria, Nsukka in 1976. The record of this demonstration supplemented with archaeological discoveries in the town have helped in our understanding of the system of bloomery iron smelting there.

During research in 1990,extensive spread of iron smelting debris were located in three sections of the community. The first area of the smelting debris is located close to Umundu market square (see fig. 3:2). Materials on this site consist of piles of slag, baked clay, furnace walls which were at the centre of the crescentic mound formed by the smelting debris. This particular site covers an area of 439 sq metres. The next smelting remains are within Umundu community secondary school. The remains at this site have been bulldozed completely before the school blocks were erected. Still there are concentrations of slags all over the school ground which cover an area of 7303 sq metres. The final smelting remains are on Umundu communal land. The remains at this site have also been badly disturbed by farming activities and the slags are piled in heaps within the communal land. The remains on this site cover an area of 603 sq metres.

Umundu iron smelting slags are aggregates in form (see plate 3:3) which indicate the way by which they were removed from the furnace (McDonnell 1986:83). As will be shown later in this section, the slags were raked from the furnace at the end of each smelt and broken up while sorting the bloom from the other smelting debris. Slags from this site were weighed in February 1990 and they weighed between 18gm and 20gm and have mean specific gravity of 3.63gm/cm^3. Tables 25-36 in Appendix 1, contain the results of analyses of Umundu slags. These results are discussed in Chapters Five and Six.

Before the present research was undertaken Umundu iron smelting site is the only one that has been both excavated and dated in Igboland. Three dates from this site range from "1775 ± 1925 AD, 1685 ± 1835 AD to 1625 ± 1775 AD." This was claimed by the excavator to fall between 1625 and 1925 AD (Anozie 1979:131). During the present research three charcoal samples collected from smelting furnaces in Umundu produced dates of 200 ± 80 BP, 205 ± 80 BP and 130 ± 80 BP. When these dates were calibrated to two sigma levels they show that iron smelting was being conducted at Umundu between about 1490 and 1950 cal. AD. Detail discussions on this are in sections 5:9 and 6:3.

Ethno-archaeological information and observations made during the re-enactment of Umundu bloomery iron smelting revealed that the three smelting sites at Umundu were originally away from the settlement areas and closer to the source of the raw materials, fuel and ore. Umundu iron smelters used charcoal derived by burning *ukpaka*, *okpenyi* and *ahaba* woods in their smelting. Charcoal from these woods burn slowly and produces very low ash. In the past, these species of trees were said to be very abundant in the area. Presently few scattered stands of these trees trying to regenerate are seen around the sites. Charcoal from these trees is currently so scarce that 20kg of charcoal from *ukpaka* wood was bought at a cost of N197. 00 (£17.91) by the writer in February 1990.

The ore used by the Umundu iron smelters was mostly haematite and goethite which are still very abundant in the area. They appear as nodules. The ores were broken up into small sizes of about 150mm thick before they were used in the smelting. These ore nodules erode from the lateritic deposits at the foot of the hills in the town from where they were collected on the ground surface or by simple open cast mining (Anozie 1979). Presently the haematite nodules are being quarried for road and house constructions. Analysis of these ores show that they have iron content of about 50 %. As will be seen in section 5:7, these ores have remarkably low silica and calcium oxide contents (see tables 62 and 127 in

Appendix 1). No further treatment of the ore was undertaken such as roasting and or washing before smelting. The wet chemical analysis for iron in the ores gives a mean value of about 36%. A mean values for FeO of about 64% was obtained for the EDX analysis of the ores because it has been assumed that they are made of stoichiometric FeO, etc. It is most likely that this discrepancy arises due to hydration of the ores.

Umundu iron smelters used a self draught shaft furnace built with puddled clay. The reconstructed furnace had an internal diameter of about 89cm at the base. Of the three iron smelting sites located at Umundu, only the site close to the market was not disturbed by building construction or farming activities. So to collect charcoal samples for dating and slags for analysis during this research, I carried out in February 1990 a one metre square sampling excavation at the centre of the "Market" crescentic debris mound where there were many fragments of furnace wall (Okafor in preparation b).

The excavation revealed three cultural layers that contained only slags, furnace walls, charcoal, roots and rootlets. The top layer was grey lateritic compact soil full of roots and rootlets, slags and furnace walls. The second layer was composed of hard dark brownish soil that contained slags and furnace fragments. Layer three was moderately loose and reddish brown in colour. It contained a furnace base that measured 62cm in diameter with a wall thickness of 8cm. Within this furnace base were slags, charcoal and furnace wall fragments which were interspersed by roots and rootlets. Charcoal samples, slags and refractory clay for analysis were collected from this pit.

The re-enactment furnace was 1.5m high with constricted top which was provided with small opening. To avoid collapse of the furnace during construction, it was never completed in a day. It was built in three stages, the top layer being tapered so as to generate the required smelting temperature. Two to four openings were made at the base of the furnace through which fire was set to the charge and through which air flowed into the furnace chamber. From these openings also the load in the furnace was off-loaded at the end of the smelt.

When all the necessary materials were ready, the actual smelting began. Umundu smelting starts in the evening and lasts more than one day (Okafor in preparation a). Usually it took between 24 and 36hrs to complete a round of Umundu iron smelting. The smelting starts with the loading of the furnace which was done through the top opening. First the base of the furnace was lined with very combustible materials such as very dry pieces of wood, some dry grasses or dry palm fruit fibre. After this the furnace was loaded with alternate layers of charcoal and ore, starting with charcoal. The furnace was never completely filled. It was filled only to two-thirds of it's capacity. In the 1976 re-enactment smelt, the volume ratio of ore to the charcoal charged into the furnace was 3:9.

The ore and the charcoal were measured with a basket, and three basketsful of ore were charged with nine basketsful of charcoal. Some charcoal must be left for intermittent charge into the furnace during the smelting. After the loading, fire was introduced to the load through the openings at the base.

As the smelting progressed, a droning sound doom-doom-doom comes from the furnace. As in the case of Orba iron smelting, this was an indication that the smelting was progressing well and such droning was hailed at Umundu with acclamation "*Nne muru oha* !!" (the mother of all races) an honorific to the God of iron (Njoku 1986).

At the end of the smelt, the furnace was off-loaded with wooden implements that were fashioned to a hook at one of their ends. The off-loading of the furnace was done through the openings at the base. The entire content of the furnace: bloom, slags, charcoal and unreduced ore were raked out. The blooms were sorted out from the rest of the smelting residues at times by breaking up the slags. The residues were thrown away on the nearby debris heaps while the blooms were carried in baskets and sold to the blacksmiths in Umundu or at Orie Orba market where blacksmiths from other parts of Nsukka Division come to purchase bloom they use in their smithing operations. Umundu iron smelters like their counter parts in Orba never went beyond the production of the bloom. The blooms were refined and forged by the blacksmiths that bought them from the Umundu iron smelters.

3:5 Bloomery Iron Smelting In Opi

Opi located 7km south east of Nsukka metropolis is on the southern border of the present Nsukka local government area (see fig. 1:2). Early iron smelting sites in Opi were discovered by the writer in August 1983 during his survey of early iron working sites in parts of Igboland (Okafor 1983). The presence of many cylindrical blocks of slags which were eroding from the ground and tuyere pieces that often associate with these slags convinced the writer that iron smelting was practised in the area in the past. Three radiocarbon dates of 2305 ± 90 BP, 2170 ± 80 BP and 2080 ± 90 BP were derived from charcoal samples from furnaces "A", "B" and "C" located at the foot of Opi hill. The calibration of these dates to one sigma level suggest that iron smelting was conducted at Opi between about 520 cal. BC and 15 cal. AD. When these dates are calibrated to two sigma levels, there is 98% probability that the period of iron smelting in Opi extends to between 765 cal. BC and 75 cal. AD. Detail discussion of the carbon-14 dates are in sections 5:9 and 6:3. An intensive survey of the town in August 1983 led to the discovery of four early iron smelting sites in Opi. These were located at Afor Opi market, Opi customary court grounds (native court), at the foot of Opi hill (see fig.3:3) and at Odinanso Idi Opi village square.

Apart from the ethnographic studies on the early iron smelting in Opi, which were carried out in the dry season of 1984/85 by the writer, no further work was done on these sites until February 1990 when these sites were surveyed, mapped and sampled. Further work on the site in April and May, 1991 involved more test excavations aimed at collecting samples for radiometric dating. At Afor Opi market these slag debris and blocks covered an area of 4779 sq metres, and at the grounds of Opi customary court, similar slag residues covered an area of 3175. sq metres. The major concentration of these residues is at the foot of Opi hill where they cover an area of 21551 sq metres. At Odinanso Idi Opi village square, which is 2km east of Afor Opi market, these residues were located, and there, they have been turned into shrines and ritual objects. As a result the chief priest of the shrines did not allow us to survey the site to find out its exact dimensions. But after much persuasion he allowed us to take photographS of parts of the site (see plate 3:4) and to collect slag samples for analyses.

Iron smelting slags in Opi are mostly dark grey cylindrical blocks that are between 54cm and 56cm in diameter and between 18cm and 21cm high. They weigh between 43kg and 47kg and have a relatively high density of 3.89g/cm^3. Some of the the cylindrical blocks of slag are found in situ in round slag pits that measure between 61cm and 64cm in diameter and between 19cm and 23cm deep. These pits were lined with clay of between 4cm and 6cm thick. The heat from the molten slag affected these clay linings so much that although they are friable, they assumed greyish-blue colour. None of the clay linings was slagged or vitrified or has any slag penetration as would have been the case if these were smelting pits (Pleiner 1978:38; Tite *et al*. 1985:50). Fifteen slag samples from Opi sites were analysed during this research. Results of the bulk analyses of the samples are in tables 37-51 in Appendix 1. These results are discussed in Chapters Five and Six.

No complete tuyere was found during the investigation of these smelting sites. But the fragments recovered during the investigation were highly baked and have twigs and straw inclusions used probably for tempering the clay.

These fragments have external diameter of about 14cm and 5cm internal bore. Their wall thickness is between 4cm and 6cm.

Due to frequent farming on these sites and the fact that many building constructions have taken place on them, no standing furnace remains were discovered on these sites. Added to this is the fact that most early iron smelters in West Africa made no effort to preserve their furnaces from one smelting season (usually the dry season) to the next, and the coarse nature of the clay used in making the furnace and the other apparatus, resulted in their total disintegration during the rains (Pole 1975:15). Added to the above is the fact that there is no demarcation in Nsukka Division between factory sites (smelting sites) and farm lands. A plot of land could serve as smelting site in the dry season and become farm land in the wet season. During this period most of the smelting residues would be destroyed or redeposited.

In spite of the above limitation, the limited evidence from the archaeological investigations and ethnographic information gathered from Opi have enabled us to reconstruct the smelting techniques used in Opi bloomery iron smelting. According to Opi tradition iron smelting developed in the town due to two main reasons (Okafor in preparation a). One was the demand for *akpaka igwe* (bloom) by blacksmiths from the neighbouring villages of Edeoballa and Ehalumona who travelled long distances to buy the bloom they use for forging tools. The second reason was that there was great abundance of *nne igwe* (iron ore) in Opi especially on the Opi hill.

These ores are mostly haematite which weather from the ferruginized capping on this hill. The smelters collected the ores from the ground surface by simple open cast mining using locally made hoes and digging sticks tipped with metal. Such open cast mines at the foot of Opi hill are still identifiable. Analyses of ores from Opi reveal that they have an average iron content of 46.87%. They have relatively high levels of titanium and alumina but very low calcium (see tables 62 and 127 in Appendix 1). These ores appear in lumps which were broken down to the size of about 150mm thick before they were smelted. As in Ama-Orba and Umundu, no further treatment of the ore (washing or roasting), was done at Opi before it was smelted.

The fuel used for the smelting was said to be very abundant in the area. Opi iron smelters used mostly charcoal from *ukpaka*, and *icheku* (***Velvet Tamarind***). They observed that charcoal from these trees could burn for many days without turning into ashes. They prepared the charcoal by felling these trees and chopping them into lumps. These lumps were left for about two native weeks (8 days; 4 days make a native week) to dry midway. Then they were piled together into a heap and palm oil poured on them before fire was set on the pile. The palm oil facilitates the burning of the woods.

Opi iron smelters used *ncheku* (furnace) for their smelting. From their descriptions, this is a forced draft furnace of an average man's height, just about 1.5 metres high. The furnace was constructed with local clay prepared in the same way as was done for mud houses. It was provided with an opening at the top and two or three openings at the base for inserting the tuyere and for tapping the *afuru* (slag). The slag holes were closed with earth which were removed at intervals for tapping the slags when smelting begins. Close to the base of the furnace were pits connected by channels from the furnace through which the slags drained out of the furnace into the pits. These pits were lined with clay. Opi iron smelters used pairs of bellows covered with goat or sheep skin tied to two sticks for blasting air into the furnace.

On the day of the smelt, the furnace was loaded through the top opening. First, dry grasses and very dry small pieces of wood were laid at the base of the furnace. Then another layer of very dry wood, ores and charcoal was laid in alternate fashion until the furnace was two-thirds full. Some charcoal must be left for charging the furnace as the smelting progressed. After the loading palm oil was poured on the entire load through the top opening and fire was set on the load through the openings at the base. At this time the slag tapping hole below must have been sealed with earth which was removed each time slag was to be tapped and replaced again after each tap.

As soon as fire was set on the load, the bellow work began and this had the effects of both raising the temperature and maintaining the heat in the furnace. Opi smelting was started by the sun set when the weather was cooling down and lasted till mid day the follwing day, that is, a round of Opi smelting lasted between 18 and 20hrs. At the end of the smelt, the bloom covered by slag now resting on the floor of the furnace, was left to cool in the furnace before it was removed with *mkpa osisi* (wooden clamp) through one of the openings at the base.

Opi iron smelters sold their blooms to blacksmiths from Edeoballa, Ehalumona, Nkwere and Awka. Opi iron smelters like their counter parts from Ama-Orba and Umundu never went beyond the stage of iron smelting. The blacksmiths that bought the blooms did the work of refining and forging the bloom into the desired tools.

3:6 Bloomery Iron Smelting In Owerre-Elu Nsukka:

One of the communities that make up Nsukka metropolis is Owerre-Elu which is separated from Opi iron smelters by Edeoballa, a town renowned for it's blacksmith~~ry~~ing. In terms of the area covered and the magnitude of the iron smelting residues at this site, Owerre-Elu appears to be the place where the early iron smelting was most intensively practised in the whole of Nsukka Division. Paradoxically, this site which is within Nsukka metropolis and which has the greatest archaeometallurgical potential, is the most destroyed archaeological site in Nsukka Division.

Owerre-Elu iron smelting site was badly if not totally destroyed by persistent farming and the other human actions, especially by the erection of the Nsukka new timber shed on the site. The constuction of this new timber shed in 1985 led to the bulldozing of the entire site, and it was this development that revealed the existence of the site to the writer in April 1987 (Okafor 1988). But by then the only tangible evidence of early iron smelting on this site was heaps of smelting residues mostly slags, tuyere fragments and baked clays which spread all over the site and beyond. Due to the level of damage that had been inflicted on this site, the reconstruction of iron smelting processes at this site, more than in any other site in the Division, will rely more on the analyses of iron smelting residues and ethnographic information collected in 1987 from the people of Owerre-Elu and Isiakpu (a neighbouring village to Owerre-Elu) who still claim to remember how their ancestors smelted iron in the past. Two radiocarbon dates 1060 ± 60 BP and 570 ± 60 BP were derived from fused wood and straw mixed with puddled clay used for preparing the tuyere, (both were covered by slags). When these dates were clibrated to two sigma levels they show that iron smelting was being done in Owerre-Elu between about 800 and 1430 AD.

The discovery of this site in 1987 was followed by the collection of ethnographic information on the techniques of iron smelting in Owerre-Elu (Okafor in preparation a). The record of the information gathered during the ethnographic investigations and few archaeological discoveries at the site have been used to reconstruct the techniques of the early iron smelting in the commuity. What emerged from this reconstruction shows, as will be seen later in this section, that Owerre-Elu iron smelting techniqes were similar in some respects to those of Opi (ibid). The proper survey and mapping of this site was done in February 1990, during which period the various samples from this site used in this research were collected (see fig. 3:4).

Slags from Owerre-Elu are tapped slags that appear in flat cake forms with elongated smooth ropy surfaces (see plate 3:5) which indicate that they were completely molten during the smelting (Tylecote 1986, 1987; McDonnell 1983, 1986). Measurements taken from the slags from this site in February 1990 reveal that they measure between 15cm by 20cm and weigh between 1.5 and 4.2kg. They have very high density of 4.24g/cm^3. Tables 1-12 in Appendix 1, contain the results of bulk analyses of slag samples from Owerre-Elu. These results are discussed in Chapters Five and Six. No complete tuyere was recovered at this site but the fragments recovered were made of local clay which was tempered with straw and twigs. One particular fragment was highly baked and slagged which probably indicates the length of insertion of these tuyere into the furnace. These tuyere fragments have an external diameter of between 40mm and 50mm with wall thickness of 30mm to 40mm. From the survey it was discovered that the entire site covered an area of 54937 sq metres (see fig. 3:4).

According to Nsukka tradition *nne nkpume* (iron stone) was the main ore smelted at Owerre-Elu. From the present research, Owerre-Elu iron ore is discovered to be mostly haematite with average iron content of 52.84%. The ore contains clay minerals which are mostly alumina, silica and small amount of iron oxide. They contain very low calcium but they have significant level of titanium (see tables 62 and 127 in Appendix 1). The ores occur in lumps which are broken up into small bits before they were smelted. These ores are mostly found at the southern part of the site where they weather from the long ridge of ferruginized out crop along the border of Owerre-Elu, Isiakpu and Ede-Oballa. The ores for smelting were collected on and at the foot of this ridge.

Charcoal was used by Owerre-Elu iron smelters. This was derived by burning woods from *ukpaka*, *okpeny* and *icheku* which were very abundant in the area. Even now many stands of these hard woods are regenerating within the site. Owerre-Elu iron smelters used charcoal in their smelting in the same way as Opi iron smelters (which was described in section 3:5). The trees were felled and cut into lumps and allowed to dry for about eight days before they were burnt with the aid of palm oil.

The furnace used by Owerre-Elu iron smelters was forced draft shaft furnace built with puddled clay from the local clay. The base of the furnace was estimated to measure about 80cm and it was about 1.5m tall. These were built close to the ore source and at the initial period of the industry the hard woods that provided the fuel were very abundant at the site. An opening was provided at the top of the furnace through which it was loaded. About two or three openings were provided at the base of the furnace through which tuyeres were inserted and for tapping the slag. The slag tapping hole was plugged with clay and ashes which were removed during each tapping and replaced after. Owerre-Elu furnaces however, have no slag pits. The slag flows out from the furnace during each tap on the ground, that is why the slags appear in flat cake forms (see plate 3:5). Bellows used by Owerre-Elu iron smelters are similar to those used by the Opi smelters. They use sheep or goat skins to cover the bellows to which were tied two sticks for easy working of the bellows and fast blasting of air into the furnace.

On the day of the smelting, the base of the furnace was lined with very dry grass and easily combustible materials. On these were loaded alternate layers of ore and charcoal till the furnace was two-thirds full. Palm oil was used according to the prevailing weather condition and the condition of the load in the furnace. On very warm days palm oil may not be poured on the load, but if the weather was wet or dull, it was necessary to pour palm oil on the load before fire was set on it through the openings at the base. The bellow work starts as soon as fire was set on the load and this went on uninterrupted till the end of the smelt which lasts about 18hrs.

At the end of the smelt the furnace was allowed to cool before the bloom was removed through one of the basal openings with *mkpa osisi* (wooden clamp). The blooms were sold to Edeoballa blacksmiths and other blacksmiths at Orie Orba market. What is emerging from Nsukka Division tended to show that the iron smelters were quite different from the blacksmiths. The smelters produced the bloom which they sold to the blacksmiths who refined and forged the bloom into tools. Owerre-Elu like Opi has no local blacksmiths unlike at Orba and Umundu Where local iron smelters and blacksmiths existed. See table 119 in Appendix 1 for the summary of techniques used by Nsukka iron smelters.

CHAPTER 4

Bloomery Iron Smelting Residues

4:1 Introduction

Iron smelting residues have been identified and classified by McDonnell (1983, 1986). Four main residues are commonly associated with bloomery smelting. These are smelting slags, cinder, fuel ash slag, or vitrified fuel ash (Tylecote 1987:292) and refractory materials (furnace walls, furnace lining and tuyere). Three of these residues, (cinder, fuel ash slag, and furnace lining) survive or disappear from the smelting sites depending on the weather condition, age of the site and the level of subsequent human activities at the site. The older the site and the more human activities that take place on the site, the less chances archaeologists have of recovering them from the site. Again, although these three residues emanate from iron smelting, it is impossible to reconstruct the smelting processes through their chemical or physical analysis since such analysis gives very biased results concerning the processes that took place at the site (Friede 1977:227).

4:2 Bloomery Iron Smelting Slags

Various definitions and descriptions of bloomery slags have been given by many people like Alexander and Steel (1946), Bachmann (1982) Friede *et al*· (1982) and Morton and Wingrove (1969a, 1969b). Slags are once molten fused by-products of metal working which comprise gangue materials from the ore, fuel and furnace materials. They are very durable and are almost indestructible. They only suffer some loss of alkali minerals through leaching when they are in water logged condition. Apart from this the only loss slags suffer is redeposition which follow from their durability and toughness. Slags vary in texture, colour, density, appearance and specific gravity. They have no definite shape or form, since they exist in diverse forms depending on how they remained in or were removed from the furnace (McDonnell 1986:83). Generally most bloomery slags are composed of iron silicate.

Slags, therefore, can be defined as a solidified once molten silicate matter that result from the reduced iron ore, which is composed of gangue material from the iron ore, fuel ash and refractory materials. They are often found discarded on metal working, smelting, smithing and refining sites. Most often they are regarded as useless and worthless by-products of a particular metal working process. The quantity of slags produced in a site depends on the particular type of metal working that was done on a site. Of all metal working processes, metal smelting produces the largest quantities of slags. It is estimated that in a very efficient bloomery smelting the ratio of metal to slag produced is 1:4 (Van der Merwe 1969:100). While the bloom produced is carried away and forged into tools which were distributed to the wider communities, the slags remain in heaps at or near the production sites ready to tell the tales of how they came about.

The quantity of slags in a site is an indication of the technological process that went on in that particular site i.e smelting, smithing or refining (Friede and Steel 1988:39). Smelting sites are easily distinguished from smithing and refining sites by the quantity of slags observed in them. Large quantities of slags in a site plus some ores are clear indications of smelting activities, while few smallish lumps of slags especially found close to settlement areas are an indication of smithing or refining activities (Tylecote 1962:193). Besides this parameter, it is generally difficult to distinguish by visual inspection a lump of smelting slag from smithing/refining slags. However, when chemically analysed, smithing slags contain higher proportion of silica than the smelting slag (Friede and Steel 1988:39), while the smelting slags have a higher proportion of manganese than the smithing slags (McDonnell 1986:170)

As observed above, bloomery slags are composed mostly of iron silicate. In the bloomery smelting some of the iron in the ore charged acted as a flux; as a result much of the iron is contained in the bloomery slags. At times up to 40 to 50% of the iron in the ore is lost to the slag (Morton and Wingrove 1969b:55). The remarkable iron silicate nature of the bloomery slags derive from this high loss of iron from the ore to the slag. This is now one of the criteria for distinguishing metallurgical slags from non- metallurgical slags. Bloomery slags are remarkable for their low melting temperature which often is between 1100 and 1200°C (Tylecote 1980:223), since the temperature at which they were produced rarely exceeded 1300 to 1400°C (Bachmann 1982:10). The low melting temperature of the dominant constituent mineral, fayalite, is responsible for this remarkable low melting point of bloomery slags (Bachmann 1982; Tylecote 1980).

In the slag study and classification much reliance is placed on the morphological study of slags. Thus the morphological examination of slags has formed the basis for distinguishing various forms of slag residues since it is impossible to do detailed chemical and physical study of all the slags found in iron smelting sites. Morphologically, there are three main distinct forms of smelting slags. These are (a) tap slags, (b) raked slags or smelting slags,and (c) furnace bottoms.

4:2:1 Tap Slags

Tap slags were produced in iron smelting operations in which the furnace was provided with an aperture for draining the molten slag out of the furnace while the smelting was in progress. The molten slag solidified outside the furnace. Often tap slags were run through a channel into containers, mostly pits, where they solidify. Such tap slags have often been confused with furnace bottoms where neither the furnace nor the channels have survived as in Leja in Nsukka, Nigeria. But they can be differenciated from furnace bottoms by the various layers which mark individual tappings from the furnace (see plate 4:1).

When such tap slags are examined with the scanning electron microscope the individual tapped slag batches are separated by white bands of magnetite which form on the cooling surface before the next tapped batch superimposed on the former one (see plate 5:11). In some cases the tapped slags have been allowed to run and solidify on the ground surface around the furnace. Such tap slags are flattish in form with lava-like ripple appearance (see plate 3:5). This type of tapped slag is generally black, and dense with smooth surface. In section such slags are bluish-grey in colour, and coarsely vesicular near the upper cooling surface with few inclusions (McDonnell 1983:82). The technique of tapping slag while the smelting was in progress has the advantage of keeping the furnace running for a long period and a large bloom can be produced without the slag reaching the tuyere zone to block it.

4:2:2 Raked Slag Or Smelting Slag

Raked slags were produced in iron smelting operations in which the furnace was not provided with an aperture for tapping the slag. Both the bloom and the slag remained in the furnace until they were removed from the furnace often by raking through the basal openings. Often the slags are broken up to separate them from the bloom. The mode of their removal from the furnace makes them survive as amorphous irregular aggregates (see plates 3:2 and 3:3). Morphologically it is often hard to distinguish raked slags from the smithing slags which also survive as aggregates. But the determination of their manganese content (% by weight) through chemical analysis is the surest way to achieve this distinction. Raked slags from iron smelting will have higher level of manganese oxide, MnO, than the smithing slag (Friede and Steel 1988:39; McDonnell 1986).

4:2:3 Furnace Bottom

Furnace bottoms are slags produced in furnaces without tapping facilities and in which the slag was not raked or removed from the furnace after the smelting. The slag accumulated, solidified and remained at the furnace bottom. They are often plano-convex in form (Tylecote 1987:312) and their external appearance varies from an agglomerated to a smooth surface which shows that the slag was molten before solidification, and that it was formed by continuous dripping and accumulation of molten slags and not by intermittent tappings. Furnace bottoms have been found to contain higher impurities from the ore, fuel and the refractory materials (Friede *et al*·1982:38).

4:3 The Composition Of Bloomery Slags:

The structure and constitution of bloomery slags have been studied by Fells (1983) and Bachmann (1982). Although Fells approached her investigation of archaeological slags from a geological and modern metallurgical point of view, the study nevertheless, revealed the basic information common to most bloomery slags. Chemically bloomery slags are composed mainly of iron oxides and silicates which together make up over 90% of the slag constituents. Other oxides commonly found in bloomery slags are aluminium oxide Al_2O_3, magnesium oxide MgO, manganese oxide MnO, calcium oxide CaO, titanium dioxide TiO_2, phosphorous pentoxide P_2O_5, potassium oxide K_2O, and venadium pentoxide V_2O_5. These chemical oxides appear in varying proportions depending on the ore composition, fuel and the refractory material compositions.

With regard to mineral composition, bloomery slags are very varied (Fells 1983:69). Their mineral compositions have been observed to depend on several factors (1) The composition of the smelted ore (2) The chemical composition of the fuel used in the smelting (3) The chemical composition of the refractory materials used in the smelting: the furnace, the furnace lining and the tuyere. (4) The chemical composition of any flux(es) used in the smelting. (5) The rate of air blast that went into the furnace during the smelting. (6) The state of the slag as it was removed from the furnace i.e. whether it was tapped in a molten state or removed after solidification in the furnace. and (7) The surrounding conditions of the slag since it was formed.

The various minerals found in bloomery slags exist as mineral phases of varying proportions (Fells op. cit; Morton and Wingrove 1969a, 1972; Todd 1976; Tylecote 1987). Of all the mineral phases found in bloomery slags, the most common ones are iron silicate, iron oxides (wustite and magnetite), and glass. In some slags other minerals such as hercynite and leucite are also present.

Iron Silicate, Fayalite $2FeO.SiO_2$: Slags from bloomery iron smelting are generally fayalitic in composition as they contain mostly iron silicate mineral known as fayalite $2FeO.SiO_2$ (Morton and Wingrove op. cit). However, there are slags in which MnO, MgO, or CaO replace the FeO in the iron silicate to form magnesium, manganous or calcium silicate. Some other metal working operations such as smithing and forging of blooms in the hearth, as well as the smelting of copper and lead ores where iron ore was used to flux the smelt, produce fayalitic slags (Tylecote 1980:223, 1962:34). But such fayalitic slags produced in the course of copper or lead smelting are distinguished by the higher copper or lead oxides levels (often 0.5% more) in the suspected slags (Rostoker and Gebhard 1981:42-43). Such slags from lead or copper production are normally found close to their ore bearing zones. Fayalitic slags from the smithing forge are distinguished from iron smelting slags by their amorphous lumpy shapes and their chemical analysis.

The fayalite mineral in the bloomery slags appear as lath-like crystals. The slags investigated in this research have massive fayalite and lath-like crystals, although some are fine in form. Spot analysis of fayalitic phases in bloomery slags show that they are made up of mainly iron oxide and silicon dioxide which are always present in the ratio of 2:1 (see tables 63 to 113 in Appendix 1).

Iron Oxides: The next common mineral found in bloomery slags are iron oxides, wustite FeO, and magnetite Fe_3O_4. The iron oxides in bloomery slags commonly exist as white dendrites of wustite. The presence of oxides in dendritic form indicates that the oxide was in solution at elevated temperatures and crystallised out of a liquid during cooling. Dendritic shapes are not normally associated with materials that under-went a solid-solid transformation (Friede et al. 1982: 47). The level of free iron oxides in slags is assumed to indicate the degree of efficiency of the particular bloomery technology (Morton and Wingrove op cit; McDonnell 1986:86). The higher the free iron oxide content in the slag, the less efficient the particular bloomery operation. The spot analysis of wustite phase in the analysed samples show that iron oxide content is above 90% (see tables 109 to 112 in Appendix 1).

The wustite in the bloomery slags display a range of forms and shapes. Most of the wustites observed in the slags investigated in this research assume dendritic forms which are fine and long or short and thick. In some slags, they appear as isolated dendritic globules (see plate 5:8).

In some slags, especially tap slags, bands of iron oxides mainly of magnetite occur on the chilled surface of the slag. This magnetite is the product of oxidation which took place as the molten slag left the reducing condition in the furnace (Morton and Wingrove 1969a). In tap slags that were run into slag pits, such bands of magnetite are observed in between each succesive tapping (see plate 5:11). The presence of this magnetite in some of the slags investigated in this reseach, confirm that they were completely molten before they were tapped from the furnace (Fells op cit:108).

Another mineral commonly found in bloomery slags is hercynite $FeO.Al_2O_3$. Morton and Wingrove (1972:483) from their study of bloomery iron slags from Medieval Iron Age sites in England, claim that the hercynite phase is found mainly in slags from lean carboniferous iron ores and that the hercynite phases are located more around the gas holes in the slags. This claim is not observed in this research, as discussed in section 6:4, the presence of hercynite was determined more by the level of alumina in the ore, to some extent the rate of cooling and fuel used in the smelting. Also the particular location of this mineral in the slags is not limited to the vesicular regions in the slags. They were oberved within the fayalite and glass matrix where they appear as dark-grey euhedral crystals (see plate 5:6b) or as angular intergrowths with fayalite. Spot analysis of hercynite phases reveal that their iron oxide and aluminium oxide ratios are approximately 1:1 (see tables 63 to 113 in Appendix 1).

Depending on the cooling rate and existing condition of the slags, the alkali oxide K_2O, in the slags form the mineral leucite $K_2O.Al_2O_3.4SiO_2$ with the excess alumina and silica. The mineral exists as intergrowth or irregular dark patches in or near air holes (see plate 5:12) often hard to distinguish from the glass phase. When spot analysed, the leucite phase has very high levels of silicon dioxide and potassium oxide and relatively low levels of iron oxide (see tables 102 to 113 in Appendix 1).

The final phase that is commonly found in bloomery slags is the dark glass phase. This is the last portion of the slag to solidify and so in it are settled most minor oxides and minerals such as the alkali minerals, from the fuel ash, phosphorous, silica, a little iron, lime and manganese (see tables 63 to 99 in Appendix 1). The glass phase in the bloomery slags often exist as intergrowths between the fayalite crystals. However in most of the slags investigated in this study, the glass phase was found more in and around the gas holes.

The quantity of glass phase in bloomery slags reflect the gangue level of the smelted ore. The more gangue materials the ore contained the higher the quantity of the glass phase. Morton and Wingrove (op cit) in their study of Medieval and Roman iron smelting slags from England claim that the glass phase of the bloomery slags is mostly anorthite mineral $CaO.Al_2O_3.2SiO_2$. But the study of oxide composition and glass phases present in most slag samples analysed in this study show that the calcium oxide CaO, levels in them are too low to support such a conclusion (see tables 57 to 60 in Appendix 1). With the exception of glass phases observed in three slag samples from Owere-Elu (Ow/9/90, Ow/10/90, Ow/11/90) in which the calcium oxide percentage varied between 6.97 wt % and 20.87 wt %, no other sample has a lime percentage above 2.33 wt %. This confirms the views expressed by Fells (op cit:178), Kresten (1986:43) and Todd (op cit) that anorthite mineral is not common in bloomery slags.

4:4 Uses Of Bloomery Slags:

The study of modern blast furnace smelting and their products reveal that most of their products are useful in various aspects of human life. These products include slags from iron smelting. The slags from modern blast furnace have been of various commercial use, as some have been used in the manufacture of railway ballast, tar macadam, cement and slag wool heat insulation (Coghlan 1977:19). But these are slags from modern blast furnaces.

To many people who may come across this thesis, the phrase, 'uses of bloomery slags' will sound strange, since the general understanding of most people about ancient slags is that they are useless and worthless products of early metal working. Even many eminent scholars who have devoted a lot of time and research on the early metal working hold this opinion about bloomery slags. It is not surprising that in the Notes on Prehistoric and Early Iron in the Old World, Coghlan (op cit) wrote, 'In prehistoric days it is probable that no use was made of slags', and Bachmann (1982:1) in The Identification of Slags from Archaeological sites observed that, 'In general terms slags are waste, i.e, discarded or left behind evidence of human activities'. David *et al*·(1989:204) while describing their present research on the early iron smelting in Mafa, northern Cameroon wrote, 'Slag is in general of no use to man or beast but the making of it consumes prodigious quantities of fuel and ore'.

Because bloomery slags are regarded as useless waste products, it has become normal for many prospecting archaeologists to simply report the presence or absence of slags in their areas of research and feel satisfied that that was the most that can be done about this category of finds. There are probably many boxes of unstudied slags locked-up in our museums and collections of individual archaeologists. The revelation of their number will astonish those who know the wealth of information that lurk in these so-called useless objects. If the people who are supposed to know more about slags view them as useless, one will not expect ordinary people or lay people in the field to have a different understanding of the subject.

Contrary to the general understanding, slags are very useful products of iron workers, which like pottery can be used in the reconstruction of the past social, technological and commercial activities of many Iron Age communities. In this respect, as will be seen below, the highest attributes of slags, toughness and durability, have been exploited to great advantage by many Early Iron Age people in various parts of the world. Other attributes of bloomery slags such as their having a very high iron content have also been exploited by a number of communities across the world who used slags as raw material in iron production.

Slags are tough and are almost indestructible. Because of these qualities they have been utilised in building and road constructions. Starting from the area of this research, Igboland in Nigeria, I observed that the foundations of many buildings at Leja, Opi and Ugwu-Ogu in Okigwe were made on slag blocks. This is very common at Afor Opi market, Idi Opi village and Dunoka quarters at Leja (see plate 4:2). In Nsukka Division as in many parts of Nigeria, erosion gullies have been posing many environmental problems to many communities. The people of Idi Opi, Aku, Leja and Ukehe have identified slags as the most adequate material for checking the erosion gullies. They now pile blocks and aggregates of these slags across and along these erosion sites (see plate 4:3). The new express way that joins northern Nigeria and the south through Makurdi and the ninth-mile-corner was completed in 1976. During the construction of this road, the slags at the iron working debris mound at Amube Amalla Orba was quarried by the construction company and used to lay the foundation of parts of this road (Okafor 1984a).

Outside Nigeria, in England, ancient slags have been used in many building projects. For example, the front walls of houses number 2, 4, 8 and 16 along Victoria Road in Sheffield 10 area, were capped with blocks of ancient slags. Also the Crozzle walls along Williams Street in the Don Valley area of Sheffield are capped with these ancient slags. At the Weald of Kent, Coghlan (op cit) and McDonnell (1986:59), reported that bloomery slags were used by the Romans for the foundation of their roads.

In many Iron Age communities, there are social roles assumed by slags. As the most durable direct product of metal production, it has been surrounded by much reverence in some societies while in some, it has become part of the local shrines through which worships and prayers are channelled to God. At Odinanso Idi Opi, slag symbolises honesty, justice and strength. To demonstrate these virtues an accused or a suspect may be expected to swear his or her innocence on a piece of slag. In the village square at Idi Opi, slag shrines are meticulously maintained and revered (see plates 4:4). Besides ritual and religious aspects, slags due to their toughness have been assembled and arranged as permanent etukwu seats, for all general gathering and assembly of the town's local people. This obtains at Leja where Anozie (1979) observed that more than 500 cylindrical blocks of slags arranged at the Otobo Dunoka (Dunoka square) have served many generations of Leja people during their political, religious and ceremonial gatherings (see plate 4:5). Some of the slags at Odinanso Idi Opi square are also serving a similar social need.

Ancient slags have also been useful in the manufacture of metal production refractories. The Early Iron Age people of Madagascar crushed slags and mixed them with clay for the construction of their special smelting furnace known as Ogival-shape furnace (Tylecote 1965:345). A similar use was made of slags by the copper smelting people at Timna. At Timna, the Iron Age copper workers made a modification of their smelting refractories by tempering them with crushed tap slags (Freestone 1989:156). The crushed slags reduced shrinkage and distortion and allowed the escape of volatiles evolving from the clay. At Buhaya kingdom of Tanzania, the ethno-archaeological and archaeological study of early iron production there by Schmidt and Avery ([1978:1086) revealed that large blocks of slags from the former workings were used as foundations for furnace chimney.

Ancient Bloomery slags are characterised by their high iron content. Tylecote (1987:310) observed that about 50% of iron charged in the bloomery furnace is lost in the slag. He observed further that bloomery slags have been resmelted in modern blast furnaces and iron in them replaced by calcium oxide to form calcium silicate. Even in the Early Iron Age period, bloomery slags were excavated and resmelted with profit. For example, in the nineteenth century when the people of Bamenda in northern Cameroon ran short of iron tools, they reverted to traditional iron smelting. But surprisingly instead of their using iron ores which were readily available in the area for the smelting, they went to the Grass Fields of northern Cameroon and excavated slags from their former working and resmelted them. The yield from the resmelted slags was so profitable that the bloom filled the shortfall in their iron supply (Rowlands 1989:276; Warnier and Fowler 1979:337).

In Ethiopia, the Dimi iron smelting community resmelted slags from their former working. The old slags were crushed and mixed with iron ore. This mix was then charged together into the furnace for a fresh smelt (Todd and Charles 1978; Todd 1979). Also in England, for more than twenty years, Yarranton and other people quarried many thousand tons of the Roman slags near the city of Worcester and smelted this in his furnace after mixing them with iron stone from the forest of Dean (Yarranton 1681). Hart (1971) also reported of smelting of old slags in the forest of Dean. About 12 cart loads of ancient slags at Hopyland were carried away in 15th century to Byrkeknott where they were used to modify bloomery iron produced at that site (Tylecote 1960:455).

Besides the direct resmelting of the bloomery slags by iron smelters, the slags have been used as flux in iron smelting furnaces, improving the yield as only the small proportion of iron in the ore would now be required to liquate the slag. In some iron smelting communities like Ola Igbi near Oyo in Nigeria, slags from the former smelts were essential for their smelting operations. They claimed that their smelting industry would cease if there was no old slag available to flux the new smelt. To confirm this point, they disclosed that when they migrated to their present settlement they had to carry with them a sufficient quantity of slags from their former smelts with which to continue the smelting industry (Bellamy and Harbord 1904:111).

Apart from the direct use of ancient slags in building and constructions and as raw material in iron smelting, they are very useful in the reconstruction of extinct technological processes. Many Early Iron Age people have no written records of when their iron smelting industry began, the area covered by the industry, the records of their raw materials: ores, fuel and flux(es) and their sources. There are also no records and descriptions of the supporting apparatus such as the furnace used in the smelting. Most importantly, there are no records of their technological efficiency by way of the level of yield from the smelts, the temperature levels attained in each process and the viscosity of the resulting slag residues which go a long way in determining the efficiency of the industry. The study and analysis of bloomery slags have been found useful in solving these problems.

Engstrand and Ostlund (1962) Krueger and Weeks (1965), Stuiver and Van der Merwe (1968) and Van der Merwe (1969) have demonstrated with success the determination of dates for Iron Age sites through the carbon-14 dating of slags. Van der Merwe (op cit:100) observed that slags are a safe source of carbon for carbon-14 dating. The type of slags recommended for this are slags rich in carbon. Most slag conglomerates entrap unburnt carbon like charcoal which can be extracted with forceps for dating (see plate 3:1). This gives a direct date for the process in question and eliminates doubts and problems of association. Since most woods that provide fuel used in the smelting are sap-woods, the age determined by the carbon-14 dating method is very close to the exact age of the process under consideration.

Some early attempts to determine the source of raw materials used in iron production have involved the study of the trace elements in the artefacts and the ore (Haldane 1970; Hedges and Salter 1979). Inspite of the problems involved in the study of the trace elements, which are often below the detection capacity of the technique employed and are introduced or affected in some way during the process (Tite 1991:143), Tylecote (1970a:21) claims that slags are an adequate substitute for ores as the right material to be analysed for provenance studies. This recommendation was taken by Todd (1976) who, in trying to determine the relationship that exists between the ores and the slags collected at the Dimi in Ethiopia, studied the major and minor oxides in the slag inclusions in the artefacts. But Fells (1983:165) in her study of the structure and constitution of slags, observed that although slag composition is to a great extent determined by the ore composition, it may be affected to a varying degree by contributions from the fuel, flux(es) (if any) and the clay of the refractory materials.

It was considered that variation in slag composition caused by contaminants and processes would significantly affect the results of a provenance study. To overcome this problem, Fells used the study of five non partitioning oxides (Al_2O_3, MnO, TiO_2, MgO and V_2O_5) present in the slags and the ores for the provenance study. In south Africa, Van der Merwe and Killick (1979) made a provenance study of slags by comparing the analysis (ratios) of a number of slags found at the 'Square' smelting site (25km south east of Phalaborwa) with analysis of the various slags and ores from other parts of the Phalaborwa-Gravelotta area. The result of this study using Fe/Ti ratios as well as Ti/Ca ratios for the comparision of the slag groups suggest that the ores used at 'Square' came from an ore body some 20 miles to the east of the smelting site (Friede *et al*. 1982:41).

An important factor that determines the success and efficiency of iron smelting is temperature. The direct determination of the temperature range of any smelting operation was impossible until the invention of the modern instruments like thermocouples. Presently one vital aspect of archaeometallurgy is the determination of the melting temperatures and temperature levels of high temperature processes, which reflect both the efficiency and advances in furnace technology. Slags from bloomery smelting have been used for this temperature determination. This has been achieved in two ways (1) the heat test methods and (2) using phase diagrams.

(1) Various heat tests such as hot-stage microscope technique or leitz heating microscope or a Du Pont differential thermal analysis cell have been used by some scholars to determine the melting temperatures of different slags (Friede *et al*. 1982:42-43). This involves the heating of the powdered slag samples and observing the successive temperatures when the initial sintering begins and the

final melting and disappearance of the last crystalline phase through a thermocouple. The heat test technique has inherent draw backs. Most bloomery slags are dark or often pitch black when cold, and even if completely molten, the thin layer of slag pool on the thermocouple used for the measurement remains opaque. As a result the melting of the last phase may not be clearly evident and may result either in low readings or in readings that are higher than the actual melting temperature of the slag.

(2) The second method is the estimation of the slag melting temperature through the simple phase diagram. In this method the major oxides in the slags analysed are normalised to 100% and plotted on the appropriate phase diagram (Morton and Wingrove 1969a, 1972). Kresten (1986) and Freestone (1989) observed that there are major variations between the estimated melting temperatures and the measured melting temperatures. These discrepancies according to them, are due to the fact that only the major phases in the slags are used in the calculations.

But Morton and Wingrove (op cit) consider phase diagrams very adequate for the estimation of the melting temperature of slags. Since slags form at temperatures below their actual melting points and become free running as the temperature increases; they assert that the estimated melting temperature of slags from phase diagrams is close to the furnace operating temperature. Many people who have done analysis of bloomery slags such as Fells (1983), Friede *et al*. (1982), Todd (1976) and Tylecote (1987) have estimated the liquidus temperature of the slags through the phase diagram. This is the method followed in this research and the melting temperature of all the slag samples investigated in this research have been estimated on a phase diagram. Details of this are in Chapter ~~s~~Six where the individual samples, their analysis and results are discussed.

4:5 Slag-Like Objects:

One of the risks ever present in the study of iron working residues is the increased chance of mistaken identity. Many burnt refractory materials used for bricks or pottery firing have often been mistaken for smelting furnace fragments. But the product of iron working that is most of the time not easily and properly identified is slag. The reason for this is that there are many objects, artificial and natural, which look like slags and have on many occasions been misidentified (Bayley 1985, Biek 1970, Butterworth 1979, Evan and Tylecote 1967, Stanley 1934). Among these pseudo-slags are cinder, fuel ash slag or vitrified fuel ash and natural objects like lava, volcanic rocks such as obsidian and at times hardened-cemented cave soils. The surest way to distinguish these pseudo slags is through physical or chemical analysis. However, because cinder contains some slag which held the components together its chemical analysis do resemble those of true slags to some extent. So to distinguish cinder from the slags, morphological investigations should supplement the chemical analysis.

4:5:1 Cinder

Many people in their writing use the word cinder interchangeably with slag. For example, Morton and Wingrove (1969a:1556) reported the carrying and smelting of cinder from Worcester by Yarranton and other people. They then made a foot note explaining that cinder in this context means slag. This mix up should be avoided. Cinder is cinder and slag is slag and each of them has some special attributes which can be recognised if a little time is spent to examine them properly. Cinder has no distinct composition, and has become a loose generic term for incompletly consumed material in the smelting furnace (McDonnell 1983:83). It is recognised by the individual lumps of ore and partially reduced ore and charcoal fused together by slag. They often exist as drossy solid material on top of molten slag, consisting of a mass of materials infusible at the working temperature of the furnace and so cinder never reached a molten state (Morton and Wingrove 1969a:55). Cinder from iron working operations weathers with increase in years and on analysis they show presence of hydrates to iron oxides that were not present when the cinder was formed (Tylecote 1962:250). Most cinders from iron smelting, because of their conglomerate nature, have generelly uneven angular surfaces.

4:5:2 Fuel Ash Slag Or Vitrified Fuel Ash

The charcoal or wood charged with ore into the furnace burns out and leaves some quantity of ash. At high temperatures that prevail within the furnace, some of the ash combines with siliceous matter from the ore, furnace and tuyere to form fuel ash slag, other wise known as vitrified fuel ash (Tylecote 1987:292). Fuel ash slag occurs both in smelting furnace and smithing hearth and this makes it unreliable evidence of iron smelting site. However, fuel ash slag when found in iron working sites, is more associated with smithing contexts. This is due to the fact that the smelting furnace is a more dynamic system in comparision to the smithing hearth. As the charge in the furnace moves down the furnace, the formation of the fuel ash slag occurs at the tuyere zone where there is higher oxidising condition. But the fuel ash will be consumed by silicate slag as the temperature increases and as the border moves away from the tuyere zone. Following from this, it is probable that lower quantities of fuel ash slag were produced in bloomery smelting than in the smithing hearth (McDonnell 1986:57).

Fuel ash slags are easily recognised by their very low density and multiple gas holes. When analysed they contain very low iron or copper but they have very high silica, phosphate and alkali oxides. Fuel ash slags acquire various colours ranging from white to grey, brown and black depending on the prevailing conditions associated with their formation, (oxidation or reducing conditions) and the temperature at which they were formed.

Fire set on many organic remains deliberately or by accident can produce very high temperatures which causes the vitrification of the organic material. Such vitrified material was reported at Thatswane Hills in Botswana, South Africa. There, the vitrified matter covered several hundreds of square meters and was thought to be smelting slags. The study of these vitrified deposits revealed that they were burnt manure of the South African cattle herders (Butterworth 1979). Their chemical analysis revealed very high silica, phosphate and alkali compounds with very low iron and copper. This demonstrates that the deposit did not result from metal working. A similar find was reported by Zeuner (1959) and Allchin (1963) of how accidental fire on the domestic fuel produced mounds of vitrified dungs and chaff at Kudatini in south India. Although these vitrified materials resemble iron smelting slags morphologically, they are not evidence of iron smelting. They are only evidence of high temperature incidents.

4:5:3 Natural Objects That Appear Like Slag

At times the geological origin or the environment of certain natural objects make them look like slag. For example, Stanley (1934) reported the find made by Dart and Grande in 1931. In an exploration in Rhodesia, Dart and Grande thought that they had found a cave in which iron smelting had been conducted in an ancient times possibly 6,000 years ago 'amidst an old Palaeolithic culture'. The confusion stemmed from a hard vescular, stony lump of dark reddish brown colour object they found in the Mumbwa cave which had a certain superficial resemblance to slags. When Stanley analysed this object, it was discovered to be a cave deposit of sand and silt cemented with calcium phosphate and carbonate (Stanley 1934:507). Other natural objects that are often mistaken for slags are solidified lava and volcanic rocks such as obsidian (Bayley 1985:43). Because obsidian is solidified molten magma from the earth, it has smooth and glassy surfaces like tap slags. However they are distinguished from slags by their smooth sections which contain few or no gas holes, and when chemically analysed, they contain very little iron or copper oxides. From the above, it is evident that much caution should be exercised before an isolated object found in a doubtful context is tagged a slag.

4:6 Refractory Materials.

From an archaeological point of view, refractory materials are all the clay apparatus used in metal working that were able to withstand temperature greater than those normally encountered in a domestic hearth. These apparatus include furnace and furnace lining, crucibles, moulds and tuyere (Freestone 1989:155). In this reasearch, we are concerned with furnace and furnace linings and tuyere. The other refractory materials like crucibles used in the melting and purification of metals and moulds used in the casting of metals and so on, were not involved in bloomery iron smelting (Tite *et al*·1985:50).

Generally these refractory materials, (furnace and furnace lining and tuyere) were made of non-conbustible earth materials like clay, stone or ceramics which contain silicates in one from or another. If these refractories are heated to a sufficiently high temperature, they will melt but the temperatures achieved in the bloomery technology (about 1300 to 1400°C) are well below their melting points. The overall effect of this high temperature is the baking of these refractories to a very hard state which enables them to survive reasonably well. However, often due to other human activities on the sites and the coarse nature of the soil used in making of these refractories, they do not always survive complete.

Although the refractory materials used in the bloomery iron smelting do not melt at the temperature level reached in the bloomery technology, at a temperature of about 1000°C and above, the silicates in their inner surface do react with the ash produced by the fuel in the furnace to form fuel ash slag or vitrified fuel ash which was discussed in section 4:5 of this chapter. Such slag-like silicate materials are not always indications of a particular type of metallurgical process as they were formed within metal smelting as well as within metal smithing operations. At best, they indicate a high temperature incident (Bayley 1985:41).

These refractory materials are characterised by their vitrified or slagged surfaces (Pleiner 1978), as well as by the slags, cinder or fuel ash slags found in association with them. They have no particular colour, but often they become dark-bluish in colour. Furnace linings resemble smithing hearth linings and they are difficult to differentiate. One cannot therefore delimit smelting sites on the basis of the presence of furnace lining alone. Similarly most tuyeres used in smelting operations, morphologically resemble those used in smithing hearths. But while any part of the smelting tuyere can survive fairly well because they were usually baked for longer period at higher temperatures, the parts of the smith tuyere that survive are the nozzle tips which were vitrified (Okafor 1984).

Apart from the use of these refractories as aids in the reconstruction of the smelting processes such as in finding the details about whether slags were tapped or not, or whether the furnace was a forced draft or a self draft; they have been most useful in two aspects of archaeological investigations (1) In the identification of what metal was smelted in a site. (2) In the estimation of the operating temperature of a furnace.

For identification of the smelted metal, a polished thin section cut perpendicular to the refractory ceramic surfaces is examined using scanning electron microscope or electron microprobe. During this examination any slag adhering to the surface is analysed and usually the results reflect the elemental components of the particular metal smelted at the site (Tylecote 1980). Again the working temperature of a particular smelting operation can be estimated through the study of these refractory ceramics. The firing temperature observed with the different degrees of vitrification on the inner surface of the refractory material can be estimated by comparing it with the vitrification developed in samples either taken from the lowest fired parts of the ceramics or taken from mineralogically similar ceramics and refired to known temperatures in a laboratory furnace. Through this technique the maximum temperature reached at the surface of the ceramic when it was in use may be estimated (Tite *et al.* 1985:50).

CHAPTER 5

NSUKKA IRON SMELTING RESIDUES: ANALYSES AND RESULTS

5:1 Introduction:

All the samples used in this research were collected from the four sites Owerre-Elu, Opi, Orba and Umundu (see fig.1:2) which were isolated for this study. The reason for using these sites were discussed in section 1:6, and their description forms the bulk of Chapter three. The study of residues from these sites will form a basis for investigating many other Early Iron Age smelting sites that are found in Nigeria and other parts of West Africa.

Each of these sites was surveyed to determine their dimensions and variations (if any) that exist within them. For sample collection, each of the sites was zoned into residue cluster areas. This was to make sure that all the parts of the site were covered. The number of samples collected from each cluster area depended on the quantity and variety of residues observed in the area. This research aimed at getting reasonable and accurate results. Therefore, as many samples as possible were taken from each site, since it is known that the more accurate the result desired, the more samples should be used (Binford, 1964; Knudson, 1978:319; Rootenberg, 1964). A total of fifty-six slag samples, nine ore samples, three refractory clay samples, three bloomery iron samples and eleven carbon samples were analysed during this research.

To avoid collecting cinder or any pseudo-slag by mistake, all the slag samples used in this study, irrespective of the technique by which they were produced during the smelting, were well formed slags that have smooth surfaces. This indicates that they were completely molten during the smelting (McDonnell 1983:81). Ore samples analysed in this research were collected directly from ore sources discovered through ethnographic investigations in the various iron smelting communities in Nsukka Division (Okafor in preparation a). This approach was adopted because most ores recovered from the smelting sites are either rejected ones or have been treated (concentrated by washing and panning, roasted or smeared with clay to up-grade the gangue matter, (Friede *et al.* 1982:42, Fells 1983:71, 162, McDonnell 1986:56, Tylecote 1975:7, Zacharias and Bachmann 1983:1). So to get as near as possible to the ore smelted at a site, it is better to collect samples for analyses from the outcrop of the ore, where this is known.

5:2 Analytical Techniques:

The main technique used in the analyses of these samples is Scanning Electron Microscopy (S.E.M) linked with a Link Energy Dispersive x-ray microanalyser (EDS). The particular microscope used is a CAMSCAN 2 housed at the Sorby Centre, School of Materials, University of Sheffield. Some researchers have studied iron smelting residues, such as slags, with optical microscope (Knudson 1978:282) or through wet chemical analysis (May 1904:20). Limitations of these techniques when compared with advantages of S.E.M were considered before the decision to use S.E.M was taken.

For example, the S.E.M with EDS has the capacity of detecting elements above about 0.25 wt %. The technique gives very reliable results especially for elements that have their atomic number above ten (Notis *et al.* 1988:36-37; Freestone 1985). The S.E.M has the capacity of detecting major and minor elements in a sample. Major elements in a sample are those that have their wt % greater than 2%, minor elements have their wt % below 2% and trace elements have their wt% less than 0.01% (Tite 1972:201).

Another advantage of S.E.M is that the samples to be analysed require minimum preparation. The samples are only polished scratch-free and carbon and silver coated. The various techniques used in preparing the samples analysed in this research are described in section 5:3. For studies that require quantitative information such as this, the sample should be sectioned and polished scratch-free, while for ordinary general studies, surface coating of the samples may suffice. The S.E.M technique is non-destructive and as a result the sample may still be used for other forms of studies and analyses after the sample has been analysed with the S.E.M. In S.E.M analysis, resolution increases with voltage but for samples like slags and iron, 20 to 30 kV was found to be suitable for all imaging and analysis (Meeks1988:23-29).

Compared with the optical microscope, the magnification range of S.E.M beam exceeds those of the optical microscope which has limit of about x1000. The S.E.M has a far greater depth of field than the optical microscope, at least by 300 times more. The S.E.M high resolution power is far ahead of those of optical microscope being in the range of about 200 angstroms in the S.E.M (Olsen 1988:3). Above all the high quality micrographs revealing various mineral phases in the samples produced by the S.E.M technique are not matched by the optical microscope photos. To crown it all, the EDS attached to the S.E.M gives the elemental composition of the samples from their x-ray spectra. This gives fast qualitative and quantitative analysis of the sample. The optical microscope was only used in this study to check and verify the observations made by S.E.M, especially the mineral phase proportions and their structure.

With regard to wet chemical analysis, the S.E.M is very time saving. The bulk and phase analyses of a sample can be completed within a few minutes, unlike wet chemical technique which can take many hours. Again while S.E.M is non-destructive of the samples, the wet chemical technique requires samples which will be disolved completely before any results can be achieved. Using wet chemical technique only average results of the analysis are achieved since only the major elements in the sample are analysed and nothing is known about the minor and trace elements in the samples (Knudson 1978:282). In fact, Knudson advised against the use of wet chemical techniques in studies where complex materials are to be analysed and detailed results needed. Using S.E.M techniques the various mineral phases in the sample are identified and their analyses related to the bulk area analysis results. This is never possible using wet chemical technique which does not reveal the various phases in the sample. Again x-rays and micrographs of the sample can be obtained using S.E.M technique and this is also not possible in wet chemical technique.

Besides my personal observation concerning the reliability of S.E.M linked with EDS to produce good results in residue analysis, most people who have undertaken iron working residue studies recently have all used S.E.M technique. For example Fells (1983), Goucher (1983), McDonnell (1986), Todd (1976) and Todd and Charles (1978) have all used S.E.M technique in their residue studies and discovered that the technique has great advantage over other techniques and produces reliable results. Each of these studies was able to establish various processes involved in the bloomery slag formation using the S.E.M technique.

In this study, the slags were analysed 'as formed' in the furnace and so only FeO was analysed and no ferric iron was analysed. The reasons for this are (1) the technique used in this analysis (S.E.M) does not detect the valency of iron and so ferric iron was not analysed. If desired this could be determined by wet chemical technique but this will require different samples whose results will be independent of the other analysis results and their necessary phase composition (McDonnell 1986:74). (2) It is known that ferric oxides in slags are a product of conditions prevailing around the slags after they were formed (Morton and Wingrove 1969a:1557; Tylecote 1987:298). Since ferric oxides in slags were not part of the slags initially when they were formed in the furnace, there was no need to investigate them further.

Most bloomery slags direct from the furnace are non -magnetic, but with exposure to atmospheric air and subsequent oxidation process, the FeO in the slag is oxidised to Fe_3O_4 and so this renders the slag magnetic. This situation can also come about as a result of corrosion effects that occur after the slag has solidified and this can lead to secondary oxidation. This aids the formation of hydrated compounds in the slags which again render them magnetic (Tylecote 1987:299). The colour of the slag samples was determined with a Munsell soil colour Chart (MC) and magnetic levels of the slags were assessed using simple magnet tests.

One major characteristic of bloomery slags observed during this study is the variability of bulk analysis results, (see tables 1-56 and 57-61 in Appendix 1). This phenomenon, McDonnell (1986:71) observed is due to the fact that ancient slags are very heterogeneous. This in essence renders a single slag bulk analysis unreliable. To overcome this problem, at least three bulk analyses of each slag sample were made and their mean used for further calculations.

5:3 Sample Preparation:

All the efforts put in the sample preparation were aimed at producing scatch-free surfaces that can be analysed using S.E.M, XRD technique and metallurgical optical microscopes. To produce such standard samples, Meeks (1988) and Claughers (1988) guide lines for preparing samples for S.E.M analysis are very valuable.

The samples (slags, ores, refractory clays and bloomery iron samples) were sliced into thin slices of about 15mm^2 and 3mm thick using a WOCO 50

Universal Cutting Machine fitted with a diamond blade. After the slicing, all the samples were transferred to the school of Materials of the University of Sheffield for subsequent preparations.

At the Sorby Centre of the School of Materials, the samples were mounted on aluminium stubs using loctite 312 and activator NF. This was followed by polishing. The polishing was started on abrasive paper 240, then on abrasive paper 400. Water was used throughout for washing the sample surfaces while cotton wool was used for drying them. After polishing on the abrasive paper 400, the samples were washed with liquid soap and water and dried with cotton wool. The samples were further washed with methanol to disolve any adhering grease and dry the water on the sample surfaces. A hot air blower was used to dry the sample further before polishing was continued on a Kent 3 lapping and polishing machine unit. First, a six micron plate was used for the polishing after it was sprayed with six micron hyprez fluid. The polishing on this plate lasted four minutes. Then the samples were washed with water and liquid soap, dried with cotton wool and then washed with methanol and dried with a hot air blower. The final polishing was done on a one micron plate which was sprayed with one micron hyprez spray diamond lapping compound and hyprez fluid. Again the polishing on this plate lasted four minutes. The samples were washed with liquid soap and water, dried with cotton wool and further washed with methanol and dried with a hot air blower.

At this stage the samples have highly polished surfaces. Conductive adhesive, silver dag, diluted with amyl acetate, was applied on two adjacent points from the polished surface of each sample down to the aluminium stub. The silver dag not only helped to hold the samples on the stubs but eliminated heating and charging of the sample surface by the electron beam. The coating also helped to ensure clear observation of the sample surface.

With the exception of the metal samples, the polished surfaces of all other samples were carbon coated using an Edwards Coating Unit 12E6 to make them conductive. This carbon coating unit applies only a very thin layer of carbon which did not mask the surface. As the samples were subjected to x-ray analyses, this thin carbon layer minimised the absorption of x-rays from the sample. The conductive layer also removed surface charging from the sample and minimised heating of the samples by the electron beam.

At this stage the samples were ready to be analysed. To avoid any surface contamination by moisture and dust, they were stored in a dessicator from where they were removed for any analysis and to which they were returned after the analysis.

5:4 Morphological Examination Of Nsukka Slags:

Iron smelting residues have been discussed in Chapter four. In this section attention is paid specifically to iron smelting residues fron Nsukka. Iron smelting residues recovered from bloomery iron smelting sites group into four broad types, slags, cinder, fuel ash slag or vitrified fuel ash and refractory materials (furnace walls, furnace lining and tuyere). In all the bloomery iron smelting sites investigated in Nsukka Division only three of these residues- slags, cinder and two types of refractory material (furnace walls and tuyere) were recovered. There is complete absence of vitrified fuel ash slag and furnace lining in these sites.

The absence of these two residues, vitrified fuel ash slag and furnace lining in Nsukka iron smelting sites is not surprising. Their survival or disappearance from smelting sites depend on the age of the site, the weather and the subsequent human activities at the site. The alternate hot-humid and torrential storms prevalent in the region, plus intensive human activities in form of farming and construction on these sites mean that there was little chance for their survival, as these factors lead to their fast weathering and disintegration. Again vitrified fuel ash slags are more commonly found in smithing sites than in smelting sites (McDonnell 1986). This is due to the fact that the smelting furnace is a more dynamic system in comparison with the smithing hearth. As the charge in the furnace moves down, the formation of vitrified fuel ash slag occurs at the tuyere zone where there is higher oxidising condition. But the fuel ash will be consumed by silicate slag as the temperature increases and as the load moves away from the tuyere zone (ibid). This accounts in part for the absence of this particular residue in the investigated sites.

Cinder is very commonly found in Nsukka iron smelting sites; but is localised only within smelting furnaces. All the cinders recovered during this research were found close to the floors of the excavated furnaces where they form an amalgam of slag, partially reduced ores and charcoal (see plate 3:1). Whenever they were found in the excavated furnaces they heralded the chance of collecting well preserved charcoal samples. Cinders from Nsukka iron smelting sites have no particular shapes, features, textures or colour. Those recovered from Umundu where there is evidence of water percolation are friable and brittle.

Their colour varies from dark-grey to yellowish-brown. As results from cinder analysis give biased information on the processes that took place on iron smelting sites (Friede 1977:227), care was taken not to include them in the analysed samples.

Slags are the best preserved iron smelting residues in Nsukka Division. There are three forms of iron slag in the Division. These are (1) cylindrical slag blocks (see plate 3:4a), (2) flattish ropy-smooth surfaced slags (see Plate 3:5) and (3) bits of slag aggregates (see plates 3:2 and 3:3). Each of these belong to one of the three phases of Nsukka iron smelting periods, and is found only in the areas covered by the particular phase.

In Nsukka Division, cylindrical blocks of slags are found in Opi, Leja and Aku. These blocks weigh between 43kg and 47kg. They have average density of 3.89g/cm^3. Their colour varies from dark brown (MC 7.5 YR 4/4) to strong brown (MC 7.5 YR 5/8). They have no rust and some of them are slightly magnetic at fresh surfaces. There are no inclusions in these slag blocks.

Before this research was undertaken, it was claimed that the cylindrical blocks of slag were slag bottoms that resulted from pit furnace operations (Anozie 1979). Evidence from the present research does not support this claim. Rather it suggests that these are slags tapped into pits through channels from shaft furnaces. On the external surface of these blocks are visible layers representing individual slag tappings superimposed on one another (see plate 4:1).

The divisions between the layers are not visible with the naked eye at the core of these blocks. But when samples from the core are examined under S.E.M, the layers are seen marked by white bands of magnetite, a product of oxidation on the slag after it was tapped from the furnace. This will be discussed in detail in section 5:5:1 below. See also plate 5:11. The layers are visible on the external surface because of the faster cooling rate and solidification in areas closer to the wall of the pit than at the centre. The slag at the centre was relatively still more molten than that closer to the wall when it was covered by the next batch of slag. Because each layer was formed under different conditions and cooled at differing rates, they have different mineral textures.

Another point that supports the evidence that these are tap slags is that none of the walls of the pits in which these slag blocks are found is slagged or vitrified or has any slag penetration as would be the case if there was any direct smelting in them (Tylecote 1962, 1980, Pleiner 1978:38, Tite *et al*. 1985:50). The walls of these pits are friable and greyish-blue in colour. The colour resulted from the heat of the molten slag. Finally, excavation of these slag pits has never produced any charcoal or other smelting residues. All the charcoal and refractory materials recovered from these sites came from within the furnaces which were surrounded by these slag pits.

The flattish ropy smooth surfaced slags belong to the middle phase of Nsukka iron smelting. They are found along the Nru-Owerre-Elu-Isiakpu area. These are typical tap slags and they survive in flat cake form that weigh between 1.5 and 4.2 kg. They are very dense and have an average density of 4.24gm/cm^3 Some of these slags are weakly magnetic at fresh surfaces. They have no rust or inclusions. They have mixed colours of dark-brown (MC 7.5 YR 3/4) and black all over (MC 7.5 YR 2/10). In section they have bluish-grey colour and are coarsely vesicular near the surface.

Nsukka slag aggregates belong to the late phase of of Nsukka iron smelting. They are found within the Orba-Umundu-Ehandiagu area. The mode of their raking from the furnace and sorting from the bloom makes them survive as amorphous irregular aggregates. They are very vesicular and less dense. The average density of slag samples from Orba is 3.98gm/cm^3 while those from Umundu have an average density of 3.63gm/cm^3. Their colour varies from dark-brown (MC 7.5 YR 3/4) to black all over (MC 7.5 YR 2/10). A few of these slags have some quartz inclusions but they have no rust. None of the slag samples from these sites ~~is~~ are magnetic.

5:5 Microscopic Examination Of Nsukka Slags:

Morphologically Nsukka slags group into three distinct categories as discussed in section 5:4 above. The morphological analysis was done through visual examinations. Microscopic and EDS analyses of slags from these categories reveal more detailed information about them. S.E.M is the major technique employed in the analysis of these slags. The reasons and the advantages of this technique over all other possible techniques were discussed in sections 1:6 and 5:2 above. Because bloomery slags are highly variable, at least three bulk analyses were conducted on each sample and the mean of the results used for calculating the basicity, viscosity coefficient and normalised values of the major oxides plotted in the phase diagrams. Various mineral phases in each sample were also analysed with the spot mode of S.E.M and EDS.

Digipad program of S.E.M was used to work out proportions of mineral phases in each sample. This relies on the grading of various grey levels exhibited by the mineral phases. During the analyses it was observed that some of the grey levels were very close to each other, and this made it difficult to accurately measure the proportions of the phases with similar grey levels. The proportions of the minerals worked out with the digipad program were used as a starting point for a better estimate of the minerals in each sample. Examination of the results of the bulk analyses of all the slags from Nsukka show that about 95% of each of the slag samples was made up of FeO, Al_2O_3 and SiO_2. A computer program was therefore written which normalised the components of these oxides to 100%. The proportions of the constituent mineral phases in each sample were estimated from the overall composition and the composition of the individual phases using the computer program.

The program estimated the proportions of the mineral phases using the analysis of the three constituent oxides, (FeO, Al_2O_3 and SiO_2) in each of the mineral phases of the samples and the densities of the main mineral phases in Nsukka slags, (fayalite, hercynite, wustite and glass). It then calculated the proportions of the mineral phases that gave rise to the overall composition of the slag. In almost all the samples analysed, the calculated phase percentage equals the measured phase percentage except in three samples where it did not work out perfectly due to the peculiar nature of the samples themselves. These anomalous samples are discussed in section 5:5:1 below.

It is realised that this program provided only an approximate phase proportion as not all the oxides in the samples were used and since only the major mineral phases were estimated. But the aim was to test whether the observed phase proportions from the digipad program were consistent with the phase and the sample analyses. In most of the samples the greys grade into one another or overlap and made separation difficult often giving misleading results.

Checks were made on the estimated phase proportions by making grid counts of the mineral phases in the photomicrographs of each slag sample. In all, the estimated proportional percentage of each mineral agrees well with those worked out with the grid count. Summaries of the mineral phases and their proportions are in tables 118 and 126 in Appendix 1. Figures 5:1, 5:2, 5:3 and 5:4 contain histograms of mineral phases in Nsukka slags. The composite plots of the minerals in Nsukka slags are shown in figure 5:5, and will be discussed in chapter Six.

5:5:1 Constitution Of Nsukka Slags:

The composition of bloomery slags has been discussed in section 4:3. Results of the analyses of Nsukka slags reveal that they are essentially fayalitic. Fayalite mineral make up more than 60% of the samples (see fig. 5:1). This agrees with the findings of Fells (1983), McDonnell (1986) and Todd (1976) which show that bloomery slags are mostly fayalitic. FeO, Al_2O_3 and SiO_2 are the major oxides in Nsukka slags. These three oxides make up more than 95% of each of the slags studied during this research. Other oxides in Nsukka slags are MgO, TiO_2, MnO, P_2O_5, CaO, S, V_2O_5 and K_2O. The levels of these eight oxides are low and varied in Nsukka slags (see tables 57 to 60 in Appendix 1). At times some of them were not detected in some samples.

Nsukka slags are characterised by their very low levels of CaO which led to low basicity of the slags. The mean and S.D for CaO in the studied slags are 0.75% S.D 0.34 for Opi, 0.39% S.D 0.25 for Owerre-Elu, 0.37% S.D 0.19 for Orba, and 0.49% S.D 0.23 for Umundu. Analyses of Nsukka iron ores produced similar low levels of CaO (see table 62 in Appendix 1). The CaO content of Nsukka iron ores was never above 0.02%. This suggests that the slags derive from ores similar to those analysed since it is the nature of CaO in the furnace charge to precipitate in the slag (Todd 1976). The low CaO levels in Nsukka slags show that no flux rich in lime was used in Nsukka iron smelting. This is in line with other bloomery operations; as yet no concrete documentation has been made for the use of lime as flux in bloomery iron smelting (Fells 1983:132).

Another characteristic of Nsukka slags is the moderate levels of TiO_2 found in them. The mean and S.D for this oxide in Nsukka slags (see tables 57-60 in Appendix 1) are Opi mean 1.15% S.D 0.13, Owerre-Elu mean 1.28% S.D 0.18, Orba mean 1.65% S.D 0.24 and Umundu mean 1.47% S.D 0.39. Again this compares with the levels of Ti in iron ore samples from Nsukka. The mean and S.D of Ti in Nsukka iron ores are for Opi 1.32% S.D 0.09, for Owerre-Elu 1.14% S.D 0.43, for Orba 1.45% S.D 0.02 and for Umundu 1.54% S.D 0.06.

Nsukka slags have also high Al_2O_3. The high Al_2O_3 levels in all the slags from the Division fit them perfectly into the $FeO-Al_2O_3-SiO_2$ ternary phase diagram as FeO, Al_2O_3 and SiO_2 are the three most abundant oxides in all the analyses (Fells 1983:82) (see tables 57 to 60 in Appendix 1). The mean and S.D for Al_2O_3 in Nsukka slags are for Opi 14.02% S.D 2.76, for Owerre-Elu 9.06% S.D 1.24, for Orba 11.18% S.D 1.41 and for Umundu 10.39 S.D 1.92. Nsukka iron ores are of similarly high levels. The mean and S.D of Al in Nsukka iron ores are Opi: 4.18% S.D 0.86, Owerre-Elu: 4.78% S.D 0.99, Orba: 5.52% S.D

0.08 and Umundu: 6.25% S.D 0.40. Al_2O_3 in the furnace charge does not partition between the iron and the slag, it precipitates in the slag (Fells 1983:132). So Al_2O_3 in the slag reflects the level in the ore. Nsukka slags have consistently low levels of S, V_2O_5, MgO, MnO, K_2O and P_2O_5. Their levels vary between slag samples and sites.

Mineral phases in these slags were identified with EDS linked with S.E.M. The analyses show that there are three major mineral phases in Nsukka slags. These are fayalite, hercynite and wustite. There are minor phases which appear with these major phases in some of the samples. These are glass, leucite and thin bands of magnetite (see figs. 5:6). The structure and texture of these minerals vary within and between sites. Factors responsible for the changes in the mineral structure of slags are discussed in section 6:4 below. The observed mineral structures in Nsukka slags are grouped and discussed below.

The most abundant mineral phase in Nsukka slags is iron silicate-fayalite $2FeO.SiO_2$ (see table 126 in Appendix 1 and fig. 5:1). EDS spectra of this phase show that it is mostly Fe, Si and Mn (see fig. 5:6a). S.E.M spot analyses of the same mineral show that it contains FeO and SiO_2 and very low Al_2O_3 (See tables 63 to 113 in Appendix 1) and is very near to pure fayalite in composition (Deer *et al*. 1985:4). In photomicrographs fayalite phase has a grey or light-grey colour (see plate 5:1). Four structural forms of fayalite are identified in Nsukka slags. These are (1) massive fayalite crystals, (2) broken lath fayalite crystals, (3) lath fayalite crystals and (4) fine tiny fayalite crystals.

Massive fayalite crystals are found mostly in slag samples from Opi. Except samples Op/8/90 and Op/9/90 all other slag samples from Opi have massive fayalite crystals. Half of the slags from Owerre-Elu have this structure. These are Ow/2/90, Ow/3/90, Ow/4/90 Ow/5/90, Ow/11/90 and Ow/12/90. Only three slag samples Um/6/90, Um/7/90 and Um/14/90 from Umundu have this massive fayalite. None of the samples from Orba ~~has~~ have this structure. The massive fayalite crystals are associated with angular dark grey hercynite. The fayalite crystals are only broken by pools and patches of glass or leucite and vesicles (see plate 5:1).

The broken lath fayalite crystals in Nsukka slags precipitate in a glass matrix and are associated with dark grey hercynite crystals (see plate 5:2). Such fayalite crystal structures are found in samples Op/8/90, Op/9/90, Ow/1/90, Ow/6/90, Ow/7/90, Ow/9/90, Ow/10/90, OR/7/90, OR/8/90,OR/11/90, OR/12/90, Um/8/90, Um/12/90, Um/15/90, Um/16/90 and Um/17/90. Lath fayalite crystals in Nsukka slags also precipitate in glass matrix. They exist in parallel or sub-parallel bundles (see plate 5:3).

Such lath fayalite crystals are found in samples Ow/8/90, OR/9/90, OR/10/90, OR/13/90, OR/14/90, OR/15/90, OR/16/90, OR/17/90, OR/18/90, Um/9/90, Um/10/90, Um/11/90 and Um/13/90. No slag from Opi has fayalite of this structure. The fine fayalite rods are found in glass matrix interlacing with the euhedral fayalite crystals. The fine fayalite rods are generally anhedral and perhaps represent the last stage of crystalization before solidification in the glass. The fine fayalite rod are common in the slag samples with broken lath and lath fayalite crystals (see plate 5:4). Few are however present in the glass of some samples with massive fayalite crystals as in samples Op/17/90, Op/18/90 Um/7/90 and Um/14/90.

Volumetric calculations of minerals in Nsukka slags show that fayalite phase make up 76.42% of the mineral phases in Owerre-Elu slags, 65.6% of the mineral phases in Opi slags, 61% of the minerals in Orba slags and 60.25% of the mineral phases in Umundu slags (see table 126 in A~~a~~ppendix 1).

The next dominant phase in Nsukka slags is hercynite FeO.Al$_2$O$_3$. EDS spectra of hercynite show that it consists essentially of Fe, Al, Ti, Cr and Mn (see fig. 5:6b). S.E.M spot analyses of hercynite also show that it is made up mostly of FeO and Al$_2$O$_3$ with very low SiO$_2$ and TiO$_2$ (see tables 63 to 117 in A~~a~~ppendix 1). The composition of this phase is not stoichiometric FeO.Al$_2$O$_3$. Hercynite crystals in Nsukka slags have dark-grey colours, and some contain light-grey zones in the periphery (see plate 5:5). The zoned areas are generally rich in FeO and TiO$_2$ when spot analysed.

Hercynite crystals in Nsukka slags appear in two structural forms (1) euhedral and (2) anhedral structures. Large bold euhedral crystals of hercynite precipitate in samples with massive or broken lath fayalite as in samples Op/19/90, Ow/10/90, OR/7/90 and OR/8/90 (see plates 5:6a-b). Some hercynite occur in glass as in sample Um/15/90 (see plate5:6b). In slag samples with parallel or sub-parallel lath bundles of fayalite, are found few crystals of small euhedral crystals and anhedral crystals of hercynite that formed in the glass or fayalite. This is evident in samples OR/9/90, OR/10/90, Um/11/90 and Um/13/90 (see plate 5:7) In some samples such as OR/7/90 OR/8/90 and Ow/1/90 the two forms of hercynite (bold euhedral and dispersed anhedral crystals) are found formed in the broken lath crystals of fayalite (see plate 5:2).

Volumetric calculations of this mineral show that it is most abundant in Opi slags where it constitutes 22.47% of the minerals in the slags from that site. It seems that the considerable levels of TiO_2 in some hercynite phase in Opi slags contributed to the high percentage of this mineral in Opi slags, as the zoned areas of the phase contain considerable amount of TiO_2. This is followed by Owerre-Elu slags where hercynite constitutes 16.17% of the minerals. Umundu slags have 12.42% and Orba slags have 11.5% of their minerals made up of hercynite.

Free iron oxide, wustite is found in some slags from Nsukka Division. This mineral is however not common in all iron smelting sites in Nsukka. Only 33% of all the slags analysed during this research contain wustite mineral. There is no wustite in all the slags from Orba and Umundu iron smelting sites. Out of the twelve slag samples studied from Owerre-Elu, only five samples (Ow/2/90, Ow/3/90, Ow/5/90, Ow/10/90 and Ow/11/90) have wustite. The wustite mineral is most abundant in Opi slags. Only three slag samples Op/5/90, Op/6/90 and Op/8/90 have no wustite, the remaining twelve samples studied from Opi sites have this mineral.

EDS spectra of wustite phase show that it is mostly Fe with little Al, Si and Ti (see fig. 5:6c). S.E.M spot analyses of wustite show that it has more than 90% FeO. Al_2O_3, SiO_2, and TiO_2 are the main impurities in the wustite phase. These impurities are consistently low constituents of all the wustite phases analysed during this research. The wustite in Nsukka slags exist as white dendrites that are found in glass and or fayalite matrix. Few of the wustite dendrites are very fine and these form in the massive fayalite crystals as in samples Ow/2/90, Ow/3/90, Ow/11/90, Op/7/90 and Op/11/90 (see plate 5:8).

The majority of the wustite dendrites in Nsukka slags are thick or rounded as in Ow/2/90, Ow/10/90, Op/19/90, Op/7/90 and Op/10/90 (see plate 5:9). They form in light grey massive fayalite crystals associated with dark grey angular hercynite crystals. A few of the slag samples have elongated wustite dendrites as in samples Ow/11/90, Op/14/90 and Op/15/90 (see plate 5:10) The elongated wustite is associated with massive fayalite crystals, bold euhedral hercynite crystals and pools of dark glass. In very few samples the wustite dendrites exist as isolated globules in the massive fayalite crystals or in the glass pools as in samples Op/11/90 and Op/19/90 (see plate 5:9).

As the free iron oxide, wustite, is the penultimate stage in the reduction of iron ore to metal iron (Morton and Wingrove 1969a:1557), the level of wustite in the slags is assumed to indicate the degree of efficiency of the particular bloomery operation. The higher the free iron oxide content of a slag the less efficient the smelting operation (McDonnell 1986:86). This means that the level of wustite in bloomery slags will vary according to the skill of the operators to work the furnace so that minimum wustite be left in the slag. Improvement in technique with time would account for increasing efficiency and less wustite in the slag (Morton and Wingrove 1972:480). Based on this assumption, it would seem that Nsukka iron smelters had a high and efficient technique especially during the latter phase of the industry represented at Orba and Umundu. As noted above the wustite phase is totally absent in slags from Orba and Umundu. It constitutes only 6.2% of the mineral phases in Opi slags and only 3.92% of the mineral phases in Owerre-Elu slags.

In four of the tap slags from leja and Owerre-Elu were observed white bands of iron oxide, which based on the information from published works of other researchers, were assumed to be magnetite, Fe_3O_4 (see plate 5:11). EDS analyses of the bands show that they consist of Fe, and Ti (see fig. 5:6d). Magnetite found in bloomery slags results from oxidation which took place as the molten slags left the reducing condition in the furnace (Morton and Wingrove 1969a, McDonnell 1986:86), hence their restriction on the chilled surfaces of the slags (Fells 1983:108). Since all the slags used in this research were analysed 'as formed' in the furnace, the observed magnetite which resulted after the slags were formed were not considered further. However, its presence in some of the slags was helpful in confirming that the slags were molten when they were formed and in proving that the slags were tapped (Fells Op cit, McDonnell Op cit).

Thirteen slag samples from Opi (Op/6/90, Op/8/90, Op/9/90, Op/10/90, Op/11/90, Op/12/90, Op/13/90, Op/14/90/, Op/15/90, Op/16/90, Op/17/90, Op/18/90 Op/19/90) contain minor mineral phase leucite, $K_2O.Al_2O_3.4SiO_2$. No other site in Nsukka has slags that contain this mineral. It constitutes 5.07% of the minerals in Opi slags. This phase exists as dark intergrowths or irregular dark patches very hard to separate from the glass (see plate 5:12). They contain white dendrites of wustite in massive fayalite crystals associated with dark grey euhedral hercynite crystals. When spot analysed, they revealed high contents of K_2O, SiO_2 and Al_2O_3 and relatively low levels of FeO. EDS analyses of leucite show that it consists of Si, K, Al, and little Fe (see fig. 5:6e).

The presence of this mineral in some slags from Opi is surprising. The analyses of iron ores from Opi show very low levels of K_2O (mean 0.04% S.D 0.02). Besides the ore, the other possible source of K_2O for the slags is fuel (the charcoal) and perhaps the refactory materials. But as this mineral is found only in some slags from Opi, it is possble that the iron smelters at Opi conducted some of their smelts with charcoal that has high potassium content which precipitated in the slag. Fells (1983:131) observed that K_2O content of fuel ash generally precipitates in the slag.

Another minor mineral phase in Nsukka slags is glass. The glass phase exist in two forms in Nsukka slags (1) as dark pools and (2) as dark matrix containing other phases. Pools of glass are found in slags that contain massive and broken lath fayalite crystals. They are found in or near air holes in these slags. Some of the slags that contain such forms of glass are Ow/2/90, Ow/3/90, Op/5/90 and Op/7/90 (see plate 5:1). Slag samples that contain parallel or sub-parallel lath crystals of fayalite and lots of fine fayalite rods have glass matrix (see plate 5:3). Glass matrix is common in slags from Orba and Umundu. Tiny rods of fayalite are found in the glass matrix, though some glass pools contain few of such tiny fayalite rods as in samples Um/7/90 and OR/9/90 (see plate 5:4).

Both EDS and S.E.M analyses of glass phase in Nsukka slags show that they are the last phase to crystalize, hence the precipitation of most alkali, lime, phosphorous and excess silica in them (see fig. 5:6f). The quantity of glass in slag depends on the quantity of alkali, lime, phosphorous and how siliceous the smelted ore was (Tylecote 1987). Results of Nsukka ore analyses show that they contain very low K, Ca. P and moderate Si (see table 62 in Appendix 1). This was perhaps responsible for the low glass contained in Owerre-Elu and Opi slags. Umundu and Orba slags have high siliceous glass. This resulted from the excess silica in the slag. In Umundu slags, glass constitute 27.33% of the mineral phases, while in Orba slags it constitutes 27.25% of the minerals. In Owerre-Elu and Opi slags glass constitute 3.42% and 0.8% of the mineral phases repectively.

During the analyses of the slags, samples Ow/12/90, Um/11/90 and Op/9/90 produced anomalous results. Sample Ow/12/90 contains 85% massive fayalite crystals, angular dark grey hercynite crystals and only 1% dark pools of glass (see plate 5:13). Because of the very low level of glass in this sample it was difficult to prevent the x-ray from interfering with the glass analysis. This resulted in the glass analysis of the sample having very high FeO (73.06%) and too low SiO_2 (7.87%) (see table 74 in Appendix 1). During the phase calculation it was difficult to use this result of the glass analysis to arrive at the average of its bulk analysis. This was overcome by calculating and using the average results of the other glass analyses from Owerre-Elu.

In the next anomalous sample Um/11/90, the hercynite phase is in the form of anhedral crystals which are found in bundles of light grey lath fayalite and dark glass (see plate 5:14). It was difficult to analyse any of the phases without the x-ray source penetrating other phases. Thus during the analysis of the fayalite, x-rays from hercynite gave an unusually high level of Al_2O_3 (8.26%) in the fayalite analysis. Also x-rays from the glass during the hercynite analysis gave the unusually high level of SiO_2 (10.46%) in the hercynite analysis (see table 92 in Appendix 1). As in sample Ow/12/90, the average results of all the other fayalite and hercynite phases from Umundu were calculated and used for determining the phase proportion of sample Um/11/90.

Sample Op/9/90 is the other anomalous sample (see plate 5:15). Some of the hercynite in this sample is in the form of anhedral crystals which are found in the fayalite and glass. This resulted in x-rays from the hercynite contributing to the fayalite analysis. This gave high Al_2O_3 (7.98%) in the fayalite analysis (see table 103 in Appendix 1). As in the other two anomalous samples above, the average results of other fayalite analyses from Opi were calculated and used for Op/9/90 phase calculations.

5:5:2 Varying Features Of Nsukka Slags:

Various analytical results from this research reveal some variations within the slag samples from Nsukka. Some of these variations exist between slags from the same site while some are found between slags from different sites. Results of morphological analyses of Nsukka slags show that they fall into three distinct groups (1) the cylindrical blocks (2) the flat smooth ropy surfaced slags and (3) the slag aggregates. The form of each of these groups of slags derive from the mode of their removal from the furnace.

The cylindrical slag blocks found within Opi and the related sites assumed their form from the slag pit into which they were tapped. The flat smooth ropy surfaced slags from Owerre-Elu-Nru-Isiakpu area were allowed to spread on the ground as they left the furnace chamber, hence their flat features. The slag aggregates found in Orba and Umundu derive their feature from the fracturing of the raked slag from the smelting furnace in order to separate the blooms from the residues. Evidence from these slags suggest that the form of bloomery slags are not dependent on the furnace used (since all Nsukka iron smelting were conducted in various forms of shaft furnace), but are dependent on the mode of their removal from the furnace and the treatment they were subjected to immediately after leaving the furnace.

Examination of these slags reveal further variations within them. The most obvious variation is the presence or absence of wustite in some of the slags. Only 33.33% of all the slags analysed contain wustite. Orba and Umundu slags differ from other Nsukka slags by not containing any wustite. The reasons for the absence of this mineral in Orba and Umundu slags is discussed in section 6:6 below. Only five samples from Owerre-Elu contain wustite (see table 126 in ~~a~~APpendix 1). It is only in Opi slag samples that wustite is found in all but three samples. On the whole, the relatively low level of free iron oxide in Nsukka slags can be seen as evidence of efficient smelting (Morton and Wingrove 1972:478).

There are considerable variations in the level and form of glass in Nsukka slags. Owerre-Elu and Opi slags contain low levels of glass. These are found as pools and patches close to the air holes in the slags. Only two samples from Opi (Op/5/90 and Op/7/90) contain glass which constitute 1% and 3% respectively of the minerals in the slag samples. Analyses of glass in the two samples show that they are highly siliceous with SiO_2 contents of 69.92% and 46.39% repectively. The glass in Op/7/90 contain high P_2O_5 (9.42%) and CaO (18.35%). These do not reflect the low levels of these two oxides in the bulk analysis of the same sample which shows P_2O_5 as constituting only 0.84% and CaO 0.43% (see table 60 in ~~a~~Appendix 1). The possible explanation for this is that the high levels of these two oxides in the glass (of Op/7/90) derived from the fuel ash which precipitated in the glass (Todd 1976). In Owerre-Elu samples, glass constitutes 3.42% of the minerals while in Opi it constitutes only 0.80%.

Three slag samples from Orba and Umundu (OR/7/90, OR/8/90 and Um/6/90) share in this characteristic of containing low glass. Sample OR/7/90 contains 9% glass, OR/8/90 has 17% glass while Um/6/90 has 5% glass. Apart from these three samples all other remaining twenty-one slags from Orba and Umundu have high glass contents that range between 20% and 44%. In the slags that have high glass content, the glass forms the matrix for other minerals. Glass constitutes 27.25% of the minerals in Orba slags and 27.33% of Umundu slag minerals.

It was observed above that the level of glass in the slags derive from the alkalis, lime, and phosphorous in the ore plus any excess silica in the smelt.

Nsukka iron ores being mostly haematite, are workable with bloomery techniques as they contain low levels of gangue material. The mean and S.D for silica in Nsukka iron ores are Opi mean 16.6% S.D 4.5, Orba mean 6.41% S.D 0.12, Owerre-Elu mean 10.08% S.D 2.44 and Umundu mean 8.71% S.D 2.0. The smelting of these ores without any flux led to the production of slags that contain very low glass. This is evident in Opi and Owerre-Elu slags. In Orba and Umundu smelts where sand was used as flux, the slags contain high levels of glass which resulted from the excess SiO_2.

Thirteen slag samples from Opi contain leucite (see table 126 in Appendix 1) which constitutes 5.07% of the minerals in Opi slags. This mineral just as glass, exists in pools and patches in and near the air holes. Opi slags are the only ones from Nsukka that contain this mineral. Apart from its high K_2O, SiO_2 and Al_2O_3 contents they are remarkably low in FeO (see tables 102 to 113 in Appendix 1). As observed in section 5:5:1, the presence of leucite in some Opi slags probably derived from the charcoal used by the smelters since the level of K_2O in Nsukka ores is generally very low (see table 127 in Appendix 1). This is more probable since K_2O content of fuel ash generally precipitates in the slag (Fells 1983).

The very low levels of CaO in Nsukka iron ores (see table 62 in Appendix 1) and the non-use of lime as flux during the smelting led to the production of slags that have very low acidic/basic oxides ratios hence the too low basicity for Nsukka slags. On the whole Nsukka slags have very low basicity. The mean and S.D of basicity calculated for slags from Owerre-Elu are mean 0.02% S.D 0.01, for Opi slags mean 0.03% S.D 0.01, Orba slags mean 0.02% S.D 0.01 and Umundu slags mean 0.02% S.D 0.02.

The melting temperature range estimated from the ternary phase diagrams for slag samples from Nsukka, (1150°C to 1450°C) (see tables 57-61 in Appendix 1 and fig. 5:5) is within the possible temperature achieved in bloomery technology (Bachmann 1982:19). However, there are variations in the melting points of some slags from different sites in Nsukka. Slag samples that have lower contents of Al_2O_3 and hercynite have their melting points ranging between 1150°C and 1280°C. But those that contain high levels of Al_2O_3 and hercynite, (see table 126 in Appendix 1) have high melting points that range between 1310°C and 1450°C. This is evident in most slags from Opi and two from Owerre-Elu. Their hercynite contents range between 21% and 34% (see table 126 in Appendix 1). Pure hercynite mineral has a high melting point of 1780°C (Deer *et al.* 1985), and the hercynite would be the first solid phase to form in the slag.

To liquate the high hercynite gangue, it was necessary to increase the temperature in the furnace, hence the high temperature associated with these slag samples. The consistent low melting points achieved for slags from Orba and Umundu resulted from the use of sand as flux to lower the melting points of the gangue. Ten Owerre-Elu and six Opi slags that have low melting points that range between 1150°C and 1280°C (see tables 57 to 60 in Appendix 1) result from their lower hercynite contents that never exceeded 18% (see table 126 in Appendix 1).

5:6 Refractory Materials From Nsukka Iron Smelting Sites

Refractory materials recovered from archaeological sites have been described in section 4:6. In this chapter attention is concentrated on the description of the three tuyere refractory samples (OP/2/90, OR/1/90 and OW/16/90) from Opi, Orba and Owerre-Elu and the results of the analyses they were subjected to. Visual examination of the three samples and other tuyere fragments from the sites show that they contain some twigs or straw which were added to either strengthen the earthen raw material or to lessen their plasticity. This appears to be a common technique among iron smelters in Nsukka Division.

Ethnographic data collected from Orba and Umundu blacksmiths (Okafor in preparation a) show that the practice of tempering puddled earth with straw or grass is still in vogue. However, some of the blacksmiths use palm nut fibre to temper the earth they use in making the tuyere. The ethnographic data also revealed that the earth used for tuyere construction was the locally available earth, and not any special type of earth or clay.

Results of analytical investigations conducted on the three refractory samples from Opi, Orba and Owerre-Elu show that they operated above their normal endurance temperatures. This resulted in bloating, massive soaking with slags and vitrification of the tuyere zones that were in contact with the slag in the furnace (see plate 5:16). **Sample OP/2/90** is a highly vitrified tuyere fragment recovered at a point 160mm below the surface on the floor of smelting furnace "A" at the foot of Opi hill (Okafor in preparation b). It was sealed by slags and compact lateritic soil.

A polished thin section removed from this sample was analysed using scanning electron microscope linked with EDS. EDS analysis of the sample shows that it consists of Si, Fe, Al and Ti. S.E.M photomicrographs of the samples revealed three phases. (1) Bloated dark grey quartz crystals and two slag phases. The bloated quartz phase is surrounded by slag zone that soaked the tuyere. Two phases observed in the slag are iron silicate (fayalite) which appears in dendritic bright colour; and the glass which is light grey in colour. Results of spot analyses of these three phases are in table 120 in Appendix 1.

The S.E.M results confirm the visual observations made on the sample i.e. that it was soaked by slag and that it was vitrified. The results of the analyses, especially the analysis of the slag inclusion, are similar to the results of phase analyses of slags from the same site (see tables 120, and 99-112 in Appendix 1). All these results show low CaO, MgO, MnO and K_2O.

Sample OR/1/90 is friable on the external surface, but the internal surface is hard and contaminated by slag. It was recovered from the trial excavation at the smelting site in Agu-Amaorba. The sample was collected at a point 120mm below the surface. It was covered by slags, charcoal and lose earth.

A polished thin section from the sample was analysed with EDS and it revealed that the sample consists of Si, Fe, Al and Ti. S.E.M. photomicrographs of the sample reveal that it consists of bloated quartz crystals which are dark grey in colour and dendritic iron silicate. The quartz crystals were highly affected by the heat and this produced some cracking in the bloated area. Results of the spot analysis of the two phases (quartz and slag) are in table 121 in Appendix 1.

Sample OW/16/90 is a vitrified tuyere fragment from a smelting debris heap behind the timber shed. It was mixed with flat pieces of ropy slags. A polished thin section from the sample was analysed with EDS and it revealed that the sample consists of Si, Fe, Al, Ti and K.

S.E.M. photomicrographs of the sample revealed three phases which consist of dark grey bloated quartz, bright iron silicate phase (fayalite) and light grey hercynite. Results of the spot analyses of these phases are in table 122 in Appendix 1. There are areas of void which are dark in colour. These resulted from burnt out straw temper.

Further tests to obtain the operating temperatures of the furnace by heat tests on the tuyere fragments were not considered necessary because the positions and points of contact of the tuyere fragments are not known. Since all the samples are in fragments it is not known if the samples were inside, outside or at the edge of the furnace when the smelting took place. With this doubt, one cannot be definite that the temperature derived from such tests will reflect the furnace operating temperature (personal communication, Bayley 1991).

Nevertheless, the deep vitrification and soaking by slags observed on the samples suggest that they were affected by a very high temperature. Experiments on smelting ceramics suggest that temperature range of 1100-1250°C was necessary for vitrification to occur in the ceramics (Craddock *et al.* 1985) and experimental studies indicate that temperatures in the centre of the furnace were higher than those at the wall by 100-300°C (Tylecote and Merkel 1985). Experiments conducted in simple shaft furnaces by Nosek (1985) show that temperatures of 1300-1400°C have been attained in bloomery smelting. This covers the temperature range estimated for Nsukka slags, especially those from Opi. In an attempt to liquate the Al_2O_3 rich iron ores without fluxing, the smelters reached high temperatures of 1350-1450°C which led to the high vitrification, bloating and soaking of the tuyere by slags.

5:7 NSUKKA IRON ORES:

Nine iron ore samples from the four sites studied during this research were analysed. As was discussed in section 1:6, the ores were collected from the various ancient iron ore mines located through ethnographic investigations conducted in the Division. The iron ores were analysed in order to establish the particular type(s) of iron ores used by Nsukka early iron smelters. This is necessary since there is as yet no concrete evidence that pin-points the type of ore used by Nsukka bloomery iron smelters.

Three principal techniques were used for the ore analysis. These are (1) XRD technique which established the particular minerals that are present in each of the ore samples. (2) wet chemical technique which revealed the elemental constituents of the ores and (3) S.E.M linked with EDS which established the qualitative and quantitative levels of the constituent oxides. Results of these analyses are in tables 62 and 127 in Appendix 1.

Two iron ore samples (OP/3/90 and OP/4/90) were collected from the two ancient iron ore mines at the base of Opi hill. XRD analysis of the two samples show that they are essentialy haematite and quartz. EDS analysis of the samples show that they are made up of Fe, Si, Al and Ti. Their minor element constituents are Mg, P and Ca.

S.E.M photomicrographs of the samples (see plate 5:17) reveal three phases. These consist of dark quartz crystals in light in colour iron oxide matrix. Few dark grey clay minerals consisting mainly of Al, Si and Fe are also in the iron oxide matrix. There are few holes that are dark in photomicrographs observed in these samples.

Two iron ore samples (OR/5/90 and OR/6/90) were collected along the dried water valley at Agu-Amaorba smelting site. These two were also analysed with XRD technique. The results of the analysis show that the samples are essentially goethite with little haematite. EDS analyses of the samples show that they consist of Fe, Si, Al and Ti. As in Opi ores,S.E.M photomicrographs of the samples revealed three phases. These consist of light in colour iron oxide matrix which contains grey clay minerals and quartz crystals. The clay minerals have high Ti level.

Three samples (OW/13/90, OW/14/90 and OW/15/90) were collected at 100 metre intervals from the eastern end of Owerre-Elu hill to its western end. Analyses of the samples with XRD technique show that they consist of haematite and little quartz. EDS analysis of the samples shows that they consist of Fe, Si, Al and Ti. S.E.M analysis of the sample revealed three phases. These consist of grey clay minerals in light grey iron oxide matrix and few quartz crystals. There are few dark angular quartz crystals in the iron oxide matrix.

From Umundu iron ore valley, two ore samples (UM/4/90 and UM/5/90) were collected. XRD analyses of the samples show that sample UM/4/90 is essentially haematite and quartz while sample UM/5/90 is a combination of haematite and goethite. EDS analysis of the samples show that they consist of Fe, Si, Al and Ti. S.E.M analysis of the samples show that they consist of three mineral phases. These are made up of light grey iron oxide matrix containing dark grey clay minerals and dark angular quartz crystals.

Results of these analyses closely reflect the geology of the area. The entire Nsukka Division is on a common geological setting (see section 2:2). This shows in the close similarity of the results of the various iron ores analysed in this research (see tables 62 and 127 in Appendix 1). Orba and Umundu iron ores consist of goethite and haematite. This reflects the micro-environments of these two iron ore mines. The two ore mines are within water valleys (though now dried). It is therefore not surprising that some of the iron ores from the two mines contain goethite 4(FeO.OH) which is a hydrated iron ore (Deer *et al.* 1985:442). The haematite ores also contain some water as the analysed oxides did not add up to 100%.

Based on the present evidence, one can now say with confidence that Nsukka early iron smelters used haematite and goethite for their iron production. The ores analysed in this research are reasonably high in Fe. Using the mean values of the bulk analyses results of the ores in table 62, the ratios of Fe : Si + Al in Orba, Owerre-Elu and Umundu are more than 3 : 1, 2 : 1 and 2 : 1 respectively. This shows that there is enough iron in these ores to liquate the gangue and form iron (Tylecote 1986:131). This further shows that it is possible to reduce these ores in the bloomery furnace (Friede and Steel 1977:234; Friede 1980).

Another evidence from the ore analyses is that Nsukka iron ores are titaniferous. They have also high alumina contents, which is reflected in the slags from the area. Tables 57-61 show that the major oxides in all the slags apart from FeO and SiO_2 are Al_2O_3 and TiO_2. This suggests that ores used in the production of these slags must have come from ores similar to the ones analysed in this research. The high levels of alumina in the ores and consequently in the slags resulted in the common presence of spinel (hercynite) which under EDS analysis consists of iron, alumina, titanium and manganese in all the slags analysed during this research (see fig. 5:6b).

5:8 NSUKKA BLOOMERY IRON SAMPLES:

Three samples from bloomery iron artefacts from Umundu from the museum of the archaeology department, University of Nigeria, Nsukka were studied during this research. These comprise two samples (UM/18/90 and UM/19/90) taken from smithing anvils and one sample (UM/20/90) taken from a smithing hammer (see plate 1:1). The artefacts were made with locally smelted blooms. The two anvils consist of thick central iron bars with three or four bands of flat iron bars welded to their flat ends on which the hammering took place. The hammer was formed in a thick cylindrical rod 50mm in diameter at its handle end, and 90mm in diameter at its contact hammering end.

The aims of the investigation were to determine the level of carbon content of the artefacts, their mode of manufacture and the nature of any slag inclusions they may contain. This is necessary since some people speculate that Nsukka early iron smelters produced 'high carbon steel' (Anozie and Ray 1983:27; Van der Merwe 1980:493). It is also necessary to know if there is any similarity between the results of the analysis of the slag inclusions and those of the slags from the area.

The samples were studied through leco carbon analysis to determine their carbon content. Their slag inclusions were analysed with the spot mode of the S.E.M and EDS. A metallurgical microscope was used in examining the samples after they were etched with 2% Nital. This revealed their microstructure and information relating to the mode of their fabrication.

The overall results of the investigations show that the samples are low carbon steel. Sample UM/18/90 has 0.20% carbon. Samples UM/19/90 and UM/20/90 have each 0.05% carbon. None of the samples has carbon content as high as 1.0%. Results of the S.E.M analyses of the samples show that they are all bloomery iron with slag inclusions. The slag inclusions in the three samples consist of wustite in a matrix of glass in which fayalite had precipitated (see plate 5:18). The fayalite phase in sample Um/18/90 has significant levels of MnO and P_2O_5 (see table 123 in Appendix 1).

Slag inclusions in sample Um/18/90 consist of isolated light grey wustite, dark glass matrix and fayalite. The fayalite has high MnO (6.94%) and P_2O_5 (6.31%) (see table 123 in Appendix 1). Sample Um/19/90 has the lowest slag inclusion, but as the other two samples, the slag inclusions in it consist of wustite, glass matrix and fayalite. The Al_2O_3 level in sample Um/19/90 inclusions in table 124 is unusually low. Results of the bulk analysis of the inclusions and their phases are in table 124 in Appendix 1. Sample Um/20/90 has many slag inclusions which also consist of wustite, glass matrix and fayalite. The fayalite contains dark areas high in TiO_2 (19.29%). Results of the bulk analysis of the slag inclusions and their phases are in table 125 in Appendix 1.

5:9 CARBON SAMPLES FROM NSUKKA IRON SMELTING SITES:

One of the major aims of this research is to establish absolute dates for iron smelting sites in parts of Igboland, particularly for the sites in Nsukka Division. Before the present research began, only one iron smelting site, Umundu, has been dated (Anozie 1979:131). The other major sites were dated through oral tradition and geneochronology (Njoku 1986). To improve the situation and place the sites in their proper chronological sequence, a range of eleven carbon samples were collected from the four sites under investigation, (Opi, Owerre-Elu, Orba and Umundu). These samples were submitted to the Oxford Accelerator Laboratory and the dates given below were obtained.

From Opi iron smelting site, three charcoal samples, (OP/1/90, OP/2/91 and OP/3/91) were collected from the excavated iron smelting furnaces "A", "B" and "C". The furnaces were located at the foot of Opi hill. The charcoal samples were sealed by slags and tuyere fragments at the floor of the furnaces (Okafor in preparation b). Sample OP/1/90 was collected in February 1990 field work, while samples OP/2/91 and OP/3/91 were collected during 1991 field work in April-May.

Samples OW/17/90 and OW/18/90 were collected from the base of the smelting furnace in the ploughed farm within the Owerre-Elu smelting site. It was thought that the sample OW/17/90 was fused wood, but during the sample preparation, it was discovered to be some amorphous carbonaceous substance (personal communication, Housley 1991). Nevertheless, a date was derived from it. Sample OW/18/90 consists of twigs and grasses used to temper the earth for the making of the tuyere.

Three charcoal samples OR/2/90, OR/3/90 and OR/4/90 were collected at various points in layer three within a badly damaged smelting furnace in the dried water valley at Agu-Amaorba smelting site. All the charcoal samples from Orba were encased in slags and cinder. The remaining three charcoal samples UM/1/90, UM/2/90 and UM/3/90 were collected from various points in layer two of the excavated furnace at the Umundu 'market' site. There were many roots and rootlets encountered during the excavation, which were removed during pre-treatment. However the three samples were well encased in slags which shielded them from contaminations.

Chronologically the dates derived from these samples by accelerator technique are as follow. All the dates were uncalibrated in radiocarbon year BP (Before present - AD 1950) using the half life of 5568.

Lab No.	**Sample No.**	**Date**
OxA-3201	OP/3/91	2305 ± 90
OxA-2691	OP/1/90	2170 ± 80
OxA-3200	OP/3/91	2080 ± 90
OxA-2738	OW/17/90	1060 ± 60
OxA-2739	OW/18/90	570 ± 60
OxA-2693	OR/2/90	300 ± 90
OxA-2694	OR/3/90	215 ± 100
OxA-2695	OR/4/90	295 ± 85
OxA-2688	UM/1/90	200 ± 80
OxA-2689	UM/2/90	205 ± 80
OxA-2690	UM/3/90	130 ± 80

When these dates were calibrated using the calibration curve of Stuiver and Pearson (1986) and the calibration program of Van der Plicht and

Mook (1989) the results are the age ranges in table 128 in Appendix 1. Figure 5:7 contains the chart of carbon-14 dates from Nsukka iron smelting sites. These dates are discussed in sections 6:3-6:6.

Five of the charcoal samples were further analysed to determine the particular tree species used during the smelting. The general opinion has been that *Ukpaka* **Pentaclethra macrophylla,** *Okpenyi* **Ricinus communis,** *Ahaba* **Acioa barteri** and *Icheku* **Dialium guineense** were the main hard woods used for fuel by iron smelters in Nsukka (Anozie 1979; Njoku 1986). To clarify this point, some portions of the charcoal samples used for dating were examined at the Royal Botanical Garden Laboratory, Kew, Richmond, Surrey for specie identificaton. The five samples examined were part of samples OR/2/90, OR/3/90, OR/4/90, UM/2/90 and UM/3/90. On the whole the examination produced inconclusive results due to insufficient preservation of the diagnostic anatomical features in the samples. The following report was received.

'We have now examined five charcoal samples from Nigeria... Unfortunately the state of preservation of the samples is such that insufficient diagnostic anatomical features are visible to allow conclusive identification to be made. Sample Um/19/90 is similar in many respects to our reference material of various species of Acacia (family Leguminosae). This is the best preserved sample. Sample OR/3/90 also possesses characters in common with some members of the Leguminosae but is not sufficiently well preserved for us to be able to assign it to any particular genus. Samples Um/3/90, OR/2/90 and OR/4/90 are certainly all woody dicotyledons but possess so little intact anatomical structure that we are not able even to hazard a guess as to from which family they originate. We can, however, say with some confidence that none of the woods which you listed as having been used in iron smelting in the past are represented in the samples (i.e. **Pentaclethra macrophylla, Ricinus communis, Acioa barteri** and **Dialium guineense**)' (personal communication, Lawrence 1991).

Pentaclethra macrophylla and **Ricinus communis** are woody dicotyledons. The poor state of the examined samples, particularly samples UM/3/90, OR/2/90 and OR/4/90 leaves us in a position in which we can not rule out completely that any of these tree species were used by Nsukka iron smelters for fuel. However, some revelations were made which suggest that other types of non-hard woods such as Acacia (Leguminosae family) might have been used by some iron smelters in Nsukka Division. There are many Acacia species in the area, but these have never been associated with iron smelting.

CHAPTER 6

DISCUSSION AND INTERPRETATION

6:1 The Aims of the Research

The aims of this research, as discussed in section 1:1, were (1) to identify developments in early iron smelting in Nsukka Division, Nigeria through results derived from analyses of iron smelting residues. (2) To add data to the limited available information on early African iron working. This was felt to be necessary for the following reasons. There are large quantities of iron smelting residues in Nsukka Division. These are indications of the presence of iron smelting activity in the area in the past. Unfortunately, except for a few scattered oral and ethnographic references, most of the present population in the area do not know how these iron smelting residues were produced.

The lack of clear knowledge of who produced the residues and how they were produced has given many people room for free interpretation and speculations concerning the nature of Nsukka early iron working. As discussed in section 1:1, much has been written about the origin of Nsukka iron working (Isichei 1976:9-16), the chronology of Nsukka iron working (Anozie 1979:131; Njoku 1986) and the techniques of Nsukka iron smelting (Anozie 1979; Anozie and Ray 1983b). Most of this was written solely using oral accounts from people of disparate backgrounds and without concrete data.

This research was therefore conceived and carried out to establish the techniques, chronology and changes that took place in Nsukka early iron smelting through the study of the surviving residues. What is available in the area as the record of this industry are oral traditions and ethnographic data from people most of whose generations never witnessed the process in action. Furthermore, there are large quantities of these smelting residues preserved at the primary sites of the industry. It was thought that the extinct processes and techniques that gave rise to them can be learnt from their study. The residues are direct results of the processes of early iron working and as such reflect the processes that gave rise to them. This view is encouraged by the fact that some people such as Allen (1981), Fells (1983), Goucher (1983), McDonnell (1986), Morton and Wingrove (1969a,1972), Tylecote (1975) and Van der Merwe and Killick (1979) have used analyses of bloomery iron smelting residues to reconstruct the processes that gave rise to them.

These residues, particularly slags are very durable and some people like Coghlan (1977:10) Tylecote (1987:291) and Van der Merwe (1969:100) claim that they are almost indestructible. Unfortunately the bloomery iron smelting residues in Nsukka are daily threatened by the rapid expansion of rural development that destroy the primary sites. Where the sites are not facing any threat of destruction, the residues are quarried and used for construction purposes. There is therefore urgent need to study these residues now as they are the only surviving link with the early iron working in Nsukka before they are either destroyed or redeposited out of their primary sites.

There is also the pressing need to establish a bigger data base for a better understanding of African iron working. The data will help in understanding how this industry was operated in the region. This is necessary as there is ample evidence for diverse techniques for iron working in Africa about which not much is yet known. The origin of African iron working has dominated the study of the African Early Iron Age without conclusive consensus. I believe that an understanding of the various techniques of iron production practised in different parts of Africa will provide a starting point for any objective study of the origin of African iron working. The problem of the origin of African iron working can only be solved through a comparative study of the nature of African iron working *per se* with that of the suggested donor zones. Without regional or area studies of various groups that practised this technology in Africa, such as the one done in this research, there will be no data base for a comprehensive study of African iron working and its origin.

6:2 Nsukka Iron Ores and Bloomery Iron Samples:

Nine iron ore samples analysed during this research show that Nsukka iron ores are predominantly haematite and goethite. The results of the analyses reflect the geology of the area. The entire Nsukka Division is on a common geological setting (Umeji 1980). This was discussed in section 2:2. This common geological setting is reflected in the close similarity of the various iron ores analysed during this research (see table 62 in Appendix 1).

Orba and Umundu iron ores are made up of goethite and haematite. This reflects the micro environment of these two ore mines. The two ore mines are within dried-out river beds. It is therefore not surprising that some of the iron ores from the two mines contain goethite 4(FeO.OH) which is a hydrated iron ore (Deer *et al.* 1985:442). There is also a significant amount of water in the non goethite ores.

Nsukka iron ores analysed during this research have high Fe contents. Using the mean values of the bulk analyses results of the ores in table 62, the ratios of Fe : Si + Al in Orba, Owerre-Elu and Umundu are more than 3 : 1, 2 : 1 and 2 : 1 respectively. This shows that there is enough iron in the ores to liquate the gangue and form iron (Tylecote 1986:131). This shows further that it is possible to reduce these ores in the bloomery furnace, as ores of poorer quality have been successfully smelted using bloomery technique (Friede and Steel 1977:234, Friede 1980).

Major elements in Nsukka iron ores apart from Fe and Si are Al and Ti. Nsukka iron ores are titaniferous with high contents of alumina. The other constituent elements Ca, Mg, Mn, P, and K are remarkably low. Fells (1983:70, 132) has shown that ore composition is reflected in the slag composition and mineralogy, and that Al, Mg, Mn and K contents of ores do not partition between the slag and the iron during smelting in the furnace but precipitate in the slag. Nsukka slags analysed during this research contain high Al_2O_3, relatively low TiO_2 but very low MgO, MnO, and K_2O (see tables 57 to 61 in Appendix 1). Relying on this evidence it is reasonable to say that the ores used in the production of these slags must have come from ores similar to the ones analysed during this research. The high levels of Al_2O_3 in the ores and consequently in the slags resulted in the common presence of hercynite in all the slags analysed during this research. Based on the present evidence, one can now safely say that Nsukka early iron smelters used local haematite and goethite for their iron production.

Description of early iron smelting in Nsukka communities in sections 3:3-3:6 show that the processes of iron production (smelting and smithing) in the Division were not done by one person or group. There were two groups, the iron smelters and the blacksmiths. The smelters produced the blooms which they sold to the blacksmiths, who then forged them into tools. Ethnographic data from Nsukka show that the production of tools from the locally smelted blooms involves initial heating and purification of the bloom to exude entrapped slags.

This was followed by further heating and hammering till the required tool was produced.

Results of the analyses of three bloomery iron samples from Umundu show clearly that some of the slags were still in the iron as inclusion and that the process of hammering which was manual did not produce uniform deformation. The inclusion analysis of the bloomery iron is somewhat like that from the Umundu slags in that FeO and SiO_2 are generally high (see tables 59, 123 to 125 in Appendix 1). MnO and P_2O_5 in the fayalite phase of Um/18/90 are significantly higher than that in the slags (see table 123 in Appendix 1) and Al_2O_3 is low in the Um/19/90 inclusion analyses (see table 124 in Appendix 1). All the other constituents are low. The forging has thus had a variable effect on the inclusion composition and this might merit further investigation.

As observed above the ordinary slag bulk analyses reflect the ore analyses. This suggests that ores similar to the ones analysed in this research were used in the production of the iron samples. The three iron samples analysed during this research were found to be low carbon steel of 0.2% and 0.05% carbon. It is difficult to suggest that this was the standard quality of iron tools produced by Nsukka early iron workers. However the fact that most of their products were tools for daily usage (for farming, hunting, wine tapping, carving, rituals, ceremonial, defence etc) suggests that their overall quality will be similar to the ones investigated in this research.

6:3 Carbon-14 Dates From Nsukka Iron Smelting Sites:

Eleven carbon-14 dates from charcoal excavated from iron smelting furnaces in Nsukka Division (Opi, Owerre-Elu, Orba and Umundu) (see section 5:9 and table 128 in appendix 1 for the list of the dates and their calibrated ranges) show that there was continuous iron smelting in the Division probably from about 765 cal. BC to about 1950 cal. AD. The dates were produced by the Oxford Accelerator Mass Spectrometry laboratory. AMS produces dates from samples that are associated closely with the archaeological context and events (Hedges 1981). Its highest merit lies in the fact that it allows the use of very much smaller (milligram-sized) samples as opposed to the (several grams) sample required for the conventional radioactive counting method for determining the carbon-14 content (Harris 1987:23, Hedges 1987:57, Tite

1991:142). All the carbon samples dated during this research were recovered from excavated iron smelting furnaces (Okafor in preparation b) where they were covered by slags, cinder and / or tuyere pieces. Most of the carbon samples were totally encased in slags and cinder which ensured that the dates from these samples are the actual dates for iron smelting done in the excavated furnaces.

Killick (1987:30) claims that there is 'old wood' problem in many early dates from iron smelting sites in sub-saharan Africa. This implies that some of the early dates produced by these sites result from the use of 'old wood' by the iron smelters. This suggestion is not supported by the ethno-archaeological evidence from many parts of Africa which show that iron smelters are very selective in the wood they use for their smelting. To a large extent the quality of fuel used in the smelting determines the temperature which can be attained in the furnace. This means that the smelting operations depend to a considerable degree upon the availabilty of a good and suitable fuel (Coghlan 1977:21).

Most early iron smelters in sub-saharan Africa made use of selected types of wood, preferably hard dense wood that could produce slow, hot burning charcoal. Van der Merwe (1980:488) observed that most iron smelters of Southern Africa select leadwood **Combretum imberbe** for their smelting because it produces slow, hot burning charcoal. In the Western and Central Sudan, Haaland (1985:55, 62-63) recorded **Acacia nilotica** and **Tamarindus indicus** as the two preferred trees for producing charcoal used in the smelting. Pole (1975:14) in the Upper Region of Ghana observed that charcoal used by iron smelters came from 'preferred species of trees'. Because of the preference for some types of trees for charcoal, it is unlikely that early iron smelters used 'old wood' in their smelting, since it is unlikely that such old wood could have produced hard charcoal able to sustain the temperature necessary for reducing the iron ore.

Coghlan (1977:21) recorded that the early metal workers used sap-wood for their charcoal production. Similar information was recorded by the writer during his ethnographic study of iron production in parts of Igboland (Okafor 1984). The elders of Amaorba led by Onyishi Peter Attama informed me that they fell *okpenye* trees and burn them after about eight days when the wood is half-dried. According to them, this was when the wood yields hard ash-free charcoal. In this case there is only an interval of about eight days between the felling of the trees and their use in the smelting. Any date obtained from such charcoal used in smelting will reflect the age of the smelting involved especially when the samples are closely related to the residues as the ones dated during this research.

The dates from Nsukka iron smelting sites cover and bracket all the accepted published radiocarbon dates for early iron smelting sites in other parts of Nigeria (Anozie 1979:131; Calvocoressi and David 1979:10-11; Connah 1968:317, 1981:146; Fagg 1969; Shaw 1969, 1978:97, 1981:630; Sutton 1976:18). During the long period of this industry in the Division, it experienced some changes in technique, each time moving towards perfection and greater efficiency. The earliest period was less efficient in iron extraction and more labour intensive, while the latter ones were more efficient in iron extraction and made less demand on labour. Nsukka iron smelting based on the data from the present research fall into three chronological phases. Each of the phases falls within the chronological sequence and is characterised by particular forms of the smelting residues.

6:4 The Early Phase Of Nsukka Iron Smelting:

The early phase of Nsukka iron smelting is represented by sites at Opi, Leja and Aku (see fig. 6:4). Three AMS radiocarbon dates, 2305 ± 90 BP, 2170 ± 80 BP and 2080 ± 90 BP (see table 128 in Appendix 1), from secure contexts, are available for this phase. Calibrated on the 1986 Stuiver and Pearson calibration curve, and using the 1989 calibration program of van der Plicht and Mook, these dates have 98% probability to fall between 765 cal. BC and cal. AD 75. This is the oldest iron smelting period in Nsukka. Iron smelting during this phase was done in forced draught shaft furnace with slag pits connected with channels (Okafor in preparation b). The furnaces measure between 0.85m and 1.25m in diameter and have thin walls measuring about 40mm. Slags from these furnaces were tapped intermittently into the slag pits through the connecting channels. The slags solidified in the pits forming cylindrical blocks.

These blocks weigh between 43kg and 47kg. They have average density of 3.89gm/cm^3. Their colour varies from dark brown (MC 7.5 YR 4/4) to strong brown (MC 7.5 YR 5/8). They have no rust and some of them are slightly magnetic at fresh surfaces. There are no inclusions in these slag blocks. Results of analysed samples from these blocks show that they have a very low basicity, with a mean of 0.03, and S.D 0.01. Their melting temperatures which were estimated from the phase diagrams (see fig. 6:1) range between 1155°C and 1450°C (see table 60 in Appendix 1).

Before this research was undertaken, it was claimed that the cylindrical blocks of slag are slag bottoms that resulted from pit furnace operations (Anozie 1979). Evidence from the present research does not support this claim. Rather it suggests that these are slags tapped into pits through channels from the shaft furnace. On the external surface of these blocks are visible layers representing individual slag tappings superimposed on one another (see plate 4:1).

The divisions between the layers are not visible with the naked eyes at the core of these blocks. But when samples from the core are examined under S.E.M, the layers are seen marked by white bands of magnetite, a product of oxidation on the slag after it was tapped from the furnace. The layers are visible on the external surface because of faster cooling rate and solidification in areas closer to the wall of the pit than at the centre. The slag at the centre was relatively still more molten than those closer to the wall when it was superimposed by the next batch of slag. Because each layer was formed under different conditions and cooled at differing rates, they have different mineral texture (see plate 5:11)

Another point that supports the evidence that these are tapped slags is that none of the walls of the pits in which the slag blocks are found is slagged or vitrified as would be the case if there was any direct smelting in them (Tylecote 1962, 1980; Pleiner 1978:38). The walls of these pits are friable and greyish-blue in colour. The colour resulted from the heat of the molten slag. Finally excavations of these slag pits have never produced any charcoal or other smelting residues. All the charcoal and refractory materials recovered from these sites came from within the furnaces which were surrounded by the slag pits.

The study of slags from the early phase Nsukka iron smelting sites with S.E.M and EDS show that they are mainly fayalitic in composition. Apart from fayalite, the other major phases are hercynite and wustite. Leucite and glass are the minor phases also found in the slags of this period. These phases exist in different structural forms and textures. Except samples Op/8/90 and Op/9/90 that contain broken lath fayalite the remaining thirteen samples that belong to this phase contain massive fayalite crystals. The massive fayalite crystals are only broken by pools and patches of glass or leucite and vesicles, while the broken lath fayalite are found in the glass.

There is as yet no firm knowledge of the factors responsible for mineral structures found in slags (McDonnell 1986:197). However, opinion based on limited experimental research on materials similar to slag suggest that the different mineral structures in slags result from different cooling rates (Donaldson 1976, Fells 1983:72, 102). Fayalite is a member of olivine group of minerals (Deer 1985:1). Experiments have been conducted on the morphological changes that take place in olivines subjected to varying rates of cooling (Donaldson op cit). The investigation produced lath structure but no massive forms. It also showed that at the slow cooling rate of 5 to 7°C/hr polyhedral and euhedral crystals of olivine were formed. But at the faster cooling rate of 40 to 300°C/hr elongated lath crystals were produced. Donaldson (op cit:202) also investigated the effects of super cooling by isothermal crystallization and found that the greater the degree of super cooling the more elongated the crystals.

If this finding is applied to Nsukka slags it would suggest that the fayalite structures in these slags were produced by varying cooling rates, slow cooling and fast cooling. The massive and the large broken lath fayalite structures are found in all the early phase slags and tap slags from Owerre-Elu. Most of the slags that exhibit massive fayalite structures are large cylindrical blocks of slags which would be expected to take a long time to cool (McDonnell 1986:197). So slow cooling rate is a factor in the formation of large fayalite crystals in these slags. Volumetric calculations of the minerals in the slags of the early phase show that 65.6% of the mineral constituents is fayalite (see table 126 in Appendix 1).

Hercynite, the next major mineral in the slags of the early phase of Nsukka iron smelting exist as large bold euhedral crystals that precipitate from the liquid and are then surrounded by massive and broken lath fayalite crystals. It was seen above that the massive fayalite crystals resulted from the low cooling rate of the cylindrical blocks of slag and tap slags. This factor is again responsible for well developed crystals of hercynite. Volumetric calculation of this mineral show that it is most abundant in Opi slags where it constitutes 22.47% of the minerals in the slags from that site. The low rate of cooling experienced by slags of this phase led to the full growth of the crystallized hercynite mineral in these slags.

Morton and Wingrove (1972:483) claim that the hercynite minerals in slags result from carboniferous ores and that the hercynite phase in slags are limited to the edge of gas holes. Discoveries from this research appear not to agree with these claims as none of the iron ores available to iron smelters at Nsukka is carboniferous (Umeji 1980). Results of the ore analyses give credence to this geological information as all the ores from the Division are high grade ores, mostly haematite or goethite, with some silica.

The nature of the gangue material of the ore and the cooling rate of slags are the probable factors responsible for the presence of this mineral in the slag. The ores had high Al_2O_3 which reacted with iron oxide to form hercynite that precipitated in the slag (Fells 1983:132). In none of the analysed slags was hercynite limited to the air holes. If it exhibited any pattern at all, they appear to associate more with fayalite matrix away from the air holes as in samples Ow/12/90, OR/7/90, Op/8/90, Op/10/90 and Op/11/90 (see plates 5:2 and 5:13). In these samples the hercynite mineral is completely within the fayalite matrix segregated from the air holes.

Wustite, free iron oxide, was found in all but three slag samples (Op/5/90, Op/6/90 and Op/8/90) from the early phase of Nsukka iron smelting. Free iron oxides, wustite, in Nsukka slags exhibit dendritic structures of varying sizes. Wustite is a high temperature mineral stable only in temperatures above 560°C. Below this temperature it decomposes into iron and magnetite at the point of equilibrium (Egundebi 1987). The presence of wustites in slags, therefore, indicates that the point of equilibrium was not reached hence the retention of the phase at high temperature (Wingrove 1970:258). The presence of oxides in dendritic form shows that the oxide was in solution at elevated temperature and crystallized out of a liquid during cooling (Friede *et al*. 1982:47). The dendritic form of wustite as exhibited in Nsukka slags therefore derives from the high temperature formation of this mineral, a high temperature mineral with several equivalent directions of high heat flow rate.

As the free iron oxide, wustite is the penultimate stage in the reduction of iron ore to metal iron (Morton and Wingrove 1969a:1557), the level of wustite in the slags is assumed to indicate the degree of efficiency of the particular bloomery operation. The higher the free iron oxide content of a slag the less efficient the smelting operation (McDonnell 1986:86). This means that the level of wustite in bloomery slags will vary according to the skill of the operators to work the furnace so that minimum wustite is left in the slag. Improvements in techniques with time would account for increasing efficiency and less wustite in the slag (Morton and Wingrove 1972:480). Based on this assumption, it would seem that iron smelters in the early phase sites were less efficient in their iron extraction when compared with the middle and the late phases of this industry in the Division. Although wustite constitutes only 6.2% of the mineral phases in Opi slags, it is still the highest level when compared with other slags from the other phases as will be seen below.

Thirteen slag samples from Opi (Op/6/90, Op/8/90, Op/9/90, Op/10/90, Op/11/90, Op/12/90, Op/13/90, Op/14/90/, Op15/90, Op/16/90, Op/17/90, Op/18/90 Op/19/90) contain the minor mineral phase leucite, $K_2O.Al_2O_3.4SiO_2$. No other site in Nsukka has slags that contain this mineral. It constitutes 5.07% of the minerals in Opi slags. This phase exists as dark intergrowths or irregular dark patches very hard to separate from the glass. When spot analysed, it revealed high contents of K_2O, SiO_2 and Al_2O_3 and relatively low levels of FeO.

The presence of this mineral in some slags from Opi is surprising. The analyses of iron ores from Opi show very low levels of K_2O (mean 0.04% S.D 0.02). Besides the ore, the other possible source of K_2O for the slags is fuel (the charcoal) and perhaps the refractory materials. But as this mineral is found only in some slags from Opi, it is possble that the iron smelters at Opi conducted some of their smelts with charcoal that has high potassium content which precipitated in the slag. Fells (1983) shows that K_2O, originating from fuel ash generally precipitate in the slag.

The dominant mineral phases in Opi slags are fayalite, hercynite and wustite. This is evident both from the microstructure and the results of plotting the normalised values of the three most abundant oxides in the bulk analyses on approprite phase diagram (see fig. 6:1). In all Nsukka slags investigated during this research; FeO, Al_2O_3 and SiO_2 are the most dominant oxides (see tables 57 to 61 in Appendix 1). These three oxides fit into $FeO-Al_2O_3-SiO_2$ phase diagram. Fells (1983:82) adopted this approach and by using it she was able to discover the various mineral groups in the slags she investigated. Some of the results from the present research suggest that this is not always the case. They show that the mineralogy of some slags is not revealed by the plot of the constituent oxides.

In this research phase diagrams were used for mineralogical grouping of the slags as well as for the estimation of their melting temperatures. The slags melting temperatures were estimated by plotting the normalised values of the component oxides in the phase diagram (Morton and Wingrove 1969a, 1972; Todd 1976). Kresten (1986:43) claimed that there are major variations between the estimated melting points from the phase diagram and the measured melting points. Friede *et al* (1982:42-43) while not refuting this claim, has discovered that even the measured melting points are not error free. This is because the dark or pitch black colour of most slags make it difficult to read the pyrometer used for the measurement as the slag remain opaque when molten. This results in obtaining melting points that are either under or above the actual melting points. Until a better system is discovered either of the techniques will continue to be used in the slag melting point estimate.

Opi slags plot within the two systems fayalite-wustite-hercynite and fayaliyte-hercynite-iron cordierite. Three samples Op/5/90, Op/6/90 and Op/8/90 contain no wustite in their microstructure. This is reflected in their plotting. The three plotted in the fayalite-hercynite-iron cordierite compatibility. Although samples OP/9/90, Op/12/90, Op/13/90, OP/14/90 and Op/17/90 contain 7%, 5%, 10%, 10% and 7% wustite respectively, they fall in fayalite-hercynite-iron cordierite triangle. This indicates that the plotting of the component oxides in slag bulk analyses does not always reflect the actual mineralogy of slags. In this case there are four phases present and the ternary diagram cannot truely represent the system.

Opi slags contain more hercynite mineral (22.47%), more than any other slag from Nsukka. Hercynite has a high melting temperature of 1780°C. Consequently all Opi slags that have high Al_2O_3 and hercynite (as in samples Op/5/90, Op/7/90, Op/8/90, Op/10/90, Op/11/90, Op/15/90, Op/16/90, Op/17/90, Op/18/90 and Op/19/90) have high melting points that range between 1350°C and 1450°C. It seems that in an attempt to liquate the high Al_2O_3 gangue, the iron smelters at Opi produced slags of high melting temperatures.

This happened incidentally during the early phase of iron smelting in Nsukka. This technique of liquating slags at high temperatures was improved upon during the late phase of Nsukka iron smelting by the use of silica as flux to lower the melting point of the gangue and extract more iron from the slag. The silica formed iron silicate with some iron oxide thereby freeing reduced iron. This produced wustite-free slags characteristic of the late phase of Nsukka iron smelting which will be discussed below.

6:5 The Middle Phase Of Nsukka Iron Smelting:

AMS produced two high-precision dates, 1060 ± 60 BP and 570 ± 60 BP, for the middle phase of Nsukka iron smelting. Calibration of the two dates to two standard deviations gives a range of 810 to 1435 cal. AD. This suggests that the middle phase of Nsukka iron smelting probably fell between 810 and 1435 cal. AD. Iron smelting sites that belong to this phase lie in the Owerre-Elu-Nru-Isiakpu axis (see fig. 6:4). During this phase, iron was still smelted in forced draught shaft furnaces. Their slags were still tapped but not into pits. The slags of this period were allowed to run out of the furnace and spread on the ground around the furnace, thereby forming flat smooth ropy surfaced slags.

These are typical tap slags and they survive in flat cake forms weighing between 1.5 and 4.2 kg. They are very dense with an average density of 4.24gm/cm^3 Some of these slags are weakly magnetic at fresh surfaces. They have no rusts or inclusions. They have mixed colours of dark-brown (MC 7.5 YR 3/4) and black all over (MC 7.5 YR 2/10). In section they have bluish-grey colour and are coarsely vesicular near the surface. Results of analysed samples of these slags show that they have very low basicity (mean 0.02, S.D 0.01).

Most slags that belong to this phase contain no free wustite. This suggests improvement and efficient mastery over the techniques and extraction of the reduced iron. The improvement was also observed in the lower melting points estimated for most of the slags of this period. 75% of the total slags from this phase analysed have melting points that are below 1200°C.

As in early phase slags, S.E.M and EDS analyses of slags that belong to the middle phase of Nsukka iron smelting show that they are made up of fayalite, hercynite and few patches of glass localised in the vesicles. Some contain white dendrites of wustite as well (see plate 5:5). The most abundant mineral in the slags of the middle phase is iron silicate, fayalite $2FeO.SiO_2$ (see table 126 in Appendix 1 and fig. 5:1). Volumetric calculations of the minerals in the middle phase slags show that fayalite phase make up 76.42% of the minerals.

Four structural forms of fayalite are identified in slags of the middle phase. These are (1) massive fayalite crystals, (2) broken lath fayalite crystals, (3) lath fayalite crystals and (4) fine fayalite crystals. Fayalite crystals in half of the slag samples from Owerre-Elu have massive structure. These are Ow/2/90, Ow/3/90, Ow/4/90 Ow/5/90, Ow/11/90 and Ow/12/90. The massive fayalite crystals are only broken by pools and patches of glass and vesicles (see plate 5:8).

The broken lath fayalite crystals in slags of the middle phase Nsukka iron smelting precipitate in a glass matrix (see plate 6:1). Such fayalite crystal structures are found in samples Ow/1/90, Ow/6/90, Ow/7/90, Ow/9/90. Lath fayalite crystals in Nsukka slags also precipitate in glass matrix. They exist in parallel or sub-parallel bundles (see plate 6:2). Such lath fayalite crystals are found in sample Ow/8/90. The fine fayalite rods are found in the glass matrix interlacing with the euhedral fayalite crystals. The fine fayalite rods are generally anhedral and perhaps represent the last stage of crystalization before solidification in the glass. The fine fayalite rods are common in the slag samples with broken lath and lath fayalite crystals. Few are however present in the glass of some samples with massive fayalite crystals.

Hercynite crystals in the slags of the middle phase appear in two structural forms (1) euhedral and (2) anhedral structures. Large bold euhedral crystals of hercynite form as a primary phase in samples with massive or broken lath fayalite as in samples Ow/9/90, Ow/10/90, Ow/11/90 and Ow/12/90 (see plates 5:5 and 5:8). In slag samples with parallel or sub-parallel lath bundles of fayalite, are found small euhedral crystals and anhedral crystals of hercynite that formed in the glass or fayalite. This is evident in sample Ow/8/90. In some samples such as Ow/1/90, and Ow/7/90 the two structures of hercynite (bold euhedral and dispersed anhedral crystals) are found in the broken lath crystals of fayalite. The hercynite in Owerre-Elu samples constitute 16.17% of the mineral phases in the analysed samples (see fig. 5:2).

Out of the twelve slag samples studied from Owerre-Elu, only five samples Ow/2/90, Ow/3/90, Ow/5/90, Ow/10/90 and Ow/1/90 have wustite. The remaining seven samples have no wustite. The wustite exists in dendritic form and constitutes 3.92% of the mineral constituents in the slags from the middle phase analysed during this research (see fig. 5:3 and plates 5:8-5:9).

All the slag samples from Owerre-Elu have fayalite as a primary phase. But they distribute within the two systems fayalite-hercynite-wustite and fayalite-hercynite-iron cordierite (see fig. 6:2). Five slag samples (Ow/2/90, Ow/3/90, Ow/5/90, Ow/10/90 and Ow/11/90) that contain free iron oxide in their microstructures plot in the fayalite-wustite-hercynite triangle while five of the samples (Ow/1/90, Ow/4/90, Ow/6/90, Ow/7/90, Ow/12/90) that have no wustite in their microstuctures plot in the fayalite-hercynite-iron cordierite compatibility triangle. Samples Ow/8/90 and Ow/9/90 have no free iron oxide in their microstuctures but they plot on the fayalite-wustite-hercynite and fayalite-hercynite-iron cordierite boundary.

The plot of Owerre-Elu slags reflect the variations evident in the slags from that site. Seven of the slags from that site contain no wustite and they have lower melting points of 1150°C compared to the five samples that have wustite whose melting points range between 1150°C and 1325°C. The information from Owerre-Elu as presented by these slags may be better understood from the evidence provided by the radiometric dates from the site. Based on carbon-14 dates, Owerre-Elu belong to the middle phase of Nsukka iron smelting. This phase lasted probably from about 800 to 1430 cal. AD. The late phase of Nsukka iron smelting (represented by Orba and Umundu sites discussed below) lasted between 1430 and 1950 cal. AD.

Following from this evidence, one can suggest that those Owerre-Elu slags that contain no wustite and have low melting points (which are very similar in this respect to Orba and Umundu slags) belong to the latter period of the middle phase, while the remaining samples that contain wustite and have higher melting points (which are similar in this respect to Opi slags) belong to the early period of the same middle phase.

6:6 The Late Phase Of Nsukka Iron Smelting:

The late phase of Nsukka iron smelting covers the period from about 1430 to about 1950 cal. AD. This is based on six high-precision AMS dates, 200 ± 80 BP, 205 ± 80 BP, 130 ± 80 BP, 300 ± 90 BP, 215 ± 100 BP and 295 ± 85 BP from secure contexts. Iron smelting sites that belong to this phase are those in the Orba-Umundu-Eha-Ndi-Agu area (see fig. 6:4). Iron slags that belong to the late phase of Nsukka iron smelting survive as aggregates in the smelting sites. The mode of their raking from the furnace and sorting from the bloom make them survive as amorphous irregular aggregates. They are very vesicular and less dense. The average density of slag samples from Orba is 3.98gm/cm^3 while those from Umundu have average density of 3.63gm/cm^3. Their colour varies from dark-brown (MC 7.5 YR 3/4) to black all over (MC 7.5 YR 2/10). A few of these slags have some quartz inclusions but they have no rusts. None of the slag samples from these sites is magnetic. As in Opi and Owerre-Elu slags, Orba and Umundu slags have very low basicity. The mean of basicity of Orba slags is 0.01 while that for Umundu slags is 0.02. The melting temperature of slag samples from Orba and Umundu range between 1150°C and 1280°C (see tables 58 and 59 in Appendix 1).

S.E.M and EDS analyses of slags from the late phase Nsukka iron smelting sites show that they are made up of fayalite, hercynite and glass. There is a total absence of free iron oxide, wustite, in the slags of the late phase. This demonstrates the high degree of efficiency in iron extraction attained by Nsukka iron smelters during the late phase of this industry. As observed in section 6:4 the level of free iron oxide in slags suggests the ability of the iron master to work the furnace so that the least amount of iron was left in the slag (Morton and Wingrove 1969a, 1972:478)

The fayalite mineral in the slags of the late phase exists mostly as needle-like laths in parallel and sub-parallel bundles. There are a few slags that contain massive and broken lath fayalite structures, and some have tiny fayalite rods that are found in the glass. Only three slag samples (Um/6/90, Um/7/90 and Um/14/90) from Umundu have this massive fayalite. None of the samples from Orba has this structure. The massive fayalite crystals are only broken by pools and patches of glass and vesicles (see plate 5:1).

The broken lath fayalite crystals in the slags of the late phase form in glass matrix containing angular dark grey hercynite (see plate 5:6b). Such fayalite crystal structures are found in samples OR/7/90, OR/8/90,OR/11/90, OR/12/90, Um/8/90, Um/12/90, Um/15/90, Um/16/90 and Um/17/90. Needle-like lath fayalite crystals in the late phase slags also precipitate in glass matrix. They exist in parallel or sub-parallel bundles.

Such lath fayalite crystals are found in samples OR/9/90, OR/10/90, OR/13/90, OR/14/90, OR/15/90, OR/16/90, OR/17/90, OR/18/90, Um/9/90, Um10/90, Um/11/90 and Um/13/90. The fine fayalite rods are found in the glass matrix interlacing with the euhedral fayalite crystals. The fine fayalite rods are generally anhedral and perhaps represent the last stage of crystalization before solidification in the glass. The fine fayalite rods are common in the slag samples with broken lath and lath fayalite crystals. Few are however present in the glass of some samples with massive fayalite crystals as in samples Um/7/90 and Um/14/90.Volumetric calculations of minerals in the slags of the late phase show that fayalite phase make up 61% of the minerals in Orba slags and 60.25% of the mineral phases in Umundu slags (see table 126 in Appendix 1 and fig. 5:1).

It was seen in section 6:4 that cooling rate affects the structure of olivine minerals, and that the massive fayalite structures common in the slags of the early phase derived from the slow cooling experienced by the large cylindrical blocks of slag. Umundu and Orba slags (late phase slags) were raked out of the furnace at the end of the smelt and broken into pieces in order to sort the bloom from the residues, as a result of this technique slags from these sites are expected to cool faster.

The majority of Orba and Umundu slags have small lath and needle-like fayalite crystals in parallel and sub-parallel bundless and greater contents of fine fayalite crystals. Although samples Um/6/90, Um/7/90 and Um/14/90 from Umundu contain massive fayalite crystals. This occurence may still reflect the circumstance of their cooling. Not all the rakes from the furnace were fractured during the sorting. Obvious lumps of slag were thrown away to the slag heaps without breaking, such lumps of slag would definitely experience slow cooling rate and that would result in the formation of fayalite crystals similar to those from slag blocks.

Hercynite crystals in the slags of the late phase appear in two structural forms (1) euhedral and (2) anhedral structures. Large bold euhedral crystals of hercynite precipitate as a primary phase in samples with massive or broken lath fayalite as in samples OR/7/90 and OR/8/90. In slag samples with parallel or sub-parallel lath bundles of fayalite, are found few crystals of small euhedral crystals and anhedral crystals of hercynite that form in the glass or fayalite. This is evident in samples OR/9/90, OR/10/90, Um/11/90 and Um/13/90. In some samples such as OR/7/90 and OR/8/90 the two forms of hercynite, bold euhedral and dispersed anhedral crystals, formed in the broken lath crystals of fayalite.

It was seen above that the massive fayalite crystals resulted from low cooling rate of the cylindrical blocks of slag and tap slags. This factor is again responsible for well developed euhedral crystals of hercynite closely associated with them. On the other hand, slags that had fast cooling rates, which produced lath and fine fayalite crystals have small crystals and anhedral crystals of hercynite associated with them. In such slags, the hercynite crystals had not enough time to develop before solidification took place. This particular hercynite form is in many slags from Orba and Umundu as evident in samples OR/9/90, OR/10/90, OR/14/90, OR/15/90, OR/18/90, Um/9/90, Um/11/90, Um/12/90 and Um/13/90.

The low rate of cooling experienced by slags from the early and the middle phase sites and three others from Umundu (Um/6/90, Um/7/90 and Um/14/90) led to full growth of the crystallized hercynite mineral in these slags. Orba and most of Umundu slags that contain poorly developed hercynite crystals have lower contents of this mineral. Umundu slags have 12.42% of hercynite while Orba slags have 11.5%.

Another mineral in the slags of the late phase Nsukka iron smelting is glass. The glass phase in these slags exist in two forms (1) as dark pools and (2) as dark matrix containing other phases. Pools of glass are found in slags that contain massive and broken lath fayalite crystals. They are found in or near air holes in these slags. Some of the slags that contain such form of glass are Um/6/90, Um/7/90 and Um/14/90. Slag samples that contain parallel or sub-parallel lath crystals of fayalite and lots of fine fayalite rods have glass matrix. Glass matrix is common in slags from Orba and Umundu (see plates 5:3-5:4). Fine rods of fayalite are found in the glass matrix, though some glass pools contain few of such tiny fayalite rods as in samples Um/6/90 and Um/7/90. Umundu and Orba slags have high siliceous glass. This resulted from the excess silica in the slag.

Ethnographic data from the area show that sand was charged into the furnace as smelting progressed perhaps to flux the smelt (Okafor 1984a:24-25). Tylecote (1987:108) observed that high grade ores that are rich in iron oxide require sand flux for a better yield of iron. The practice of fluxing bloomery smelts with sand has been documentd in many early metal working sites (Fells 1983:132). During his research on the early iron working sites at Nebersdorf in Austria, Sperl (1980:61-74) discovered excess quartz in the slags from that site which might have been used as flux. In South Africa, iron smelters at the site near Phalaborwa fluxed their smelts with silica (Van der Merwe and Killick 1979:89). The charged sand produced the excess SiO_2 that gave rise to high glass in the slags from Umundu and Orba sites. In Umundu slags, glass constitutes 27.33% of the mineral phases, while in Orba slags, it constitutes 27.25% of the minerals (see fig. 5:4).

All Orba and Umundu slags fall within the fayalite-hercynite-iron cordierite triangle (see fig. 6:3) with none of their samples in the Fayalite-hercynite-wustite triangle. This reflects the results of micro analyses of these slags which did not detect wustite in slags from Orba and Umundu. The estimated melting temperatures for slags from Orba and Umundu range between 1150°C and 1280°C. The melting temperatures of slags from these two sites are within the minimum temperature range necessary for bloomery iron smelting (Van der Merwe 1969:17; Tylecote 1987:296). The aid for the production of these wustite-free slags with low melting points is the silica which Orba and Umundu iron smelters used in fluxing their smelts. The silica reduced the melting points of the gangue and combined with some iron oxide to free reduced iron.

The late phase of Nsukka iron smelting marks the final phase and the zenith of iron smelting in the Division in terms of efficiency of iron extraction and lowest labour demand. None of the slags from this phase contains wustite which suggests that there was total extraction of iron from the the smelt. The phase also witnessed the use of sand as flux to lower the melting point of the gangue in the smelt. The sand formed iron silicate with iron oxide thereby freed the reduced iron. Slags of this phase produced lowest melting points enough for any bloomery smelting.

This phase also witnessed a move towards labour conservation. The furnace in use was still shaft furnace. But the ones used during this phase were self-induced shaft furnaces. The furnaces were not tapped during smelting. The entire load of the furnace was raked out at the end of the smelting and the blooms sorted from the residues. Although the labour need was reduced by this technique, it was achieved at the expense of time (see table 119 in Appendix 1). The use of self draught shaft furnaces demands longer smelting time which on the positive side allowed the smelters to charge the furnace as many times as they desired since this enabled them to produce bigger blooms in one smelting round.

6:7 Interpretation

The developments in Nsukka furnaces discussed above suggest continuous progress towards conservation of labour. After the early phase, the slag pits were abandoned and slags were tapped straight onto the ground surface during the middle phase. In the late phase, the necessity to use bellows, was removed by the introduction of self-draught furnace. The time and labour spent in tapping the slags were also saved by not tapping the slags as smelting progressed. Instead the furnace was off-loaded at the end of the smelt. It is evident that there were steady changes in the furnace designs used during bloomery iron smelting in Nsukka; each successive change aimed at perfecting the technique and reducing the labour need of the industry.

Based on the carbon-14 dates available at present, one can observe a gap of several hundred years between the early phase and the middle phase of Nsukka iron smelting. It seems to me, that this is largely due to chances of sampling and data collection. It is hoped that the apparent gap will be filled when more dates are processed from sites that belong to one of these two phases. It is possible that the additional dates will either extend the early phase further into the first millennium AD or project the beginnings of the middle phase back into the last centuries BC. Morphological and physical analyses of smelting residues from Nsukka show clearly that they fall into three definit groups outlined by the three phases, thus eliminating any possibility of a fourth phase since each of the groups is differentiated by distinct technological attributes.

Evidence from slag analyses, as well as from archaeological and ethno-archaeological information from Nsukka show that there are concrete variations in the technique of iron smelting in Nsukka Division. The variations also exist in the three phases isolated above. This raises the question of how Nsukka iron smelting was organised. The questions that arise from the data are: (1) were there individual iron producing communities in Nsukka Division who were practising and producing their iron independently of the other groups?. (2) why does each phase represent a different technological phase of the industry?, and (3) why are the three observed phases not represented in any one site?.

The question of whether the various iron producing communities in Nsukka were producing their iron independently, would imply that each of these communities produced their iron only within one of the three phases observed, and did not do any production either before or after that phase. It is unlikely that this was the situation in Nsukka, since the available dating evidence and the results of residue analyses suggest a continuous development.

With regard to the next question, two points would explain why each phase of Nsukka iron smelting was technologically different from the others. There is clear evidence that shows that the late phase smelting operations were better and more efficient than the earlier ones. The changes in technology that differentiate each of the phases of Nsukka iron smelting can be explained, in part, as resulting from several decades of experience in this industry. This is more probable since carbon-14 dates from the four sites studied demonstrate a clear progress from about 760 BC to 1950 AD. This progress was also observed in the results of the residue analyses.

Secondly, it is observed from the furnace operations associated with each of the phases, that they show a steady move towards labour reduction. Two factors, economic and political, identified by other researchers (Afigbo 1973a, Oguagha 1982, Shaw 1970), that were operative in the northern Igboland between the middle and the late phase of Nsukka iron smelting could lead to labour shortage in the Division. Shaw (1970:285) observed that among the trade goods the Igbo exported in the 9th century AD to her northern neighbours were slaves. Afigbo (1973a:79-80) observed also that in the nineteenth century, slave trading was one of Nsukka's most established commercial engagements. He spotted Nkwo-Ike, Ozalla, (in Nsukka division) as a renowned slave market. The fear of slave raids and the actual fall in population due to exportation of able bodied young men must have affected the essential labour needed by the iron smelting industry.

Other threats to the available labour at this period in Nsukka Division, were raids and wars which the Igala mounted on Nsukka. The Nsukka-Igala wars have been dated to between 18th and 19th centuries AD (Oguagha 1982:58). These were prolonged wars, which at their peak left most of Nsukka under Igala influence. It is likely that the technological changes that differentiate each of the phases of Nsukka iron smelting were concrete response by the iron masters to the shortage of labour.

This was evident in the middle phase of Nsukka iron smelting, where the slag pits were abandoned and slags tapped straight on the ground surface. Improvement and experience over several years within the industry plus increasing labour shortage led to the abandonment of forced-draught furnace. They further led to not tapping the slags as smelting progressed. The furnace was off-loaded once at the end of the smelt.

Probably due to scarcity of labour and insecure situation resulting from slave raiding and Nsukka-Igala wars, some iron smelters used wood directly in their smelting without spending time to burn the wood in order to produce charcoal. Ethnographic data from Ama-Orba suggest that this was the technique used by the Ama-Orba iron smelters during the late phase of Nsukka iron smelting (Okafor 1984a:23). This reduced the labour needed for the industry and produced a change in the prevalent technology.

The differences in techniques of iron smelting observed in each of the three phases of Nsukka iron smelting are, therefore, reflections of the state of the economic and political climate in the Division during each of the phases. The early phase of Nsukka iron smelting appears to have been peaceful, thus the industry was very elaborate and complex during this phase. The iron smelters had enough labour and time to build and use forced-draught shaft furnaces. They also dug slag pits, lined them with clay and provided connecting channels to the pits during this phase.

But during the middle phase, the labour available was threatened by the sale of slaves to the Arabs in the 9th century AD (Shaw 1970:285). The iron smelters responded by abandoning the use of slag pits and channels for collecting slags. Slags from the smelts of the middle phase sites were tapped onto the ground surface where they survive as flat smooth ropy surfaced slags.

A further severe threat to available labour by both the Atlantic and the internal slave trading and the insecurity created by the Nsukka-Igala wars forced other technological changes on the industry. This is evident in the late phase of Nsukka iron smelting. As a result of the loss of labour and insecurity, the forced-draught shaft furnace technique was abandoned and self-draught furnace technique adopted in its place, thus eliminating the jobs of bellowsmen. The slags ceased to be tapped as the smelting progressed instead the entire load of the furnace was off-loaded once at the end of the smelt. At times, when severely pressed by the above constraints, the late phase iron smelters made further adjustments in their technology by smelting directly with wood instead of charcoal, thus reducing the time and labour invested on charcoal production.

For an understanding of why the three phases are not represented in any one site, it will be vital to know what was happening in northern Igboland (which includes Nsukka Division) during the Early Iron Age. From the study of glottochronology, Igbo as a separate ethnic group started to emerge from the Kwa linguistic sub-family about six thousand years ago (Armstrong 1962:284, 1964:22-23; Afigbo 1973:8). This view is supported by archaeological results from the area. Analyses of archaeological materials excavated from sites in the north of Igboland, particularly from Nsukka, by Professor D. Hartle suggest that the area was occupied before the third millennium BC. The study of pottery recovered from the excavated sites show that there are close similarities in colour, form and decoration between the excavated sherds and those still in use in the area today (Hartle 1967:134-143).

Available historical and ethnographic data from Igboland show that when the Igbo emerged as ethnic group, they first settled on the northern Igbo area. From there they evolved their culture and expanded to other areas they now inhabit. It is relevant to observe that even now many Igbo groups claim that their ancestors lived originally on the northern Igbo plateau before moving out in search of unoccupied land (Afigbo 1973:9).

A number of factors attracted the ancestors of Igbo to the northern Igbo plateau. The area, although originally in the forest zone, was at the fringe of the savannah belt. This, according to Uzoezie (1972), made exploitation and farming feasible with the limited tools at their disposal. Again the area, particularly during the latter period, is on the major trade routes that join the Igala and the Idoma in the north with the Igbo, the Ijaw and the Cross River areas in the south.

Finally on this northern Igbo plateau, mainly on the Nsukka-Udi Cuesta, are the most abundant deposits of iron ores. The iron ores cap the residual hills in the area and most of them contain up to 50% iron (Umeji 1980). It is probable that during the long period of existence on this plateau, some group of Igbo people learnt how to smelt the iron ores. Afigbo (1973:16) observed that, 'the northern Igbo plateau and its extension to Bende is rich in iron ore deposits. And it is here that smelting and iron working were most highly developed in Igboland. The iron tools and implements that were produced on the plateau were in great demand over the rest of Igboland and beyond'.

It would appear from the carbon-14 evidence that this iron producing group possibly from as early as 8th century BC first settled and exploited the ores and woods at those sites that are within the early phase (Opi, Leja and Aku sites) (see fig. 6:4). Based on the evidence from slag analyses, the loss of iron to slags prevalent at the sites of this period suggests that the producers were still trying to improve their mastery over the ores and the techniques they were using. This phase, may have ended around the first century AD according to Carbon-14 dates. It is probable that the exhaustion of resources, ores and fuel, at the sites of the early phase and the need for iron tools led to a search for new source of raw materials. This made the group or their decendants to move into middle phase area (Owerre-Elu-Nru-Isiakpu) (see fig. 6:4) where there were rich iron ore capping on the Nru-Isiakpu ridge and also rich hard wood for fuel. Carbon-14 dates show that these sites were exploited probably between 800 and 1450 AD.

Probably around 1430 AD, the necessary resources for iron smelting at middle phase sites were diminishing and the need for iron tools led to the exploitation of the resources in the late phase areas (Umundu-Orba-Eha-Ndi-Agu) (see fig. 6:4). As observed above this was the last phase of iron smelting in Nsukka Division. Six carbon-14 dates show that iron was smelted in this area between 1430 and 1950 AD.

From the foregoing evidence, I conclude that the bloomery iron smelting in Nsukka Division was conducted by a specialised group of craftsmen and their descendants, who moved from one location to another as they exhausted available raw materials at each site. Archaeological evidence discussed above demonstrates cultural continuity in the area from the third millennium BC to the present (Hartle 1967). This shows that the ancestors of the present Nsukka communities had always lived in the area. Ethno-archaeological evidence from the *Eguru*, Nsukka blacksmiths, show that iron working had been a craft for segregated groups not open to everybody. It had been a craft for close caste that jealously guarded its privileges and duties and shunned admittance of non-members (Okafor 1984a:69-76).

Evidence from linguistic study in Nsukka Division seem to support this conclusion. There are common words for the key items that relate to iron production in all the communities in the Division. For example, furnace is generally known as *itoro* or *utu*, bloom is known as *aga*, slags are known as *afuru* or *nsi igwe* and iron ores are known as *nne igwe* or *nne nkwume*. The uniform vocabulary for all the key elements in iron production is a strong point which negates multiple distinct practitioners but favours "a group" practice.

This concluson is also supported by the fact that although all Nsukka people know these key elements of iron smelting by their names, they do not know how they came about. For example, most people that live within the early and the middle phase areas do not know how the smelting debris around them originated. Even the oldest man in Opi, Onyishi Abonyi Nnamani of Umugedu village, Idi Opi, told me that the cylindrical blocks of slag in Opi are "small hills" germinating from the ground. He believes that these were not made by man. A similar story was told at Owerre-Elu where Onyishi Ozioko Ugwu described the flat tap slags as *Nsi igwe* given to Owerre-Elu people by God to defeat their Edeoballa enemies. This, I learnt, referred to Owerre-Elu Edeoballa wars of the 19th century during which Owerre-Elu people inflicted heavy casualties on their enemy by throwing slags on them.

Finally a tradition recorded by C. Onyeke (1986:14) from Idi Opi clan claims that Opi people never smelted iron themselves. It claims that iron smelting at Opi was done by outsiders who came and lived with them and left at the end of their work. Bloomery iron smelting is a highly specialised trade. In many artisan communities like Igboland, the presence of iron smelters would be very valuable for the production of raw materials for making most of the tools needed by the community. It is therefore unlikely that any community or clan would object to smelters camping and doing their smelting where ever they locate the raw materials. The tradition of locating smelting sites close to the source of iron ore would explain why the smelting group moved to fresh ore sources when they exhausted the available ore in a location. Njoku (1986) recorded that more consideration was given to the location of available ore while chosing smelting sites than to wood in the north of Igboland. However where the condition was favourable both were taken into account.

CHAPTER 7

CONCLUSIONS

Developments in the bloomery iron smelting in Nsukka Division have been identified through the study of iron smelting residues. During the study, various techniques of investigation were used. These are ethnographic investigation, archaeological and analytical techniques. The analyses were conducted at two levels, morphological and microscopic. These investigations discovered several hitherto unknown aspects of early iron smelting in Nsukka. Based on the discoveries, the following conclusions were reached about Nsukka bloomery iron smelting.

1. Nsukka bloomery iron smelting took place from about 765 cal. BC to about 1950 cal. AD. During this long period, the industry passed through three phases. Each successive phase represented a technological change in the industry.

2. On the whole, Nsukka bloomery technology was very efficient in terms of iron extraction. Very little free iron oxide was left in the slags. In this regard, the earliest period of this industry was least efficient, while the latter period was most efficient as it had almost total extraction of iron from the slags and reduced labour need for the industry.

3. Nsukka bloomery iron smelting was conducted in various forms of shaft furnace. Forced draught shaft furnaces were used from the beginning of the industry in Nsukka till about 1435 AD. The slags from these furnaces were tapped as smelting progressed. The forced draught shaft furnaces were replaced with self draught shaft furnaces which were in use between about 1430 AD and 1950 AD. The slags of these furnaces were not tapped as smelting progressed.

4. No pit or bowl furnace was found in Nsukka Division. What were thought to be bowl furnaces by some people (Anozie 1979), were discovered to be slag pits into which slags were tapped as smelting progressed.

5. Nsukka bloomery iron smelters smelted local goethite and haematite. Nsukka iron ores are rich in Al_2O_3 with significant levels of TiO_2. Initially the high presence of Al_2O_3 in the iron ores led to the use of high temperatures in smelting them. Later on, the temperature was lowered by the use of sand flux.

6. Major mineral phases in Nsukka slags are fayalite, hercynite and wustite. Minor phases present in some of these slags are glass, leucite and magnetite.

7. Nsukka slags have very low basicity. Nsukka bloomery iron smelters did not use lime or any lime-rich flux in their smelting.

8. The presence and the location of iron ores determined the site of the industry. The sites were relocated when the ores were exhausted.

9. Bloomery iron smelting in Nsukka was conducted by a specialised group of craftsmen, who moved from one location to another as they exhausted available raw materials on each site.

10. Nsukka bloomery iron smelters limited themselves to the production of blooms. They never refined or forged the blooms they produced. The blooms were sold to blacksmiths who refined and forged them into tools.

11. The prevalent economic and political situations in the region controlled the development of the various technological phases of the industry. The threat to labour need of the industry in the middle and the late phases of the industry was overcome by adopting straight smelting techniques which had the least labour need.

SUGGESTIONS FOR FURTHER WORK

This research has not only identified developments in Nsukka bloomery iron smelting but has also opened up wider areas for research in Igbo prehistory.

1. Before this research was undertaken, Professor Shaw (1970) published his archaeological discoveries in Igbo-Ukwu, a town about 98 km south of Nsukka. Excavations at Igbo-Ukwu discovered complex metallurgical objects mainly copper and bronzes. Evidence for iron working discovered during the excavation include iron objects and slags. According to carbon-14 dates, Igbo-Ukwu metallurgical industry can be dated to about the 9th century AD.

Since its discovery, the Igbo-Ukwu metallurgical complex has remained isolated in time and space. No archaeological site in Eastern Nigeria has produced similar complex metal objects. According to the carbon-14 dates of the present research, Igbo-Ukwu dates fit into the middle phase of Nsukka iron smelting. For a better understanding of Igbo civilisation, there is a need to know if and how the iron produced in Nsukka aided the growth of Igbo-Ukwu metallurgy and civilisation.

2. The earliest widely accepted site for iron working in sub-saharan Africa is at Taruga in central Nigeria. Taruga sites belong to the Nok culture which has its southern most sites in Katsina-Ala. Earliest dates for iron working at Taruga belong to the period 400-200 BC (Tylecote 1975a:1). Katsina-Ala is about 350 km north-east of Nsukka.

Carbon-14 dates from the present research show that there was iron smelting in Nsukka from about 765 BC-75 AD during the early phase of Nsukka iron smelting. The present dates from Nsukka have raised the question of where iron was first smelted in West Africa i.e. within the Nok culture area or Nsukka Division. This is a crucial issue which future research on iron working in Nigeria should address itself; to find out what relation (if any) existed between the 8th and 5th centuries BC iron smelting within Nsukka Division and the Nok culture respectively.

3. The present research has identified the constitution of Nsukka slags. This is a data base that will aid any research on Nsukka trade links with her neighbours during the Early Iron Age. It is known that Nsukka was and still is on

the major trade routes that link the Igala and the Idoma in the north with the other Igbo peoples, the Ibibio and the Ijaw in the south. The study of slag inclusions in bloomery iron tools recovered from these Nsukka neighbours and beyond can determine how far blooms and iron tools manufactured in Nsukka were distributed. As seen in this research, and discovered also by Todd (1976), the composition of slag inclusion in metals are similar to the composition of the furnace slags.

4. Carbon-14 dates from this research show that iron was smelted in Nsukka for about 2000 years. From the composition of Nsukka slags most of the iron in the smelted ores was extracted. For a better understanding of Nsukka and Igbo prehistory, further research is necessary to discover how the iron produced during this long period was consumed and what effects its consumption had on the societies that used it.

5. Using the mean values of Fe in the iron ores from Opi in table 62, the ratio of Fe : Si is about 2 : 1. The value of iron in Opi ores is too low and would not be possible to smelt in bloomery process since some of the iron present in the ore must be lost in liquating the slag. This raises some questions. (1) Did Opi iron smelters pre-treat their ore in order to concentrate it before smelting?. (2) It was argued in Chapter Six that the smelters relocated their smelting sites whenever they exhausted available raw materials in an area. Does the analysed samples from Opi represent low grade ores rejected by Opi iron smelters?. These are questions which future research in the area should try to clarify.

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APPENDIX 1

TABLE OF BULK ANALYSES

OWERRE-ELU SLAG BULK ANALYSES

TABLE 1

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/1/90	FeO	61.49	60.83	60.47	60.93	0.42
	Al_2O_3	8.53	10.27	7.56	8.79	1.12
	MgO	N.D	0.27	N.D	0.09	0.13
	SiO_2	26.07	24.53	27.89	26.16	1.37
	T_1O_2	1.25	1.20	0.93	1.13	0.14
	MnO	1.21	1.20	1.38	1.26	0.08
	P_2O_5	0.76	0.97	0.85	0.86	0.09
	CaO	0.65	0.33	0.53	0.50	0.13
	S	N.A	N.A	N.A	N.A	N.A
	V_2O_5	N.A	N.A	N.A	N.A	N.A
	K_2O	0.04	0.40	0.39	0.28	0.17
	Total	100	100	100	99.99	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.83	

TABLE 2

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/2/90	FeO	64.92	62.95	66.33	64.73	1.39
	Al_2O_3	8.86	11.86	8.27	9.66	1.57
	MgO	N.D	0.93	0.49	0.47	0.38
	SiO_2	20.93	19.23	19.28	19.81	0.79
	TiO_2	0.91	1.30	1.54	1.25	0.26
	MnO	3.45	2.82	3.08	3.12	0.26
	P_2O_5	0.37	0.57	0.79	0.58	0.17
	CaO	0.19	0.15	0.01	0.12	0.08
	S	0.22	0.10	N.D	0.11	0.09
	V_2O_5	0.16	0.10	0.23	0.16	0.05
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	100	100	100	100.01	
	$\frac{CaO}{SiO_2}$				0.01	
	K				2.33	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 3

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/3/90	FeO	65.75	66.17	64.82	65.58	0.56
	Al_2O_3	8.60	8.69	9.02	8.77	0.18
	MgO	0.02	0.44	0.33	0.26	0.18
	SiO_2	22.05	20.54	21.72	21.44	0.65
	TiO_2	1.28	1.35	1.52	1.38	0.10
	MnO	1.63	1.04	1.20	1.29	0.25
	P_2O_5	0.46	0.98	0.69	0.71	0.21
	CaO	0.09	0.57	0.55	0.40	0.22
	S	0.13	0.20	0.10	0.14	0.04
	V_2O_5	N.D	0.03	0.05	0.03	0.02
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				2.24	

TABLE 4

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/4/90	FeO	63.10	61.66	61.11	61.96	0.84
	Al_2O_3	7.62	8.12	8.54	8.09	0.38
	MgO	0.50	0.55	N.D	0.35	0.25
	SiO_2	24.47	25.92	26.20	25.53	0.76
	TiO_2	1.35	1.15	1.19	1.23	0.09
	MnO	1.14	1.03	1.45	1.21	0.18
	P_2O_5	0.93	0.85	0.89	0.89	0.03
	CaO	0.70	0.58	0.51	0.60	0.08
	S	0.10	0.12	0.13	0.12	0.01
	V_2O_5	0.10	0.01	N.D	0.04	0.04
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	100	100	100	100.02	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.92	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 5

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/5/90	FeO	64.25	61.47	62.49	62.74	1.15
	Al_2O_3	8.00	13.50	14.76	12.09	2.94
	MgO	0.39	1.31	0.44	0.71	0.42
	SiO_2	23.03	19.77	19.33	20.71	1.65
	TiO_2	1.42	1.50	1.42	1.45	0.04
	MnO	1.27	1.50	1.40	1.39	0.09
	P_2O_5	0.80	0.38	0.04	0.41	0.31
	CaO	0.47	0.30	0.08	0.28	0.16
	S	0.23	0.10	N.D	0.11	0.09
	V_2O_5	N.D	0.14	N.D	0.05	0.07
	K_2O	0.14	0.03	0.04	0.07	0.05
	Total	100	100	100	100.01	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.98	

TABLE 6

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/6/90	FeO	60.67	61.16	60.69	60.84	0.23
	Al_2O_3	9.55	9.95	9.60	9.70	0.18
	MgO	N.D	N.D	0.54	0.18	0.25
	SiO_2	26.80	25.80	27.22	26.61	0.60
	TiO_2	1.26	1.24	1.11	1.20	0.07
	MnO	0.41	0.34	0.19	0.31	0.09
	P_2O_5	0.27	0.60	0.25	0.37	0.16
	CaO	0.26	0.11	0.33	0.23	0.09
	S	0.10	N.D	N.D	0.03	0.05
	V_2O_5	0.28	0.44	N.D	0.24	0.18
	K_2O	0.40	0.36	0.07	0.28	0.15
	Total	100	100	100	99.9	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.71	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 7

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/7/90	FeO	61.02	60.42	59.97	60.47	0.43
	Al_2O_3	9.71	7.58	10.12	9.14	1.11
	MgO	N.D	0.24	0.10	0.11	0.10
	SiO_2	26.78	28.11	26.69	27.19	0.65
	TiO_2	1.38	1.22	1.16	1.25	0.09
	MnO	0.49	0.55	0.48	0.51	0.03
	P_2O_5	0.27	0.80	0.61	0.53	0.22
	CaO	0.12	0.45	0.22	0.26	0.14
	S	N.D	0.15	0.21	0.12	0.09
	V_2O_5	0.15	0.04	0.17	0.12	0.06
	K_2O	0.09	0.44	0.36	0.30	0.15
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				0.71	

TABLE 8

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/8/90	FeO	65.21	62.62	64.78	64.20	1.13
	Al_2O_3	7.89	9.17	8.32	8.46	0.53
	MgO	N.D	0.24	N.D	0.08	0.11
	SiO_2	23.89	24.70	24.23	24.27	0.33
	TiO_2	1.37	1.57	1.27	1.40	0.12
	MnO	0.50	0.46	0.25	0.40	0.11
	P_2O_5	0.61	0.48	0.44	0.51	0.07
	CaO	0.30	0.30	0.32	0.31	0.03
	S	0.08	N.D	N.D	0.03	0.04
	V_2O_5	N.D	0.16	0.08	0.08	0.07
	K_2O	0.16	0.29	0.31	0.25	0.07
	Total	100.01	99.99	100	99.99	
	$\frac{CaO}{SiO_2}$				0.01	
	K				2.01	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 9

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/9/90	FeO	64.28	64.54	64.19	64.34	0.15
	Al_2O_3	9.20	7.92	9.04	8.72	0.57
	MgO	0.19	N.D	0.08	0.09	0.08
	SiO_2	23.60	24.07	23.56	23.74	0.23
	TiO_2	1.34	1.81	1.45	1.53	0.20
	MnO	0.36	0.51	0.38	0.42	0.07
	P_2O_5	0.26	0.72	0.52	0.50	0.19
	CaO	0.26	0.26	0.25	0.26	03
	S	0.03	N.D	0.22	0.08	0.10
	V_2O_5	0.19	N.D	N.D	0.06	0.09
	K_2O	0.28	0.16	0.33	0.26	0.07
	Total	99.99	99.99	99.02	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				2.03	

TABLE 10

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/10/90	FeO	68.66	64.78	66.65	66.70	1.58
	Al_2O_3	7.75	11.89	11.59	10.41	1.88
	MgO	0.29	N.D	0.62	0.30	0.25
	SiO_2	19.60	19.79	17.71	19.03	0.94
	TiO_2	1.55	1.69	1.55	1.60	0.07
	MnO	0.65	0.66	0.41	0.57	0.12
	P_2O_5	0.82	0.54	0.82	0.73	0.13
	CaO	0.43	0.55	0.60	0.53	0.07
	S	N.D	N.D	N.D	N.D	N.D
	V_2O_5	0.14	0.05	N.D	0.06	0.06
	K_2O	0.11	0.06	0.03	0.07	0.03
	Total	100	100.01	99.98	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				2.33	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 11

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/11/90	FeO	65.94	66.59	66.32	66.28	0.27
	Al_2O_3	7.98	6.16	7.64	7.26	0.79
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	20.77	22.20	21.21	21.39	0.60
	TiO_2	0.96	0.86	1.10	0.97	0.10
	MnO	1.73	1.38	1.55	1.55	0.14
	P_2O_5	0.76	0.91	0.78	0.82	0.06
	CaO	1.14	1.13	0.90	1.06	0.11
	S	N.D	0.14	N.D	0.05	0.07
	V_2O_5	0.24	0.10	N.D	0.11	0.10
	K_2O	0.48	0.53	0.50	0.50	0.02
	Total	100	100	100	99.99	
	$\frac{CaO}{SiO_2}$				0.05	
	K				2.45	

TABLE 12

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OW/12/90	FeO	69.04	68.97	68.28	68.76	0.34
	Al_2O_3	6.96	7.79	8.25	7.67	0.53
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	22.06	21.44	21.58	21.70	0.26
	TiO_2	0.81	1.17	1.15	1.04	0.17
	MnO	0.04	0.10	0.02	0.05	0.03
	P_2O_5	0.50	0.34	0.54	0.46	0.09
	CaO	0.19	0.14	0.18	0.17	0.02
	S	N.D	N.D	N.D	N.D	N.D
	V_2O_5	0.40	0.05	N.D	0.15	0.18
	K_2O	N.D	N.D	N.D	N.D	N.D
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				2.36	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

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TABLE 13

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/7/90	FeO	58.62	61.72	60.08	60.14	1.27
	Al_2O_3	12.73	10.32	12.92	11.99	1.18
	MgO	0.26	N.D	0.11	0.12	0.11
	SiO_2	26.91	26.10	24.91	25.97	0.82
	TiO_2	0.91	1.37	1.26	1.18	0.20
	MnO	0.08	N.D	0.01	0.03	0.04
	P_2O_5	0.45	0.38	0.42	0.42	0.02
	CaO	0.03	0.11	0.20	0.11	0.07
	S	N.D	N.D	0.09	0.03	0.04
	V_2O_5	N.D	N.D	N.D	N.D	N.D
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	99.99	100	100	99.99	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				1.60	

TABLE 14

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/8/90	FeO	57.65	61.06	57.85	58.85	1.56
	Al_2O_3	13.01	10.03	12.72	11.92	1.34
	MgO	0.72	N.D	N.D	0.24	0.34
	SiO_2	26.45	26.80	27.30	26.85	0.35
	TiO_2	1.19	1.33	1.00	1.17	0.14
	MnO	0.11	0.10	0.13	0.11	0.01
	P_2O_5	0.63	0.52	0.72	0.63	0.09
	CaO	N.D	0.06	0.11	0.06	0.04
	S	0.02	0.10	N.D	0.04	0.05
	V_2O_5	N.D	N.D	N.D	N.D	N.D
	K_2O	0.22	N.D	0.17	0.13	0.09
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				1.54	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 15

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/9/90	FeO	52.81	51.97	49.51	51.43	1.40
	Al_2O_3	11.07	12.00	13.36	12.14	0.94
	MgO	0.24	N.D	0.05	0.10	0.10
	SiO_2	32.68	32.62	33.61	32.97	0.45
	TiO_2	1.79	1.98	1.83	1.87	0.08
	MnO	N.D	0.11	0.13	0.08	0.06
	P_2O_5	0.92	0.95	0.55	0.81	0.18
	CaO	0.27	0.20	0.29	0.25	0.04
	S	0.11	N.D	0.08	0.06	0.05
	V_2O_5	N.D	N.D	0.43	0.14	0.20
	K_2O	0.11	0.17	0.15	0.14	0.02
	Total	100	100	99.99	99.99	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.17	

TABLE 16

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/10/90	FeO	50.19	49.86	49.38	49.81	0.33
	Al_2O_3	12.98	12.92	13.60	13.16	0.31
	MgO	N.D	0.35	0.67	0.34	0.27
	SiO_2	33.95	33.70	32.84	33.50	0.48
	TiO_2	1.67	1.86	1.56	1.70	0.12
	MnO	0.08	N.D	0.30	0.13	0.13
	P_2O_5	0.66	0.69	1.26	0.86	0.28
	CaO	0.10	0.18	0.15	0.14	0.03
	S	0.16	0.20	0.02	0.13	0.08
	V_2O_5	0.09	N.D	N.D	0.03	0.04
	K_2O	0.12	0.25	0.22	0.20	0.06
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				1.08	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 17

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/11/90	FeO	53.16	53.48	51.64	52.76	0.80
	Al_2O_3	12.14	10.95	14.87	12.65	1.64
	MgO	0.08	N.D	0.21	0.10	0.09
	SiO_2	31.61	32.42	29.73	31.25	1.13
	TiO_2	1.65	1.69	1.83	1.72	0.08
	MnO	N.D	0.02	N.D	0.01	0.03
	P_2O_5	0.73	0.77	1.06	0.85	0.15
	CaO	0.34	0.48	0.45	0.42	0.06
	S	0.12	0.05	0.09	0.09	0.03
	V_2O_5	0.10	N.D	0.02	0.04	0.04
	K_2O	0.07	0.13	0.10	0.10	0.02
	Total	100	99.9	100	99.99	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.20	

TABLE 18

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/12/90	FeO	54.69	56.60	54.61	55.30	0.92
	Al_2O_3	11.63	12.58	14.03	12.75	0.99
	MgO	0.57	0.03	N.D	0.20	0.26
	SiO_2	29.87	27.46	27.99	28.44	1.03
	TiO_2	1.83	1.72	1.73	1.76	0.05
	MnO	N.D	0.03	N.D	0.01	0.01
	P_2O_5	0.70	0.78	0.88	0.79	0.07
	CaO	0.40	0.40	0.44	0.41	0.02
	S	0.18	0.07	0.05	0.10	0.06
	V_2O_5	N.D	0.15	0.03	0.06	0.06
	K_2O	0.13	0.18	0.23	0.18	0.04
	Total	100	100	99.99	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.40	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 19

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/13/90	FeO	54.96	54.57	54.07	54.53	0.36
	Al_2O_3	10.62	11.76	11.10	11.16	0.47
	MgO	N.D	0.09	1.01	0.37	0.46
	SiO_2	30.64	29.83	30.24	30.24	0.33
	TiO_2	1.54	1.47	1.71	1.57	0.10
	MnO	0.05	N.D	N.D	0.02	0.02
	P_2O_5	1.31	0.98	1.20	1.16	0.14
	CaO	0.30	0.53	0.45	0.43	0.10
	S	0.06	0.19	0.03	0.09	0.07
	V_2O_5	0.13	0.12	N.D	0.08	0.06
	K_2O	0.39	0.46	0.19	0.35	0.12
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.3	

TABLE 20

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/14/90	FeO	54.25	52.22	53.55	53.34	0.84
	Al_2O_3	10.94	10.81	10.35	10.70	0.25
	MgO	0.03	N.D	0.77	0.26	0.36
	SiO_2	30.69	32.76	31.26	31.57	0.87
	TiO_2	1.55	1.51	1.64	1.56	0.05
	MnO	0.04	0.02	N.D	0.02	0.02
	P_2O_5	1.44	1.48	1.54	1.49	0.04
	CaO	0.64	0.55	0.60	0.60	0.04
	S	0.10	0.01	0.03	0.05	0.04
	V_2O_5	0.12	0.20	N.D	0.11	0.08
	K_2O	0.20	0.44	0.26	0.30	0.10
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.3	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 21

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/15/90	FeO	60.49	59.14	59.39	59.67	0.59
	Al_2O_3	8.22	9.16	9.33	8.90	0.49
	MgO	N.D	0.09	N.D	0.03	0.04
	SiO_2	27.36	27.99	27.38	27.57	0.29
	TiO_2	1.64	1.83	1.83	1.76	0.09
	MnO	0.13	0.11	0.14	0.13	0.01
	P_2O_5	1.10	0.98	0.95	1.03	0.06
	CaO	0.54	0.44	0.50	0.49	0.04
	S	0.10	N.D	N.D	0.03	0.05
	V_2O_5	0.12	N.D	0.07	0.06	0.05
	K_2O	0.30	0.26	0.41	0.33	0.06
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.68	

TABLE 22

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/16/90	FeO	59.56	58.40	59.00	58.98	0.47
	Al_2O_3	9.04	9.55	9.45	9.35	0.22
	MgO	0.68	0.24	N.D	0.31	0.28
	SiO_2	27.55	27.73	27.33	27.54	0.16
	TiO_2	1.29	2.05	1.78	1.71	0.31
	MnO	0.14	0.12	0.29	0.18	0.08
	P_2O_5	0.84	1.16	1.18	1.06	0.15
	CaO	0.56	0.34	0.46	0.45	0.09
	S	0.08	0.08	0.23	0.13	0.07
	V_2O_5	0.06	0.08	0.18	0.11	0.06
	K_2O	0.20	0.25	0.10	0.18	0.06
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.65	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 23

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/17/90	FeO	56.51	56.52	59.15	57.39	1.24
	Al_2O_3	9.89	10.92	9.66	10.16	0.55
	MgO	N.D	0.09	0.06	0.05	0.04
	SiO_2	28.60	27.98	27.22	27.93	0.56
	TiO_2	1.98	2.02	1.96	1.99	0.02
	MnO	N.D	N.D	0.10	0.03	0.05
	P_2O_5	1.93	1.25	1.10	1.43	0.36
	CaO	0.67	0.72	0.53	0.64	0.08
	S	0.02	0.12	N.D	0.05	0.05
	V_2O_5	N.D	0.06	N.D	0.02	0.03
	K_2O	0.40	0.32	0.22	0.31	0.07
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.57	

TABLE 24

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OR/18/90	FeO	59.60	57.74	58.57	58.63	0.76
	Al_2O_3	8.73	10.54	8.77	9.35	0.84
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	27.53	27.99	28.68	28.07	0.47
	TiO_2	1.82	1.87	2.00	1.90	0.08
	MnO	0.08	N.D	N.D	0.03	0.04
	P_2O_5	1.24	1.15	0.84	1.08	0.17
	CaO	0.43	0.45	0.71	0.53	0.13
	S	0.19	N.D	0.01	0.06	0.09
	V_2O_5	N.D	N.D	N.D	N.D	N.D
	K_2O	0.38	0.26	0.42	0.35	0.07
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.62	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

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TABLE 25

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/6/90	FeO	64.12	60.06	61.13	61.77	1.72
	Al_2O_3	5.14	12.46	11.20	9.60	3.20
	MgO	0.86	N.D	0.25	0.37	0.36
	SiO_2	28.32	24.39	25.70	26.13	1.63
	TiO_2	0.76	1.95	1.17	1.30	0.49
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.36	0.39	0.34	0.36	0.02
	CaO	N.D	0.42	0.13	0.18	0.17
	S	0.24	0.10	N.D	0.12	0.10
	V_2O_5	0.13	N.D	N.D	0.04	0.06
	K_2O	0.08	0.23	0.08	0.13	0.07
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.75	

TABLE 26

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/7/90	FeO	56.16	53.24	53.25	54.22	1.17
	Al_2O_3	10.59	11.10	11.19	10.96	0.26
	MgO	N.D	N.D	0.30	0.10	0.14
	SiO_2	28.56	30.83	30.75	30.05	1.05
	TiO_2	2.90	2.82	2.33	2.68	0.25
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.67	0.89	0.92	0.83	0.11
	CaO	0.68	0.77	0.73	0.73	0.04
	S	0.10	0.10	0.21	0.13	0.05
	V_2O_5	N.D	N.D	0.05	0.02	0.02
	K_2O	0.34	0.25	0.27	0.28	0.04
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.37	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 27

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/8/90	FeO	53.70	54.58	57.61	55.30	1.67
	Al_2O_3	14.94	11.88	11.77	12.86	1.47
	MgO	N.D	0.22	N.D	0.07	0.10
	SiO_2	28.58	30.51	28.12	29.07	1.04
	TiO_2	1.50	1.21	1.38	1.36	0.12
	MnO	N.D	0.02	N.D	0.01	0.03
	P_2O_5	0.42	0.59	0.29	0.43	0.12
	CaO	0.36	0.47	0.31	0.38	0.07
	S	N.D	0.02	N.D	0.01	0.03
	V_2O_5	N.D	0.12	0.16	0.09	0.07
	K_2O	0.49	0.38	0.36	0.41	0.06
	Total	99.99	100	100	99.99	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.35	

TABLE 28

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/9/90	FeO	48.49	48.35	47.06	47.97	0.64
	Al_2O_3	12.70	14.01	12.92	13.21	0.57
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	35.80	34.86	36.92	35.86	0.84
	TiO_2	1.26	1.38	1.41	1.35	0.06
	MnO	0.09	0.16	N.D	0.08	0.07
	P_2O_5	0.57	0.28	0.54	0.46	0.13
	CaO	0.53	0.46	0.64	0.54	0.07
	S	0.10	N.D	0.04	0.05	0.04
	V_2O_5	0.07	0.04	N.D	0.04	0.03
	K_2O	0.39	0.46	0.46	0.44	0.03
	Total	100	100	99.99	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.01	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 29

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/10/90	FeO	63.38	63.56	61.28	62.74	1.03
	Al_2O_3	7.00	6.50	10.78	8.09	1.91
	MgO	N.D	0.24	N.D	0.08	0.11
	SiO_2	27.50	27.98	26.00	27.16	0.84
	TiO_2	1.11	1.04	0.85	1.00	0.11
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.34	0.30	0.82	0.49	0.24
	CaO	0.30	0.07	N.D	0.12	0.13
	S	0.05	0.14	0.11	0.10	0.04
	V_2O_5	0.15	0.11	0.04	0.10	0.05
	K_2O	0.17	0.06	0.12	0.12	0.04
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				1.8	

TABLE 30

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/11/90	FeO	49.32	49.04	49.92	49.43	0.37
	Al_2O_3	13.98	14.12	13.91	14.00	0.09
	MgO	N.D	0.62	N.D	0.21	0.29
	SiO_2	33.99	33.17	33.48	33.55	0.34
	TiO_2	1.51	1.26	1.52	1.43	0.12
	MnO	0.16	0.25	0.09	0.17	0.07
	P_2O_5	0.39	0.34	0.29	0.34	0.04
	CaO	0.37	0.47	0.26	0.37	0.09
	S	N.D	0.11	0.17	0.09	0.07
	V_2O_5	N.D	0.30	0.16	0.15	0.12
	K_2O	0.28	0.32	0.20	0.26	0.05
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.06	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 31

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/12/90	FeO	59.33	61.10	59.54	59.99	0.79
	Al_2O_3	9.59	8.88	9.62	9.36	0.34
	MgO	0.27	0.18	0.27	0.24	0.04
	SiO_2	27.14	26.71	26.88	26.91	0.18
	TiO_2	1.51	1.45	1.57	1.51	0.05
	MnO	0.02	N.D	0.02	0.01	0.01
	P_2O_5	0.73	0.53	0.26	0.51	0.19
	CaO	0.76	0.63	0.95	0.78	0.13
	S	0.20	N.D	0.15	0.12	0.08
	V_2O_5	0.11	N.D	0.18	0.10	0.07
	K_2O	0.33	0.52	0.55	0.47	0.10
	Total	99.99	100	99.99	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.70	

TABLE 32

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/13/90	FeO	56.34	57.78	58.53	57.55	0.91
	Al_2O_3	10.24	9.75	9.36	9.78	0.36
	MgO	N.D	0.22	0.15	0.12	0.09
	SiO_2	30.46	29.44	29.56	29.82	0.46
	TiO_2	1.33	1.24	0.97	1.18	0.15
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.55	0.53	0.42	0.50	0.06
	CaO	0.89	0.83	0.67	0.80	0.09
	S	0.12	0.02	N.D	0.05	0.05
	V_2O_5	N.D	N.D	N.D	N.D	N.D
	K_2O	0.07	0.19	0.34	0.20	0.11
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.49	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 33

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/14/90	FeO	65.45	61.75	60.73	62.64	2.03
	Al_2O_3	8.68	9.22	9.18	9.03	0.25
	MgO	N.D	0.08	N.D	0.03	0.04
	SiO_2	24.48	26.14	27.00	25.87	1.05
	TiO_2	0.97	1.45	1.87	1.43	0.37
	MnO	0.02	N.D	0.18	0.07	0.08
	P_2O_5	0.11	1.06	0.55	0.57	0.39
	CaO	0.16	0.18	0.35	0.23	0.08
	S	0.06	0.04	0.04	0.05	0.01
	V_2O_5	N.D	0.03	0.09	0.04	0.04
	K_2O	0.07	0.05	0.01	0.04	0.02
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.82	

TABLE 34

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/15/90	FeO	59.89	61.80	58.82	60.17	1.23
	Al_2O_3	9.39	8.66	9.74	9.26	0.45
	MgO	N.D	N.D	0.01	N.D	N.D
	SiO_2	27.53	26.77	27.80	27.37	0.44
	TiO_2	1.44	1.27	1.42	1.38	0.08
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.54	0.51	0.83	0.63	0.14
	CaO	0.59	0.61	0.63	0.61	0.02
	S	0.10	N.D	0.16	0.09	0.07
	V_2O_5	N.D	N.D	0.22	0.07	0.10
	K_2O	0.52	0.38	0.36	0.42	0.07
	Total	100	100	99.99	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.69	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 35

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/16/90	FeO	61.49	62.08	60.47	61.35	0.67
	Al_2O_3	6.99	8.37	8.24	7.87	0.62
	MgO	N.D	N.D	0.40	0.13	0.19
	SiO_2	28.42	26.77	27.69	27.63	0.68
	TiO_2	1.61	1.38	1.52	1.50	0.09
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.54	0.46	0.32	0.44	0.09
	CaO	0.61	0.54	0.77	0.64	0.10
	S	N.D	0.07	0.20	0.09	0.08
	V_2O_5	N.D	0.06	N.D	0.02	0.03
	K_2O	0.34	0.27	0.39	0.33	0.05
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.77	

TABLE 36

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
UM/17/90	FeO	59.82	59.47	58.60	59.30	0.51
	Al_2O_3	9.67	10.84	11.35	10.62	0.70
	MgO	0.45	0.44	N.D	0.30	0.21
	SiO_2	27.55	25.30	26.83	26.56	0.94
	TiO_2	1.28	1.53	1.57	1.46	0.13
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	0.03	1.13	0.50	0.55	0.45
	CaO	0.59	0.54	0.60	0.58	0.03
	S	0.12	0.17	0.06	0.12	0.04
	V_2O_5	N.D	0.28	0.06	0.11	0.12
	K_2O	0.49	0.30	0.43	0.40	0.08
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.64	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

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TABLE 37

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/5/90	FeO	61.49	56.73	61.47	59.90	2.24
	Al_2O_3	11.18	19.51	11.88	14.19	3.77
	MgO	N.D	0.16	N.D	0.05	0.08
	SiO_2	23.40	21.29	24.90	23.20	1.48
	TiO_2	1.36	0.99	0.47	0.94	0.37
	MnO	0.06	0.25	N.D	0.10	0.11
	P_2O_5	1.02	0.29	0.45	0.59	0.32
	CaO	0.95	0.64	0.49	0.69	0.19
	S	0.10	N.D	0.01	0.04	0.04
	V_2O_5	0.44	0.13	0.33	0.30	0.13
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	100	99.99	100	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.64	

TABLE 38

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/6/90	FeO	58.18	58.10	56.38	57.55	0.83
	Al_2O_3	11.65	12.00	14.12	12.59	1.09
	MgO	N.D	0.97	N.D	0.32	0.46
	SiO_2	25.59	25.54	25.52	25.55	0.03
	TiO_2	1.36	1.40	1.53	1.43	0.07
	MnO	0.08	N.D	0.02	0.03	0.03
	P_2O_5	0.98	0.81	1.03	0.94	0.09
	CaO	0.99	0.59	0.70	0.77	0.17
	S	N.D	N.D	N.D	N.D	N.D
	V_2O_5	0.18	0.10	N.D	0.09	0.07
	K_2O	0.98	0.50	0.70	0.73	0.20
	Total	99.99	100.01	100	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.57	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 39

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/7/90	FeO	62.20	59.75	58.82	60.26	1.43
	Al_2O_3	13.48	15.93	18.44	15.95	2.02
	MgO	0.76	0.51	N.D	0.42	0.32
	SiO_2	20.48	20.62	19.66	20.25	0.42
	TiO_2	1.11	1.20	1.49	1.27	0.16
	MnO	0.03	0.25	0.19	0.16	0.09
	P_2O_5	1.12	0.74	0.66	0.84	0.20
	CaO	0.45	0.52	0.32	0.43	0.08
	S	N.D	0.16	0.17	0.11	0.08
	V_2O_5	0.13	N.D	0.12	0.08	0.06
	K_2O	0.24	0.32	0.13	0.23	0.08
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.71	

TABLE 40

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/8/90	FeO	52.53	52.95	56.04	53.84	1.57
	Al_2O_3	16.53	14.97	15.32	15.61	0.67
	MgO	0.59	1.07	0.74	0.80	0.20
	SiO_2	25.71	26.59	23.85	25.38	1.14
	TiO_2	1.31	0.93	1.13	1.12	0.16
	MnO	0.09	0.15	0.17	0.14	0.03
	P_2O_5	1.43	1.32	0.86	1.20	0.25
	CaO	0.74	1.11	0.95	0.94	0.16
	S	0.14	0.04	0.04	0.07	0.05
	V_2O_5	0.19	0.02	0.25	0.15	0.10
	K_2O	0.74	0.85	0.66	0.75	0.08
	Total	100	100	100.01	100	
	$\frac{CaO}{SiO_2}$				0.04	
	K				1.39	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 41

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/9/90	FeO	57.16	57.35	56.63	57.04	0.30
	Al_2O_3	9.44	9.70	9.23	9.46	0.19
	MgO	0.08	N.D	0.74	0.27	0.33
	SiO_2	27.62	27.93	28.03	27.86	0.17
	TiO_2	0.99	1.08	0.92	1.00	0.07
	MnO	0.11	0.16	N.D	0.09	0.07
	P_2O_5	1.73	1.62	1.86	1.73	0.09
	CaO	1.56	1.14	1.38	1.36	0.17
	S	0.10	N.D	0.16	0.09	0.07
	V_2O_5	N.D	N.D	0.05	0.02	0.02
	K_2O	1.21	1.02	1.00	1.08	0.09
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.05	
	K				1.64	

TABLE 42

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/10/90	FeO	58.92	64.86	63.02	62.27	2.48
	Al_2O_3	17.45	11.28	12.30	13.68	2.70
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	19.83	21.53	18.09	19.82	1.40
	TiO_2	1.25	1.13	1.39	1.25	0.11
	MnO	0.15	0.23	N.D	0.13	0.10
	P_2O_5	1.10	0.22	2.91	1.42	1.11
	CaO	0.41	0.27	1.67	0.78	0.63
	S	0.12	N.D	0.16	0.09	0.07
	V_2O_5	0.12	0.35	N.D	0.16	0.15
	K_2O	0.64	0.13	0.45	0.40	0.21
	Total	99.99	100	99.99	100	
	$\frac{CaO}{SiO_2}$				0.04	
	K				1.90	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 43

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/11/90	FeO	58.99	60.75	60.31	60.02	0.75
	Al_2O_3	17.26	17.50	17.50	17.42	0.11
	MgO	0.62	N.D	0.02	0.21	0.29
	SiO_2	20.22	19.45	19.70	19.79	0.32
	TiO_2	1.52	1.13	1.15	1.26	0.18
	MnO	N.D	0.09	N.D	0.03	0.04
	P_2O_5	0.68	0.70	0.54	0.65	0.08
	CaO	0.30	0.25	0.35	0.30	0.04
	S	0.02	N.D	0.08	0.03	0.03
	V_2O_5	0.14	N.D	0.30	0.15	0.13
	K_2O	0.25	0.13	0.05	0.14	0.08
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.64	

TABLE 44

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/12/90	FeO	55.07	57.46	54.52	55.68	1.28
	Al_2O_3	11.70	10.15	10.70	10.85	0.64
	MgO	0.57	N.D	0.82	0.46	0.34
	SiO_2	28.12	28.27	29.58	28.66	0.66
	TiO_2	1.02	0.96	0.96	0.98	0.03
	MnO	0.22	0.16	0.28	0.22	0.05
	P_2O_5	1.30	0.95	0.78	1.01	0.22
	CaO	0.94	0.95	1.08	0.99	0.06
	S	0.10	0.14	N.D	0.08	0.06
	V_2O_5	0.10	0.14	N.D	0.08	0.06
	K_2O	0.84	0.81	1.29	0.98	0.22
	Total	99.98	99.99	100.01	99.99	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.49	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 45

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/13/90	FeO	57.31	53.31	57.32	55.94	1.95
	Al_2O_3	10.17	10.88	9.62	10.22	0.52
	MgO	0.42	0.05	0.02	0.16	0.18
	SiO_2	26.80	29.30	28.56	28.22	1.04
	TiO_2	1.07	1.15	0.87	1.03	0.12
	MnO	N.D	0.09	0.02	0.04	0.04
	P_2O_5	1.70	1.81	1.54	1.68	0.11
	CaO	1.40	1.53	0.82	1.25	0.31
	S	N.D	N.D	N.D	N.D	N.D
	V_2O_5	0.07	N.D	0.40	0.16	0.18
	K_2O	1.06	2.01	0.83	1.30	0.51
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.04	
	K				1.57	

TABLE 46

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/14/90	FeO	56.36	57.18	56.94	56.83	0.34
	Al_2O_3	10.42	8.90	13.63	10.98	1.97
	MgO	0.36	0.34	N.D	0.23	0.17
	SiO_2	27.55	28.85	24.93	27.11	1.63
	TiO_2	1.11	1.06	1.07	1.08	0.02
	MnO	N.D	N.D	N.D	N.D	N.D
	P_2O_5	1.24	1.45	1.21	1.30	0.10
	CaO	1.34	1.19	1.06	1.20	0.11
	S	0.02	N.D	0.12	0.05	0.05
	V_2O_5	0.06	0.18	N.D	0.08	0.07
	K_2O	1.54	0.84	1.04	1.14	0.29
	Total	100	99.99	100	100	
	$\frac{CaO}{SiO_2}$				0.04	
	K				1.59	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 47

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/15/90	FeO	59.87	63.33	61.78	61.66	1.42
	Al_2O_3	15.96	13.66	13.86	14.49	1.04
	MgO	0.40	0.73	0.33	0.49	0.17
	SiO_2	20.90	19.13	20.32	20.12	0.74
	TiO_2	1.18	1.70	1.18	1.35	0.25
	MnO	N.D	0.30	0.40	0.23	0.17
	P_2O_5	0.92	0.70	0.84	0.82	0.09
	CaO	0.46	0.41	0.66	0.51	0.11
	S	0.14	N.D	0.10	0.08	0.05
	V_2O_5	0.06	0.03	0.24	0.11	0.09
	K_2O	0.11	0.01	0.29	0.14	0.12
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.80	

TABLE 48

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/16/90	FeO	61.34	57.44	60.98	59.92	1.76
	Al_2O_3	16.05	22.53	16.17	18.25	3.03
	MgO	0.21	0.31	0.16	0.23	0.06
	SiO_2	19.08	16.90	20.10	18.69	1.33
	TiO_2	1.09	1.15	1.24	1.16	0.06
	MnO	0.16	0.23	0.36	0.25	0.08
	P_2O_5	0.90	0.82	0.50	0.74	0.17
	CaO	0.37	0.19	0.23	0.26	0.08
	S	0.09	0.09	0.03	0.07	0.03
	V_2O_5	0.30	0.04	0.13	0.15	0.11
	K_2O	0.41	0.30	0.10	0.28	0.12
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.01	
	K				1.66	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 49

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/17/90	FeO	54.21	54.49	56.83	55.17	1.17
	Al_2O_3	13.03	12.79	11.77	12.53	0.55
	MgO	0.63	0.89	0.40	0.64	0.20
	SiO_2	27.70	27.41	27.01	27.37	0.28
	TiO_2	1.06	0.92	1.10	1.03	0.08
	MnO	N.D	0.10	0.38	0.16	0.16
	P_2O_5	0.84	1.20	0.84	0.96	0.17
	CaO	0.92	0.94	0.83	0.90	0.05
	S	N.D	N.D	0.03	0.01	0.01
	V_2O_5	0.24	0.31	0.08	0.21	0.10
	K_2O	1.37	0.95	0.73	1.02	0.27
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.03	
	K				1.46	

TABLE 50

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/18/90	FeO	59.63	62.04	61.71	61.12	1.07
	Al_2O_3	20.50	17.52	14.98	17.66	2.26
	MgO	0.48	N.D	0.69	0.39	0.29
	SiO_2	16.87	17.02	19.57	17.82	1.24
	TiO_2	1.08	1.05	1.40	1.18	0.16
	MnO	0.15	0.17	0.04	0.12	0.06
	P_2O_5	0.61	1.04	0.83	0.83	0.18
	CaO	0.30	0.52	0.22	0.35	0.13
	S	0.02	0.17	N.D	0.06	0.08
	V_2O_5	0.20	0.28	0.38	0.29	0.07
	K_2O	0.16	0.19	0.18	0.18	0.01
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.76	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 51

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
OP/19/90	FeO	62.22	48.06	43.20	51.16	8.07
	Al_2O_3	16.66	8.22	24.50	16.46	6.65
	MgO	N.D	N.D	N.D	N.D	N.D
	SiO_2	18.50	36.53	26.59	27.20	7.37
	TiO_2	1.24	1.06	1.29	1.20	0.10
	MnO	0.13	1.78	2.67	1.53	1.05
	P_2O_5	0.73	2.43	1.48	1.55	0.70
	CaO	0.42	1.07	0.27	0.58	0.35
	S	N.D	0.11	N.D	0.04	0.05
	V_2O_5	N.D	N.D	N.D	N.D	N.D
	K_2O	0.09	0.76	N.D	0.28	0.34
	Total	99.99	100.02	100	100	
	$\frac{CaO}{SiO_2}$				0.02	
	K				1.26	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

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TABLE 52

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
Lej/1/90	FeO	58.76	59.98	59.53	59.42	0.50
	Al_2O_3	7.83	11.17	9.45	9.48	1.36
	MgO	0.55	0.37	N.D	0.31	0.23
	SiO_2	27.27	24.12	26.41	25.93	1.33
	TiO_2	1.03	1.01	0.97	1.00	0.02
	MnO	0.20	N.D	0.14	0.11	0.08
	P_2O_5	1.45	1.44	1.08	1.32	0.17
	CaO	1.70	1.11	1.40	1.41	0.24
	S	0.16	0.11	0.26	0.18	0.06
	V_2O_5	0.06	0.09	0.01	0.05	0.03
	K_2O	0.99	0.60	0.75	0.78	0.16
	Total	100	100	100	99.99	
	$\frac{CaO}{SiO_2}$				0.05	
	K				1.78	

TABLE 53

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
Lej/2/90	FeO	64.51	65.15	65.85	65.17	0.55
	Al_2O_3	7.66	6.73	6.73	7.04	0.44
	MgO	0.51	0.56	0.42	0.50	0.06
	SiO_2	25.34	25.34	24.56	25.08	0.37
	TiO_2	0.76	0.90	0.77	0.81	0.06
	MnO	0.41	0.05	0.10	0.18	0.16
	P_2O_5	0.77	1.08	0.98	0.95	0.12
	CaO	0.01	0.12	0.22	0.11	0.09
	S	N.D	N.D	0.26	0.08	0.12
	V_2O_5	0.03	N.D	0.08	0.04	0.03
	K_2O	N.D	0.07	0.05	0.04	0.03
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				2.07	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 54

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
Lej/3/90	FeO	59.77	62.21	61.88	61.29	1.08
	Al_2O_3	10.38	8.78	12.36	10.51	1.46
	MgO	0.04	0.01	0.18	0.08	0.07
	SiO_2	23.40	24.53	22.84	23.59	0.70
	TiO_2	1.29	0.50	0.94	0.91	0.32
	MnO	0.02	0.16	N.D	0.06	0.07
	P_2O_5	1.25	1.17	0.44	0.95	0.36
	CaO	2.21	1.39	1.00	1.54	0.49
	S	0.20	0.34	0.01	0.18	0.14
	V_2O_5	0.28	0.23	0.13	0.21	0.06
	K_2O	1.17	0.68	0.20	0.68	0.40
	Total	100.01	100	100	100	
	$\frac{CaO}{SiO_2}$				0.07	
	K				1.89	

TABLE 55

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
Lej/4/90	FeO	67.18	64.32	64.54	65.35	1.30
	Al_2O_3	5.60	7.88	8.43	7.30	1.23
	MgO	N.D	0.77	0.41	0.39	0.31
	SiO_2	24.93	25.21	24.89	25.01	0.14
	TiO_2	0.68	0.74	0.75	0.72	0.03
	MnO	0.41	0.32	0.21	0.31	0.08
	P_2O_5	0.83	0.51	0.55	0.63	0.14
	CaO	0.04	0.01	0.17	0.07	0.07
	S	0.23	0.07	N.D	0.10	0.10
	V_2O_5	0.09	0.17	0.05	0.10	0.05
	K_2O	N.A	N.A	N.A	N.A	N.A
	Total	100	100	100	99.98	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				2.05	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 56

Sample Number	Wt. % of Oxides	Area A	Area B	Area C	Mean	S.D.
Lej/5/90	FeO	65.19	63.75	60.67	63.20	1.89
	Al_2O_3	6.43	7.52	7.62	7.19	0.54
	MgO	0.02	0.80	0.40	0.41	0.32
	SiO_2	27.07	26.74	2.04	27.62	1.02
	TiO_2	0.52	0.63	0.64	0.60	0.05
	MnO	0.21	0.19	0.36	0.25	0.08
	P_2O_5	0.52	0.37	1.12	0.67	0.32
	CaO	0.03	N.D	0.15	0.06	0.06
	S	N.A	N.A	N.A	N.A	N.A
	V_2O_5	N.A	N.A	N.A	N.A	N.A
	K_2O	0.01	N.D	N.D	N.D	N.D
	Total	100	100	100	100	
	$\frac{CaO}{SiO_2}$				<0.01	
	K				1.84	

N.A = Not Analysed
N.D = Not Detected
K = Viscosity Coefficient

TABLE 57 **Owerre-Elu Slag Analysis**

Oxides	OW/ 1/90	OW/ 2/90	OW/ 3/90	OW/ 4/90	OW/ 5/90	OW/ 6/90	OW/ 7/90	OW/ 8/90	OW/ 9/90	OW/ 10/90	OW/ 11/90	OW/ 12/90	Mean	SD
FeO	60.93	64.73	65.58	61.96	62.74	60.84	60.47	64.20	64.34	66.70	66.28	68.76	63.96	2.52
Al_2O_3	8.79	9.66	8.77	8.09	12.09	9.70	9.14	8.46	8.72	10.41	7.26	7.67	9.06	1.24
MgO	0.09	0.47	0.26	0.35	0.71	0.18	0.11	0.08	0.09	0.30	N.D	N.D	0.22	0.20
SiO_2	26.16	19.81	21.44	25.53	20.71	26.61	27.19	24.27	23.74	19.03	21.39	21.70	23.13	2.69
TiO_2	1.13	1.25	1.38	1.23	1.45	1.20	1.25	1.40	1.53	1.60	0.97	1.04	1.28	0.18
MnO	1.16	3.12	1.29	1.21	1.39	0.31	0.51	0.40	0.42	0.57	1.55	0.05	1.00	0.80
P_2O_5	0.86	0.58	0.71	0.89	0.41	0.37	0.53	0.51	0.50	0.73	0.82	0.46	0.61	0.17
CaO	0.50	0.12	0.40	0.60	0.28	0.23	0.26	0.31	0.26	0.53	1.06	0.17	0.39	0.25
S	N.A	0.11	0.14	0.12	0.11	0.03	0.12	0.03	0.08	N.D	0.05	N.D	0.07	0.05
V_2O_5	N.A	0.16	0.03	0.04	0.05	0.24	0.12	0.08	0.06	0.06	0.11	0.15	0.10	0.06
K_2O	0.28	N.A	N.A	N.A	0.07	0.28	0.30	0.25	0.26	0.07	0.50	N.D	0.22	0.14
Total	99.99	100.01	100	100.02	100.01	99.99	100	99.99	100	100	99.99	100	100.04	
$\frac{CaO}{SiO_2}$	0.02	0.01	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.03	0.05	0.01	0.02	0.01
K	1.83	2.33	2.24	1.92	1.98	1.71	1.71	2.01	2.03	2.33	2.45	2.36	2.08	0.25
Melting Temperature	1150°C	1280°C	1180°C	1150°C	1325°C	1150°C	1150°C	1150°C	1150°C	1310°C	1150°C	1150°C		

N.A. = Not Analysed
N.D. = Not Detected
K = Viscosity Coefficient

TABLE 58

Orba Slag Analysis

Wt. % of Oxides	OR/ 7/90	OR/ 8/90	OR/ 9/90	OR/ 10/90	OR/ 11/90	OR/ 12/90	OR/ 13/90	OR/ 14/90	OR/ 15/90	OR/ 16/90	OR/ 17/90	OR/ 18/90	Mean	SD
FeO	60.14	58.85	51.43	49.81	52.76	55.30	54.53	53.34	59.67	58.98	57.39	58.63	55.90	3.36
Al_2O_3	11.99	11.92	12.14	13.16	12.65	12.75	11.16	10.70	8.90	9.35	10.16	9.35	11.18	1.41
MgO	0.12	0.24	0.10	0.34	0.10	0.20	0.37	0.26	0.03	0.31	0.05	N.D	0.17	0.12
SiO_2	25.97	26.85	32.97	33.50	31.25	28.44	30.24	31.57	27.57	27.54	27.93	28.07	29.32	2.38
TiO_2	1.18	1.17	1.87	1.70	1.72	1.76	1.57	1.56	1.76	1.71	1.99	1.90	1.65	0.24
MnO	0.03	0.11	0.08	0.13	0.01	0.01	0.02	0.02	0.13	0.18	0.03	0.03	0.06	0.05
P_2O_5	0.42	0.63	0.81	0.86	0.85	0.79	1.16	1.49	1.03	1.06	1.43	1.08	0.97	0.30
CaO	0.11	0.06	0.25	0.14	0.42	0.41	0.43	0.60	0.49	0.45	0.64	0.55	0.37	0.19
S	0.03	0.04	0.06	0.13	0.09	0.10	0.09	0.05	0.03	0.13	0.05	0.06	0.07	0.03
V_2O_5	N.D	N.D	0.14	0.03	0.04	0.06	0.08	0.11	0.06	0.11	0.02	N.D	0.05	0.04
K_2O	N.A	0.13	0.14	0.20	0.10	0.18	0.35	0.30	0.33	0.18	0.31	0.35	0.23	0.09
Total	99.99	100	99.99	100	99.99	100	100	100	100	100	100	100	99.97	
$\frac{CaO}{SiO_2}$	<0.01	<0.01	0.01	<0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02	0.02	0.01	
K	1.60	1.54	1.17	1.08	1.20	1.40	1.30	1.30	1.68	1.65	1.57	1.62	1.43	0.20
Melting Temperature	1280°C	1250°C	1190°C	1209°C	1215°C	1270°C	1190°C	1150°C	1150°C	1150°C	1200°C	1150°C		

N.A. = Not Analysed
N.D. = Not Detected
K = Viscosity Coefficient

TABLE 59 Umundu Slag Analysis

Wt. % of Oxides	UM/ 6/90	UM/ 7/90	UM/ 8/90	UM/ 9/90	UM/ 10/90	UM/ 11/90	UM/ 12/90	UM/ 13/90	UM/ 14/90	UM/ 15/90	UM/ 16/90	UM/ 17/90	Mean	SD
FeO	61.77	54.22	55.30	47.97	62.74	49.43	59.99	57.55	62.64	60.17	61.35	59.30	57.70	4.78
Al_2O_3	9.60	10.96	12.86	13.21	8.09	14.00	9.36	9.78	9.03	9.26	7.87	10.62	10.39	1.92
MgO	0.37	0.10	0.07	N.D	0.08	0.21	0.24	0.12	0.03	N.D	0.13	0.30	0.14	0.11
SiO_2	26.13	30.05	29.07	35.86	27.16	33.55	26.91	29.82	25.87	27.37	27.63	26.56	28.83	2.96
TiO_2	1.30	2.68	1.36	1.35	1.00	1.43	1.51	1.18	1.43	1.38	1.50	1.46	1.47	0.39
MnO	N.D	N.D	0.01	0.08	N.D	0.17	0.01	N.D	0.07	N.D	N.D	N.D	0.02	0.05
P_2O_5	0.36	0.83	0.43	0.46	0.49	0.34	0.51	0.50	0.57	0.63	0.44	0.55	0.51	0.13
CaO	0.18	0.73	0.38	0.54	0.12	0.37	0.78	0.80	0.23	0.61	0.64	0.58	0.49	0.23
S	0.12	0.13	0.01	0.05	0.10	0.09	0.12	0.05	0.05	0.09	0.09	0.12	0.09	0.04
V_2O_5	0.04	0.02	0.09	0.04	0.10	0.15	0.10	N.D	0.04	0.07	0.02	0.11	0.06	0.04
K_2O	0.13	0.28	0.41	0.44	0.12	0.26	0.47	0.20	0.04	0.42	0.33	0.40	0.29	0.14
Total	100	100	99.99	100	100	100	100	100	100	100	100	100	99.99	
$\frac{CaO}{SiO_2}$	0.01	0.02	0.01	0.02	<0.01	0.01	0.03	0.03	0.01	0.02	0.02	0.02	0.02	
K	1.75	1.37	1.35	1.01	1.80	1.06	1.70	1.49	1.82	1.69	1.77	1.64	1.54	0.27
Melting Temperature	1190°C	1190°C	1270°C	1175°C	1160°C	1210°C	1180°C	1150°C	1190°C	1155°C	1155°C	1210°C		

N.A. = Not Analysed
N.D. = Not Detected
K = Viscosity Coefficient

TABLE 60 **Opi Slag Analysis**

Wt. % of Oxides	OP 5/90	OP 6/90	OP 7/90	OP 8/90	OP 9/90	OP 10/90	OP 11/90	OP 12/90	OP 13/90	OP 14/90	OP 15/90	OP 16/90	OP 17/90	OP 18/90	OP 19/90	MEAN	SD
FeO	59.90	57.55	60.26	53.84	57.04	62.27	60.02	55.68	55.94	56.83	61.66	59.92	55.17	61.12	51.16	57.89	3.08
Al_2O_3	14.19	12.59	15.95	15.61	9.46	13.68	17.42	10.85	10.22	10.98	14.49	18.25	12.53	17.66	16.46	14.02	2.76
MgO	0.05	0.32	0.42	0.80	0.27	N.D	0.21	0.46	0.16	0.23	0.49	0.23	0.64	0.39	N.D	0.31	0.22
SiO_2	23.20	25.55	20.25	25.38	27.86	19.82	19.79	28.66	28.22	27.11	20.12	18.69	27.37	17.82	27.20	23.80	3.84
TiO_2	0.94	1.43	1.27	1.12	1.00	1.25	1.26	0.98	1.03	1.08	1.35	1.16	1.03	1.18	1.20	1.15	0.13
MnO	0.10	0.03	0.16	0.14	0.09	0.13	0.03	0.22	0.04	N.D	0.23	0.25	0.16	0.12	1.53	0.21	0.36
P_2O_5	0.59	0.94	0.84	1.20	1.73	1.42	0.65	1.01	1.68	1.30	0.82	0.74	0.96	0.83	1.55	1.08	0.36
CaO	0.69	0.77	0.43	0.94	1.36	0.78	0.30	0.99	1.25	1.20	0.51	0.26	0.90	0.35	0.58	0.75	0.34
S	0.04	N.D	0.11	0.07	0.09	0.09	0.03	0.08	N.D	0.05	0.08	0.07	0.01	0.06	0.04	0.05	0.03
V_2O_5	0.30	0.09	0.08	0.15	0.02	0.16	0.15	0.08	0.16	0.08	0.11	0.15	0.21	0.29	N.D	0.13	0.08
K_2O	N.A	0.73	0.23	0.75	1.08	0.40	0.14	0.98	1.30	1.14	0.14	0.28	1.02	0.18	0.28	0.57	0.42
Total	100	100	100	100	100	100	100	99.99	100	100	100	100	100	100	100	99.96	
$\frac{CaO}{SiO_2}$	0.03	0.03	0.02	0.04	0.05	0.04	0.02	0.03	0.04	0.04	0.03	0.01	0.03	0.02	0.02	0.03	0.01
K	1.64	1.57	1.71	1.39	1.64	1.90	1.64	1.49	1.57	1.59	1.80	1.66	1.46	1.76	1.26	1.60	0.15
Melting Temperature	1350°C	1295°C	1410°C	1350°C	1155°C	1380°C	1430°C	1200°C	1195°C	1230°C	1390°C	1450°C	1280°C	1450°C	1340°C		

N.A. = Not Analysed
N.D. = Not Detected
K = Viscosity Coefficient

TABLE 61 **Leja Slag Analysis**

Wt. % of Oxides	Lej/1/90	Lej/2/90	Lej/3/90	Lej/4/90	Lej/5/90	Mean	SD
FeO	59.42	65.17	61.29	65.35	63.05	62.85	2.27
Al_2O_3	9.48	7.04	10.51	7.30	7.27	8.32	1.41
MgO	0.31	0.50	0.08	0.39	0.31	0.32	0.14
SiO_2	25.93	25.08	23.59	25.01	27.66	25.45	1.33
TiO_2	1.00	0.81	0.91	0.72	0.73	0.83	0.11
MnO	0.11	0.18	0.06	0.31	0.24	0.18	0.09
P_2O_5	1.32	0.95	0.95	0.63	0.65	0.90	0.25
CaO	1.41	0.11	1.54	0.07	0.07	0.64	0.68
S	0.18	0.08	0.18	0.10	N.A	0.13	0.05
V_2O_5	0.05	0.04	0.21	0.10	N.A	0.10	0.07
K_2O	0.78	0.04	0.68	N.A	0.02	0.38	0.35
Total	99.99	100	100	99.98	100	100.10	
$\frac{CaO}{SiO_2}$	0.05	<0.01	0.07	<0.01	<0.01	0.02	0.03
K	1.78	2.07	1.89	2.05	1.83	1.92	0.12
Melting Temperature	1180°C	1155°C	1280°C	1150°C	1160°C		

N.A. = Not Analysed
N.D. = Not Detected
K = Viscosity Coefficient

TABLE 62

Analyses of Iron Ores from Nsukka Division
(Leja, Orba, Owerre-Elu and Umundu)

W % of Elements	OP 3/90	OP 4/90	Mean	S.D.	OR 5/90	OR 6/90	Mean	S.D.	OW 13/90	OW 14/90	OW 15/90	Mean	S.D.	UM 4/90	UM 5/90	Mean	SD
Fe	34.8	27.5	31.15	3.45	41.4	41.0	41.2	0.2	33.20	33.80	35.50	34.20	0.94	42.9	37.6	40.25	2.65
Si	12.1	21.1	16.6	4.5	6.29	6.52	6.41	0.12	12.0	11.60	6.64	10.08	2.44	6.71	10.7	8.71	2.0
Al	5.03	3.32	4.18	0.86	5.59	5.44	5.52	0.08	3.44	5.07	5.82	4.78	0.99	5.85	6.65	6.25	0.40
Ti	1.41	1.23	1.32	0.09	1.43	1.47	1.45	0.02	0.53	1.41	1.48	1.14	0.43	1.48	1.60	1.54	0.06
Ca	0.02	0.02	0.02	0	0.02	0.02	0.02	0	0.02	0.02	0.02	0.02	0	0.02	0.02	0.02	0
Mg	0.01	0.01	0.01	0	0.02	0.02	0.02	0	0.01	0.01	0.02	0.01	4.71	0.02	0.02	0.02	0
P	0.18	0.13	0.16	0.03	0.30	0.26	0.28	0.02	0.04	0.16	0.09	0.10	0.05	0.18	0.06	0.12	0.06
Mn	<0.02	0.22	0.12	0.1	<0.02	<0.02	0.01	0	<0.02	<0.02	<0.02	0.01	0	<0.02	<0.02	0.01	0
Type of Iron Ore	H	H			G	G			H	H	H			G	H		

< = Less than
H = Haematite
G = Goethite

ANALYSES OF MINERAL PHASES IN THE VARIOUS SLAG SAMPLES

OWERRE-ELU SLAGS: PHASE ANALYSES

TABLE 63

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/1/90	FeO	65.50	50.03	26.00
	Al_2O_3	0.74	44.72	15.59
	MgO	0.81	N.D	0.36
	SiO_2	30.53	0.75	48.53
	TiO_2	0.31	3.62	0.45
	MnO	1.39	0.72	0.98
	P_2O_5	0.67	0.08	3.33
	CaO	0.05	0.03	3.37
	S	N.A	N.A	N.A
	V_2O_5	N.A	N.A	N.A
	K_2O	N.D	0.06	1.41
	Total	100	100	100.01

TABLE 64

Sample Number	Wt. % of Oxides	Fayalite	Wustite	Hercynite	Glass
OW/2/90	FeO	62.87	94.61	48.27	41.24
	Al_2O_3	0.45	0.95	48.27	12.85
	MgO	N.D	0.10	N.D	0.32
	SiO_2	31.21	1.34	0.21	39.07
	TiO_2	0.23	1.79	1.41	1.03
	MnO	4.25	1.00	1.53	0.82
	P_2O_5	0.71	0.19	N.D	1.48
	CaO	0.17	0.03	N.D	2.61
	S	N.D	N.D	N.D	0.05
	V_2O_5	0.14	N.D	0.31	0.30
	K_2O	N.A	N.A	N.A	N.A
	Total	100	100	99.99	99.77

N.D. = Not Detected
N.A. = Not Analysed

TABLE 65

Sample Number	Wt. % of Oxides	Fayalite	Wustite	Hercynite	Glass
OW/3/90	FeO	67.01	95.44	51.58	38.81
	Al_2O_3	N.D	0.86	44.57	16.32
	MgO	0.30	N.D	0.59	0.45
	SiO_2	29.75	1.33	0.17	29.40
	TiO_2	0.21	1.52	2.07	2.50
	MnO	1.93	0.41	0.46	3.89
	P_2O_5	0.41	0.44	0.10	4.65
	CaO	0.21	N.D	N.D	1.70
	S	0.12	N.D	N.D	0.52
	V_2O_5	0.08	N.D	0.46	1.69
	K_2O	N.A	N.A	N.A	N.A
	Total	100	100	100	99.93

TABLE 66

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/4/90	FeO	65.29	45.76	35.39
	Al_2O_3	0.51	50.29	4.45
	MgO	0.42	0.77	N.D
	SiO_2	31.35	N.D	35.66
	TiO_2	N.D	1.28	0.56
	MnO	1.52	0.78	0.83
	P_2O_5	0.91	N.D	10.85
	CaO	N.D	N.D	11.83
	S	N.D	0.21	0.42
	V_2O_5	N.D	0.90	N.D
	K_2O	N.A	N.A	N.A
	Total	100	99.99	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 67

Sample Number	Wt. % of Oxides	Fayalite	Hercyrite	Wustite	Glass
OW/5/90	FeO	66.45	50.54	96.09	34.20
	Al_2O_3	0.63	47.04	0.59	15.52
	MgO	0.79	N.D	0.07	0.42
	SiO_2	30.46	0.13	1.16	39.47
	TiO_2	0.02	1.23	1.34	1.02
	MnO	1.38	0.56	0.24	0.64
	P_2O_5	0.13	N.D	0.15	4.77
	CaO	N.D	0.04	0.11	3.41
	S	0.07	N.D	0.12	0.21
	V_2O_5	N.D	0.44	0.13	0.31
	K_2O	0.07	0.02	N.D	0.03
	Total	100	100	100	100

TABLE 68

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/6/90	FeO	67.85	52.42	29.36
	Al_2O_3	0.73	37.10	18.16
	MgO	N.D	0.27	0.28
	SiO_2	30.40	4.40	43.05
	TiO_2	0.15	4.37	3.70
	MnO	0.54	0.05	0.01
	P_2O_5	0.20	0.18	1.36
	CaO	0.10	0.13	0.90
	S	N.D	N.D	N.D
	V_2O_5	N.D	0.65	0.13
	K_2O	0.03	0.43	3.05
	Total	100	99.93	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 69

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/7/90	FeO	68.04	47.39	22.86
	Al_2O_3	0.06	48.93	18.57
	MgO	0.21	N.D	0.37
	SiO_2	31.21	0.61	46.25
	TiO_2	N.D	2.19	2.06
	MnO	0.41	0.25	0.07
	P_2O_5	N.D	0.02	4.04
	CaO	0.06	0.08	2.33
	S	N.D	0.07	0.18
	V_2O_5	N.D	0.46	N.D
	K_2O	0.02	N.D	3.27
	Total	100	100	100

TABLE 70

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/8/90	FeO	70.28	51.25	29.24
	Al_2O_3	0.37	43.51	18.39
	MgO	0.40	N.D	N.D
	SiO_2	27.04	1.07	41.74
	TiO_2	0.37	2.22	1.88
	MnO	0.97	0.45	N.D
	P_2O_5	0.48	0.06	2.46
	CaO	0.02	0.19	1.64
	S	N.D	0.03	0.53
	V_2O_5	N.D	1.16	0.15
	K_2O	0.06	0.07	3.97
	Total	99.99	100.01	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 71

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/9/90	FeO	69.98	52.57	24.21
	Al_2O_3	0.86	41.95	12.91
	MgO	0.13	N.D	N.D
	SiO_2	27.33	0.45	41.60
	TiO_2	0.30	2.83	2.57
	MnO	0.58	0.46	N.D
	P_2O_5	0.43	0.01	6.11
	CaO	0.12	0.01	6.97
	S	N.D	0.05	1.15
	V_2O_5	0.17	1.67	N.D
	K_2O	0.10	N.D	4.49
	Total	100	100	100.01

TABLE 72

Sample Number	Wt. % of Oxides	Fayalite	Wustite	Hercynite	Glass
OW/10/90	FeO	70.53	97.46	57.44	31.69
	Al_2O_3	1.43	0.37	36.73	8.67
	MgO	0.21	0.30	0.14	N.D
	SiO_2	26.35	0.18	0.85	18.78
	TiO_2	0.45	1.33	4.17	N.D
	MnO	0.52	0.22	0.24	N.D
	P_2O_5	0.19	0.02	N.D	17.11
	CaO	0.11	0.07	N.D	20.87
	S	N.D	N.D	N.D	1.14
	V_2O_5	0.13	0.05	0.35	0.10
	K_2O	0.07	N.D	0.09	1.62
	Total	99.99	100	100.01	99.98

N.D. = Not Detected
N.A. = Not Analysed

TABLE 73

Sample Number	Wt. % of Oxides	Fayalite	Wustite	Hercynite	Glass
OW/11/90	FeO	69.05	96.82	50.97	34.87
	Al_2O_3	0.56	0.90	45.50	7.69
	MgO	0.88	N.D	0.98	0.35
	SiO_2	26.33	0-.53	0.13	27.44
	TiO_2	0.39	1.30	0.98	0.63
	MnO	1.90	0.13	0.41	0.68
	P_2O_5	0.39	0.03	N.D	7.79
	CaO	0.26	0.28	0.05	11.69
	S	0.18	0.01	0.06	1.07
	V_2O_5	N.D	N.D	0.93	N.D
	K_2O	0.07	0.02	N.D	7.82
	Total	100.01	100.02	100.01	100.03

TABLE 74

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OW/12/90	FeO	70.01	54.35	73.06
	Al_2O_3	0.60	41.02	15.44
	MgO	0.03	0.37	0.42
	SiO_2	28.24	0.57	7.87
	TiO_2	0.12	2.30	2.74
	MnO	N.D	0.21	N.D
	P_2O_5	0.56	0.03	0.13
	CaO	N.D	0.02	0.09
	S	0.16	0.10	N.D
	V_2O_5	0.17	1.05	0.25
	K_2O	0.11	N.D	N.D
	Total	100	100.02	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 75

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/7/90	FeO	67.45	45.78	34.23
	Al_2O_3	0.46	50.92	14.63
	MgO	N.D	0.49	N.D
	SiO_2	31.55	0.07	43.18
	TiO_2	N.D	1.84	1.73
	MnO	N.D	0.01	0.11
	P_2O_5	0.39	N.D	4.45
	CaO	0.02	0.05	1.01
	S	0.11	0.01	0.66
	V_2O_5	0.02	0.83	N.D
	K_2O	N.A	N.A	N.A
	Total	100	100	100

TABLE 76

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/8/90	FeO	66.52	43.66	37.64
	Al_2O_3	4.12	52.96	15.65
	MgO	N.D	0.20	N.D
	SiO_2	27.94	0.51	39.94
	TiO_2	0.97	1.62	2.21
	MnO	0.04	N.D	0.02
	P_2O_5	0.41	0.09	2.84
	CaO	N.D	N.D	0.91
	S	N.D	0.16	0.65
	V_2O_5	N.D	0.74	N.D
	K_2O	N.D	0.06	0.14
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 77

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/9/90	FeO	66.49	46.12	37.91
	Al_2O_3	0.70	52.02	31.96
	MgO	0.56	N.D	N.D
	SiO_2	31.60	1.10	24.20
	TiO_2	0.32	0.65	5.08
	MnO	N.D	N.D	0.12
	P_2O_5	0.15	0.02	0.03
	CaO	0.18	N.D	0.10
	S	N.D	0.01	0.01
	V_2O_5	N.D	0.08	0.31
	K_2O	N.D	N.D	0.28
	Total	100	100	100

TABLE 78

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/10/90	FeO	67.20	49.10	30.05
	Al_2O_3	N.D	45.91	17.26
	MgO	0.29	N.D	0.09
	SiO_2	31.55	0.55	47.55
	TiO_2	0.29	3.52	1.82
	MnO	N.D	N.D	0.12
	P_2O_5	0.42	N.D	1.39
	CaO	N.D	0.14	1.03
	S	0.12	0.01	0.31
	V_2O_5	0.05	0.77	0.03
	K_2O	0.08	N.D	0.36
	Total	100	100	99.98

N.D. = Not Detected
N.A. = Not Analysed

TABLE 79

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/11/90	FeO	66.37	47.50	38.72
	Al_2O_3	0.05	45.30	15.11
	MgO	0.16	0.60	0.52
	SiO_2	32.71	1.41	39.18
	TiO_2	0.16	4.62	3.67
	MnO	N.D	N.D	N.D
	P_2O_5	0.35	N.D	1.64
	CaO	0.02	0.21	0.99
	S	N.D	0.02	0.05
	V_2O_5	0.18	0.28	N.D
	K_2O	N.D	0.06	0.12
	Total	100	100	100

TABLE 80

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/12/90	FeO	67.07	44.07	33.08
	Al_2O_3	0.67	53.30	16.33
	MgO	N.D	N.D	N.D
	SiO_2	31.43	0.06	42.23
	TiO_2	0.26	1.97	2.35
	MnO	N.D	0.02	N.D
	P_2O_5	0.41	N.D	3.10
	CaO	0.06	N.D	2.13
	S	0.02	N.D	0.25
	V_2O_5	0.06	0.56	0.14
	K_2O	0.02	0.02	0.39
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 81

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/13/90	FeO	66.86	44.28	35.64
	Al_2O_3	0.15	51.62	13.52
	MgO	0.92	0.81	N.D
	SiO_2	31.10	0.57	41.64
	TiO_2	0.27	1.68	1.40
	MnO	0.12	0.02	0.05
	P_2O_5	0.45	0.11	4.68
	CaO	0.13	0.18	1.74
	S	N.D	0.03	0.41
	V_2O_5	N.D	0.70	0.07
	K_2O	N.D	N.D	0.85
	Total	100	100	100

TABLE 82

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/14/90	FeO	66.75	43.66	27.76
	Al_2O_3	0.33	53.25	20.44
	MgO	0.38	N.D	N.D
	SiO_2	31.33	0.37	43.67
	TiO_2	0.31	1.63	4.12
	MnO	N.D	0.07	0.13
	P_2O_5	0.67	0.11	1.62
	CaO	0.08	0.07	0.83
	S	0.04	N.D	0.03
	V_2O_5	0.08	0.84	N.D
	K_2O	0.03	N.D	1.40
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 83

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/15/90	FeO	72.18	51.58	24.44
	Al_2O_3	0.48	44.40	19.60
	MgO	N.D	N.D	N.D
	SiO_2	26.47	N.D	49.49
	TiO_2	0.27	2.60	3.26
	MnO	0.03	0.20	N.D
	P_2O_5	0.54	N.D	0.56
	CaO	N.D	N.D	1.06
	S	0.03	0.10	N.D
	V_2O_5	N.D	1.11	N.D
	K_2O	N.D	0.01	1.59
	Total	100	100	100

TABLE 84

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/16/90	FeO	70.02	49.38	37.92
	Al_2O_3	0.04	47.20	13.19
	MgO	0.49	N.D	0.74
	SiO_2	27.76	0.76	40.53
	TiO_2	0.30	1.71	0.84
	MnO	0.03	0.02	0.26
	P_2O_5	0.96	0.28	3.28
	CaO	0.20	0.04	2.57
	S	0.01	N.D	0.37
	V_2O_5	0.15	0.59	0.04
	K_2O	0.04	N.D	0.26
	Total	100	99.98	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 85

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/17/90	FeO	69.76	49.23	32.98
	Al_2O_3	0.90	46.50	16.64
	MgO	0.39	0.06	N.D
	SiO_2	27.84	0.19	41.53
	TiO_2	0.21	2.07	2.41
	MnO	0.12	0.33	N.D
	P_2O_5	0.58	0.22	2.56
	CaO	0.08	0.05	2.72
	S	0.10	N.D	0.26
	V_2O_5	N.D	1.34	0.11
	K_2O	0.02	N.D	0.78
	Total	100	99.99	99.99

TABLE 86

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OR/18/90	FeO	70.57	49.94	29.15
	Al_2O_3	N.D	45.47	18.79
	MgO	N.D	0.64	N.D
	SiO_2	28.28	0.45	43.83
	TiO_2	0.13	2.50	4.65
	MnO	0.40	N.D	N.D
	P_2O_5	0.36	0.10	0.92
	CaO	0.26	0.01	0.90
	S	N.D	0.01	0.09
	V_2O_5	N.D	0.88	N.D
	K_2O	N.D	N.D	1.67
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 87

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/6/90	FeO	67.86	45.06	32.81
	Al_2O_3	0.48	51.76	14.20
	MgO	N.D	0.15	0.03
	SiO_2	30.50	0.32	44.06
	TiO_2	0.29	1.89	1.45
	MnO	0.36	N.D	0.42
	P_2O_5	0.27	0.13	1.62
	CaO	0.11	N.D	2.84
	S	0.13	0.11	0.21
	V_2O_5	N.D	0.58	0.30
	K_2O	N.D	N.D	2.06
	Total	100	100	100

TABLE 88

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/7/90	FeO	68.08	48.45	30.56
	Al_2O_3	0.14	46.65	17.83
	MgO	N.D	N.D	N.D
	SiO_2	30.75	0.86	43.05
	TiO_2	0.24	3.56	3.27
	MnO	N.D	N.D	N.D
	P_2O_5	0.70	0.19	2.15
	CaO	0.08	0.15	1.61
	S	0.01	0.13	0.45
	V_2O_5	N.D	N.D	0.15
	K_2O	N.D	N.D	0.91
	Total	100	99.99	99.98

N.D. = Not Detected
N.A. = Not Analysed

TABLE 89

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/8/90	FeO	67.50	53.75	30.52
	Al_2O_3	0.60	42.25	16.50
	MgO	N.D	N.D	N.D
	SiO_2	31.52	0.14	45.39
	TiO_2	N.D	3.63	1.86
	MnO	N.D	0.01	0.35
	P_2O_5	0.38	0.04	1.70
	CaO	N.D	0.03	2.19
	S	N.D	0.07	0.39
	V_2O_5	N.D	0.06	N.D
	K_2O	N.D	0.01	1.10
	Total	100	99.99	100

TABLE 90

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/9/90	FeO	65.69	44.64	25.31
	Al_2O_3	0.79	51.92	17.70
	MgO	N.D	N.D	N.D
	SiO_2	32.65	0.60	51.55
	TiO_2	0.04	2.33	2.04
	MnO	N.D	N.D	0.06
	P_2O_5	0.59	0.14	0.96
	CaO	N.D	N.D	0.97
	S	N.D	N.D	0.15
	V_2O_5	0.24	0.31	N.D
	K_2O	N.D	0.05	1.26
	Total	100	99.99	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 91

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/10/90	FeO	67.36	46.50	28.02
	Al_2O_3	0.04	52.00	20.19
	MgO	0.36	N.D	0.03
	SiO_2	31.42	0.02	39.35
	TiO_2	0.08	0.99	4.62
	MnO	0.22	N.D	0.10
	P_2O_5	N.D	0.01	0.86
	CaO	0.29	N.D	1.45
	S	0.01	N.D	1.63
	V_2O_5	0.19	0.41	0.08
	K_2O	0.03	0.06	3.67
	Total	100	99.99	100

TABLE 92

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/11/90	FeO	62.10	43.96	27.37
	Al_2O_3	8.26	40.44	20.63
	MgO	N.D	N.D	N.D
	SiO_2	27.43	10.46	46.03
	TiO_2	1.02	4.45	3.21
	MnO	0.34	0.04	0.13
	P_2O_5	0.61	0.18	0.53
	CaO	0.01	0.20	1.03
	S	N.D	0.10	0.31
	V_2O_5	0.12	0.17	N.D
	K_2O	0.11	N.D	0.76
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 93

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/12/90	FeO	70.65	50.64	31.88
	Al_2O_3	1.79	45.97	15.43
	MgO	0.18	0.24	N.D
	SiO_2	26.67	0.48	43.12
	TiO_2	0.23	1.62	2.04
	MnO	N.D	0.03	N.D
	P_2O_5	0.22	0.16	1.54
	CaO	0.18	0.03	3.84
	S	N.D	N.D	0.10
	V_2O_5	0.05	0.83	0.17
	K_2O	0.03	N.D	1.88
	Total	100	100	100

TABLE 94

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/13/90	FeO	72.12	57.44	39.99
	Al_2O_3	0.80	30.73	12.89
	MgO	N.D	N.D	N.D
	SiO_2	26.27	5.88	41.19
	TiO_2	0.38	4.95	1.42
	MnO	N.D	0.05	0.20
	P_2O_5	0.29	0.01	1.30
	CaO	N.D	0.16	2.70
	S	N.D	0.08	0.06
	V_2O_5	N.D	0.57	N.D
	K_2O	0.14	0.13	0.25
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 95

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/14/90	FeO	73.88	52.27	41.16
	Al_2O_3	0.08	45.07	11.82
	MgO	0.06	0.04	0.09
	SiO_2	25.55	N.D	41.07
	TiO_2	0.19	1.11	2.30
	MnO	0.06	0.25	0.24
	P_2O_5	N.D	0.06	1.87
	CaO	0.05	N.D	0.88
	S	N.D	N.D	0.20
	V_2O_5	0.13	1.18	N.D
	K_2O	N.D	0.02	0.37
	Total	100	100	100

TABLE 96

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/15/90	FeO	72.27	56.46	38.47
	Al_2O_3	0.32	39.91	11.53
	MgO	N.D	N.D	N.D
	SiO_2	26.87	0.26	43.23
	TiO_2	0.22	2.04	1.57
	MnO	N.D	N.D	N.D
	P_2O_5	0.09	0.36	1.74
	CaO	0.23	N.D	2.02
	S	N.D	0.07	0.18
	V_2O_5	N.D	0.84	0.10
	K_2O	N.D	0.06	1.16
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 97

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/16/90	FeO	72.80	58.25	40.80
	Al_2O_3	0.28	36.66	11.12
	MgO	N.D	0.48	0.47
	SiO_2	26.41	0.57	41.30
	TiO_2	0.13	3.95	1.33
	MnO	0.01	N.D	0.04
	P_2O_5	0.12	0.01	1.64
	CaO	0.05	0.04	1.67
	S	0.09	N.D	0.29
	V_2O_5	0.05	N.D	0.04
	K_2O	0.06	0.04	1.30
	Total	100	100	100

TABLE 98

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
UM/17/90	FeO	72.34	55.45	32.82
	Al_2O_3	0.21	41.55	12.70
	MgO	0.29	N.D	0.55
	SiO_2	26.94	N.D	46.46
	TiO_2	N.D	1.77	1.45
	MnO	N.D	0.04	N.D
	P_2O_5	N.D	N.D	1.74
	CaO	0.17	0.12	2.74
	S	0.05	0.07	0.24
	V_2O_5	N.D	1.00	N.D
	K_2O	N.D	N.D	1.80
	Total	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 99

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Glass
OP/5/90	FeO	67.90	46.79	4.94
	Al_2O_3	0.25	50.34	24.25
	MgO	0.43	0.13	N.D
	SiO_2	30.85	0.16	69.92
	TiO_2	N.D	1.78	0.18
	MnO	0.27	N.D	N.D
	P_2O_5	0.05	N.D	N.D
	CaO	0.23	0.07	0.65
	S	N.D	0.01	0.06
	V_2O_5	0.02	0.71	N.D
	K_2O	N.A	N.A	N.A
	Total	100	99.99	100

TABLE 100

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Leucite
OP/6/90	FeO	64.53	42.80	2.40
	Al_2O_3	0.68	54.17	22.83
	MgO	0.29	1.01	0.79
	SiO_2	33.07	0.01	57.25
	TiO_2	N.D	0.72	0.21
	MnO	0.26	0.10	0.08
	P_2O_5	0.49	N.D	0.63
	CaO	0.51	0.08	N.D
	S	0.01	N.D	N.D
	V_2O_5	0.10	1.08	N.D
	K_2O	0.08	0.02	15.81
	Total	100.02	99.99	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 101

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Glass
OP/7/90	FeO	65.89	45.63	96.72	19.21
	Al_2O_3	0.60	51.88	0.33	5.39
	MgO	0.86	N.D	N.D	N.D
	SiO_2	31.31	0.19	1.16	46.39
	TiO_2	0.12	1.25	1.01	N.D
	MnO	0.27	0.31	0.12	0.08
	P_2O_5	0.50	0.07	0.27	9.42
	CaO	0.37	0.01	0.20	18.35
	S	0.02	N.D	0.08	0.23
	V_2O_5	0.06	0.53	0.10	N.D
	K_2O	N.D	0.12	N.D	0.93
	Total	100	99.99	99.99	100

TABLE 102

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Leucite
OP/8/90	FeO	64.22	44.66	13.19
	Al_2O_3	1.01	51.88	18.62
	MgO	1.15	0.80	N.D
	SiO_2	32.39	0.42	51.06
	TiO_2	0.36	0.39	0.80
	MnO	N.D	N.D	0.03
	P_2O_5	0.43	0.31	2.29
	CaO	0.24	0.13	3.85
	S	0.17	0.04	0.12
	V_2O_5	N.D	1.32	N.D
	K_2O	0.02	0.05	10.04
	Total	99.99	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 103

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/9/90	FeO	62.04	48.20	96.01	2.51
	Al_2O_3	7.98	48.08	0.29	22.34
	MgO	N.D	0.29	1.04	0.02
	SiO_2	27.34	0.63	0.65	56.44
	TiO_2	1.48	2.08	0.87	0.19
	MnO	0.10	0.07	N.D	N.D
	P_2O_5	0.56	0.10	0.35	1.21
	CaO	0.40	N.D	0.52	0.93
	S	0.10	N.D	N.D	0.07
	V_2O_5	N.D	0.40	0.02	N.D
	K_2O	N.D	0-.14	0.25	16.29
	Total	100	99.99	100	100

TABLE 104

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/10/90	FeO	67.27	45.79	96.08	1.14
	Al_2O_3	0.66	51.79	1.22	24.10
	MgO	1.16	0.02	0.35	0.02
	SiO_2	29.97	N.D	1.17	57.09
	TiO_2	o.16	1.21	0.64	N.D
	MnO	0.07	0.13	N.D	0.09
	P_2O_5	0.60	N.D	0.33	0.38
	CaO	N.D	N.D	0.08	N.D
	S	N.D	0.10	N.D	0.05
	V_2O_5	0.10	0.87	0.12	N.D
	K_2O	N.D	0.08	0.01	17.13
	Total	99.99	99.99	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 105

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/11/90	FeO	66.85	43.95	98.7	13.33
	Al_2O_3	0.42	54.01	0.20	21.39
	MgO	0.35	N.D	N.D	N.D
	SiO_2	31.34	0.10	0.42	52.82
	TiO_2	0.15	0.70	0.65	0.31
	MnO	N.D	N.D	N.D	N.D
	P_2O_5	0.57	0.40	0.07	1.77
	CaO	0.15	0.04	N.D	N.D
	S	0.13	0.09	0.05	0.32
	V_2O_5	0.04	0.66	0.24	N.D
	K_2O	0.01	0.04	N.D	10.06
	Total	100.01	99.99	100	100

TABLE 106

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/12/90	FeO	61.81	43.07	97.58	8.45
	Al_2O_3	0.61	53.28	0.58	22.60
	MgO	1.37	1.32	0.06	1.11
	SiO_2	34.67	0.06	0.25	51.89
	TiO_2	0.17	0.85	1.53	0.48
	MnO	0.50	0.14	N.D	0.32
	P_2O_5	0.21	N.D	N.D	1.24
	CaO	0.56	N.D	N.D	0.01
	S	N.D	0.06	N.D	0.42
	V_2O_5	0.05	1.20	N.D	N.D
	K_2O	0.05	0.02	N.D	13.48
	Total	100	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 107

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/13/90	FeO	62.01	52.12	80.86	5.58
	Al_2O_3	0.26	42.89	6.84	22.14
	MgO	0.85	0.19	N.D	0.48
	SiO_2	34.31	0.53	2.67	55.48
	TiO_2	0.30	3.61	9.07	0.29
	MnO	0.40	N.D	N.D	0.07
	P_2O_5	1.32	0.10	0.42	0.31
	CaO	0.27	N.D	0.04	N.D
	S	0.03	0.04	0.05	0.15
	V_2O_5	0.21	0.52	0.06	0.13
	K_2O	0.03	N.D	N.D	15.39
	Total	99.99	100	100.01	100.02

TABLE 108

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/14/90	FeO	63.13	46.74	94.28	1.60
	Al_2O_3	0.71	48.79	1.75	22.88
	MgO	1.26	1.23	N.D	0.05
	SiO_2	33.29	0.54	1.18	57.41
	TiO_2	0.30	1.82	2.23	0.23
	MnO	0.05	N.D	N.D	0.10
	P_2O_5	0.86	N.D	0.38	0.56
	CaO	0.40	0.12	0.14	N.D
	S	N.D	N.D	N.D	0.06
	V_2O_5	N.D	0.76	N.D	0.03
	K_2O	N.D	N.D	0.04	17.10
	Total	100	100	100	100.02

N.D. = Not Detected
N.A. = Not Analysed

TABLE 109

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/15/90	FeO	62.21	44.63	96.91	6.72
	Al_2O_3	N.D	51.81	1.14	23.05
	MgO	0.78	1.12	N.D	0.06
	SiO_2	35.88	0.47	0.98	51.75
	TiO_2	0.08	1.40	0.96	0.62
	MnO	0.39	0.05	N.D	0.33
	P_2O_5	0.47	N.D	N.D	0.44
	CaO	0.10	0.02	0.01	0.08
	S	0.03	0.08	N.D	0.08
	V_2O_5	0.04	0.42	N.D	N.D
	K_2O	0.02	N.D	N.D	16.94
	Total	100	100	100	100

TABLE 110

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/16/90	FeO	65.07	49.57	97.63	11.98
	Al_2O_3	1.49	47.32	0.66	20.03
	MgO	0.67	N.D	0.14	0.57
	SiO_2	31.37	0.31	0.76	50.23
	TiO_2	0.15	2.28	0.49	0.54
	MnO	0.21	0.01	N.D	0.15
	P_2O_5	0.62	N.D	0.01	2.07
	CaO	0.32	0.02	0.10	0.06
	S	0.08	N.D	0.03	0.12
	V_2O_5	N.D	0.49	0.18	N.D
	K_2O	N.D	N.D	N.D	14.25
	Total	99.98	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 111

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/17/90	FeO	62.85	42.74	95.97	8.57
	Al_2O_3	0.98	54.07	1.23	21.39
	MgO	0.94	1.22	0.01	N.D
	SiO_2	33.42	0.08	1.00	56.48
	TiO_2	0.42	0.91	1.41	0.21
	MnO	0.18	0.03	N.D	0.11
	P_2O_5	0.74	0.02	0.28	0.78
	CaO	0.33	N.D	0.08	0.07
	S	0.14	0.04	N.D	0.05
	V_2O_5	N.D	0.89	N.D	N.D
	K_2O	N.D	N.D	0.02	12.34
	Total	100	100	100	100

TABLE 112

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/18/90	FeO	66.88	53.64	97.76	4.42
	Al_2O_3	0.41	39.77	0.43	19.03
	MgO	0.77	N.D	N.D	N.D
	SiO_2	31.22	0.58	0.60	46.16
	TiO_2	0.08	5.52	0.93	0.13
	MnO	0.23	0.16	N.D	0.16
	P_2O_5	0.20	0.05	0.12	7.90
	CaO	0.11	0.02	N.D	8.57
	S	0.01	0.02	N.D	N.D
	V_2O_5	0.02	0.19	0.16	N.D
	K_2O	0.07	0.05	N.D	13.63
	Total	100	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 113

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Leucite
OP/19/90	FeO	68.80	49.27	95.29	13.77
	Al_2O_3	0.34	46.53	1.59	16.78
	MgO	N.D	1.13	N.D	0.02
	SiO_2	29.37	0.13	0.47	42.48
	TiO_2	0.39	2.13	2.50	0.30
	MnO	0.37	0.08	N.D	0.10
	P_2O_5	0.50	0.08	0.13	7.60
	CaO	0.21	N.D	N.D	8.30
	S	0.01	0.06	N.D	0.25
	V_2O_5	0.03	0.59	0.01	N.D
	K_2O	N.D	N.D	0.01	10.40
	Total	100.02	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 114

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Glass
Lej/1/90	FeO	65.42	48.20	95.40	41.10
	Al_2O_3	0.39	45.97	1.58	13.16
	MgO	0.22	0.32	0.55	N.D
	SiO_2	32.94	0.82	0.85	30.32
	TiO_2	N.D	3.62	1.60	1.42
	MnO	0.30	0.32	0.02	N.D
	P_2O_5	0.34	0.27	N.D	3.11
	CaO	0.35	0.03	N.D	5.69
	S	N.D	0.08	N.D	0.87
	V_2O_5	0.04	0.14	N.D	N.D
	K_2O	N.D	0.15	N.D	4.31
	Total	100	99.92	100	99.98

TABLE 115

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Glass
Lej/2/90	FeO	68.61	49.58	98.03	64.30
	Al_2O_3	0.60	46.82	N.D	3.48
	MgO	N.D	N.D	0.20	N.D
	SiO_2	29.99	0.57	0.66	17.79
	TiO_2	N.D	1.73	0.53	0.65
	MnO	0.40	0.30	0.12	0.35
	P_2O_5	0.25	0.05	0.46	11.86
	CaO	0.06	N.D	N.D	0.69
	S	0.9	0.05	N.D	0.82
	V_2O_5	N.D	0.90	N.D	N.D
	K_2O	N.D	N.D	N.D	0.06
	Total	100	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

TABLE 116

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Glass
Lej/3/90	FeO	66.25	48.98	97.28	27.26
	Al_2O_3	0.50	46.98	0.26	12.07
	MgO	1.07	0.60	N.D	0.20
	SiO_2	31.05	0.75	1.04	27.24
	TiO_2	0.20	1.95	1.12	0.89
	MnO	N.D	0.08	0.04	0.10
	P_2O_5	0.26	0.12	0.02	11.86
	CaO	0.49	0.10	0.12	12.01
	S	0.04	N.D	N.D	0.94
	V_2O_5	0.12	0.44	N.D	0.24
	K_2O	0.02	N.D	0.11	7.19
	Total	100	100	99.99	100

TABLE 117

Sample Number	Wt. % of Oxides	Fayalite	Hercynite	Wustite	Glass
Lej/5/90	FeO	62.76	48.13	77.62	29.28
	Al_2O_3	0.89	48.02	15.23	16.56
	MgO	N.D	N.D	N.D	0.36
	SiO_2	34.66	0.61	3.41	48.10
	TiO_2	0.24	2.83	3.60	0.23
	MnO	0.38	0.29	0.14	0.37
	P_2O_5	1.06	0.12	N.D	3.17
	CaO	N.D	N.D	N.D	1.68
	S	0.01	N.D	N.D	0.25
	V_2O_5	N.A	N.A	N.A	N.A
	K_2O	N.A	N.A	N.A	N.A
	Total	100	100	100	100

N.D. = Not Detected
N.A. = Not Analysed

Table 118 Mineral Phases in Nsukka Slags Samples

Site & Samples	Fayalite	Wustite	Hercynite	Leucite	Glass	Others
Owerre-Elu Samples						
OW/1/90	82% grey broken lath crystals	AB	15% dark grey euhedral crystals	AB	3% dark siliceous pools	
OW/2/90	62% massive grey crystals	18% white dendrites	17% dark grey euhedral crystals	AB	3% dark pools in air holes	
OW/3/90	70% massive grey crystals	11% white dendrites	16% dark grey euhedral crystals	AB	3% dark pools in air holes	
OW/4/90	86% massive grey crystals	AB	13% dark grey euhedral crystals	AB	1% dark pools in air holes	
OW/5/90	69% massive grey crystals	8% white dendrites	22% dark grey euhedral crystals	AB	1% dark pools in air holes	
OW/6/90	76% grey broken lath	AB	18% dark grey euhedral crystals	AB	6% dark matrix	
OW/7/90	79% grey broken lath	AB	15% dark grey euhedral crystals	AB	6% dark siliceous matrix	
OW/8/90	80% fine grey lath crystals	AB	15% dark grey euhedral crystals	AB	5% dark matrix very siliceous	
OW/9/90	81% massive grey crystals	AB	15% dark grey euhedral crystals	AB	4% dark pools in air holes	thin bands of magnetite
OW/10/90	71% massive grey crystals	4% white dendrites	21% dark grey euhedral crystals	AB	4% dark siliceous pools	
OW/11/90	76% massive grey crystals	6% fine white dendrites	13% dark grey euhedral crystals	AB	4% dark pools in air holes	thin bands of magnetite
OW/12/90	85% grey broken laths	AB	14% dark grey euhedral crystals	AB	1% dark siliceous matrix	

AB=Absent

Mineral Phases in Nsukka Slags Samples

Site & Samples	Fayalite	Wustite	Hercynite	Leucite	Glass	Others
ORBA Samples						
OR/7/90	73% grey lath crystals	AB	18% dark grey euhedral crystals	AB	9% dark siliceous matrix	
OR/8/90	72% grey broken lath crystals	AB	11% dark grey euhedral crystals	AB	17% dark matrix	
OR/9/90	54% grey lath crystals	AB	10% dark grey euhedral crystals	AB	36% dark matrix	
OR/10/90	46% grey lath crystals	AB	12% dark grey euhedral crystals	AB	42% dark matrix	
OR/11/90	48% grey broken lath crystals	AB	13% dark grey euhedral crystals	AB	39% dark matrix	
OR/12/90	60% grey lath crystals	AB	14% dark grey euhedral crystals	AB	26% dark siliceous matrix	
OR/13/90	53% grey lath crystals	AB	11% dark grey euhedral crystals	AB	33% dark siliceous matrix	
OR/14/90	63% grey lath crystals	AB	8% dark grey euhedral crystals	AB	29% dark siliceous matrix	
OR/15/90	70% grey lath crystals	AB	10% dark grey euhedral crystals	AB	20% dark siliceous matrix	
OR/16/90	63% grey lath crystals	AB	11% dark grey euhedral crystals	AB	26% dark matrix	
OR/17/90	64% grey lath crystals	AB	10% dark grey euhedral crystals	AB	26% dark siliceous matrix	
OR/18/90	66% grey lath crystals	AB	10% dark grey euhedral crystals	AB	24% dark siliceous matrix	

AB=Absent

Mineral Phases in Nsukka Slags Samples

Site & Samples	Fayalite	Wustite	Hercynite	Leucite	Glass	Others
UMUNDU Samples						
UM/6/90	80% massive grey crystals	AB	15% dark grey euhedral crystals	AB	5% dark pools in air holes	
UM/7/90	62% massive grey crystals	AB	12% dark grey euhedral crystals	AB	26% dark siliceous pools	
UM/8/90	57% grey lath crystals	AB	17% dark grey euhedral crystals	AB	26% siliceous matrix	
UM/9/90	51% fine grey lath crystals	AB	10% dark grey euhedral crystals	AB	39% dark siliceous matrix	
UM/10/90	64% grey lath crystals	AB	13% dark grey euhedral crystals	AB	23% dark matrix	
UM/11/90	46% fine grey lath crystals	AB	10% dark grey euhedral crystals	AB	44% dark siliceous matrix	
UM/12/90	69% light grey lath crystals	AB	10% dark grey euhedral crystals	AB	21% dark siliceous matrix	
UM/13/90	51% light grey lath crystals	AB	10% dark grey euhedral crystals	AB	39% dark matrix	
UM/14/90	63% massive light grey crystals	AB	12% dark grey euhedral crystals	AB	25% dark siliceous matrix	
UM/15/90	58% light grey lath crystals	AB	13% dark grey euhedral crystals	AB	29% dark siliceous matrix	
UM/16/90	59% light grey lath crystals	AB	11% dark grey euhedral crystals	AB	30% dark siliceous matrix	
UM/17/90	63% light grey lath crystals	AB	16% dark grey euhedral crystals	AB	21% dark siliceous matrix	

AB=Absent

Mineral Phases in Nsukka Slags Samples

Site & Samples	Fayalite	Wustite	Hercynite	Leucite	Glass	Others
OPI Samples						
OP/5/90	75% massive grey crystals	AB	25% dark grey euhedral crystals	AB	1% dark pools in air holes	
OP/6/90	77% massive grey crystals	AB	18% dark grey euhedral crystals	5% dark pools	AB	
OP/7/90	65% massive grey crystals	5% white dendrites	27% dark grey euhedral crystals	AB	3% dark pools in air holes	
OP/8/90	68% grey lath crystals	AB	24% dark grey euhedral crystals	8% dark matrix	AB	
OP/9/90	69% massive grey crystals	7% white dendrites	13% dark grey euhedral crystals	11% dark pools in air holes	AB	
OP/10/90	74% massive grey crystals	3% white dendrites	22% dark grey euhedral crystals	2% dark pools in air holes	AB	
OP/11/90	65%massive grey crystals	5% white dendrites	29% dark grey euhedral crystals	1% dark pools in air holes	AB	
OP/12/90	76% massive grey crystals	5% white dendrites	13% dark grey euhedral crystals	6% dark pools in air holes	AB	
OP/13/90	64% massive grey crystals	10% white dendrites	15% dark grey euhedral crystals	11% dark pools in air holes	AB	
OP/14/90	65% massive grey crystals	10% white dendrites	14% dark grey euhedral crystals	11% dark pools	AB	

AB=Absent

Mineral Phases in Nsukka Slags Samples

Site & Samples	Fayalite	Wustite	Hercynite	Leucite	Glass	Others
OP/15/90	53% massive grey crystals	19% white dendrites	24% dark grey euhedral crystals	4% dark pools in air holes	AB	
OP/16/90	59% massive grey crystals	5% white dendrites	33% dark grey euhedral crystals	3% dark pools in air holes	AB	
OP/17/90	66% massive grey crystals	7% white dendrites	16% dark grey euhedral crystals	11% dark pools	AB	
OP/18/90	55% massive grey crystals	8% white dendrites	34% dark grey euhedral crystals	3% dark matrix	AB	
OP/19/90	53% massive grey crystals	9% white dendrites	30% dark grey euhedral crystals	8% siliceous dark pools	AB	
Leja Samples						
Lej/1/90	68% massive grey crystals	5% white dendrites	16% dark grey euhedral crystals	AB	11% dark siliceous pools	
Lej/2/90	85% massive grey crystals	AB	13% dark grey euhedral crystals	AB	2% dark pools in air holes	Thin bands of magnetite
Lej/3/90	66%massive grey crystals	6% white dendrites	18% dark grey euhedral crystals	AB	18% dark pools in air holes	
Lej/5/90	86% massive grey crystals	3% white dendrites	10% dark grey euhedral crystals	AB	1% dark pools in air holes	Thin bands of magnetite

AB=Absent

TABLE 119 BLOOMERY IRON SMELTING SITES IN NSUKKA

	Processes	OPI	Owerre-Elu	Orba	Umundu
1	Age of the site	C.765BC-75AD	C.810-1435AD	C.1430-1950AD	C.1490-1950AD
2	Area covered by the site	More than 29506.4 sq. metres	54937.6 sq. metres	4431 sq. metres	8346.0 sq. metres
3	Types of iron ore at these sites	Haematite	Haematite	Goethite	Goethite and Hamatite
4	Methods of ore collection	from ground surface and open cast mining	from hill surface, slopes and open cast mining	from dried stream valley, and open cast mining	from valley slopes, gullies and open cast mining
5	Ore preparation before smelting	broken into small bits of about 1.5 cm thick	broken into small bits of about 1.5 cm thick	broken into small bits of about 1.5 cm thick	broken into small bits of about 1.5 cm thick
6	Fuel types used in the smelting	Charcoal prepared from hard woods	Charcoal prepared from hard woods	Hard woods were used direct for the smelting	Charcoal prepared from hard woods
7	Location of the smelting sites	Away from home near the ore and fuel source	Away from home near the ore and fuel source	Away from home near the ore and fuel source	Away from home near the ore and fuel source

Processes	OPI	Owerre-Elu	Orba	Umundu
8 Furnace type	forced draught shaft furnace	forced draught shaft furnace	Natural draught shaft furnace with internal wooden frame	Natural draught shaft furnace
9 Methods of loading the furnace	Dry leaves and grasses were laid at the furnace base; On this alternate layers of ore and charcoal were laid up to $^2/_3$ capacity of the furnace	The furnace base was lined with very combustible materials. On this were laid alternate layers of ore and charcoal up to $^2/_3$ capacity of the furnace	The base of the furnace was lined with dry materials on which were laid alternate layers of wood and ore. The furnace was loaded to $^3/_4$ of its capacity	The base of the furnace was lined with very dry materials on which alternate layers of charcoal and ore were laid up to $^2/_3$ of the furnace capacity
10 Length of firing and smelting	between 18 and 20 hrs.	about 18 hrs.	about 36 hrs.	between 24 and 36 hrs.
11 Methods of off-loading the furnace	The bloom was removed with a wooden clamp through one of the openings at the base of the furnace	The bloom was extracted with wooden clamp through the openings at the furnace base	At the end of the smelt the contents of the furnace, bloom, slag and charcoal were raked out with wooden hook	The contents of the furnace were raked out at the end of the smelt with wooden hook
12 Bloom refining after the smelting	The bloom was sold to blacksmiths who refined and forged it into tools	The bloom was sold to blacksmiths who refined and forged it into tools	The bloom were separated from the slags and was sold to the blacksmiths	The bloom was sold to the blacksmith who refined and forged it into tools

Processes	OPI	Owerre-Elu	Orba	Umundu
13 Types of slags at these sites	Cylindrical slag blocks tapped into slag pits	flattish tap slags with smooth ropy surface	raked bits of aggregates of slag	raked furnace slag in aggregate form
14 Others	Palm oil was poured on the load in the furnace before smelting began	At times palm oil was poured on the load in the furnace before smelting began	The smelters fed sand into the charge in the furnace during the smelting	

Table 120 Spot Analysis of Tuyere fragment from OPI

Sample Number	Wt. % of Oxides	Quartz	Fayalite	Glass
OP/2/90	FeO	0.72	50.44	32.48
	Al_2O_3	0.62	8.89	16.58
	MgO	N.D.	0.09	0.17
	SiO_2	98.18	37.87	48.41
	TiO_2	0.04	1.09	1.34
	MnO	0.01	0.34	0.17
	P_2O_5	0.32	0.64	0.36
	CaO	N.D.	0.29	0.08
	S	0.03	N.D.	N.D.
	V_2O_5	0.08	N.D.	0.29
	K_2O	N.D.	0.35	0.12
	Total	**100**	**100**	**100**

Table 121 Spot Analysis of Tuyere fragment from ORBA

Sample Number	Wt. % of Oxides	Quartz	Slag
OR/1/90	FeO	0.46	45.51
	Al_2O_3	N.D.	12.37
	MgO	N.D.	N.D.
	SiO_2	98.82	39.88
	TiO_2	N.D.	1.90
	MnO	N.D.	0.17
	P_2O_5	0.29	N.D.
	CaO	0.29	0.19
	S	0.02	0.04
	V_2O_5	0.12	0.04
	K_2O	N.D.	N.D.
	Total	**100**	**100**

N.D. = NOT DETECTED

Table 122 Spot Analysis of Tuyere fragment from Owerre-Elu

Sample Number	Wt. % of Oxides	Quartz	Fayalite	Hercynite
OW/16/90	FeO	21.92	52.34	48.71
	Al_2O_3	9.33	8.07	48.78
	MgO	0.58	N.D.	N.D.
	SiO_2	65.76	35.99	0.03
	TiO_2	1.95	1.46	1.77
	MnO	0.04	0.30	N.D.
	P_2O_5	N.D.	0.86	N.D.
	CaO	0.21	0.58	0.07
	S	0.02	0.09	N.D.
	V_2O_5	0.02	0.07	0.64
	K_2O	0.17	0.24	N.D.
	Total	100	100	100

Table 123 Analysis of Slag Inclusions in Bloomery Iron from Umundu

Sample Number	Oxides analysed	Bulk analysis of the inclusions	Wustite	Fayalite
UM/18/90	FeO	52.34	95.79	62.83
	Al_2O_3	8.07	0.92	0.71
	MgO	N.D.	N.D.	1.01
	SiO_2	35.99	0.29	19.82
	TiO_2	1.46	0.51	0.28
	MnO	0.31	1.67	6.94
	P_2O_5	0.86	0.11	6.31
	CaO	0.58	0.07	1.65
	S	0.08	N.D.	0.10
	V_2O_5	0.07	0.56	0.10
	K_2O	0.24	0.08	0.25
	Total	100	100	100

N.D. = NOT DETECTED

Table 124 Analysis of Slag Inclusions in Bloomery Iron from Umundu

Sample Number	Oxides analysed	Bulk analysis of the inclusions	Wustite	Fayalite
UM/19/90	FeO	72.45	97.37	68.11
	Al_2O_3	1.79	1.06	0.42
	MgO	N.D.	N.D.	0.69
	SiO_2	21.84	0.52	26.93
	TiO_2	0.21	0.34	0.14
	MnO	1.42	0.49	2.3
	P_2O_5	0.55	0.06	0.36
	CaO	0.76	0.10	0.64
	S	0.18	0.06	0.09
	V_2O_5	N.D.	N.D.	N.D.
	K_2O	0.80	N.D.	0.32
	Total	**100**	**100**	**100**

Table 125 Analysis of Slag Inclusions in Bloomery Iron from Umundu

Sample Number	Oxides analysed	Bulk analysis of the inclusions	Wustite	Glass	High TiO_2 Fayalite
UM/20/90	FeO	66.29	97.76	49.53	61.90
	Al_2O_3	8.30	0.51	9.16	11.82
	MgO	0.02	0.42	1.30	0.16
	SiO_2	22.80	0.56	33.27	5.88
	TiO_2	1.65	0.31	1.29	19.29
	MnO	0.82	N.D.	0.23	N.D.
	P_2O_5	0.05	0.18	2.78	0.83
	CaO	0.03	N.D.	1.96	N.D.
	S	N.D.	N.D.	0.06	0.12
	V_2O_5	N.D.	0.17	0.01	N.D.
	K_2O	0.04	0.09	0.41	N.D.
	Total	**100**	**100**	**100**	**100**

N.D. = NOT DETECTED

Table 126 **Summary of Mineral Phase Proportions in Nsukka Slags in %**

Site	Sample Number	OW/1	OW/2	OW/3	OW/4	OW/5	OW/6	OW/7	OW/8	OW/9	OW/10	OW/11	OW/12	Mean	S.D.
Owerre-Elu	Phases														
	Fayalite	82	62	70	86	69	76	79	80	81	71	76	85	76.42	6.9
	Wustite	AB	18	11	AB	8	AB	AB	AB	AB	4	6	AB	3.92	5.6
	Hercynite	15	17	16	13	22	18	15	15	15	21	13	14	16.17	2.76
	Glass	3	3	3	1	1	6	6	5	4	4	4	1	3.42	1.71
ORBA	Sample Number	OR/7	OR/8	OR/9	OR/10	OR/11	OR/12	OR/13	OR/14	OR/15	OR/16	OR/17	OR/18	Mean	S.D.
	Fayalite	73	72	54	46	48	60	53	63	70	63	64	66	61.	8.64
	Wustite	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB		
	Hercynite	18	11	10	12	13	14	11	8	10	11	10	10	11.5	2.47
	Glass	9	17	36	42	39	26	33	29	20	26	26	24	27.25	9.01
Umundu	Sample Number	UM/6	UM/7	UM/8	UM/9	UM/10	UM/11	UM/12	UM/13	UM/14	UM/15	UM/16	UM/17	Mean	S.D.
	Fayalite	80	62	57	51	64	46	69	51	63	58	59	63	60.28	8.61
	Wustite	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB		
	Hercynite	15	12	17	10	13	10	10	10	12	13	11	16	12.42	2.36
	Glass	5	26	26	39	23	44	21	39	25	29	30	21	27.33	9.86

AB = ABSENT

Table 126 (cont'd) **Summary of Mineral Phase Proportions in Nsukka Slags in %**

Opi	Sample Number	OP/5	OP/6	OP/7	OP/8	OP/9	OP/10	OP/11	OP/12	OP/13	OP/14	OP/15	OP/16	OP/17	OP/18	OP/19	Mean	S.D.
	Fayalite	75	77	65	68	69	74	65	76	64	65	53	59	66	55	53	65.6	7.71
	Wustite	AB	AB	5	AB	7	3	5	5	10	10	19	5	7	8	9	6.2	4.74
	Hercynite	25	18	27	24	13	22	29	13	15	14	24	33	16	34	30	22.47	7.04
	Leucite	AB	5	AB	8	11	2	1	6	11	11	4	3	11	3	8	5.07	4.19
	Glass	1	AB	3	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	AB	0.8	2.07

AB = ABSENT

Table 126 (cont'd)

Summary of Mineral Phase Proportions in Nsukka Slags in %

Leja	**Sample Number**	**Lej/1**	**Lej/2**	**Lej/3**	**Lej/4**	**Mean**	**S.D.**
	Fayalite	68	85	66	86	76.25	9.28
	Wustite	5	AB	6	3	3.50	2.29
	Hercynite	16	13	18	10	14.25	3.03
	Glass	11	2	18	1	8	6.96

AB = ABSENT

Table 127

Analysis of Iron Ores from Nsukka
(OPI, ORBA, Owerre-Elu, Umundu)

Oxides	OP/3/90	OP/4/90	Mean	SD	OR/5/90	OR/6/90	Mean	SD	OW/13/90	OW/14/90	OW/15/90	Mean	SD	UM/4/90	UM/5/90	Mean	SD
FeO	66.13	54.47	60.3	5.83	66.25	66.65	66.45	0.2	68.73	68.	67.22	67.98	0.62	70.27	58.77	64.52	5.75
Al_2O_3	10.13	6.99	8.56	1.57	13.7	13.69	13.69	5.	12.95	11.67	11.5	12.04	0.65	12.64	16.8	14.72	2.08
MgO	0.08	0.19	0.14	0.06	N.D.	0.35	0.18	0.18	0.1	0.03	0.04	0.05	0.03	0.2	0.22	0.21	0.01
SiO_2	20.62	34.24	27.43	6.81	17.34	16.46	16.9	0.44	17.23	16.29	16.69	16.73	0.38	14.37	20.86	17.62	3.25
TiO_2	2.67	3.38	3.03	0.36	1.86	2.31	2.09	0.23	0.57	3.42	4.26	2.75	1.58	2.14	3.02	2.58	0.44
MnO	N.D.	0.12	0.06	0.06	0.02	0.04	0.03	0.01	0.01	0.01	N.D.	0.03	0.03	N.D.	N.D.	N.D.	N.D
P_2O_5	0.21	0.38	0.3	0.09	0.6	0.21	0.41	0.2	0.12	0.37	0.06	0.18	0.13	0.25	0.06	0.16	0.1
CaO	N.D.	0.02	0.01	0.01	0.1	0.02	0.06	0.04	0.1	0.01	0.08	0.06	0.04	0.05	0.14	0.1	0.05
S	0.03	0.04	0.35	0.01	0.05	0.06	0.06	5.	0.07	0.09	0.03	0.06	0.02	0.02	0.02	0.02	0.0
V_2O_5	0.11	0.12	0.12	0.01	0.06	0.21	0.14	0.08	0.1	0.08	0.07	0.08	0.01	0.04	0.05	0.04	0.01
K_2O	0.02	0.05	0.04	0.02	0.02	N.D.	0.01	0.01	0.02	0.02	0.04	0.03	0.03	0.02	0.06	0.04	0.02
Total	100.	100.	100.34		100.	100.	100.02		100.	99.99	99.99	99.96		100.	100.	100.01	
Total Fe	51.4	42.33			51.49	51.81			53.42	52.86	52.25			54.62	45.69		

N.D. = NOT DETECTED

Table 128 Carbon-14 Dates from Nsukka Iron Smelting Sites

Lab Number	Sample Number	14 C Date BP	S.D.	Calibrated Dates *	
				One Sigma	Two Sigma
OXA - 3201 Opi	OP/3/91	2305	90	520-200 cal BC	765-120 cal BC
OXA - 2691 Opi	OP/1/90	2170	80	380 - 110 cal BC	400 - 20 cal BC
OXA - 3200 Opi	OP/2/91	2080	90	345 cal BC - cal AD 15	370 cal BC - cal AD 75
OXA - 2738 Owerre-Elu	OW/17/90	1060	60	895 - 1020 cal AD	810 - 1155 cal AD
OXA - 2739 Owerre-Elu	OW/18/90	570	60	1300 - 1420 cal AD	1285 - 1435 cal AD
OXA - 2688 Umundu	UM/1/90	200	80	1640 - 1950 cal AD	1490 - 1950 cal AD
OXA - 2689 Umundu	UM/2/90	205	80	1640 - 1950 cal AD	1490 - 1950 cal AD
OXA - 2690 Umundu	UM/3/90	130	80	1660 - 1950 cal AD	1640 - 1950 cal AD
OXA - 2693 Amaoba	OR/2/90	300	90	1460 - 1950 cal AD	1430 - 1950 cal AD
OXA - 2694 Amaoba	OR/3/90	215	100	1520 - 1950 cal AD	1450 - 1950 cal AD
OXA - 2695 Amaoba	OR/4/90	295	85	1460 - 1950 cal AD	1430 - 1950 cal AD

* The dates were calibrated using the calibration curve of Stuiver and Pearson (1986) and the calibration program of Van der Plicht and Mook (1989).

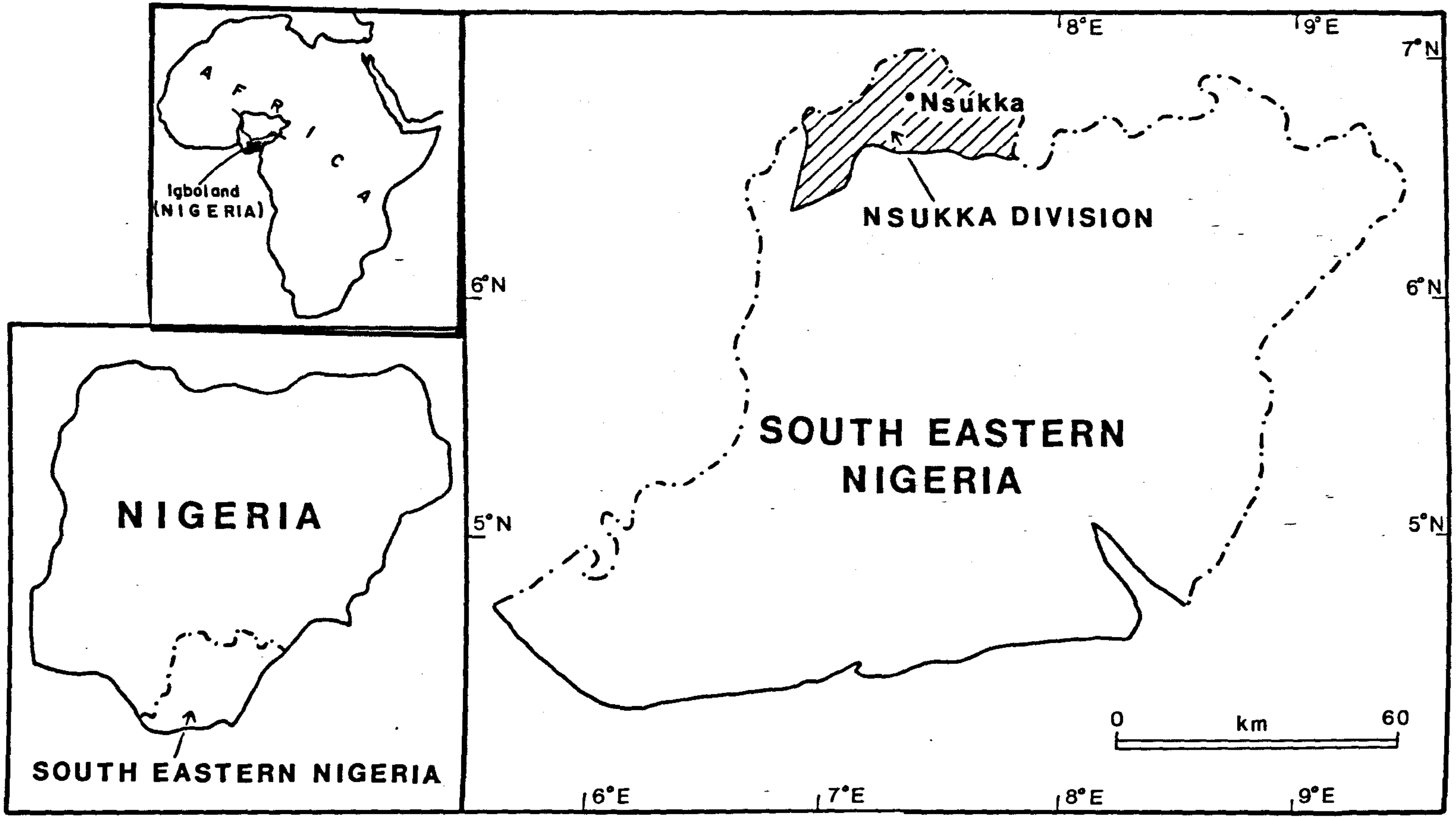


Fig. 1:1 Location of Nsukka.

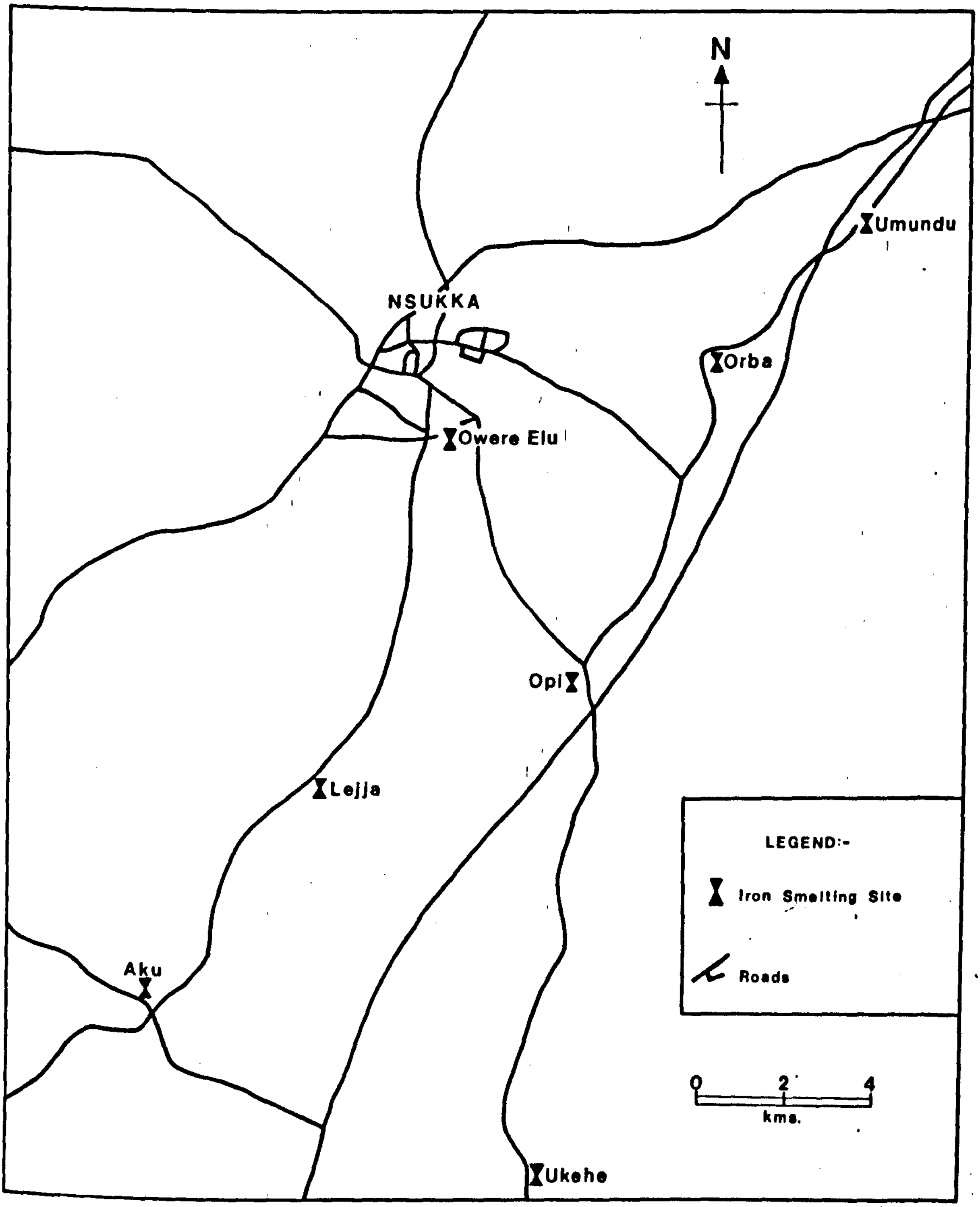


Fig. 1:2 Nsukka iron smelting sites: (Adapted from Nigeria 1:100,000 Nsukka and Igbo-Etiti local government areas: first edition).

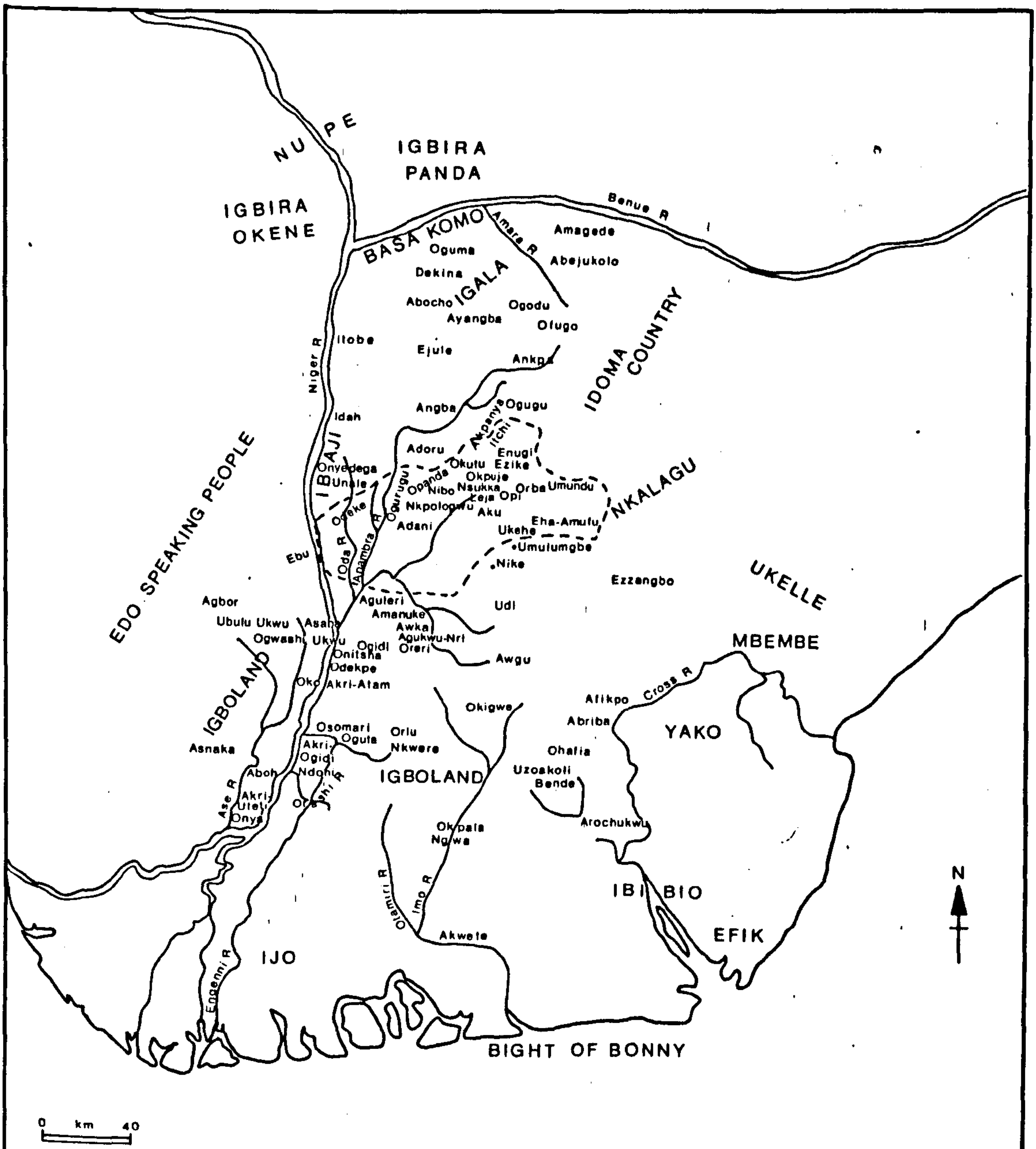


Fig. 2:1 Nsukka and her neighbours: (Adapted from road map of Nigeria, Federal Surveys, Lagos).

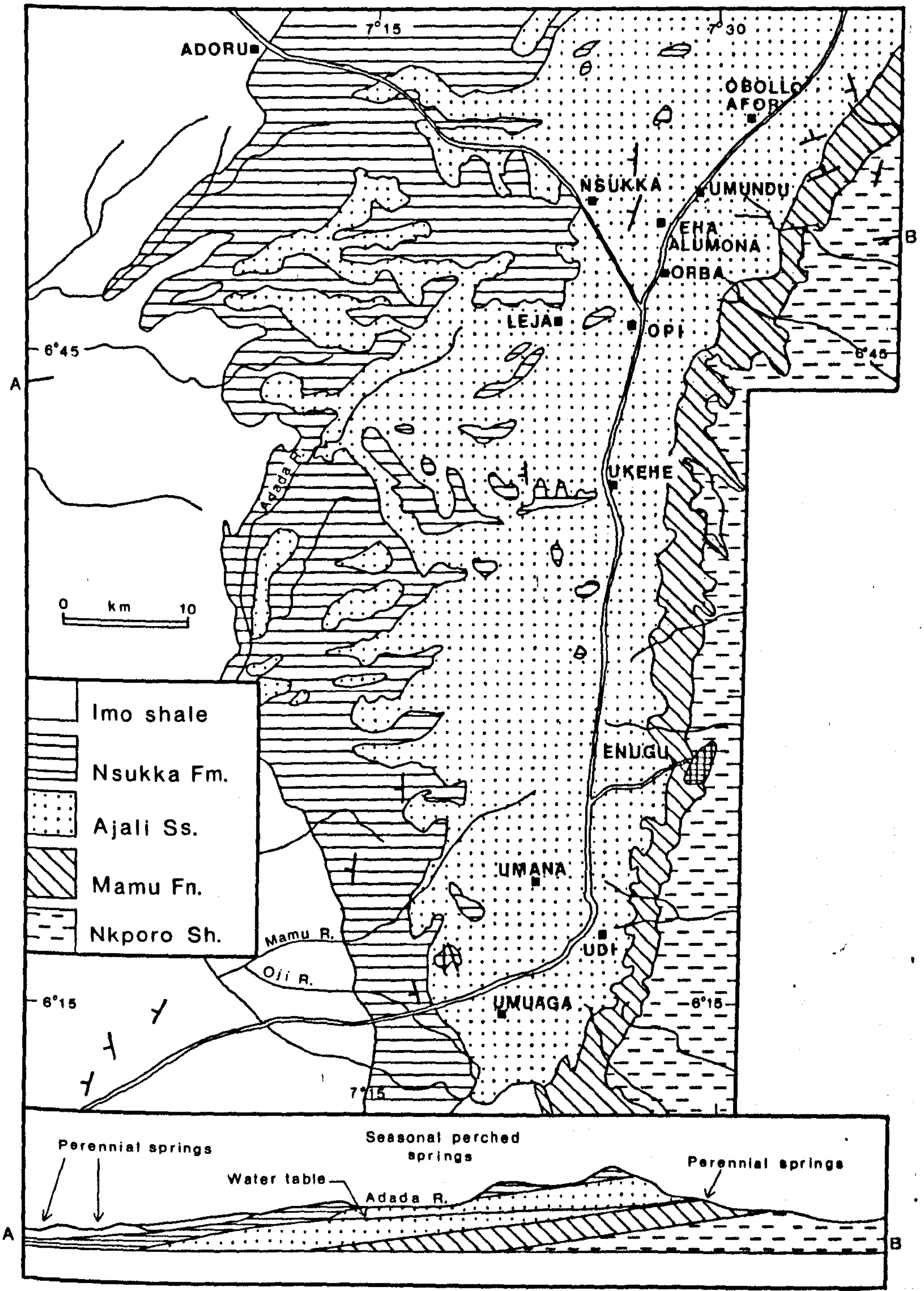


Fig. 2:2 Geological map of Nsukka Division: Fm-formation, Ss-sand stone, Fn-formation, Sh-shale (adapted from Umeji 1980:112).

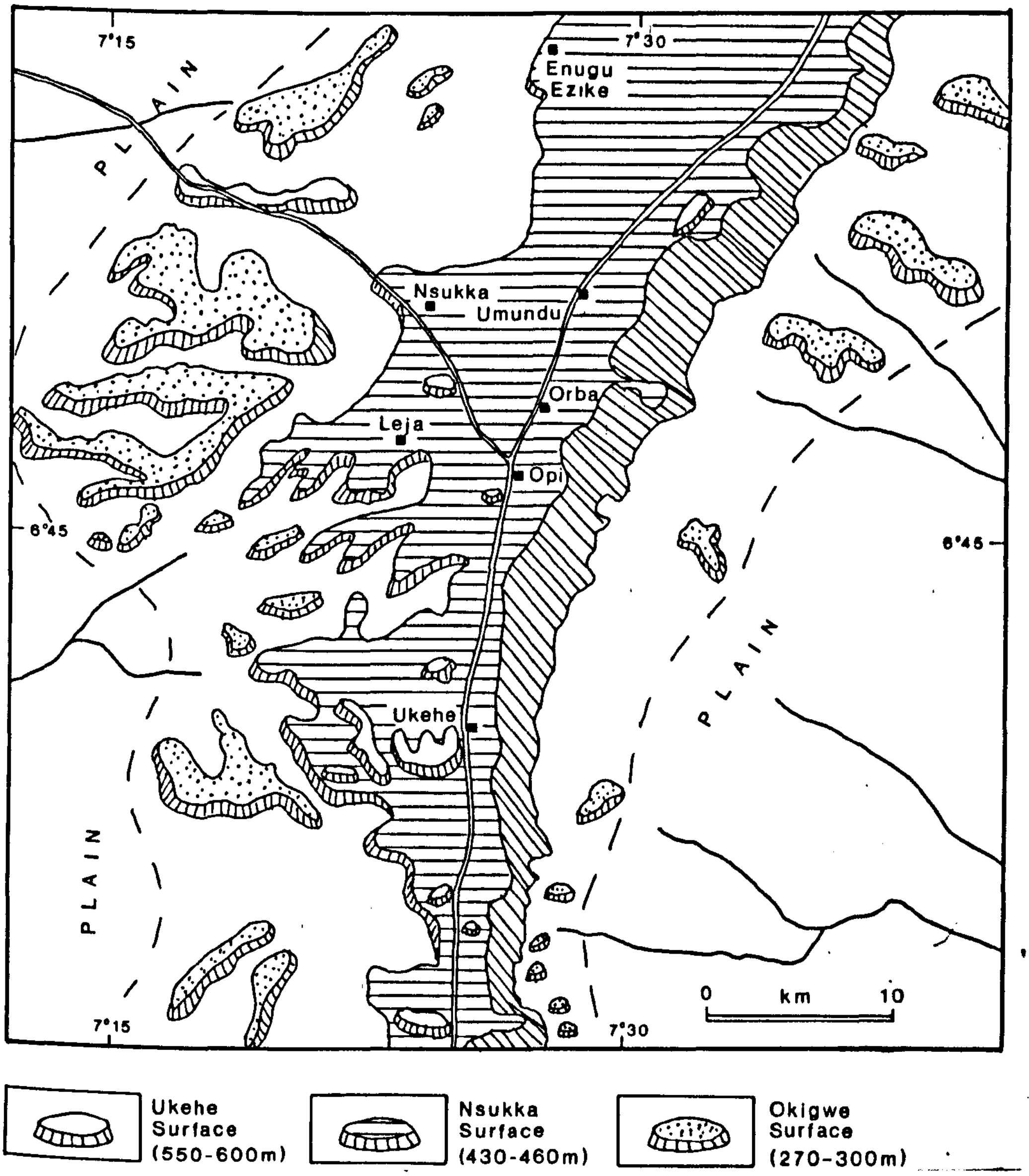


Fig. 2:3 Planation surfaces in Nsukka Division: (adapted from Umeji 1980:112).

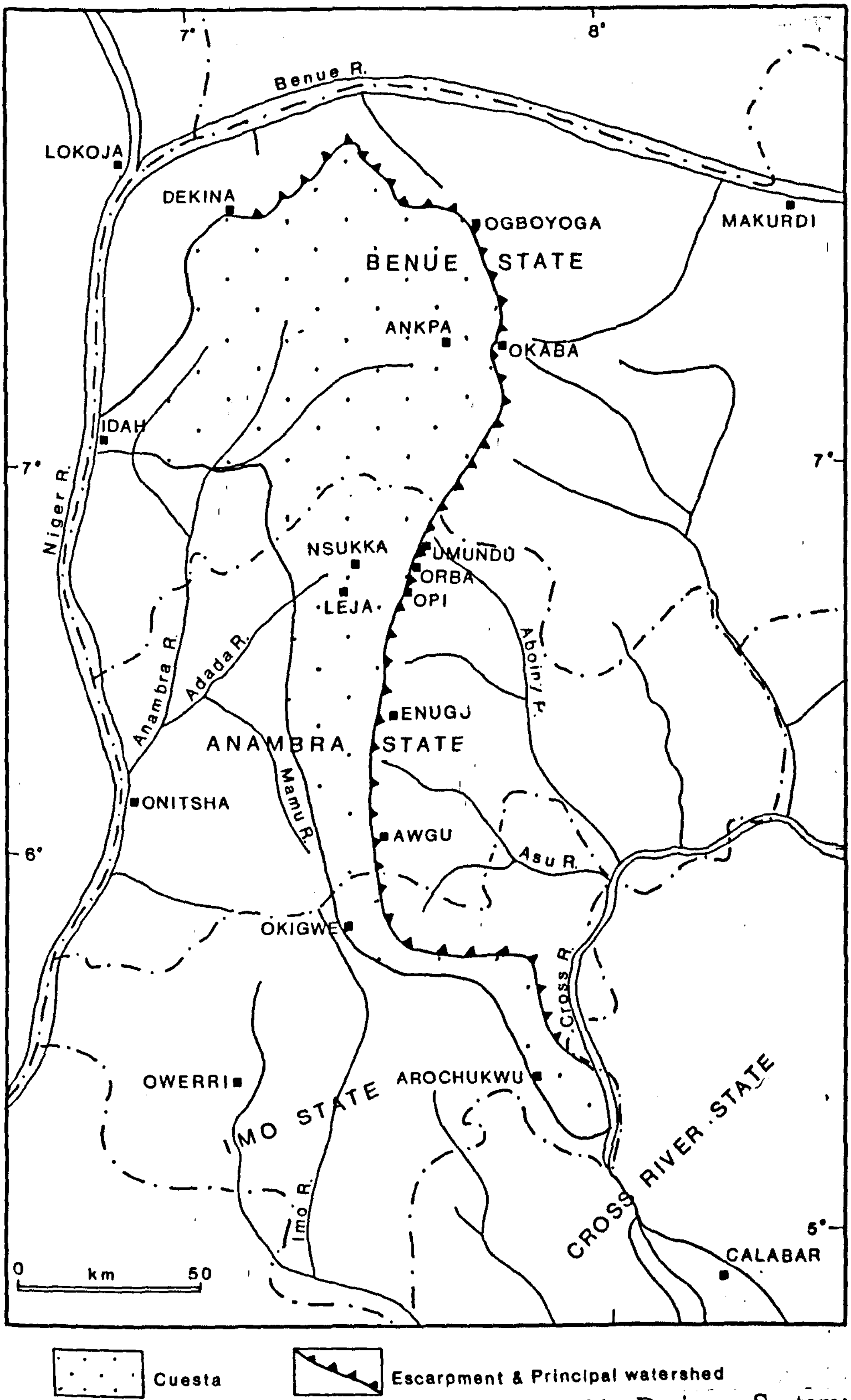


Fig. 2:4 Nsukka-Udi-Arochukwu Cuesta and Nsukka Drainage System: (adapted from Umeji 1980:110).

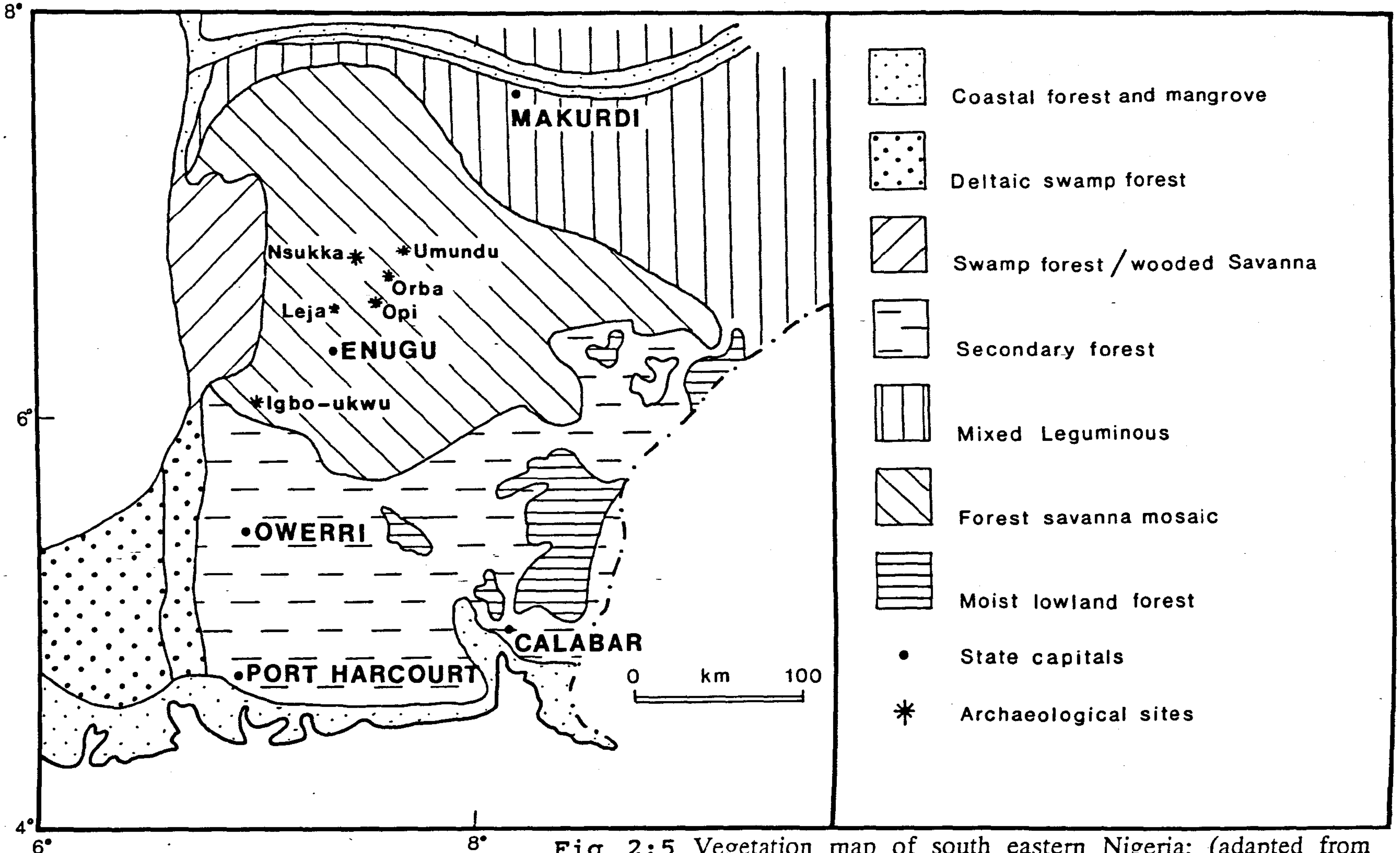


Fig. 2:5 Vegetation map of south eastern Nigeria: (adapted from Oguagha, P. A. and Okpoko, A. I. 1984:127).

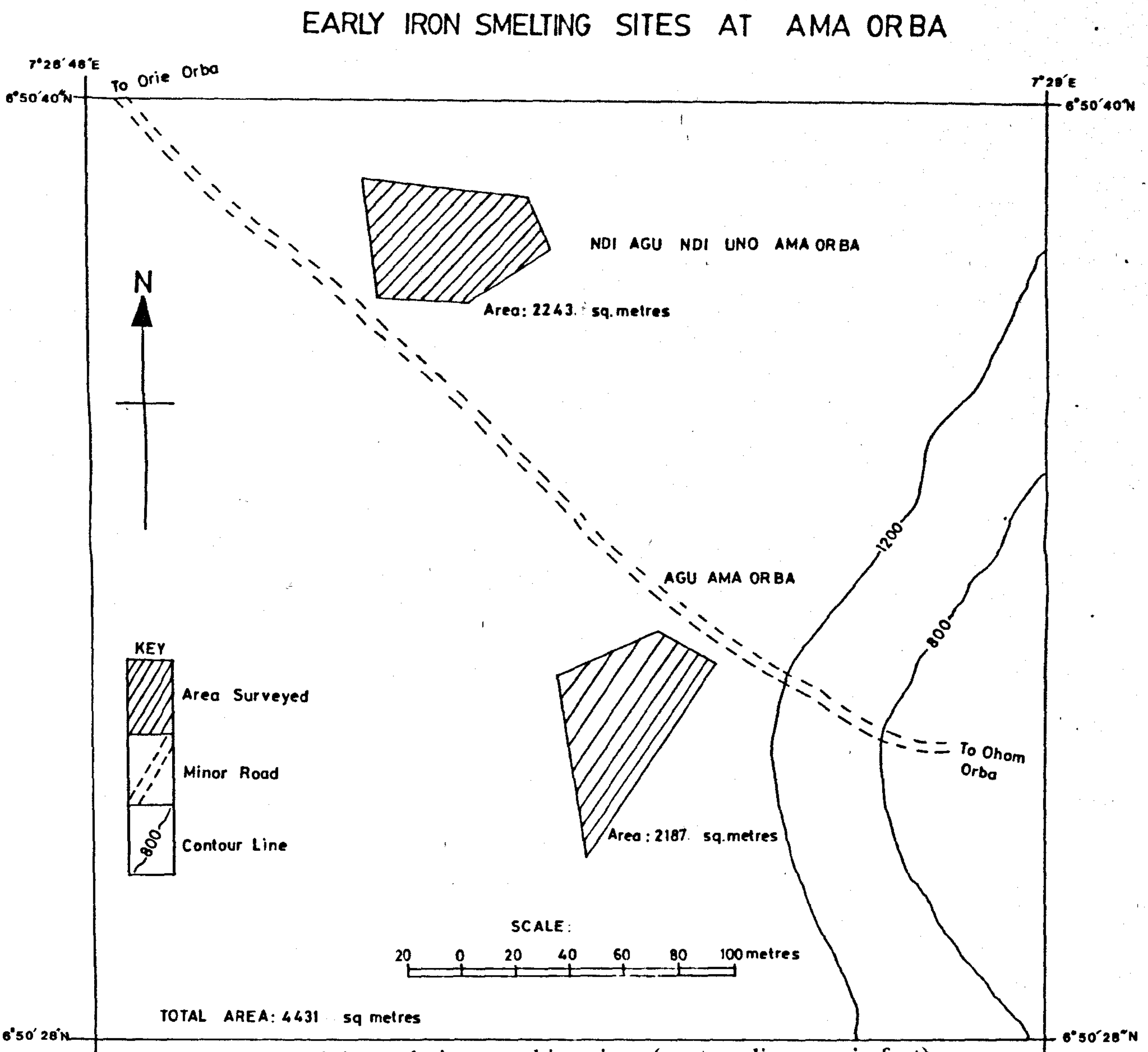


Fig. 3:1 Orba early iron smelting sites: (contour lines are in feet).

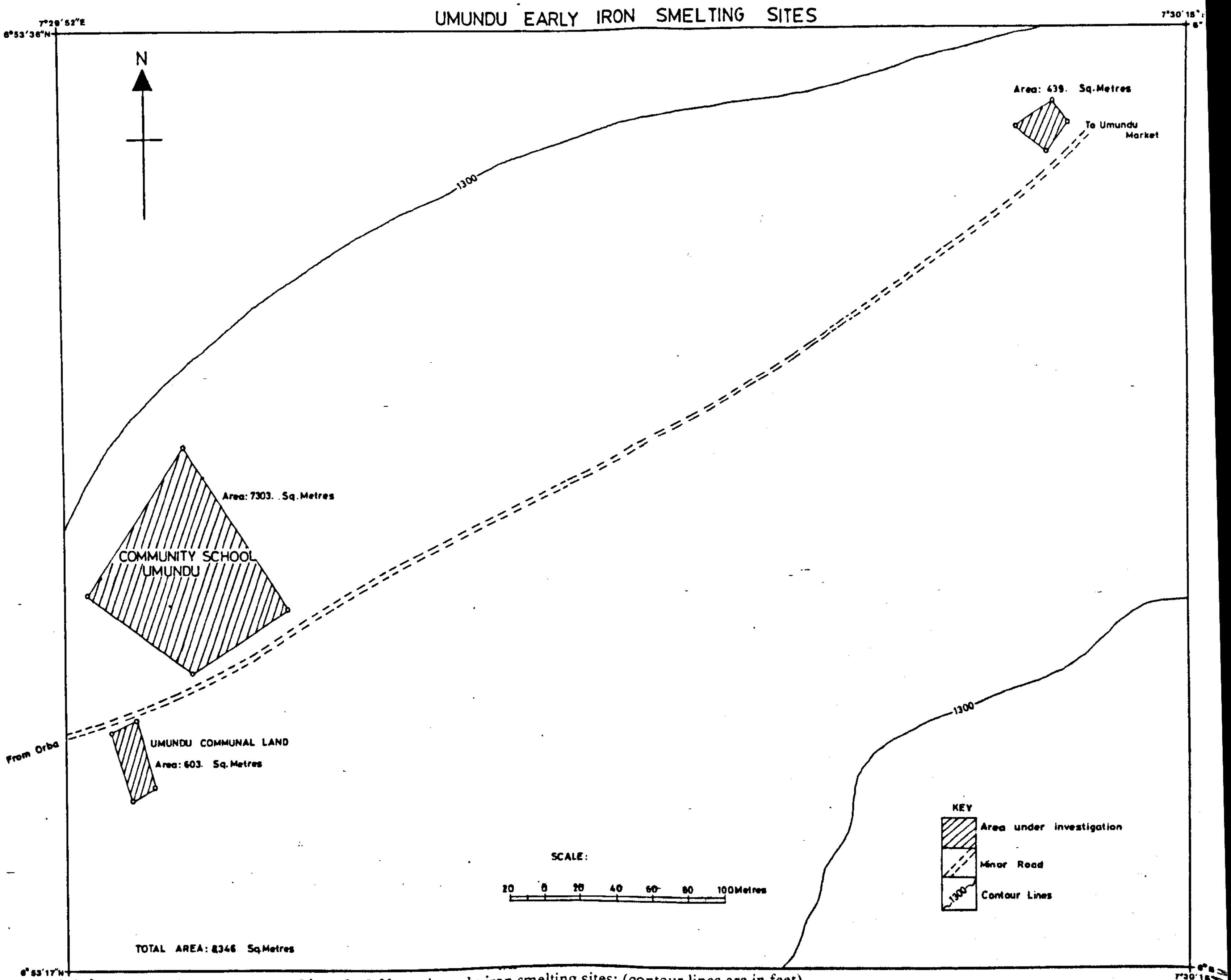


Fig. 3:2 Umundu early iron smelting sites: (contour lines are in feet).

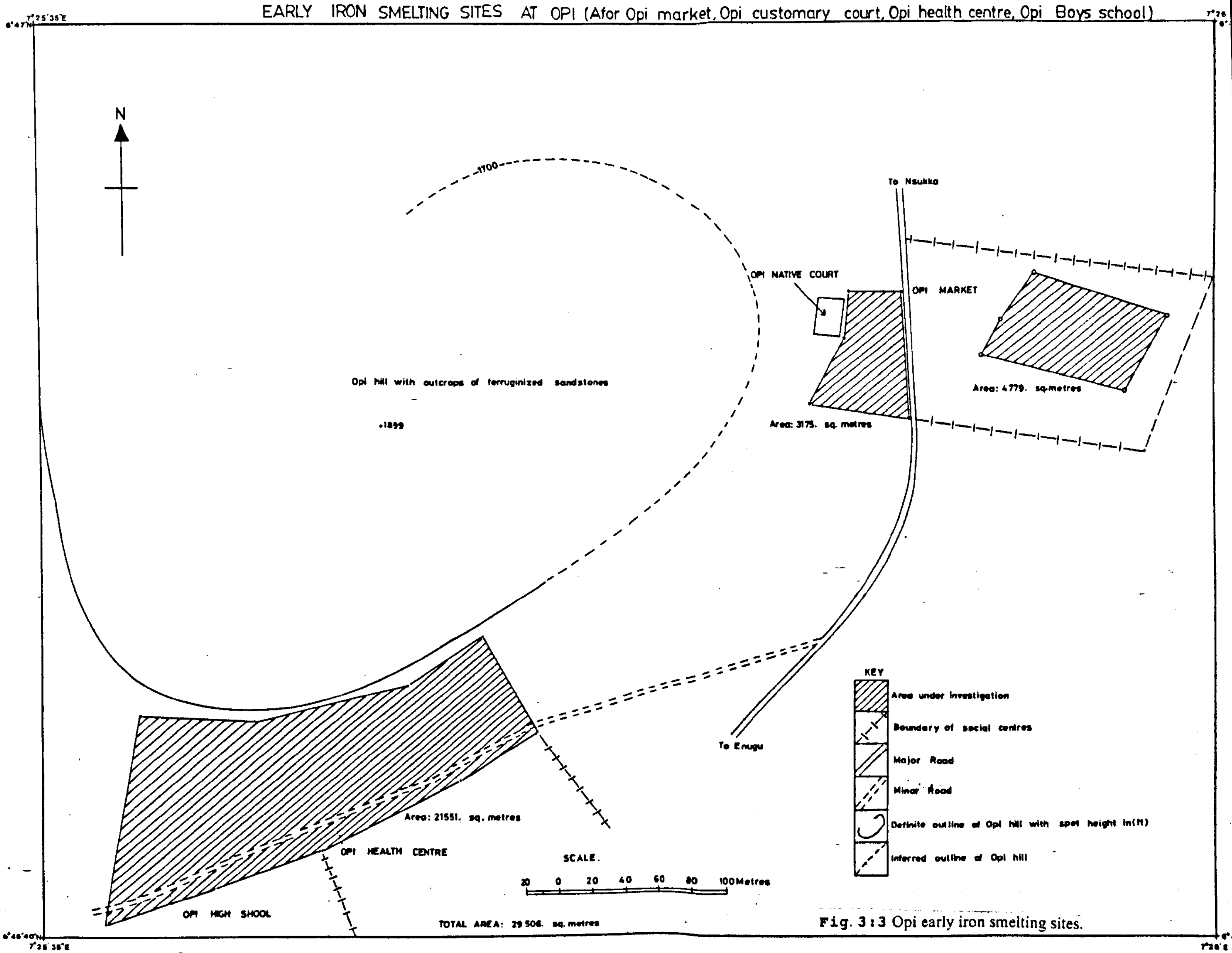


Fig. 3:3 Opi early iron smelting sites.

OWERE - ELU EARLY IRON SMELTING SITE

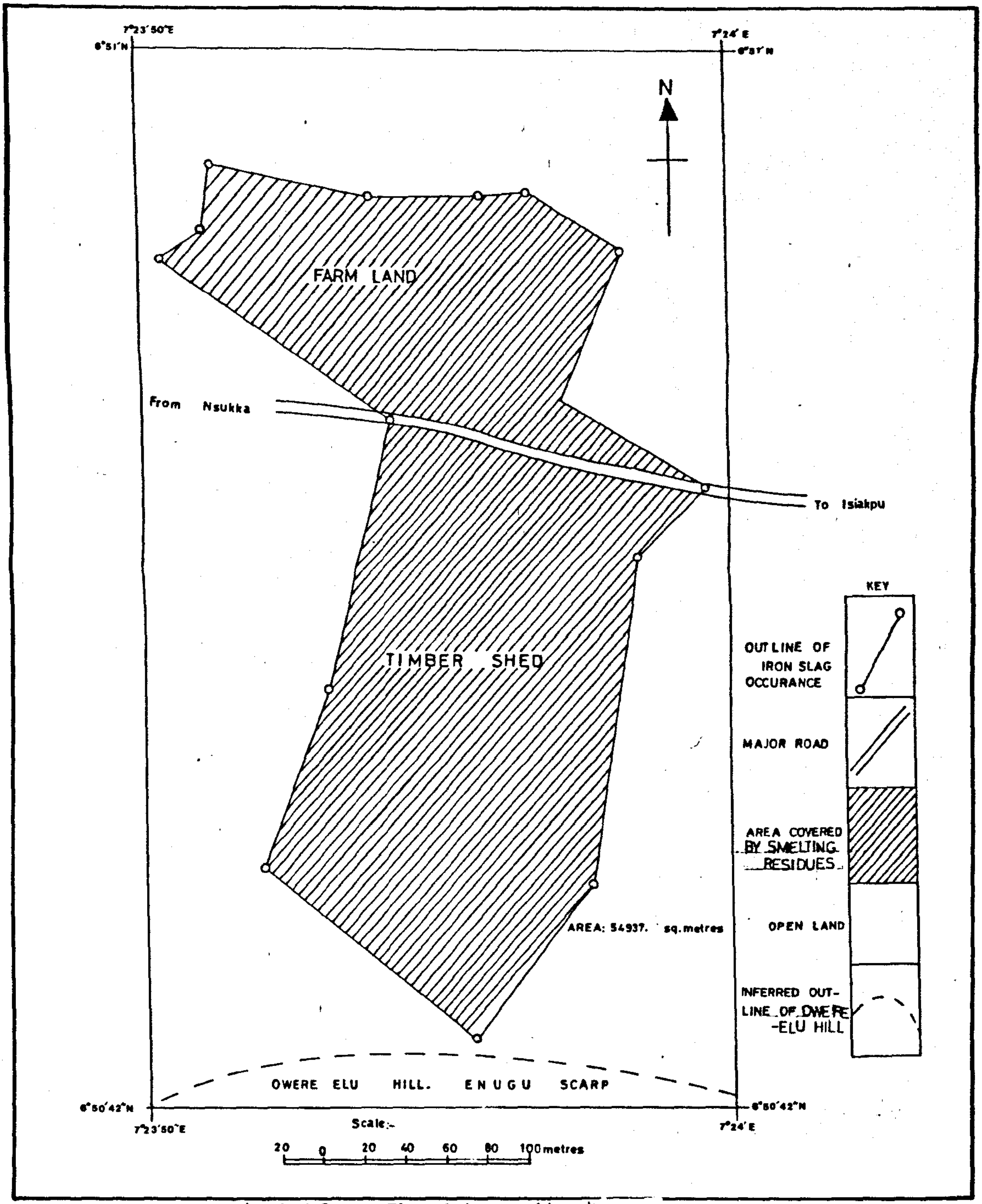


Fig. 3:4 Owerre-Elu early iron smelting site

Fig. 5:1 Fayalite in Nsukka Slags

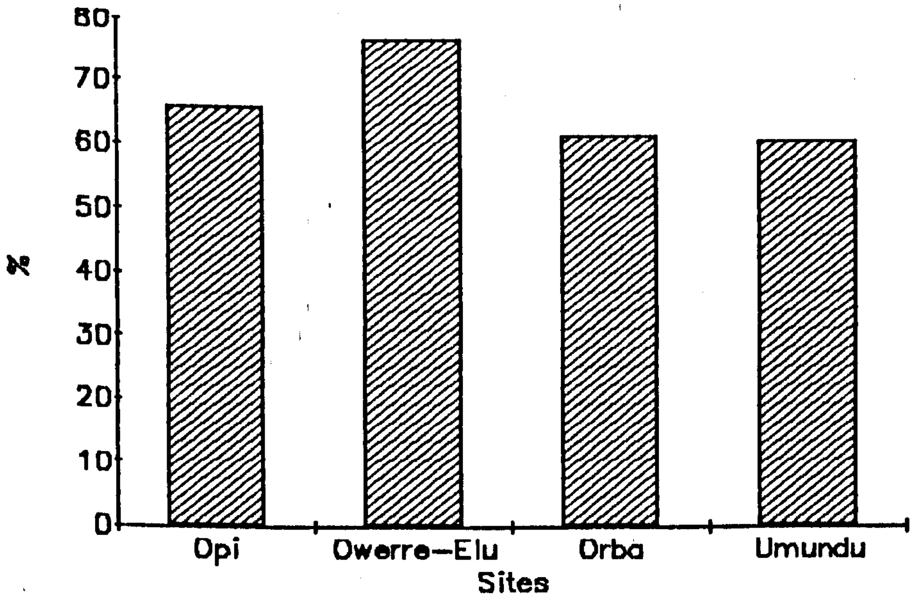


Fig. 5:2 Hercynite in Nsukka Slags

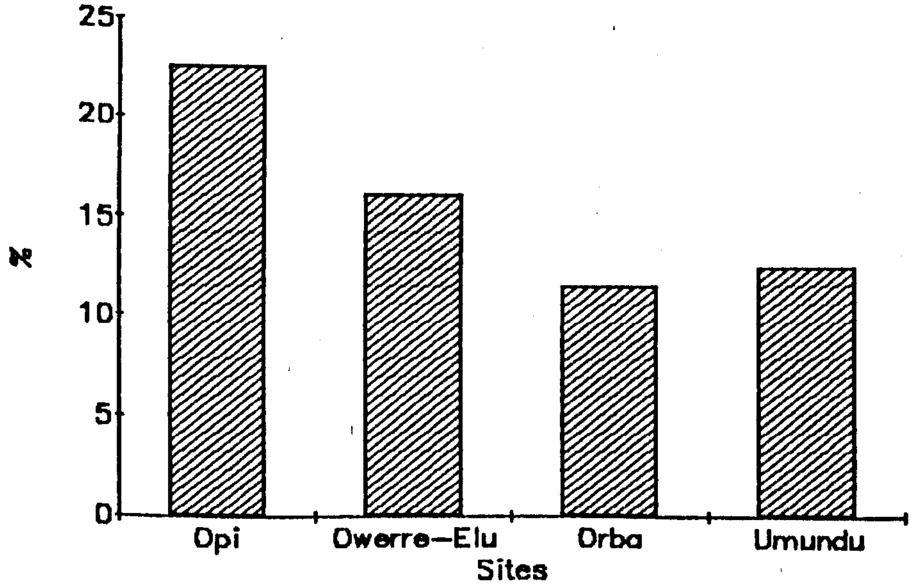


Fig. 5:3 Wustite in Nsukka Slags

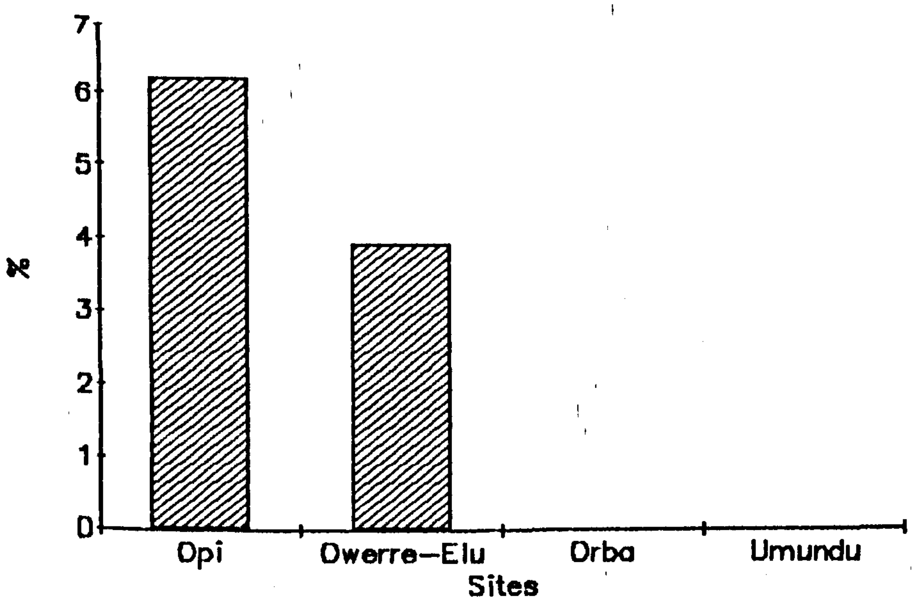
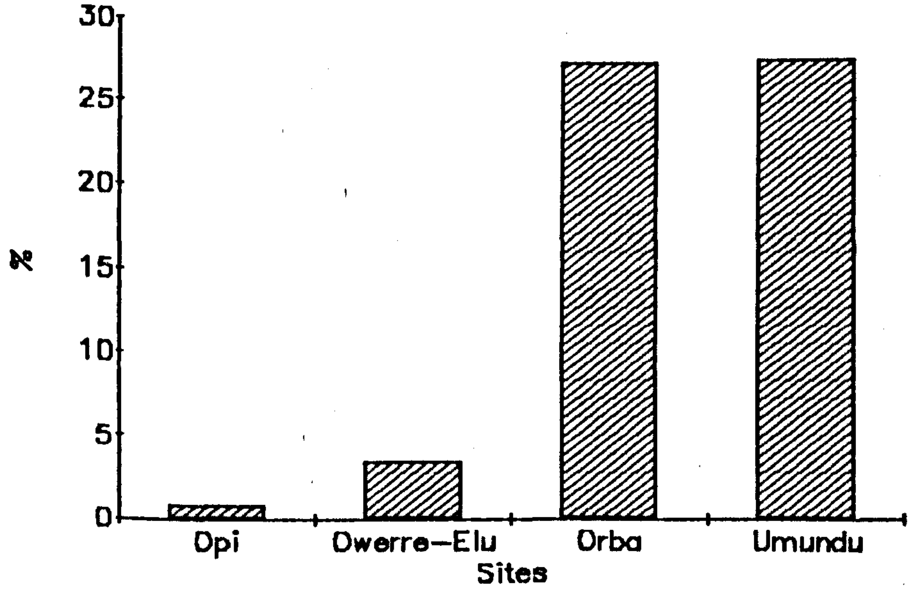
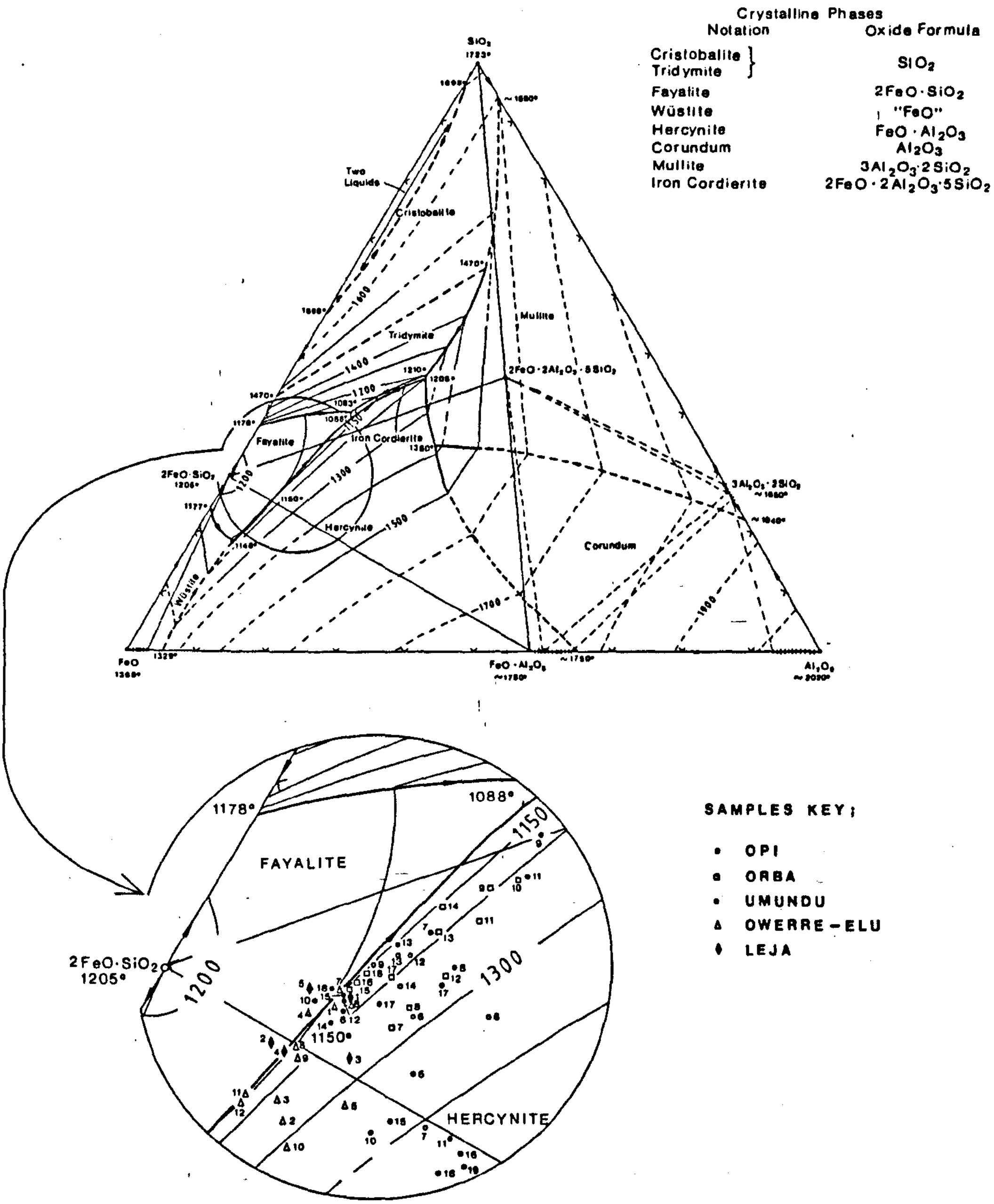


Fig. 5:4 Glass in Nsukka Slags



Liquidus Temperature of Slags from Early Iron Smelting Sites in NSUKKA on FeO – SiO$_2$–Al$_2$O$_3$ Phase Diagram using the Major Constituents of the Bulk Analyses



Crystalline Phases

Notation	Oxide Formula
Cristobalite Tridymite	SiO$_2$
Fayalite	2FeO·SiO$_2$
Wüstite	"FeO"
Hercynite	FeO·Al$_2$O$_3$
Corundum	Al$_2$O$_3$
Mullite	3Al$_2$O$_3$·2SiO$_2$
Iron Cordierite	2FeO·2Al$_2$O$_3$·5SiO$_2$

SAMPLES KEY;

- ● OPI
- ○ ORBA
- ▫ UMUNDU
- Δ OWERRE – ELU
- ♦ LEJA

Fig. 5:5aComposite plot of Nsukka slags in the FeO-Al$_2$O$_3$-SiO$_2$ ternary phase diagram: (Adapted from Phase Diagram for Ceramists: (eds) Levin, E. M. et al 1974).

Concentration of Slags from Early Iron Smelting Sites in NSUKKA on FeO–SiO$_2$–Al$_2$O$_3$ Phase Diagram

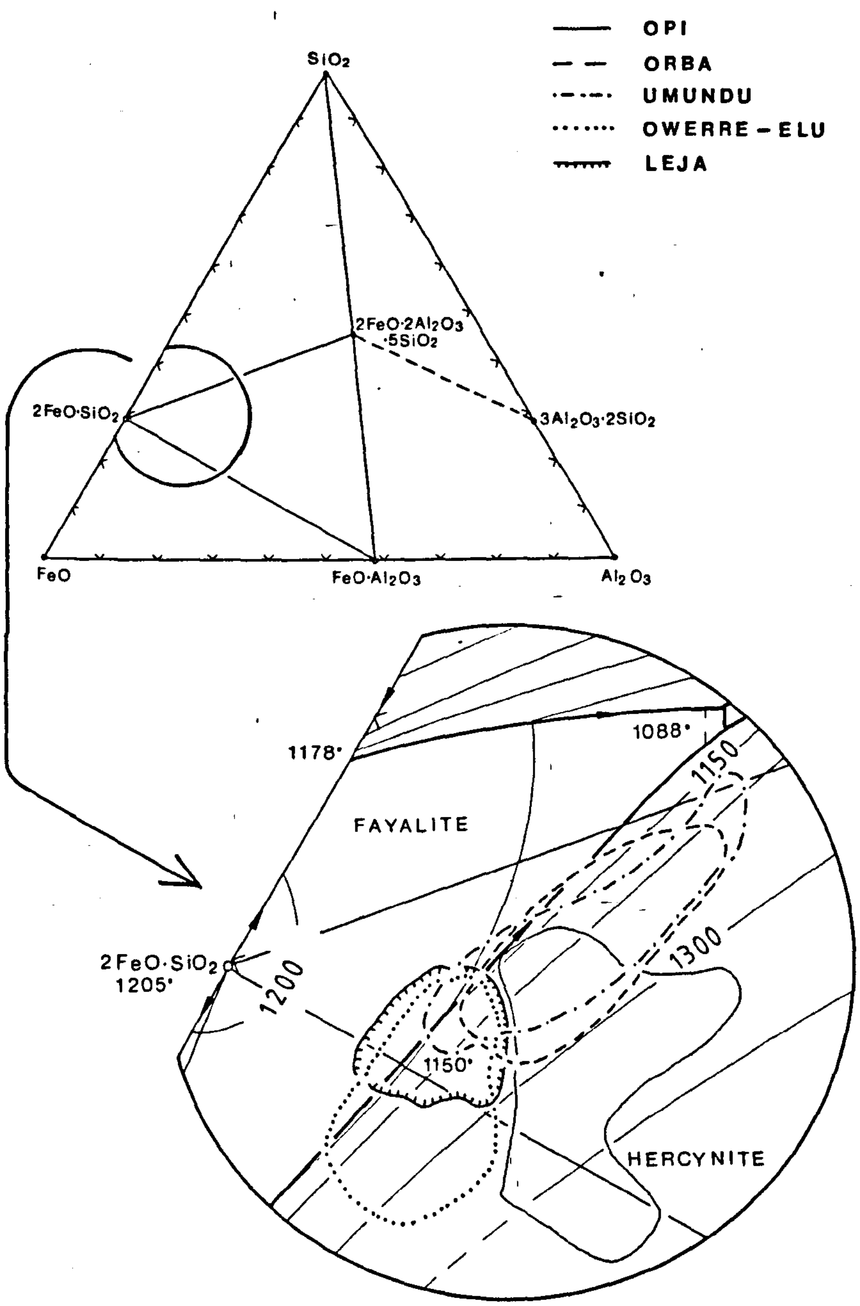


Fig. 5:5b Composite plot of Nsukka slags in the FeO-Al$_2$O$_3$-SiO$_2$ ternary phase diagram: (Adapted from Phase Diagram for Ceramists: (eds) Levin, E. M. et al 1974).

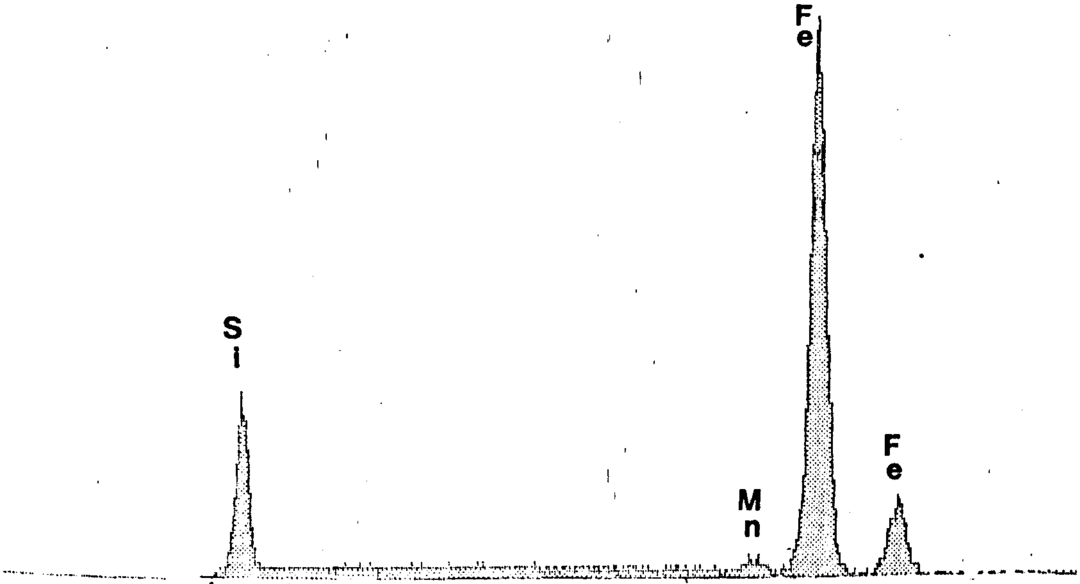


Fig. 5:6 (a) x-ray spectra of fayalite.

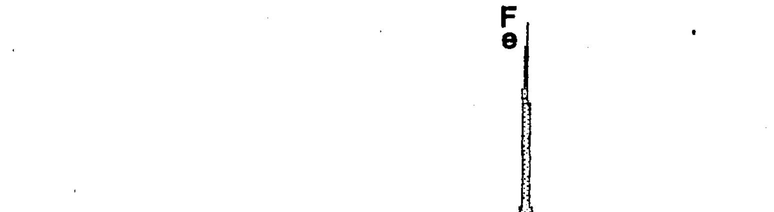


Fig. 5:6 (b) x-ray spectra of hercynite.

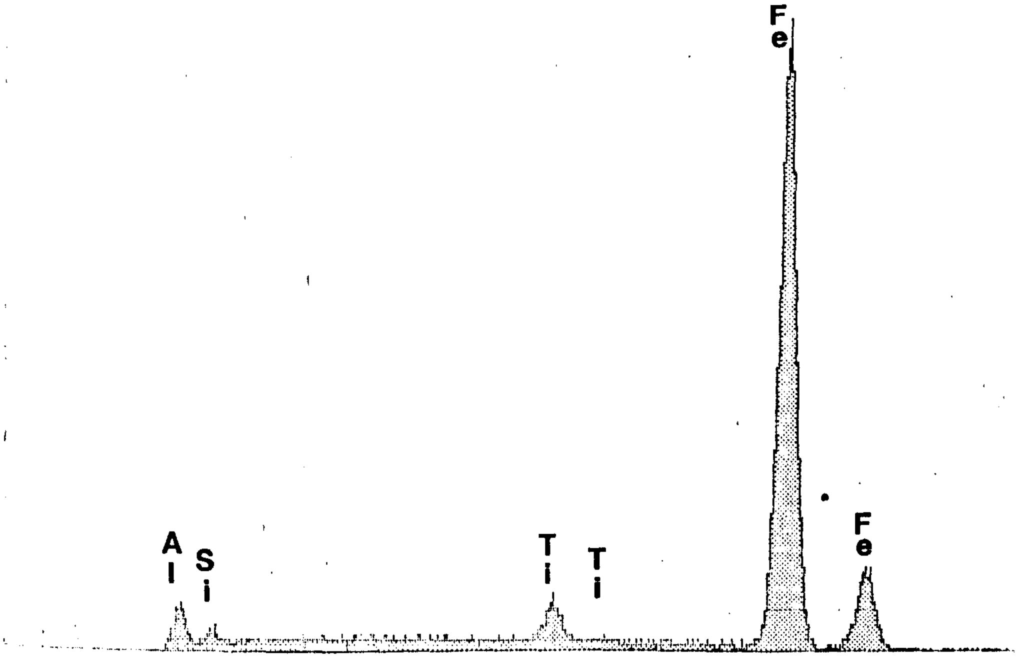


Fig. 5:6 (c) x-ray spectra of wustite.

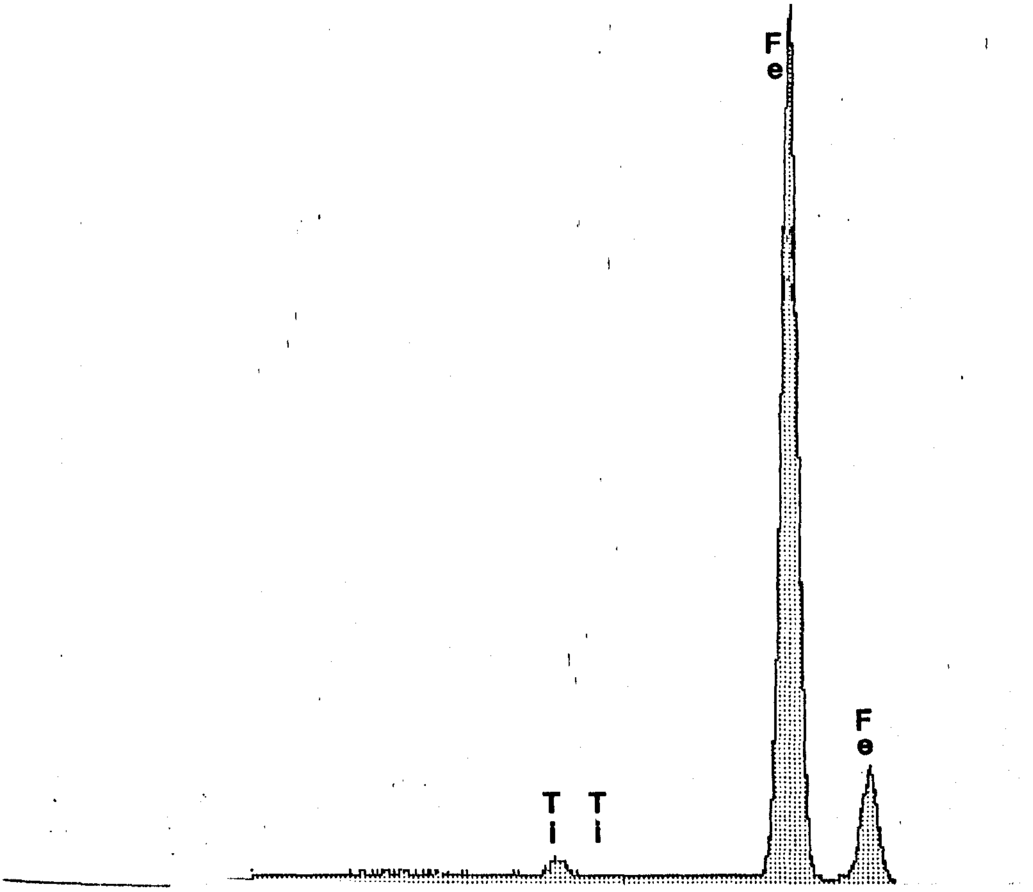


Fig. 5:6 (d) x-ray spectra of magnetite.

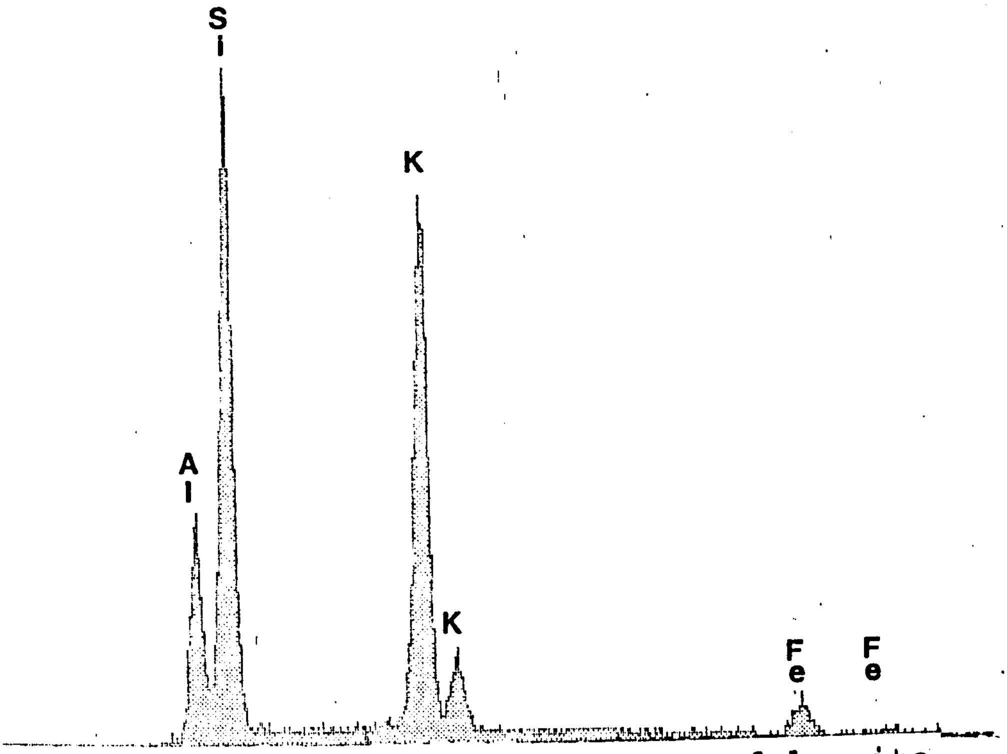


Fig. 5:6 (e) x-ray spectra of leucite

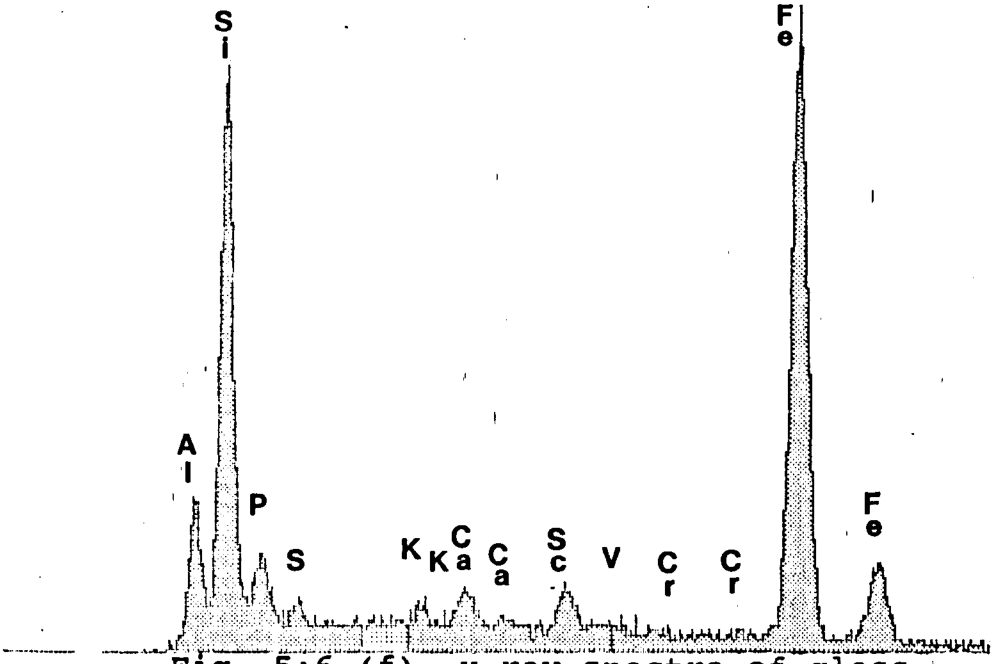


Fig. 5:6 (f) x-ray spectra of glass.

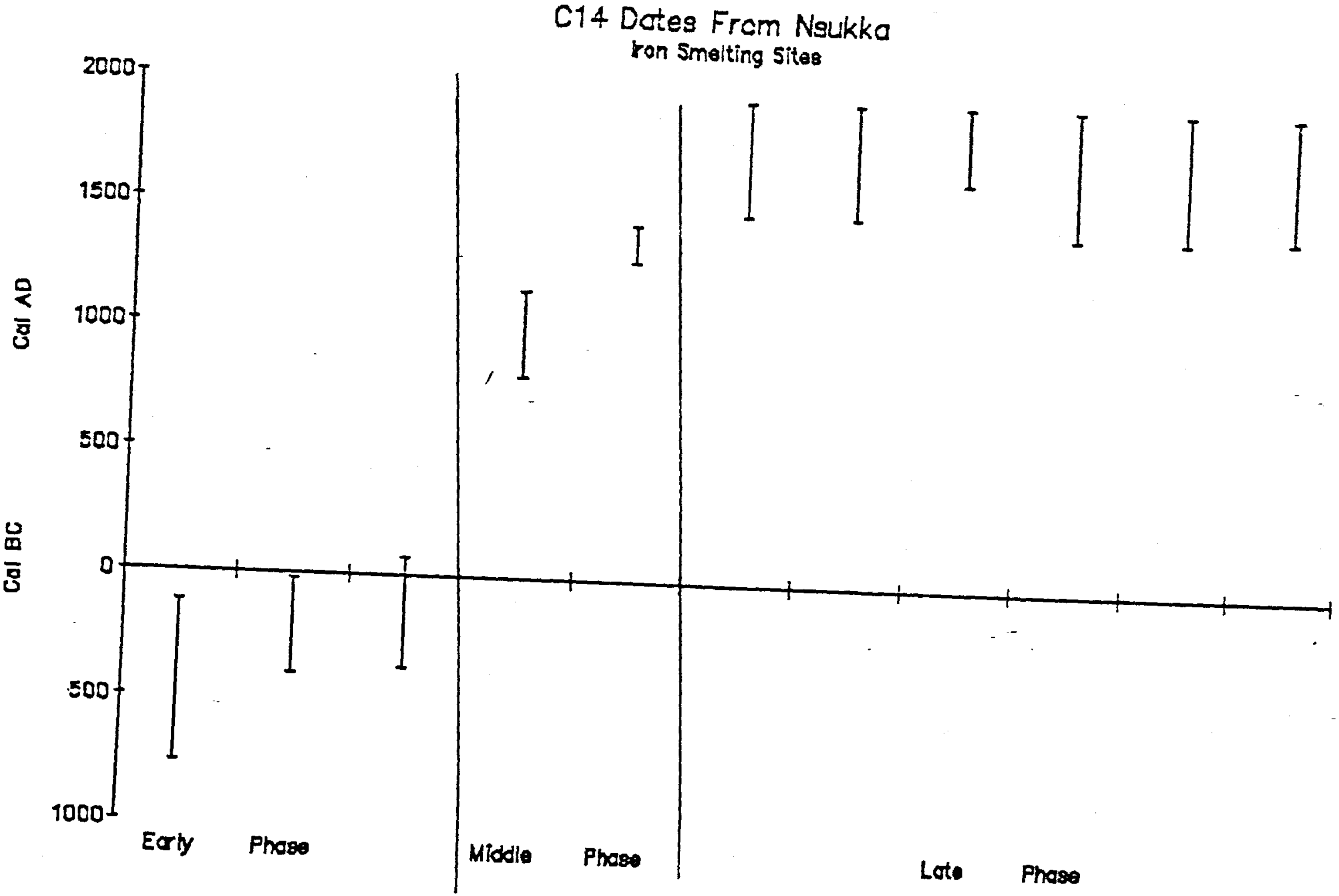
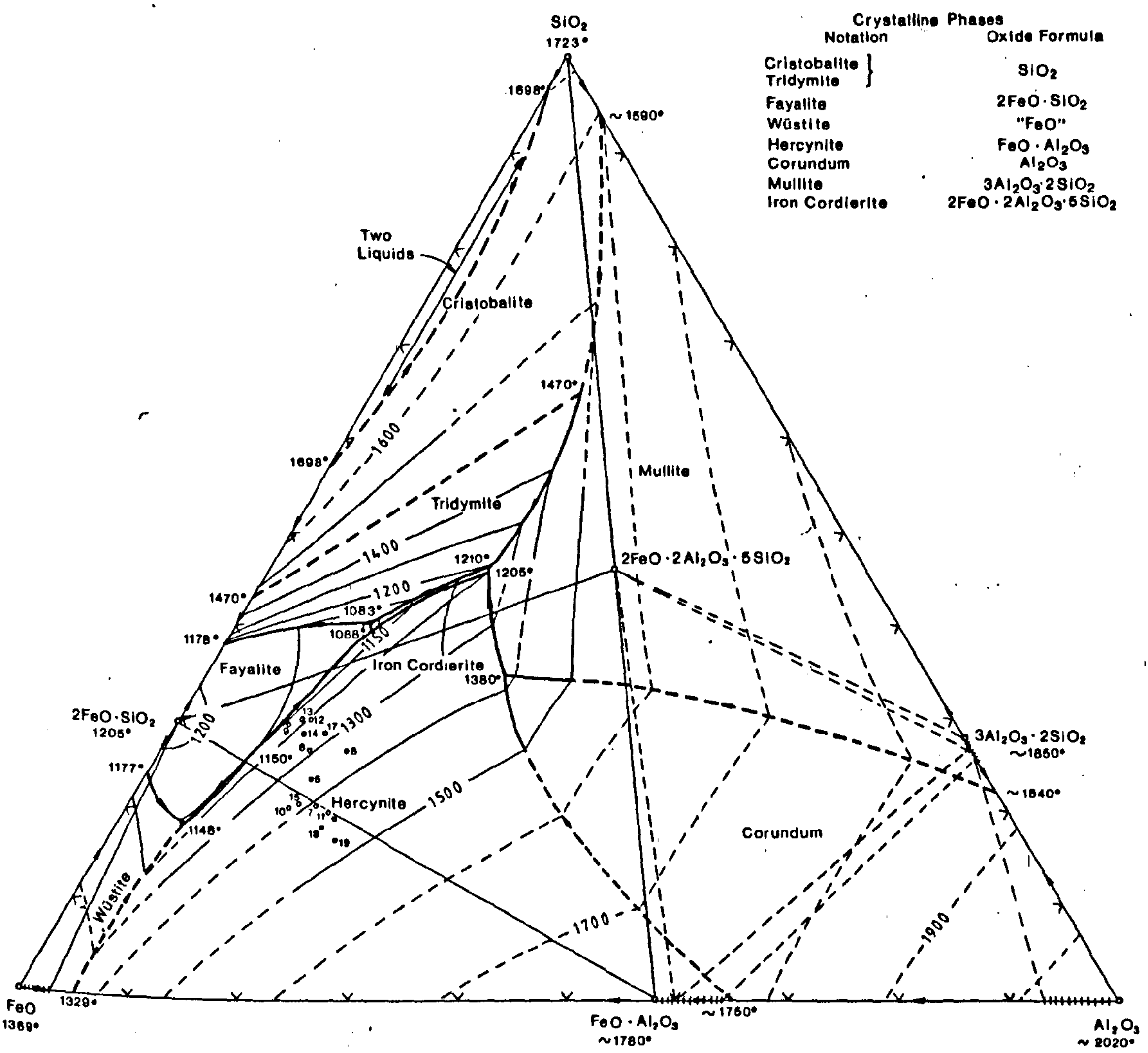


Fig. 5:7 Chart of C-14 dates from Nsukka iron smelting sites calibrated to two standard deviation.

Liquidus Temperature of Slags from OPI-NSUKKA on FeO-SiO_2-Al_2O_3 Phase Diagram using the Major Constituents of the Bulk Analyses



Crystalline Phases	
Notation	Oxide Formula
Cristobalite Tridymite	SiO_2
Fayalite	$2FeO\cdot SiO_2$
Wüstite	"FeO"
Hercynite	$FeO\cdot Al_2O_3$
Corundum	Al_2O_3
Mullite	$3Al_2O_3\cdot 2SiO_2$
Iron Cordierite	$2FeO\cdot 2Al_2O_3\cdot 5SiO_2$

Fig.6.1 —System "FeO"—Al_2O_3—SiO_2 ; composite.

Liquidus Temperature of Slags from OWERRE-ELU-NSUKKA on FeO-SiO_2-Al_2O_3 Phase Diagram using the Major Constituents of the Bulk Analyses

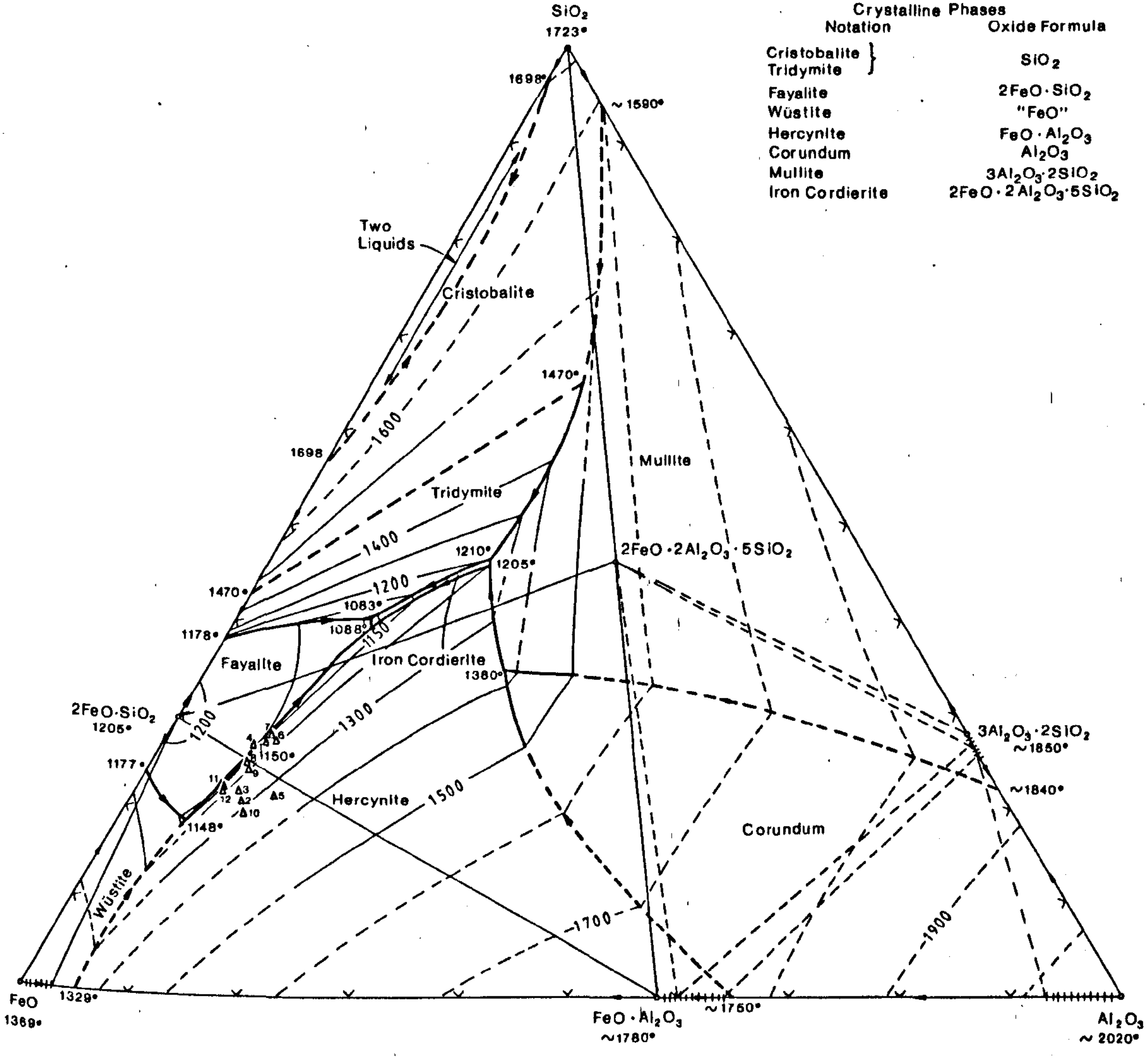
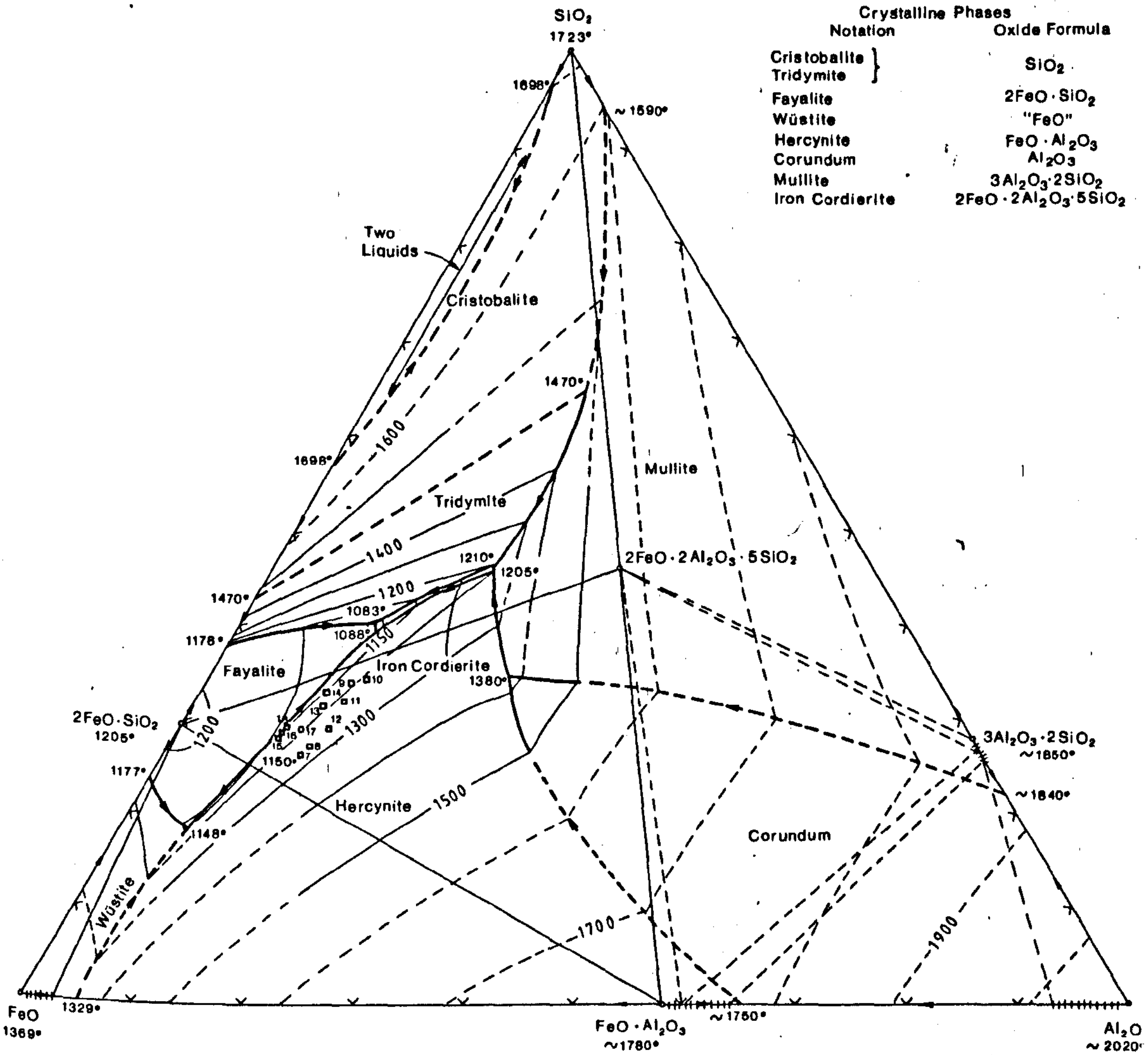


Fig.6.2 — System "FeO"—Al_2O_3—SiO_2 ; composite.

Liquidus Temperature of Slags from ORBA-NSUKKA on FeO-SiO_2-Al_2O_3 Phase Diagram using the Major Constituents of the Bulk Analyses

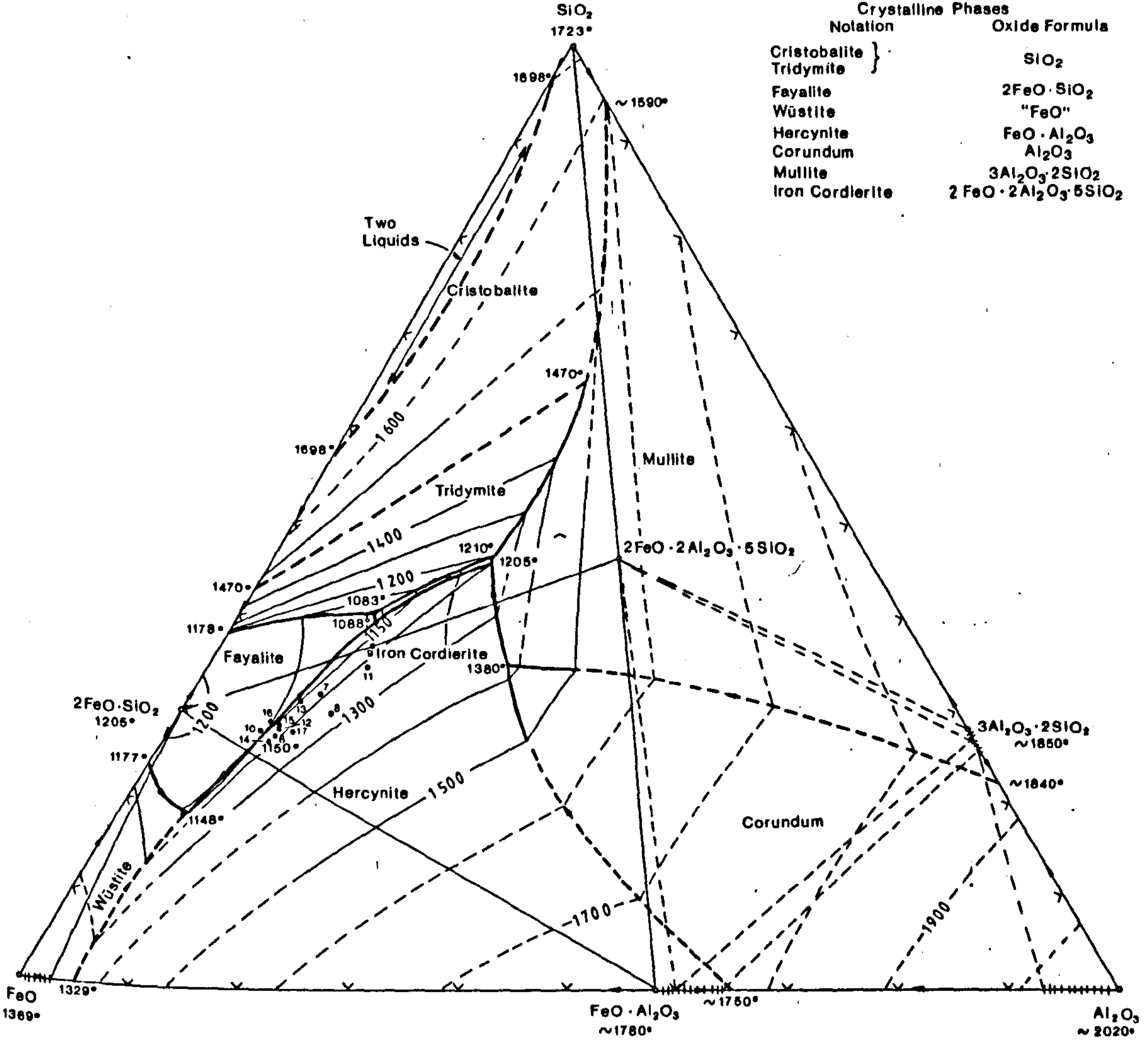


Crystalline Phases

Notation	Oxide Formula
Cristobalite Tridymite	SiO_2
Fayalite	$2FeO\cdot SiO_2$
Wüstite	"FeO"
Hercynite	$FeO\cdot Al_2O_3$
Corundum	Al_2O_3
Mullite	$3Al_2O_3\cdot 2SiO_2$
Iron Cordierite	$2FeO\cdot 2Al_2O_3\cdot 5SiO_2$

Fig.6.3a-System "FeO"–Al_2O_3—SiO_2 ; composite.

Liquidus Temperature of Slags from UMUNDU-NSUKKA on FeO-SiO_2-Al_2O_3 Phase Diagram using the Major Constituents of the Bulk Analyses



Crystalline Phases

Notation	Oxide Formula
Cristobalite Tridymite	SiO_2
Fayalite	2FeO·SiO_2
Wüstite	"FeO"
Hercynite	FeO·Al_2O_3
Corundum	Al_2O_3
Mullite	3Al_2O_3·2SiO_2
Iron Cordierite	2 FeO·2Al_2O_3·5SiO_2

Fig.6.3 b System "FeO"—Al_2O_3—SiO_2 ; composite.

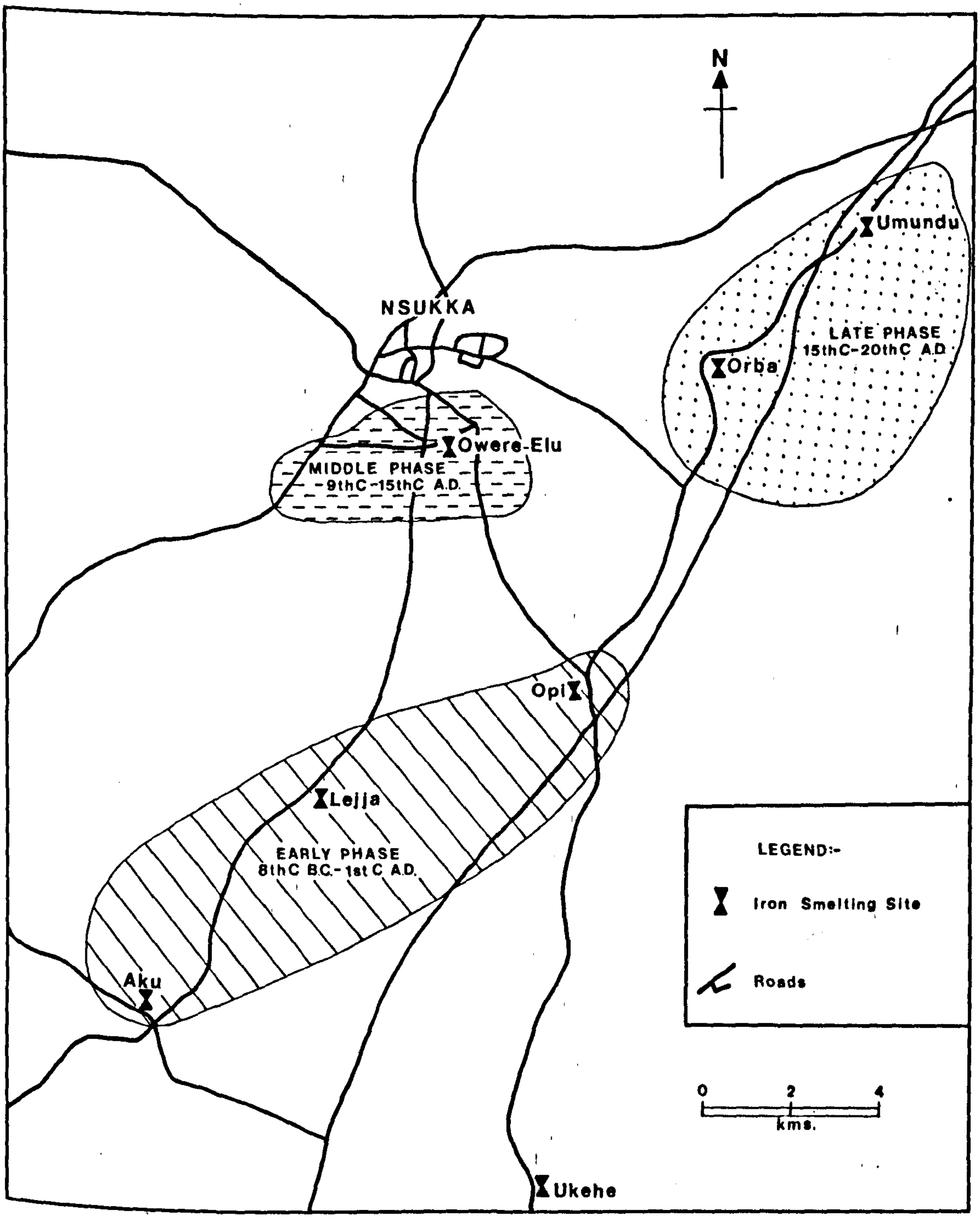


Fig. 6:4 Areas covered by the three phases of Nsukka iron smelting and their probable dates: (Adapted from Nigeria 1:100,000 Nsukka and Igbo-Etiti local government areas: first edition).

Plate 1:1 Hammer and anvils made from Nsukka bloomery iron.

Plates 1:2 Nsukka slag remains: (a) cylindrical blocks (b) flat ropy surfaced and (c) mounds of slag aggregate.

Plate 2:1 Exposed haematite lumps on the Nru-Isiakpu-Owerre-Elu ridge.

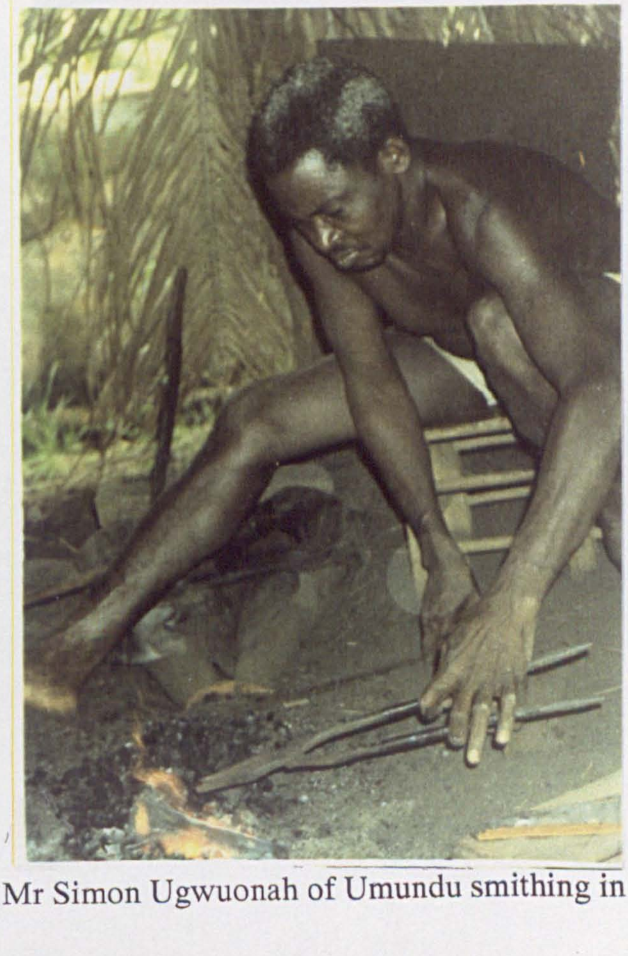


Plate 2:2 Mr Simon Ugwuonah of Umundu smithing in his workshop.

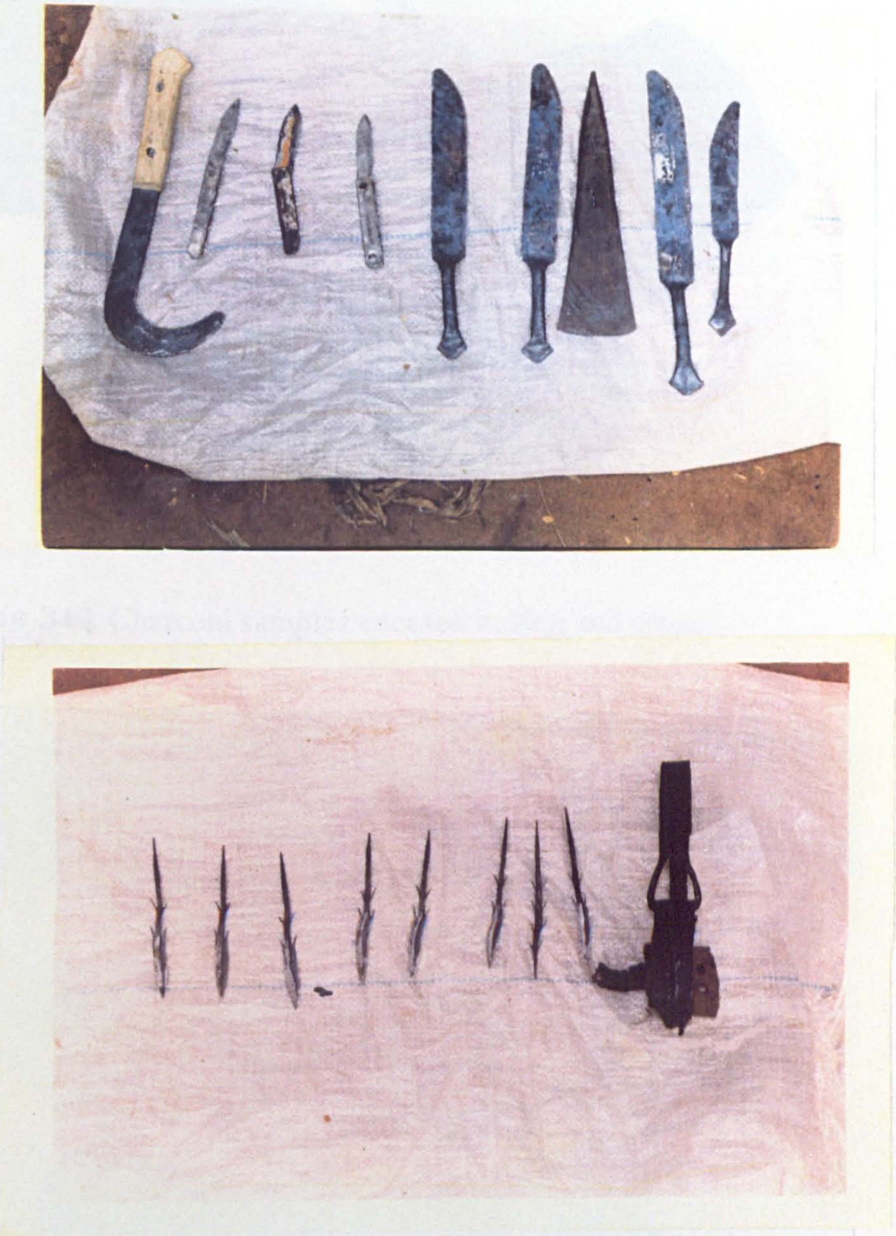


Plate 2:3 Some of the products of Nsukka blacksmiths.

Plate 3 : 1 Charcoal samples encased in slag and cinder.

Plate 3 : 2 Orba slag aggregates.

Plate 3:3 Umundu slag aggregates.

Plate 3:4 (a) Opi cylindrical blocks of slags at Odinanso Idi Opi Village square and shrines (b) Slag samples from Nsukka cylindrical blocks of slag.

Plate 3:5 Smooth ropy surfaced slags from Owerre-Elu.

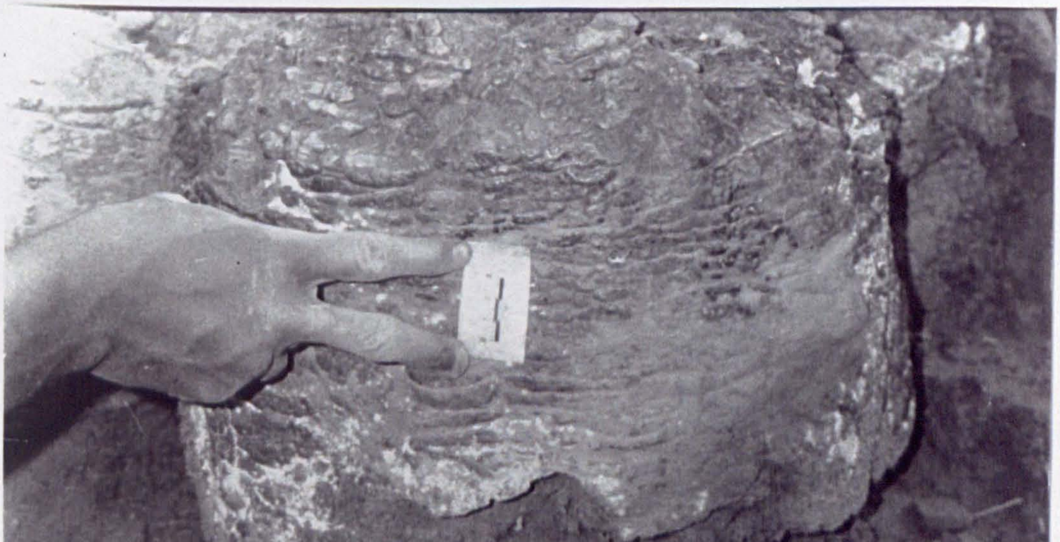
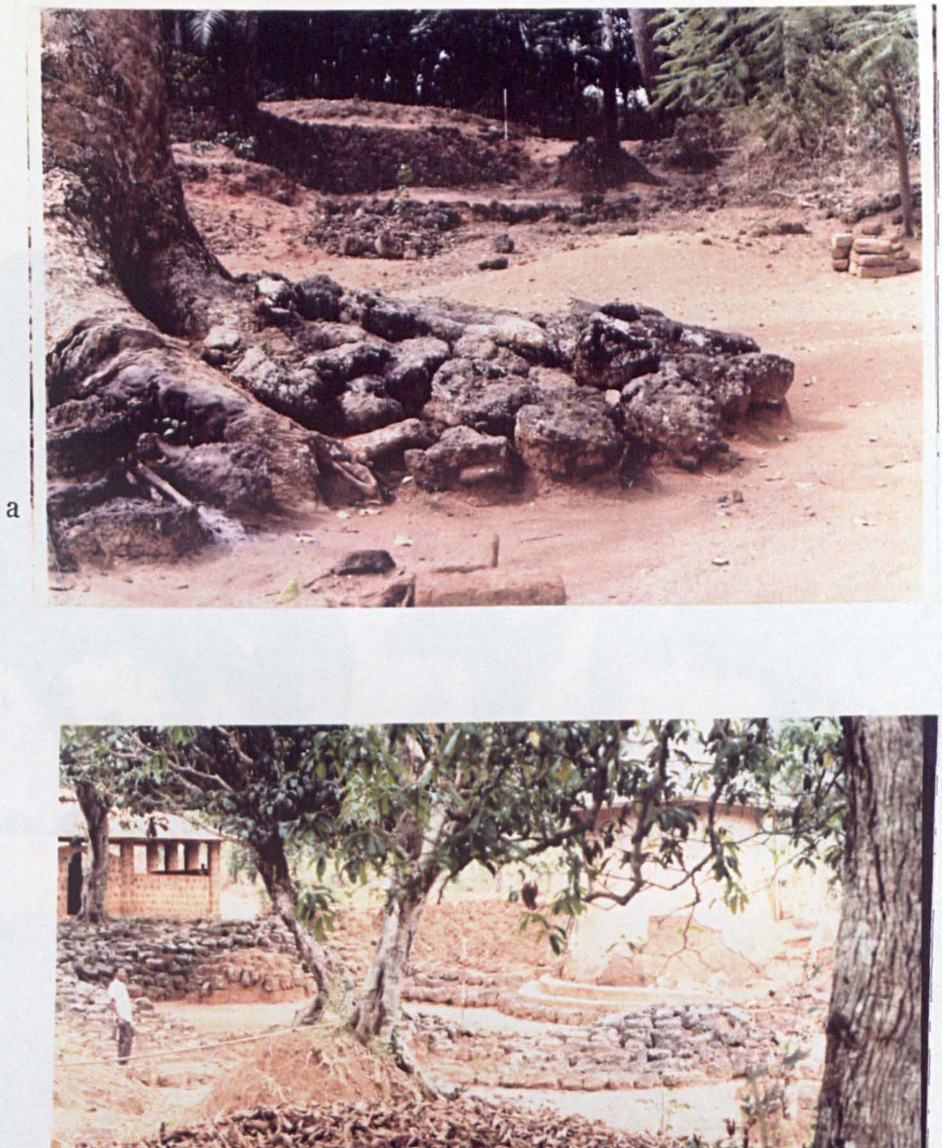


Plate 4:1 Various layers of individual tap slags that form the cylindrical slag blocks.

Plate 4:2 Slags used as building foundations.

Plate 4:3 Slags used in erosion control.



Plates 4:4 Slag shrines at (a) Opi (b) Leja.

Plate 4:5 Slags as seats at Leja.

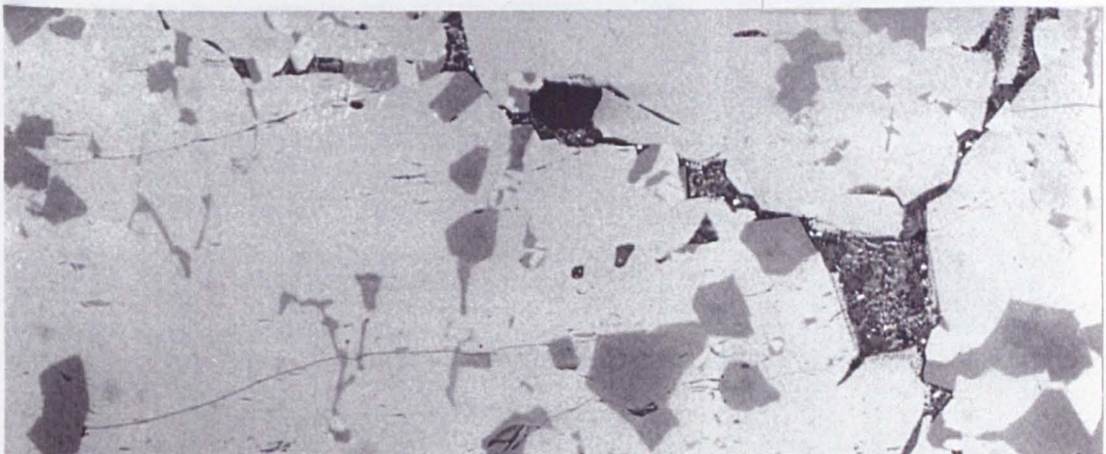


Plate 5:1 Massive light grey fayalite crystals broken by few patches of glass and angular dark grey hercynite crystals.

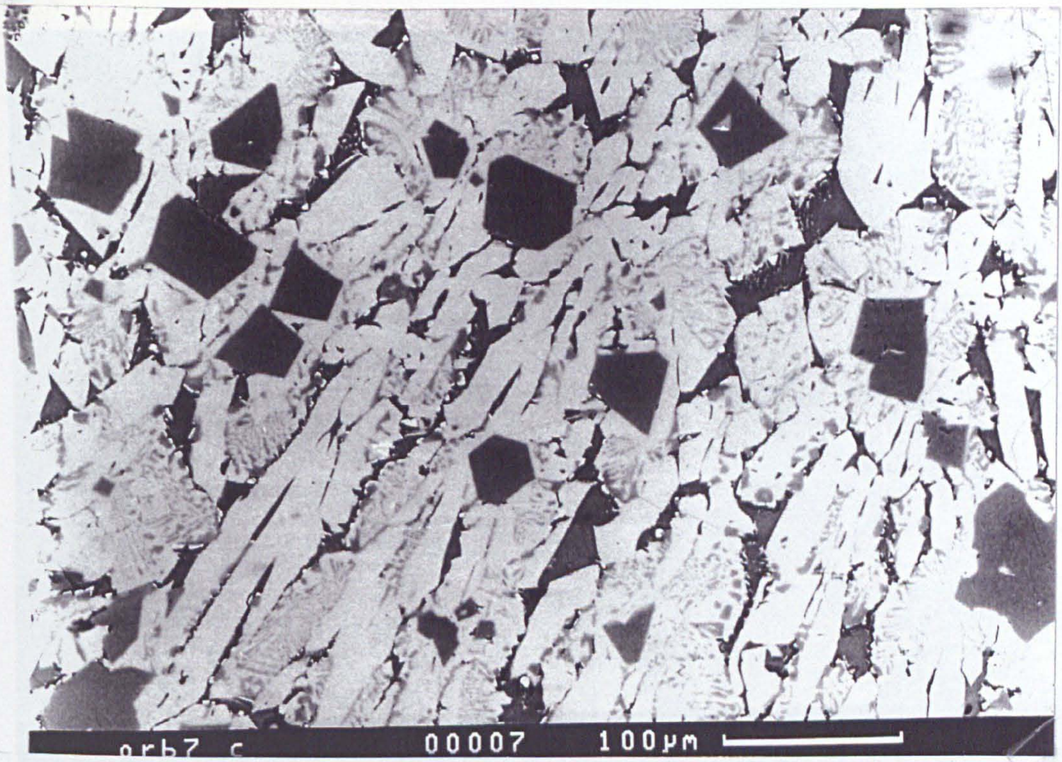


Plate 5:2 light grey broken-lath fayalite crystals associated with dark grey euhedral hercynite crystals in dark glass matrix.

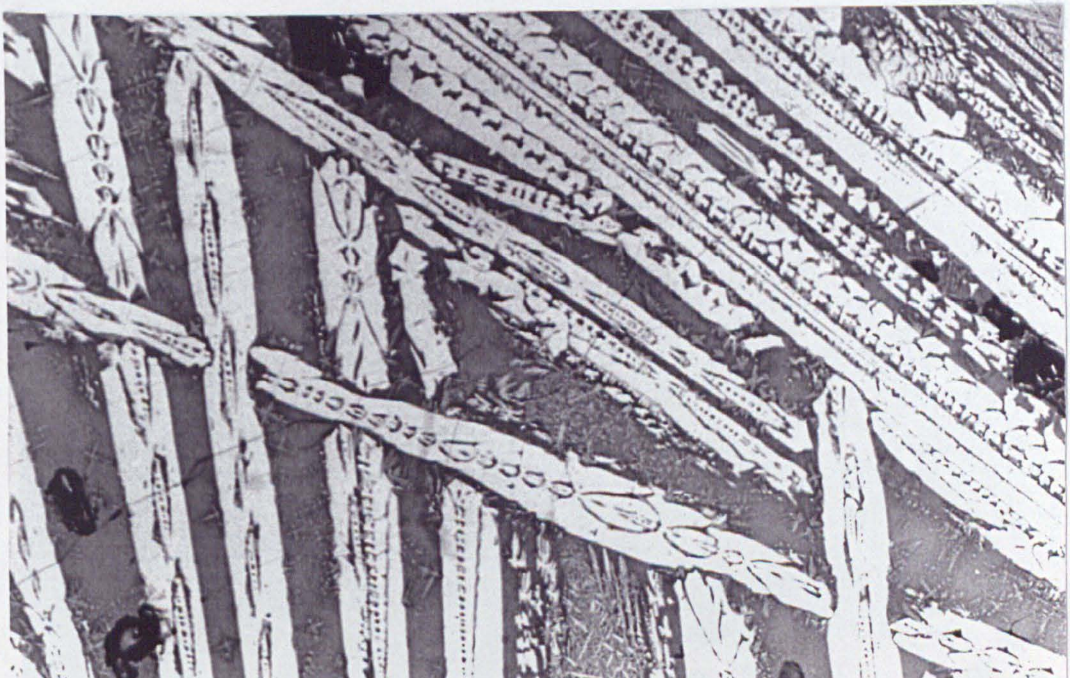


Plate 5:3 Parallel and sub-parallel light grey fayalite lath crystals in dark glass matrix.

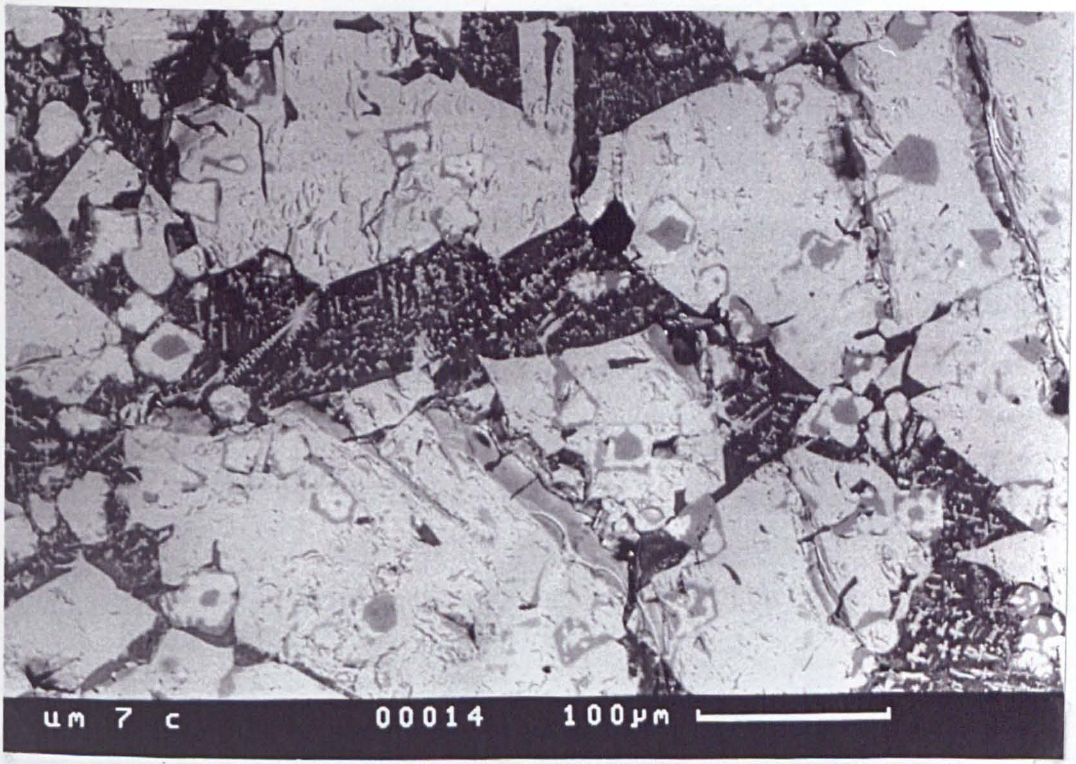


Plate 5:4 Fine rods of light grey fayalite in glass matrix containing light grey broken fayalite and dark grey euhedral crystals of hercynite.

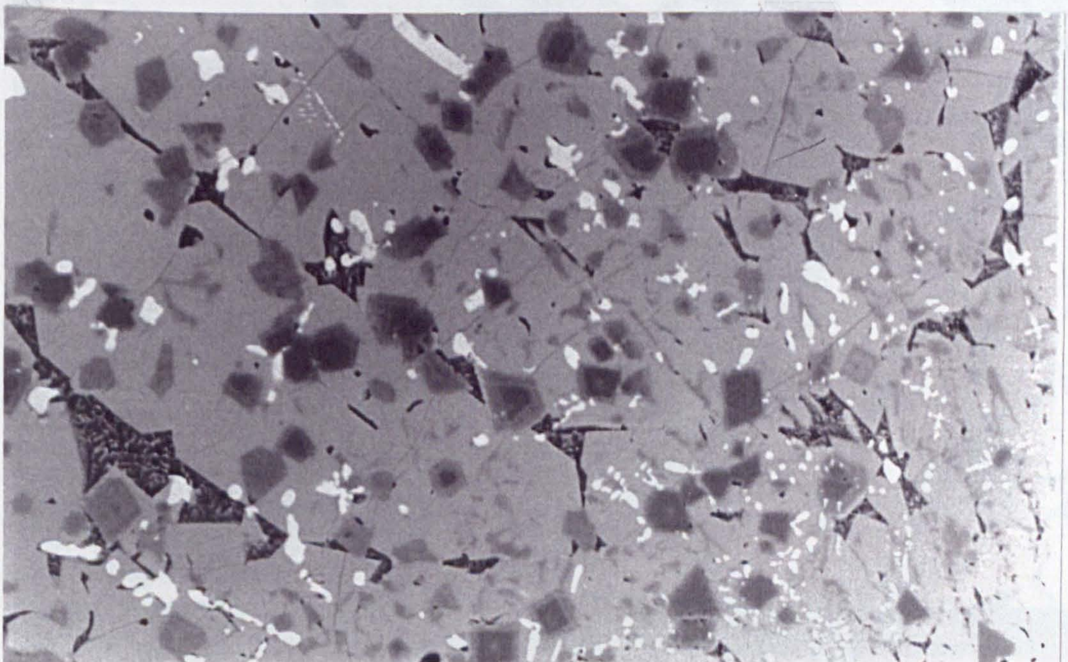
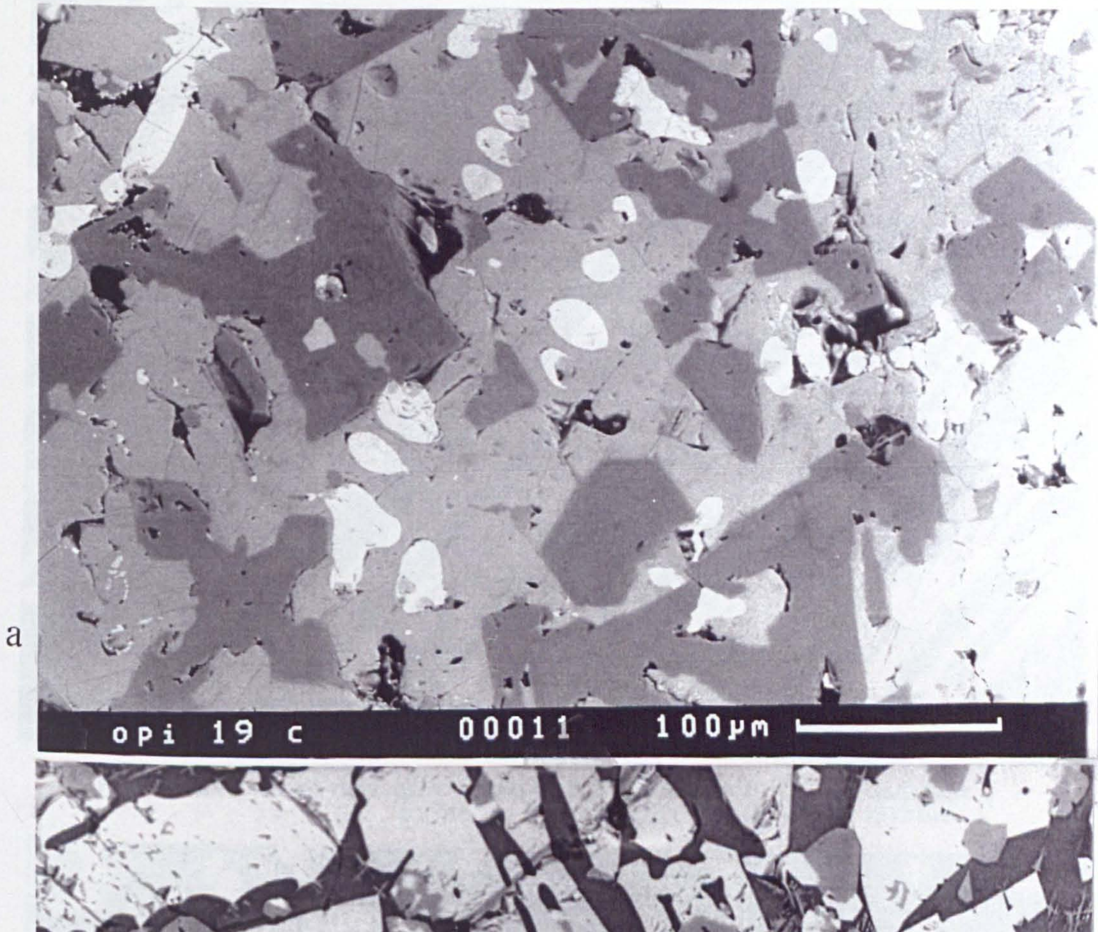


Plate 5:5 Dark grey euhedral hercynite crystals with zoned light grey periphery high in TiO_2. The hercynite associates with massive light grey fayalite and white dendrites of wustite.



Plates 5:6 Dark grey bold euhedral crystals of hercynite in (a) light grey massive fayalite containing isolated white globules of wustite (b) light in colour broken-lath fayalite crystals and dark glass matrix.

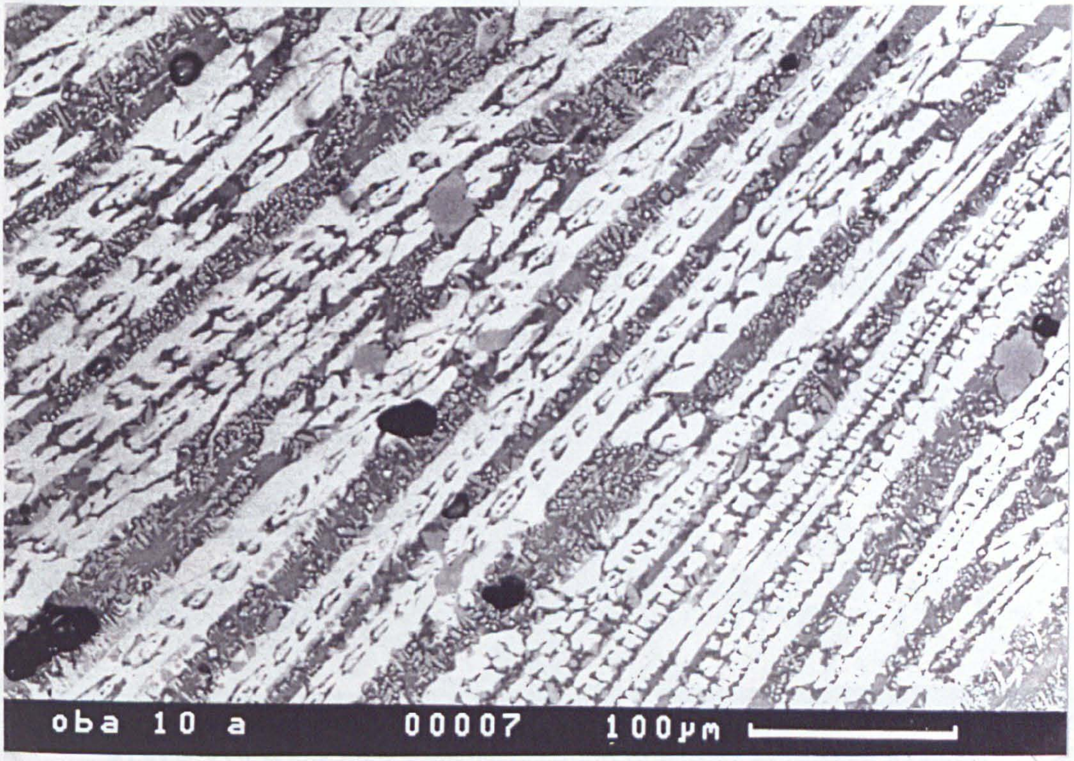


Plate 5:7 Small dark grey euhedral and anhedral crystals of hercynite formed in the dark glass and light grey fayalite.

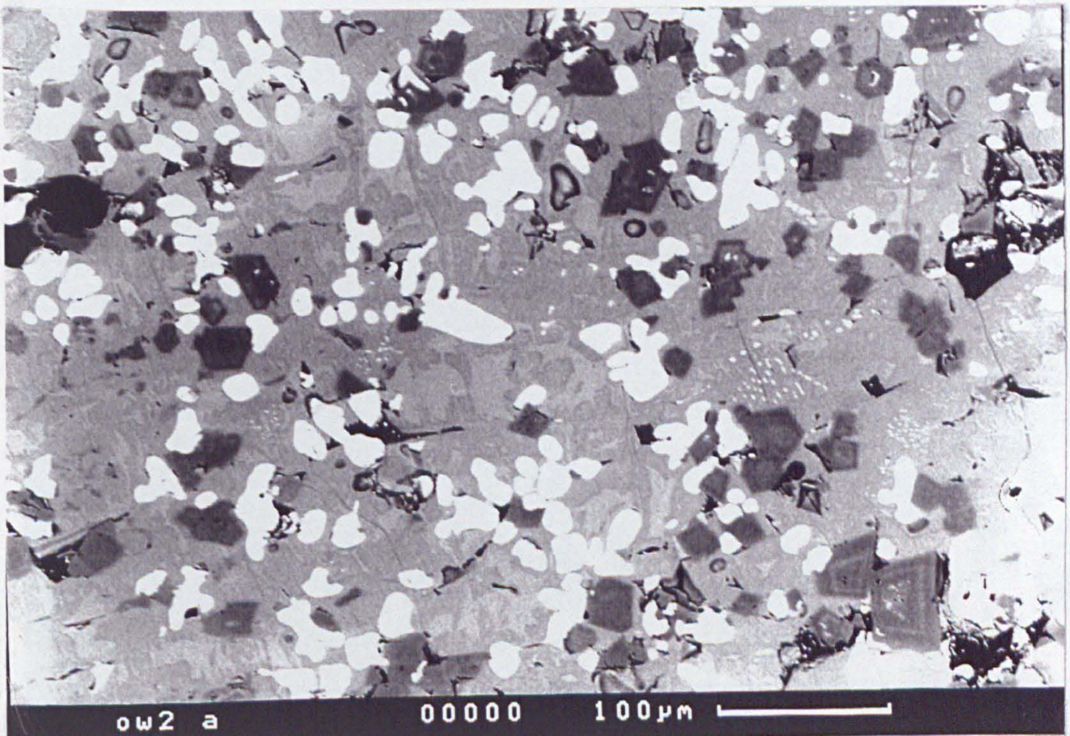


Plate 5:8 White dendrites and very fine wustite formed in light grey massive fayalite crystals containing angular dark grey hercynite crystals and pools of dark glass.

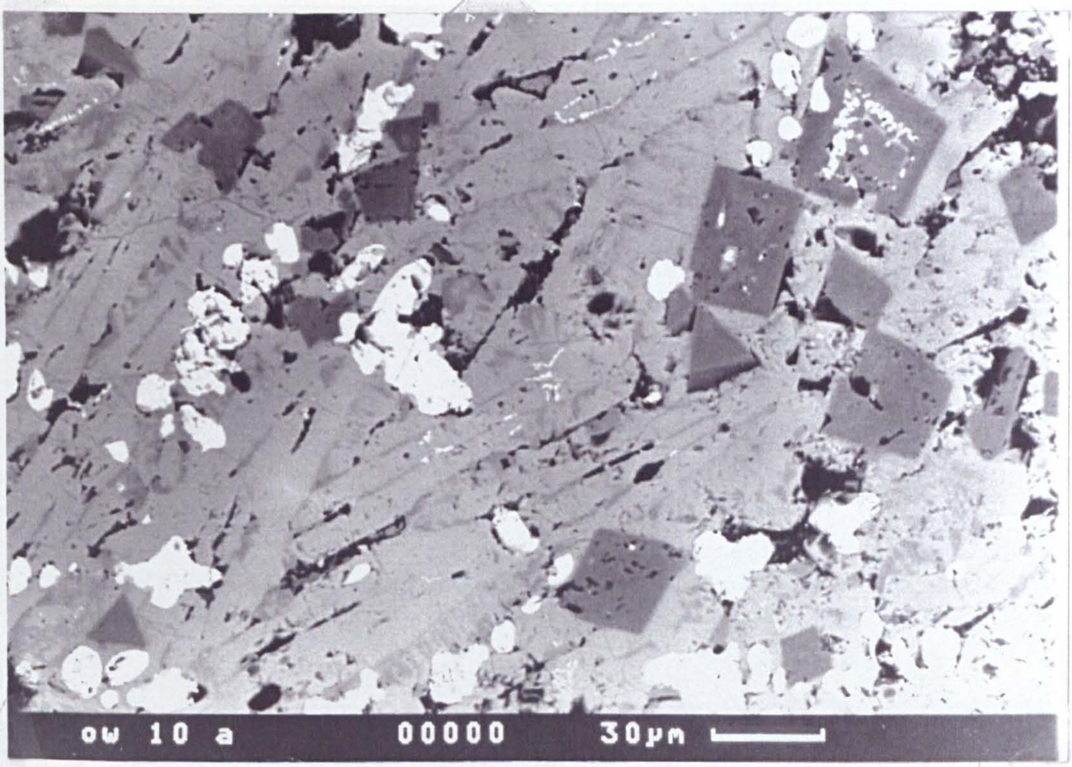


Plate 5:9 White thick/rounded dendrites of wustite as isolated globules in grey lath fayalite crystals associated with dark grey angular hercynite crystals in glass matrix.

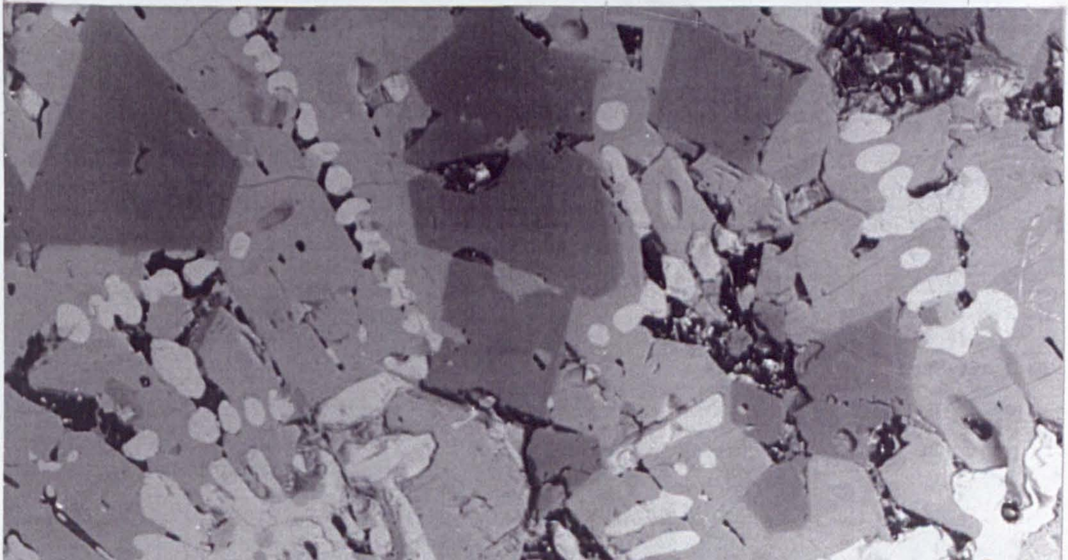


Plate 5:10 White elongated wustite dendrites in light grey massive fayalite crystals associated with bold dark grey euhedral hercynite crystals and pools of dark glass.

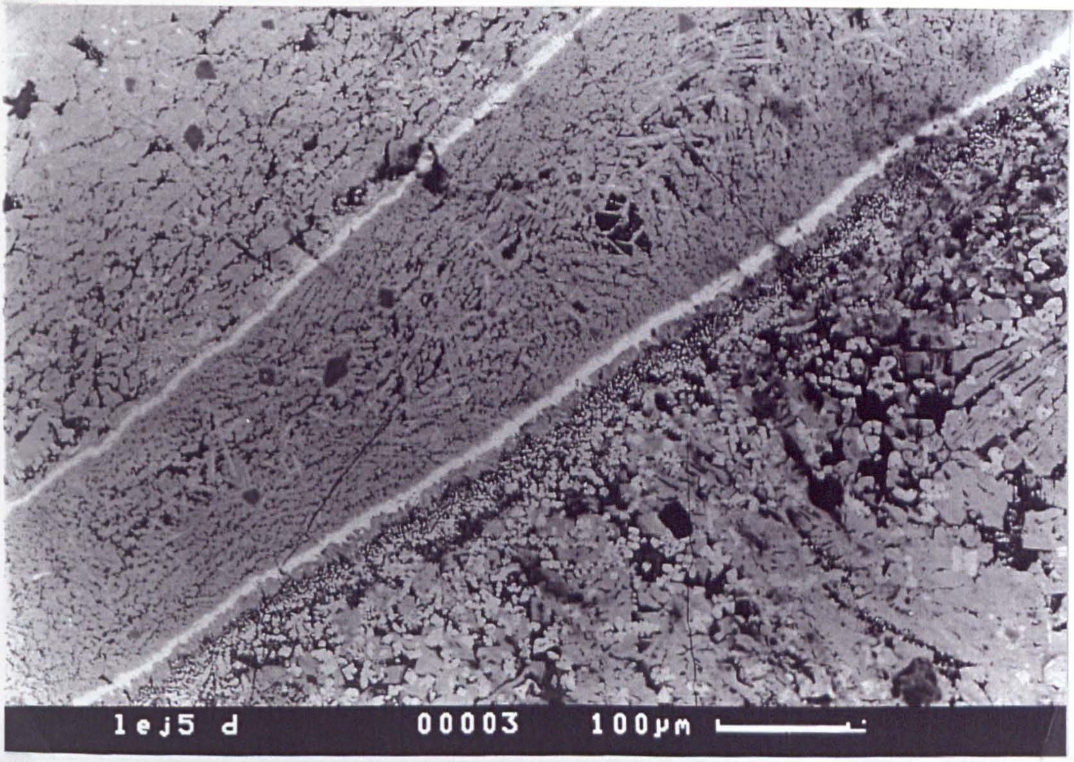


Plate 5:11 White bands of magnetite separating layers of tapped slags with varying mineral textures.

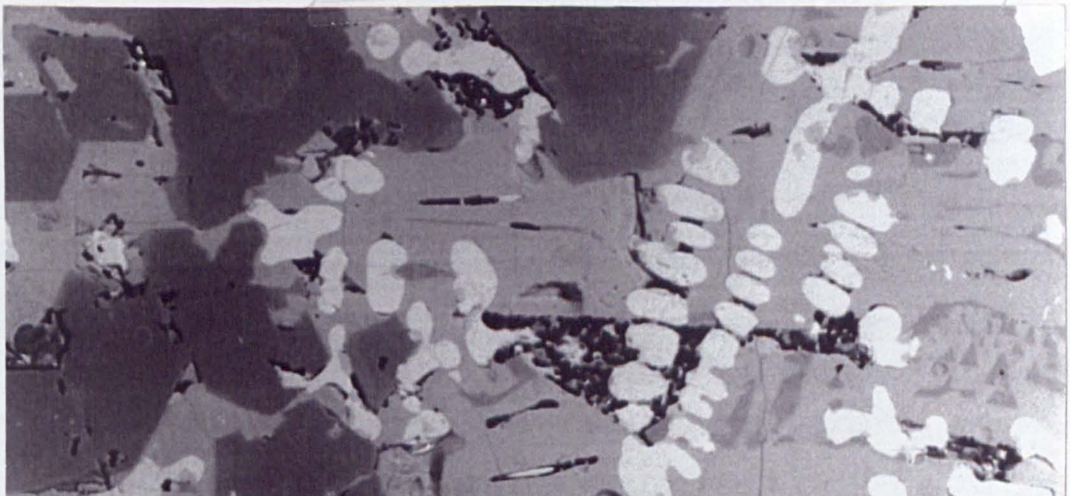


Plate 5:12 Localised dark patches of leucite in and around air holes containing white dendrites of wustite. These associate with light grey massive fayalite and bold euhedral dark grey hercynite crystal.

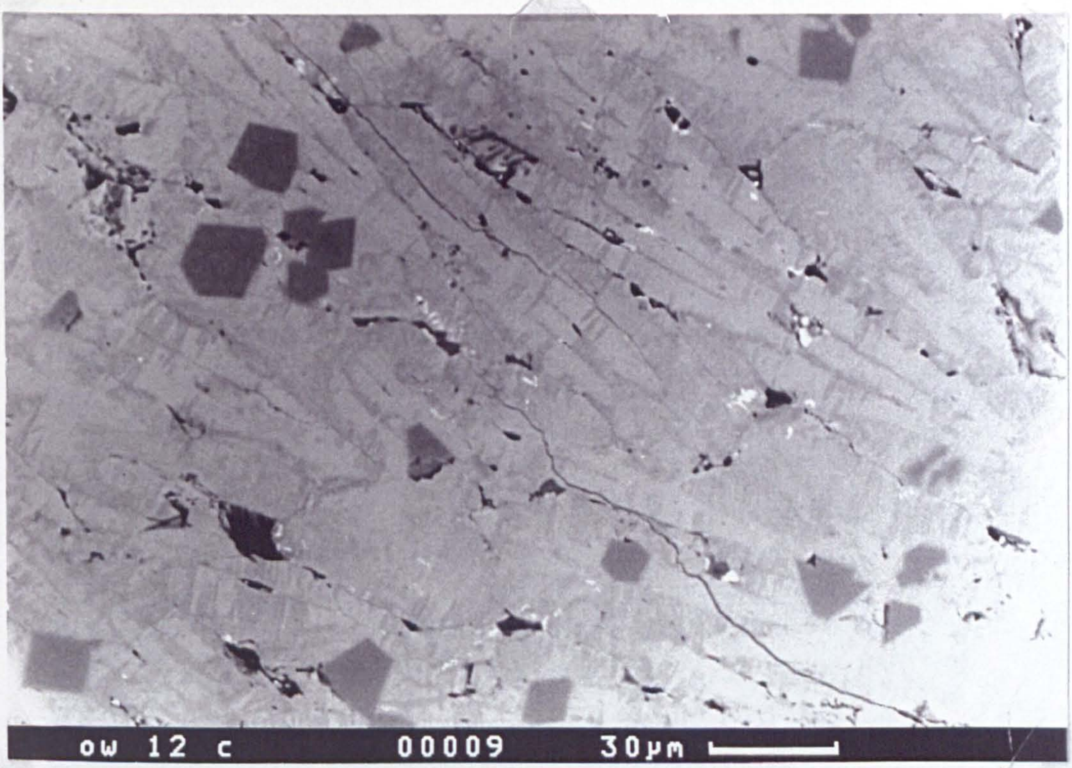


Plate 5:13 Lath grey fayalite crystals with angular dark grey hercynite crystals and few pools of glass.

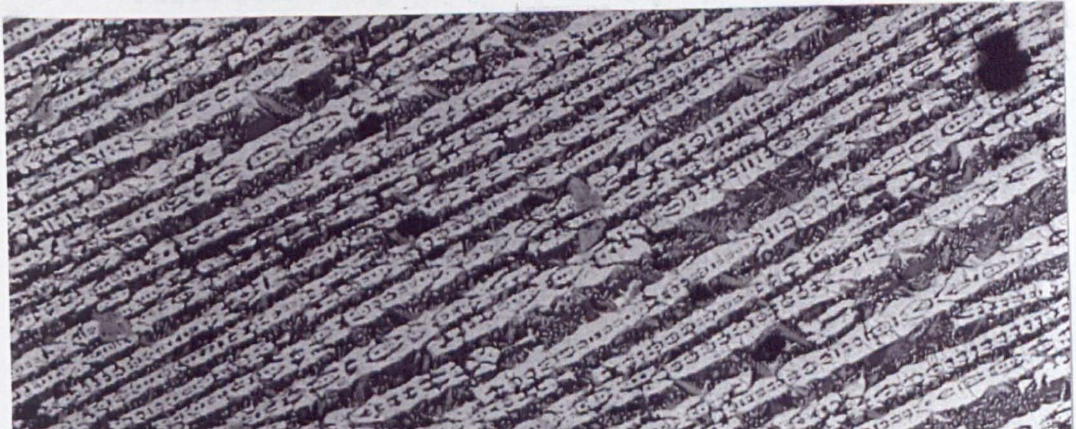


Plate 5:14 Dark grey anhedral crystals of hercynite in light grey bundles of lath fayalite crystals and dark glass matrix.

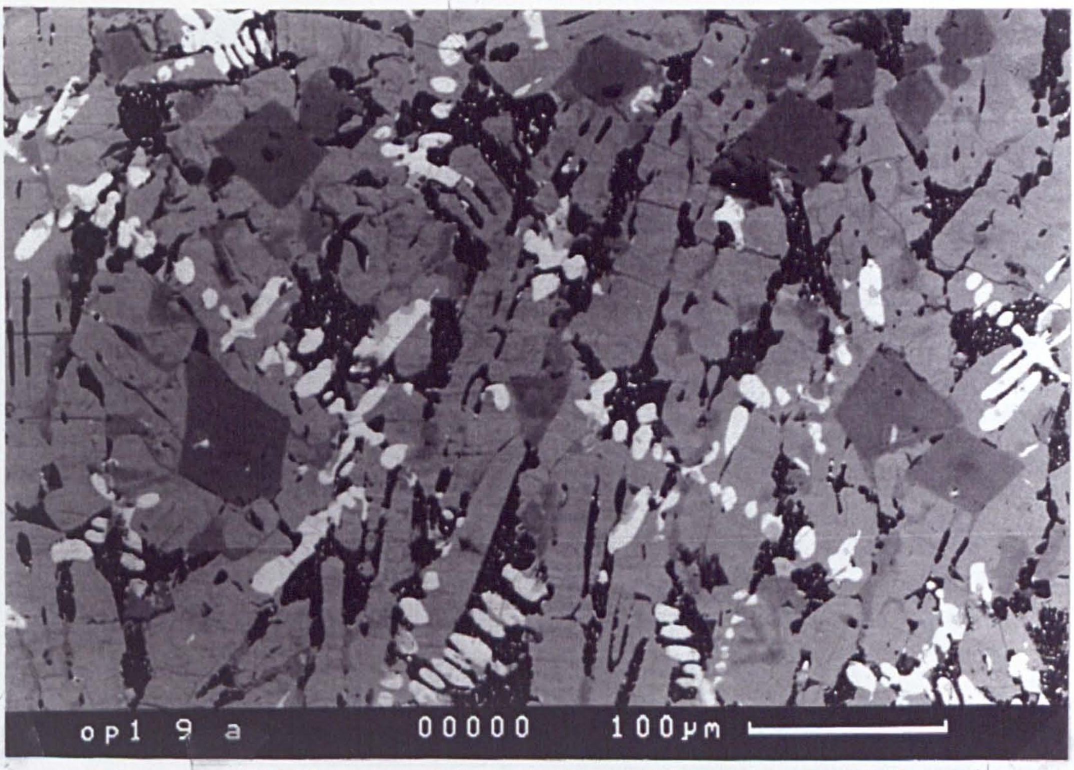


Plate 5:15 Dark grey angular and dispersed hercynite crystals formed in massive grey fayalite associated with white dendrites of wustite and dark pools of leucite.

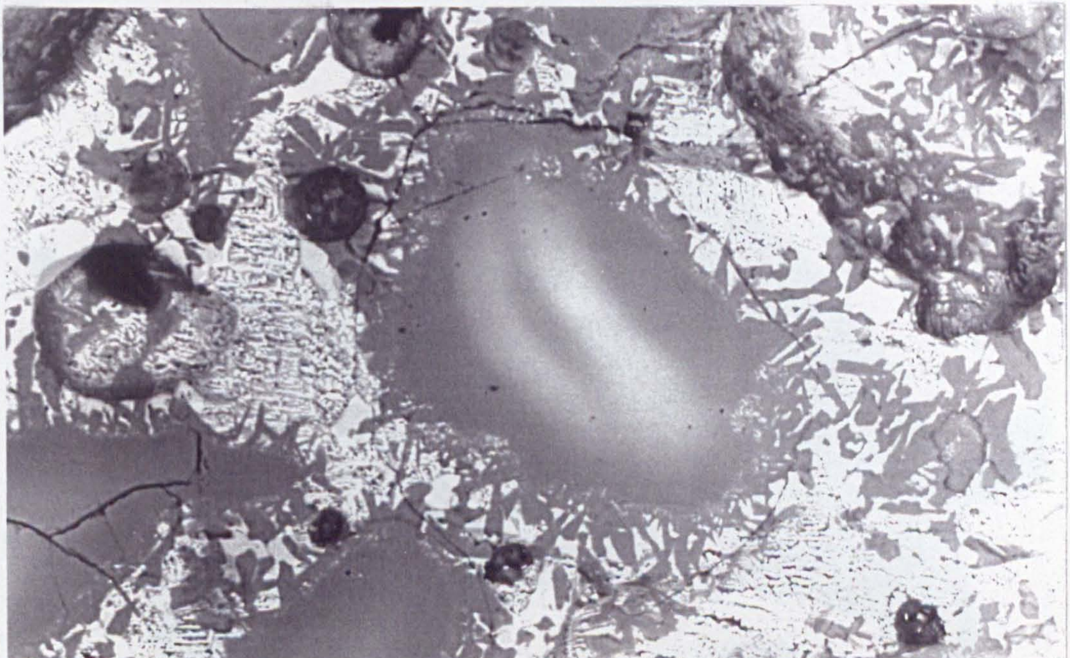


Plate 5:16 Dark grey cracked and bloated quartz crystals and light grey slag phases impregnating tuyere fabric.

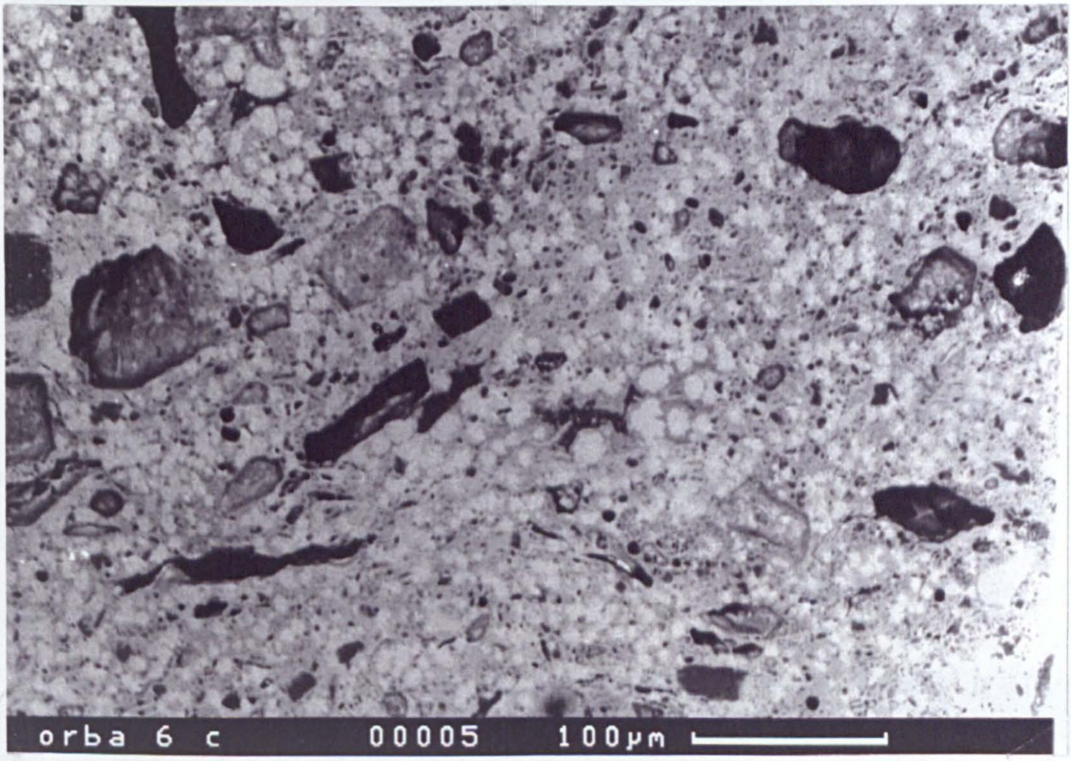


Plate 5:17 Dark grey quartz crystals in light in colour iron oxide matrix associated with dark grey clay minerals and dark holes.

Plate 5:18 Slag-inclusions in bloomery iron samples consisting of white isolated globules of wustite in grey glass matrix. and fayalite.

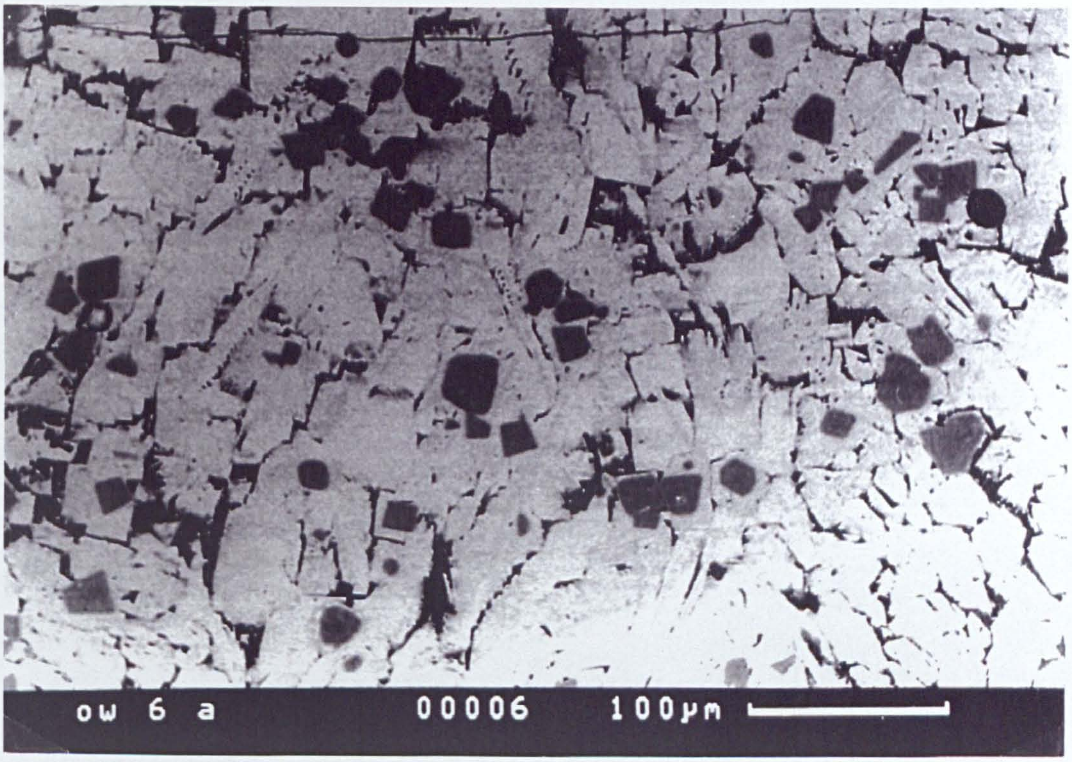


Plate 6:1 Grey broken lath fayalite crystals associated with dark grey angular hercynite crystals in dark glass matrix.

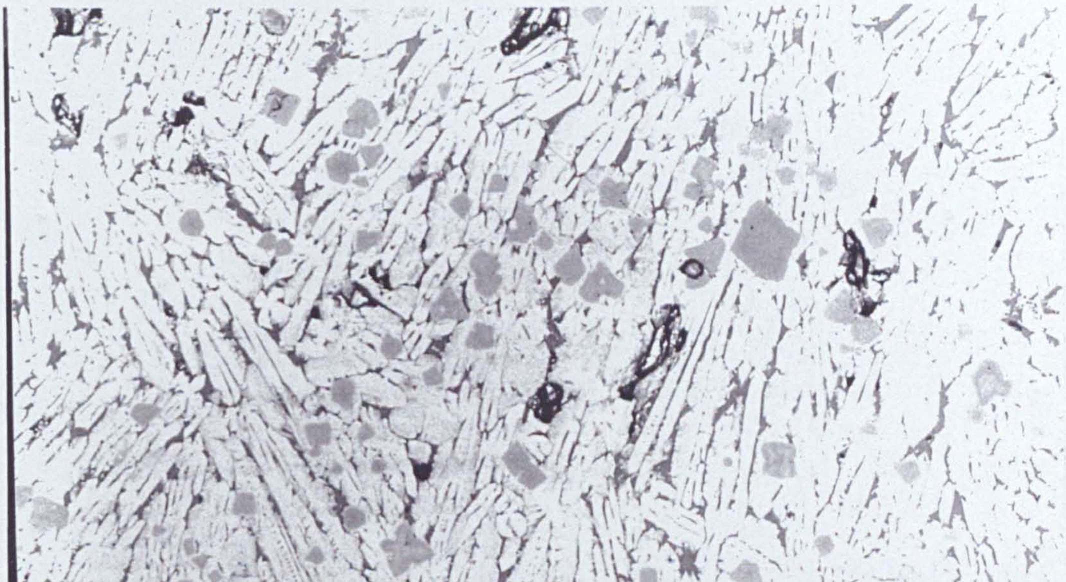


Plate 6:2 Parallel and sub-parallel grey lath fayalite crystals containing dark grey euhedral hercynite crystals in dark glass matrix.