Magnetic Particles Study and Their Applications

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Abstract

Magnetic fluids have been developed for decades and show lots of advantages in different areas of applications. This project was focusing mainly on two popular magnetic fluids: 1) the Ferrofluid (FF) which can be used in vacuum sealing and medical purpose, etc. 2) the Magnetorheological (MR) fluid which has the capability of changing material properties by applying an external magnetic field, which can be used to replace hydraulic oil in traditional shock absorber with a more advanced absorber or seismic damper.

During this project, I had successfully fabricated the magnetic nano particles, the Ferrofluid and the MR fluid. In order to study the reasons causing the change of yield stress and the reaction conditions effect on the final particle products and also the factors effect on particle settling speed in the MR fluid, we designed and carried out several experiments by using crashed glass, lithium grease as additives, changing particle ratio, controlling different reaction conditions and other methods to achieve the goals. The measurements included Scanning Electron Microscope, Energy Dispersive X-ray analysis, Vibrating Sample Magnetometer, yield stress tests and particle settling tests had carried out to study the properties of magnetic fluids. The results of the experiments showed additives like crashed glass cannot have the same effect as carbonyl iron powder does in the MR fluid, and the concentration of reagents and reaction temperature will significantly affect the produce of magnetic nano particles. Also the 3D graph of a self-designed MR fluid based car shock absorber had been plotted by Autodesk software.

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Declaration:

This thesis is a result of all author's research work regarding to the topic.

The author declare that this work does not infringe upon anyone’s copyright nor violate any proprietary rights and that any ideas, techniques, quotations, or any other material from the work of other people included in this thesis, published or otherwise, are fully acknowledged in accordance with the standard referencing practices, and this work has not previously been submitted for any other degree.

1. Research Objectives

The purpose of research was to prepare the magnetic liquids including ferrofluid and Magnetorheological (MR) fluid, study their properties and explore their potential applications. The primary objectives of this work were as the following:

- Prepare the nano size magnetic particles, do the particles coating with surfactant, and produce water based ferrofluid.

Try the most common used method, the co-precipitation method to prepare magnetic nano particles by using clean room techniques and facilities.

- By changing different reaction conditions to study their influences on the final particle products.

In order to obtain the good quality magnetic nano particles, this is an important process to get the advantage recipe of preparing particles.

- Synthesis MR (Magneto) fluid by mixing carbonyl iron powder (CIP) with oil.

- By changing CIP concentration in the fluid and adding different additives to study the reasons causing the change of yield stress.

The results obtained from this were to form the basis which would prove whether the MR fluid is good enough to be part of a car suspension system.

- Particle settling tests of MR fluids by self-designed instruments.

A designed instrument will be introduced in this section to monitor the settling of CIP in the MR fluid, this result will help us improve the stability of MR fluid.

- Get familiar with measurement tools like SEM, EDX, VSM, and the procedures doing experiments in clean room.

The measurements help us to require the particles size, which is an important aspect to be considered while making a MR fluid, and elemental analysis by using an Energy Dispersive X-ray spectroscopy will give us vital information about the impurities in the samples, also the Vibrating Sample Magnetometer will help in calculations of important properties such as coercivity and plotting hysteresis loops.

- Understand the principle of car shock absorber and modified it from traditional hydraulic oil shock absorber to MR fluid shock absorber, give the designed model or blueprint.

- Further work on combination of the carbon nanotubes with nano size magnetic particles.

A kind of new material named as carbon nanotubes (CNT) shows lots of special characteristics, especially on electrical and mechanical properties. We want to combine CNT and magnetic nano particles together to see if there are property changes of magnetic nano particles.

2. Introductions

The technological advances made in the last few years have greatly increased the demands for magnetic materials. Among which the magnetic fluids are smart liquid materials whose properties can be manipulated by applying a magnetic field. When we subject the fluids to an external field, the surface viscosity of the fluids will change according to the strength of the field and result a reformation of the liquids. And when we withdraw the external magnetic field, the reformation relaxes and the fluids return back to its original state. The properties of smart fluid have been known for more than sixty years and subjected to sporadic research up until the 1990s. When an MR fluid based suspension system model was used in 2002, it boosted the market and drew many research interests [1].

Typical magnetic fluid consists of three main elements: magnetic particles, carrier liquid and surfactant. The quality of each determines the performance of the final product. The technological advances made in the last few years have greatly increased the quality of magnetic fluids and variously enhanced properties to serve the requirements of brakes, seismic dampers [2], human prosthesis [3], etc.

The whole family of magnetic fluid covers a vast number of different types. The two most popular magnetic fluids that have been widely used commercially are ferrofluid (FF) and Magneto-rheological fluid (MR fluid). Another smart fluid, the Electrorheological fluid (ER fluid) which has almost the same function as MR fluid will be detailed introduced in the following chapters.

2.1 Ferrofluid (FF)

In the 1960's, the scientists of NASA tried to explore how to control various liquid in outer space, then the solution in the form of a new idea of magnetic fluid emerged [4]. Generally, the ferrofluid is a colloidal solution. In the solution are the single domain magnetic nano particles. The particles are dispersed stably and uniformly in a carried liquid. Those nano particles are thoroughly coated with surfactants, the ferrofluid will not coalesced or precipitated under the influence of magnetic force, gravity field, even the centrifugal force [5].

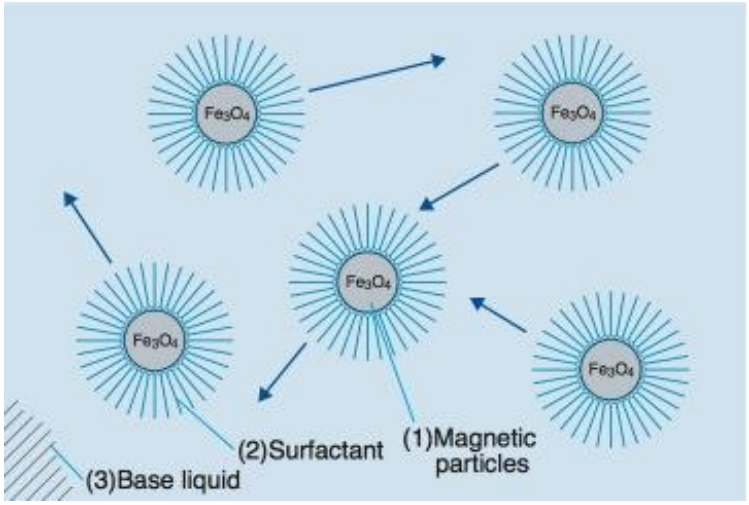

Figure 1: The Typical Ferrofluid

Image courtesy: Eagle Industry Liquid Seal Brochure

Figure 1 illustrated the basic composition of ferrofluid. Typically it is 5% magnetic solids, 10% surfactant and 85% carrier, by volume [6][7]. The base/carrier liquid is usually organic solvent or simply water. The surfactant is the compound that lowers the surface tension of liquid, ensuring that the particles do not form aggregates that become too heavy to be held in suspension by Brownian motion. Surfactant is very useful in prolonging the settling speed in ferrofluid, the Vander Waals force exists in surfactant is sufficient enough to prevent particles from settling. Some commonly used surfactants are oleic acid and aerosol sodium, di-2 ethylhexyl-sulfosuccinate.

High-quality ferrofluid is very stable. The particles in the fluids always stay separated and never agglomerate even in extremely strong magnetic fields or other external force. However, because the effect of surfactant tends to wear off over

time and eventually the nano particles will get clumped and no longer contribute to the fluid’s magnetic response.

2.1.1 Application of Ferrofluid

Ferrofluid has a wide application area. In the biomedical area, ferrofluid coated medication injection is an innovating method of delivering drugs specifically to certain tissues. The fluid can be modified to enhance its response to external field for precise delivery; and cause no reaction to the rest of human body [8]. After the treatment, the fluid coating will disperse and leave no remaining in the system [9]. For MRI imaging techniques, ferrofluid is usually implemented as a contrast enhancement agent, as the image was generated according to the magnetic relaxation times of tissue structures. Other application such as magnetic hyperthermia is also accepted more and more to help people living a better life [10].

In mechanical area, ferrofluid is famous of being used in sealing applications. The ferrofluid is suspended in place by a strong magnet, the magnetic liquid seal operate with extraordinary reliability and extremely low leakage in a very wide range of mediums, and it is the most often packaged in rotary feedthrough. As illustrated in Figure 2 and Figure 3, the ferrofluid acts as multiple magnetic O-rings around the rotary shaft, it is the best solution of offering seal and rotation at the same time to date [11].

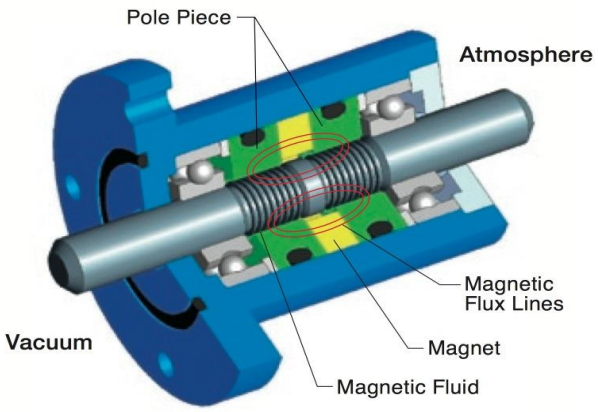

Figure 2: Ferrofluid Liquid Seal

Image courtesy of: Eagle Industry Liquid Seal Brochure

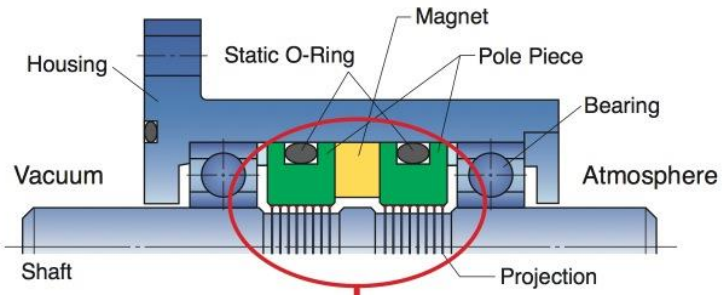

Figure 3: Details of Ferrofluid seal

Image courtesy of: Eagle Industry Liquid Seal Brochure

Ferrofluid also has numerous optical applications because of its refractive properties. These applications usually require viscosity measurements of the liquid and a set of laser generator with polarisers and analysers illuminated by a laser [12].

2.2 Magneto Rheological Fluid (MR Fluid)

A Magnetorheological fluid, also known as Bingham magnetic fluid, is a kind of materials of which the viscosity can be reversibly adjusted by the magnetic field. The first development of Magnetorheological fluid (MR Fluid) was credited to Jacob Rainbow in 1949 at the US national Bureau of Standards [13]. The MR fluid responses to an applied field and is capable of changing from a linear viscous fluid to a semi-solid state fluid.

Generally MR Fluid is a smart material that exhibit fast, reversible and tunable transition from a free-flowing state to a semi-solid state. The transition usually takes place in less than 10 milliseconds [14]. This property makes MR fluid a good candidate for applications in mechanical systems that require active control of vibrations or the transmission of torque. The conventional MR Fluid are two-phase fluids prepared by dispersing large amounts of solid, micro-scaled magnetisable particles in a base carrier liquid, additives are also necessarily added to inhibit sedimentation or aggregation and at the same time provide lubrication [15]. The most commonly used magnetic particle is iron powder obtained from the thermal decomposition of iron pentacarbonyl. Figure 4, Figure 5 shows the different appearances of MR fluid when subjected under an external magnetic field.

Figure 4: MR Fluid without the influence of magnetic field

Image courtesy of:

http://appsvr.cintec.cuhk.edu.hk/exhibition/project-quicklook.php?pid=191

Figure 5: MR fluid exposed to a magnet at the bottom of a beaker

Image courtesy of:

http://www.appsvr.cintec.cuhk.edu.hk/exhibition/project-quicklook.php?pid=191

The cause of this phenomenon happened is because when there is no magnetic field applied, the micro scale spherical particles are suspended within the carrier liquid and distributed evenly. When applied a magnetic field, the particles align themselves along the lines of magnetic flux and thickening the fluid dramatically, which cause the spikes.

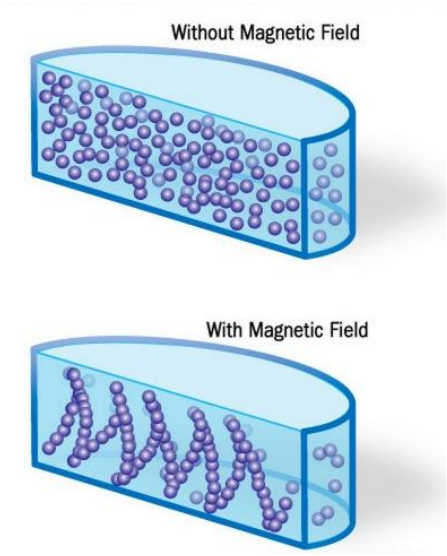

Figure 6: with and without the influence of magnetic field

Image courtesy of: http://science.howstuffworks.com/liquid-body-armor2.htm

2.2.1 Working Mode of MR fluid

MR Fluid has three main operation modes. The first is flow mode or valve mode: fluid flowing as a result of pressure gradient between two stationary plates. Second is shear mode: fluid between two plates moving relative to each other. And the last is squeeze mode: fluid between two plates moving in the direction perpendicular to their planes. In all cases, the magnetic field is applied perpendicularly to the planes of the plates, in order to restrict fluid in the direction parallel to the plates. Each different operation mode has numerous applications, depending on the requirements of the situation [16].

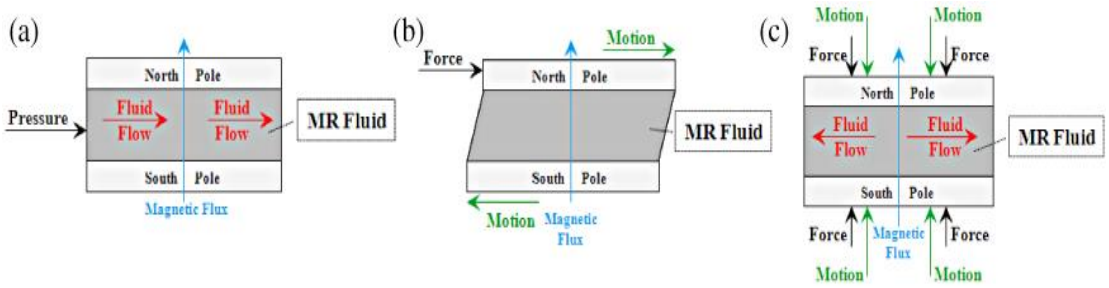

Figure 7: Working mode of MR Fluid

Image courtesy of: http://iopscience.iop.org/0964-1726/22/1/015012/article

2.2.2 Differences between Ferro Fluid and Magnetorheological Fluid

An effective way to distinguish MR fluid and Ferro fluid is to compare the size of the magnetic particles. MR fluid usually has its particle sizes vary at a few microns while nano-scale particles are used to make Ferro fluid. This difference determines the effect of Brownian motion within the fluid, and resulting only MR fluid to express yield stress under an external field.

2.2.3 Applications of MR fluid

The modern auto industry considers MR fluid a replacement of traditional hydraulic oil in the shock absorbers of a vehicle's suspension systems since it offers millisecond level response time in either light and heavy duty situation [17]. Figure 8 shows the difference between cars with and without MR fluid suspension under the same road condition. As we can see from the following figures, the burgundy car with a MR suspension system will react to a bumpy road remarkably, but the yellow car is bounced up by the road.

Figure 8: Comparison with two different suspension system

Image from internet

This technology also extends to the seismic dampers of buildings. Material scientists, mechanical and civil engineers are collaborating to develop stand-alone seismic dampers located at specific positions of buildings and bridges to avoid being damaged in earthquake [18].

Figure 9: Damper been used in civil engineering to protect the structure from earthquake

Image courtesy of: Seismic strengthening projects, Residence seismic renovation, Seattle, WA, 2002

Another important application of MR Fluid is human prosthesis. Small scale MR Fluid dampers are utilized in semi-active prosthetic legs, much like those used in military and commercial helicopters. MR fluid offers adjustable motions of prosthesis and allows the patients to move comfortably and willingly [19].

2.2.4 Current Problems and Future of MR Fluid

Since typical magnetic particles employed in MR fluid are in the range of micro meters, they are easily magnetized and oxidized for the main composition of MR fluid is iron powder. "This is contrarily to ferrofluid consists of nano particles which show a permanent magnetic moment as a consequence of their magnetic mono-domain character where the Neel relaxation mechanism dominates." [20]. The price of MR fluid is also expensive due to the sophisticated manufacturing process, which in a way slows down the popularization process.

The research of mono dispersed systems with typical sizes in between these two extremes (easily magnetized and oxidesed) has not been done yet in details mainly due to the absence of appropriate chemical approaches to prepare kinetically stable and mono dispersed magnetic colloids (Prof. Daniel J.Klingenberg, 2011) [21], But by using chemical precipitation techniques and polyol processes, we

can prepare these particles suitable for MR fluid according to M.Andres Verges and R.Costo in 2008 [22].

2.3 Electrorheological fluid

In 1947, the American inventor Willis Winslow produced suspended fine particles sized around 0.1-100 micro meters in an electrically insulating fluid [23]. The apparent viscosity of the fluids changes reversibly in response to an electric field, and he named it Electrorheological fluid (ER Fluid). The physical appearance of the fluid can go from the consistency of a liquid to that of a gel, and vice versa. The response time is in the order of milliseconds, and this effect is called the Winslow Effect [24].

The change in apparent viscosity is dependent on the applied electric field. When subjected to an electric field, ER fluid behaves as Bingham plastic (a viscoplastic material that is rigid under low pressure but liquid under high pressure). The yield point is determined by the electric field strength, after reach this critical point, the ER fluid will shears as a fluid.

The particles used in ER fluid are electrical field responsive because they are made of conducting material coated with an electro-osmotic insulator. All the particles in the fluid align themselves along the field. This initiated formation of particle chains that bridge the electrodes. Since opposite poles attract each other, all the positive particles in the fluid get attracted to the negative electrode of the field and the same way around. The formation results in the ER fluid becoming semi-solid, similar to MR fluid when subjected with a magnetic field.

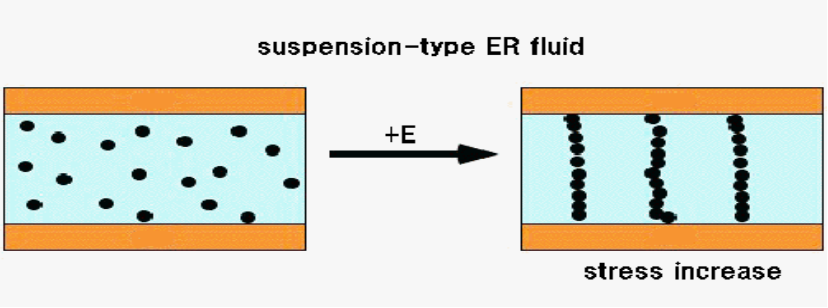

Figure 10: Two conditions of ER fluid, before and after an Electric field been applied

Image courtesy of:http://ckpmac7.yz.yamagata-u.ac.jp/E-Page/Project/research/e-rheology/electrorheo_E.htm

2.3.1 Applications of ER fluid

ER fluid can be used in brakes and shock absorber similar to what MR fluid is used for. In addition, hydraulic valves and clutches based on ER fluid are commonly seen. The difference to MR fluid is mainly reflected by the energy consumption and MR fluid offers greater efficiency on [25].

2.3.2 Current Problems and Future

ER device can control considerably more mechanical power than the electrical power used to control the effect. The main advantage is the instant switch of electric field, but it consumes more energy compared to MR fluid.

Particle suspension is a common problem shared by applications based on either of the fluid. Over time, the particle would settle and cause the fluid to lose its homogeneity. Devices would then require maintenance to replace the used fluid.

Research into Electrorheological fluid is stuck at a point where both theoretical and applied approaches are facing great challenges like huge energy consumption. With extensive research and investment, it is hopeful of receiving break through progresses in the near future.

2.4 Comparison of Three Smart Fluids

	Ferro fluid	MR fluid	ER fluid
Particles	Magnetite, Iron, ferrites, etc.	Carbonyl iron powder, cobalt, nickel, etc.	Zeolites, Polymers, SiO2, BaTiO3
Particle Size	2-10nm	1-10μm	1-10μm
Carrier Liquid	Oil based, water based, etc.	Oils, Polar Liquid, water	Oils
Density (g/cc)	1-2	3-10	1-3
Required Field	Magnetic Field	Magnetic Field	Electric Field
Operating Temperature	-40 to 150℃	-40 to 150℃	-10 to 125℃

Table 1: Comparison of Three Smart Fluids

The above table listed the major difference among three smart fluids, comparing them from particle sizes, density and operational temperatures. [26] This only covers the common applications of each, sometimes if we want to use it in the extremely environment like high temperature or low temperature and vacuum or dust environment, then changes would be made to fit those specific requirements.

3. Theoretical Knowledge

This chapter introduces the background theories including magnetic domains, characterization of magnetism, the commonly used mathematical equations, surfactant, rheology, circuits, and measurement instrumentations during the work.

3.1 Magnetic Domain

A magnetic domain is a region within a magnetic material that has uniform magnetization. Each individual magnetic moment of the atoms is aligned with one another and pointing to the same direction. While the neighboring domains have different aligning direction, resulting the bulk material expressing a neutral net magnetic state.

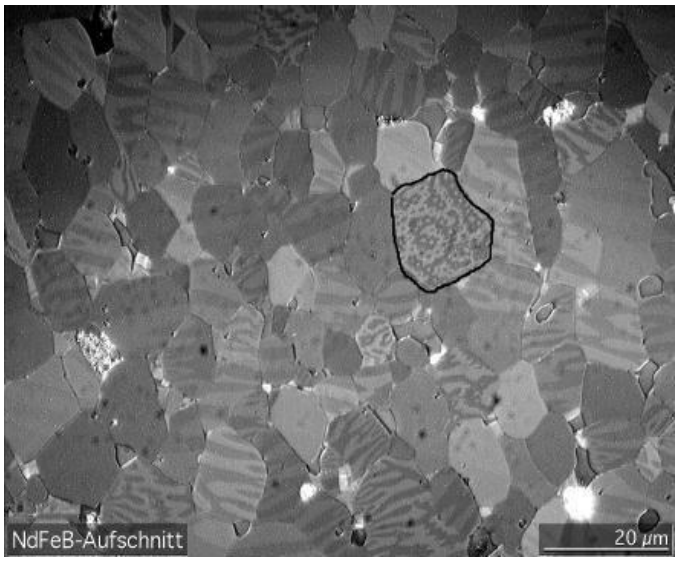

Figure 11: NdFeB domains under a Kerr microscope, the domains are the light and dark stripes visible within each grain.

Image courtesy of: Wikipedia-Magnetic Domain

3.2 Magnetism

Thales of Miletus is the first to describe the classic physical phenomena as magnetism [27]. It is the fundamental magnetic moments of elementary particles which give rise to a magnetic field that acts on other currents and moments. All materials will be influenced by a magnetic field to some extent. There is a variety of magnetism materials mainly can be distinguished as four kinds.

3.2.1 Ferromagnetism

Ferromagnetism is the basic mechanism by a certain material such as iron forms permanent magnets. Ferromagnetism is the strongest type of magnetism, and it is the only one that creates forces strong enough to be felt. A common example of ferromagnetism is compass which has been used as a navigator for sailing. Materials that can be magnetized by an external magnetic field and remain magnetized after the external field is removed is either ferromagnetic or ferrimagnetic. The most common known examples are iron, nickel, cobalt and several rare earth minerals. Ferromagnetism is very important in industry and modern technology, and is the basis for many electrical and electromechanical devices as generators, transformers, and magnetic storage such as tape recorders and hard disks.

Figure 12: Magnetic moment of Ferromagnetism

Image courtesy of:

http://www.physics.tutorvista.com/electricity-and-magnetism/properties-of-magnets.html

It can be seen from Figure 12 that the magnetic moments in a ferromagnetic material become aligned parallel to each other when magnetic field is applied and they retain their property even after removal of the applied field.

3.2.2 Diamagnetism

Diamagnets were first discovered when Sebald Justinus Brugmans observed in 1778 that bismuth and antimony were repelled by magnetic fields, since diamagnetism to a greater or lesser degree is a property of all materials and always makes a weak contribution to the material's response to a magnetic field. Diamagnetic materials create a magnetic field in opposition to an externally applied magnetic field. In other words, unlike the way a magnet attracts metals like iron, it would repeal a diamagnetic substance. Some commonly known diamagnetic materials are superconductor, Bismuth, Mercury, Silver, lead, etc.

Properties of Diamagnetic Materials are:

1. The magnetic moment and the applied magnetic field are repelled in a Diamagnetic Material.
2. Under a uniform magnetic field, the longest axis of a diamagnetic material is present at right angles to the direction of the applied field while the shortest axis is along the direction of the field.
3. Under a non-uniform magnetic field, the diamagnetic materials show movement from stronger parts of the field towards the weaker parts.

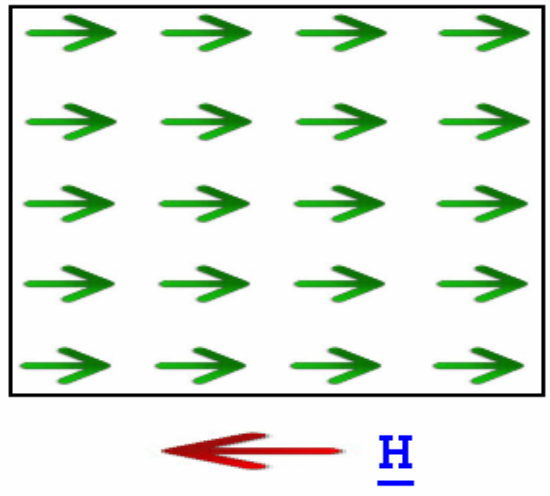

Figure 13: Magnetic moment of Diamagnetism

Image courtesy of:

http://www.physics.tutorvista.com/electricity-and-magnetism/properties-of-magnets.html

We can see Figure 13 that here the magnetic moment will be in the direction opposite to the applied field, when magnetic field is removed these materials do not retain their magnetic properties.

3.2.3 Paramagnetism

Paramagnetism is kind of magnetism that materials are attracted by an external applied magnetic field. As mentioned above, paramagnetism is different with diamagnetism, actually it is in contrast with diamagnets' behavior which is repelled by magnetic fields. These materials have very little susceptibility towards the applied magnetic field and these materials do not retain the magnetic properties when the magnetic field is removed.

Paramagnetic materials include most chemical elements and some compounds such as aluminum, sodium, calcium, oxygen and copper chloride. These substances get weakly magnetized when placed in an external magnetic field. They have tendency to move from a region of weak magnetic field to strong magnetic field.

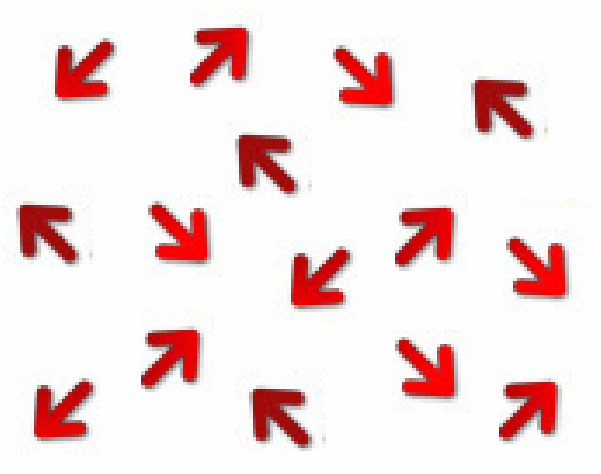

Figure 14: Magnetic moment of Paramagnetism

Image courtesy of:

http://www.physics.tutorvista.com/electricity-and-magnetism/properties-of-magnets.html

As we can see from Figure 14, the Magnetic moments tend to be orientated randomly when no magnetic field is applied. When there is a magnetic field the magnetic moments start to align parallel to the field that makes magnetization of the material proportional to the applied field.

3.2.4 Super-Paramagnetism

This is a kind of special paramagnetism that appears in small (nano scale) ferromagnetic or ferrimagnetic particles. In some materials magnetic moments change their direction behave like a paramagnet even the temperature is below curie temperature, when there is no magnetic field applied and at the same time they show high magnetic susceptibility like a ferromagnet. And the Magnetization is the extent at which a substance gets magnetized and magnetic susceptibility is how much magnetization of a material takes place with respect to an applied magnetic field.

We can consider some ferromagnetic or ferrimagnetic material if it has zero magnetization, and no magnetic field is applied below Curie temperature, we could call it as paramagnetic substance, but the some paramagnetic materials show high magnetic susceptibilities at nano scale. So these materials may also be called as Ferromagnetic materials. This paramagnetic behavior is observed only at nano scale.

3.3 Hysteresis Loop

When a material is magnetized and saturated, it will not relax back to zero magnetization when the imposed magnetizing field is removed. A reversed field would be required to neutralise the magnetization. If an alternating magnetic field is applied to the material, its magnetization process will form a loop, which is called hysteresis loop. Hysteresis loop shows the relationship between the induced magnetic flux density (B) and the magnetizing force (H). It is often referred to as the B-H loop. The following picture is an example of classic hysteresis loop.

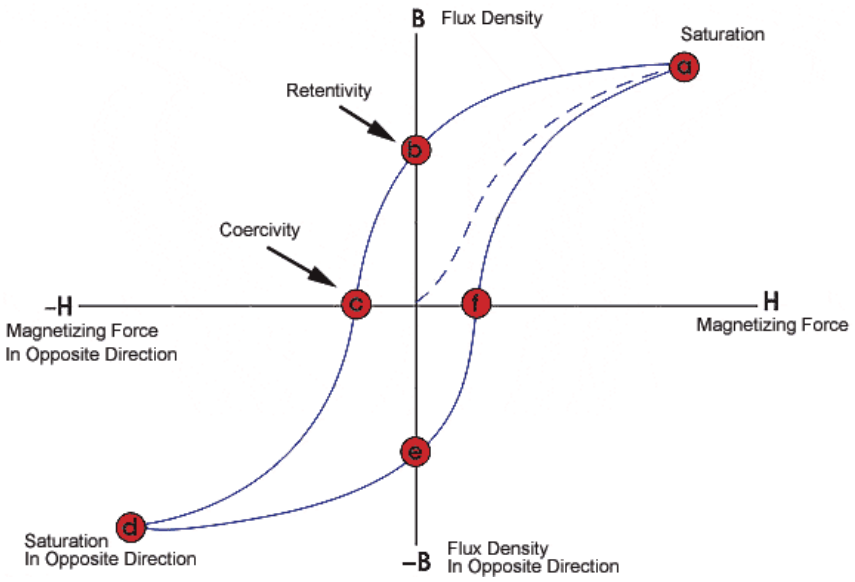

Figure 15: Hysteresis loop

Image courtesy of:

http://www.ndt-ed.org/EducationResources/CommunityCollege/MagParticle/Physics/HysteresisLoop.htm

As we can see from the Figure 15, there are several important points of the hysteresis loop that illustrate the properties of a magnetic material.

Saturation: At this particular point, the material has become completely magnetized.

Retentivity: Means residual flux density corresponding to the induction of a magnetic material’s saturation, it is a material's ability to retain a certain amount of residual magnetic field when the magnetizing force is removed.

Coercivity: Reverse magnetic field strength needed for a magnetic material to make the magnetic flux return to zero.

A hysteresis loop can tell us the basic information about the magnetic property of a magnetic material. Usually it is measured using VSM (Vibrating Sample Magnetometer) or MOKE (Magneto-Optical Kerr Effect)

3.4 Mathematical Formulas of Magnetism Study

The next section defines and explains mathematical terms related to the research.

3.4.1 Magnetic Flux Density

$$B = \mu H \qquad (3.1)$$

μ is a material dependent parameter called the permeability. In some cases the permeability may be a second rank tensor so that H may not point in the same direction as B (Tesla). In a vacuum, the permeability is a constant, $\mu_0 = 4\pi \times 10^{-7} \mathrm{N/A^{-2}}$.

3.4.2 Ampere's Law

Ampere is also known as the right-hand screw rule which is used to describe the relation between current and the magnetic field direction of field created by the current. James Clerk Maxwell derived it, and it is now one of the Maxwell Equations.

The integral form of Ampere's Law is:

$$\oint H \cdot dl = \mu_0 \iint J \cdot ds = \mu_0 I_{enclosed} \qquad (3.2)$$

Where J is the total current density, unit is ampere per square meter, $\mathrm{Am^{-2}}$, $I_{enclosed}$ is the current of enclosed wire, μ_0 is the magnetic constant.

The Law can be interpreted as the closed line integral around the closed curve, dl is an infinitesimal of the curve which equals to a 2 dimension surface integral over surface, ds is the vector area of an infinitesimal of surface S.

The differential form is:

$$\nabla \times B = \mu J \qquad (3.3)$$

If a current through a wire a magnetic field is produced which circles the wire and we sum all of the points around the magnetic field, and they will add up to the

total current passing through the wire, for the radius of the circulating magnetic field will be symmetrical we can also infer that the magnitude of the magnetic field is:

$$H = \frac{I_{enclosed}}{2\pi r} \qquad (3.4)$$

Where r is the radius of the circulating magnetic field.

3.4.3 Faradays Law

Faraday equation is one of the four Maxwell's equations which play a fundamental role in the theory of classical electromagnetism, its integral form is:

$$\oint_{\sum} \mathrm{E} \cdot \mathrm{dl} = -\oint_{\sum} \frac{\partial \mathrm{B}}{\partial \mathrm{t}} \cdot \mathrm{dA} \qquad (3.5)$$

$\sum$ is the surface bounded by the closed contour $\partial\sum$, E is the electric field, B is the magnetic field, dl is an infinitesimal vector element of the contour $\partial\sum$, and dA is an infinitesimal vector element of surface $\sum$.

The differential form is:

$$\nabla \times \mathrm{E} = -\frac{\partial \mathrm{B}}{\partial \mathrm{t}} \qquad (3.6)$$

From faradays law we can know an electric current gives rise to a magnetic field, and the magnetic fields around a circuit will give rise to an electrical current.

3.4.4 Biot-Savart Law

This is an equation describing the magnetic field generated by an electric current which relates the magnetic field to the magnitude, direction, length and electric current. The equation is:

$$\mathrm{B} = \frac{\mu_0}{4\pi} \int_{\mathrm{C}} \frac{\mathrm{Idl} \times \mathrm{r}}{|\mathrm{r}|^3} \qquad (3.7)$$

Describe a resultant magnetic field B at position r generated by a steady current I, a continual flow of charges which is constant in time and the charge neither accumulates nor depletes at any point. The law is a physical example of a line integral: evaluated over the path C the electric currents flow.

In the equation r is the full displacement vector from the wire element to the point at which the field is being computed, dl is a vector whose magnitude is the length of the differential element of the wire, in the direction of conventional current, and μ0is the magnetic constant.

3.4.5 Electric Flux Density

Electric flux is the flow rate of the electric field through a given area. Electric flux is proportional to the number of electric field lines going through a virtual surface. If the electric field is uniform, the electric flux passing through a surface of vector area S is:

$$\Phi_E = E \cdot S = ES\cos\theta \qquad (3.8)$$

E is the electric field, unit is V/m, S is the area of the surface, and θ is the angle between the electric field lines and normally perpendicular to S.

3.4.6 Electric Current Density

Current density is the electric current per unit area of cross section. It is defined as a vector and its magnitude is the electric current per cross-sectional area at a given point in space which is given by:

$$J = \lim_{n\to 0} \frac{I}{A} = \sigma E \qquad (3.9)$$

J is the electric current I (A) per unit area A (m^2), σ is known as the conductivity of the material.

3.5 Surfactant

A surfactant is defined as a material that can greatly reduce the surface tension or interfacial tension of liquids, it can also be used between a liquid and a solid. The molecular structure of the surfactant having amphiphilic, of which one end of is hydrophilic group (water love), and the other end is a hydrophobic group (water hate) [28]. Hydrophilic group is usually a polar group such as carboxylic acid, sulfonic acid, sulphuric acid; or amine group such as hydroxyl, an amide group, an ether bond and etc. Therefore, a surfactant contains both a water insoluble or oil soluble component and a water soluble component. Surfactant will diffuse in water and absorb at interfaces between air and water or at the interfaces between oil and water.

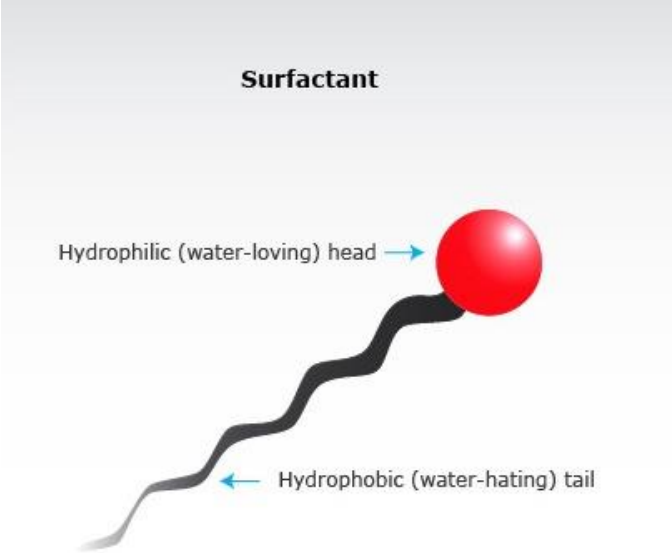

Figure 16: Structure of Surfactant

Image courtesy of:

http://www.sciencelearn.org.nz/Science-Stories/Where-Land-Meets-Sea/Sci-Media/Images/Surfactants

3.5.1 Classification of Surfactant

There are lots of methods to classify surfactants. According to its chemical structure is considered more appropriate. When the surfactant is dissolved in water, judging by whether the ion generating electrical or not, surfactant can be divided into ionic surfactants and non-ionic surfactants.

3.5.2 Surfactants Effect on the Magnetic Fluid

Particles in ferrofluid keep suspending because their Brownian (See **3.6.1**) motion and also their nano scale size will almost not be affected by the gravity force. It is though very hard to keep particles not being aggregate together over time. Because of the surfactant, we can solve this problem by coating surfactant around each nano particles (ideally) to allow them suspend well for a very long time. Later we will discuss how to perform the surfactant coating.

3.6 Rheology

Rheology is the study of the flow of matter, primarily in the liquid state or in a soft solid state in which they respond with plastic flow rather than deforming elastically in response to an applied field. It is important to understand some basic knowledge of rheology before get into further study of the characteristics of MR fluid, ferrofluid and also ER fluid.

3.6.1 Brownian Motion

Brownian motion is the random motion of particles suspended in a fluid or gas resulting from their collision with the neighboring atoms or molecules. Brownian motion is among the simplest of the continuous-time stochastic, and it is the limit of both simpler and more complicated stochastic processes [29]. Brownian motion is the reason why nano particles in the ferrofluid suspend so well in the carrier liquid.

3.6.2 Viscosity

The viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress (defined as the component of stress coplanar with a material cross section. Shear stress arises from the force vector component parallel to the cross section. Normal stress, on the other hand, arises from the force vector component perpendicular to the material cross section on which it acts) or tensile stress. In liquids, it corresponds to the informal notion of "thickness". All fluids have positive viscosity. If the viscosity is very high, for instance in pitch, the fluid will appear to be a solid in a considerably long period of time [30].

An MR fluid contains solid magnetic particles as the main constituent so that the viscosity of the fluid can be altered by an applied magnetic field, from a Newtonian fluid to a non-Newtonian fluid.

3.6.2.1 Newtonian Fluid

A Newtonian fluid is a fluid continues to flow regardless of any forces acting to it. The most common example is water. And Newtonian model gives a good representation of how an MR fluid behaves when there is no applying field.

If a fluid is Newtonian, it defines as one that its shear stress is directly proportional to shear rate, which can use a mathematical express as: y=kx+c. Where x is the shear stress and y is the shear rate, and they are of linear relationship against each other.

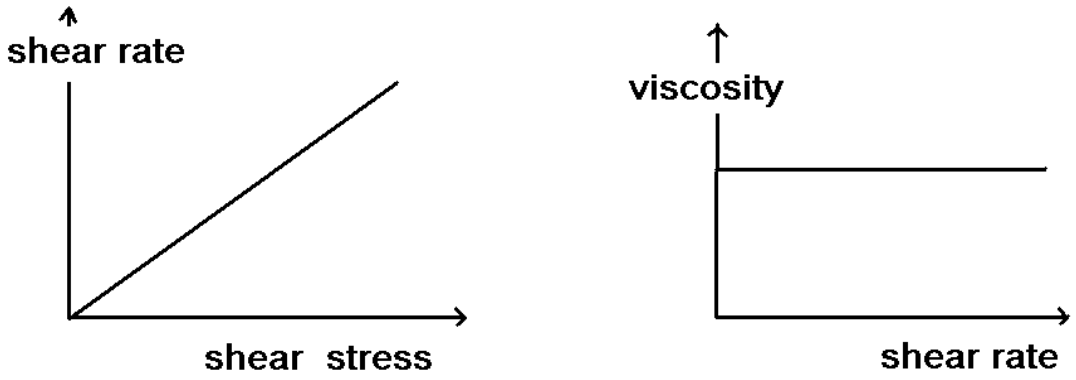

Figure 17: Newtonian Fluid

Image courtesy of: http://people.sju.edu/~phabdas/physics/rheo.html

As we can see from the about figure, the shear rate and shear stress are proportional to each other, which shows a linear graph and no matter how shear rate changes, the viscosity is always a constant.

3.6.2.2 Non-Newtonian Fluid

A non-Newtonian fluid is a fluid whose flow properties will be influenced by the applying forces. It is therefore time dependent and shear rate dependent. This model describes the property of MR fluid when a certain magnetic field is applied. Some common seen non-Newtonian fluids are honey and ketchup.

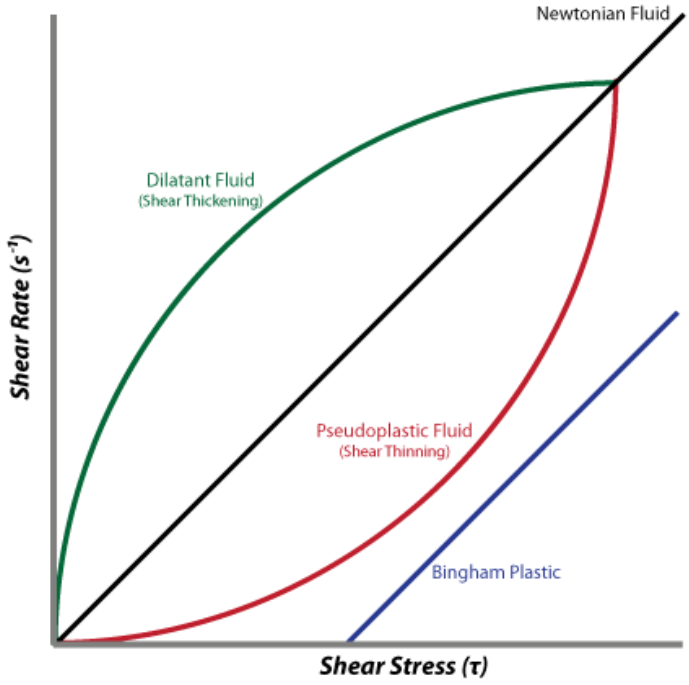

Figure 18: Relationship between Shear Rate and Shear Stress of Newtonian Fluid and non-Newtonian Fluid

Image courtesy of: Wikipedia non-Newtonian fluid

3.7 Yield Stress

When many forces are applied to an MR fluid, there will be a change in the fluid movement on application of continuous pressure in the presence of a magnetic field, the continuous pressure is termed as yield stress. Controllable yield stress is what makes MR fluid and also ER fluid very special and smart to be adaptable to a wide range of applications. MR fluids exhibit an extremely high yield stress because the existence of iron particles inside the fluid and hence makes them the great choice for big and hard impacting applications.

Generally, yield stress is defined as the point a material ceases to behave elastically. On another words, the stress divided by the strain is no longer constant. The point at which this occurs is known as the yield point. Put it in a simpler way the yield strength is the amount of pressure force needed to induce flow in the material.

The total yield stress of MR fluid can be express by this equation [31]:

$$\tau_{total} = \tau(\mathrm{H}) + \eta\Upsilon \qquad (3.10)$$

In the equation $\tau(\mathrm{H})$ is the yield stress as the function magnetic field strength (H field), η is the independent plastic viscosity and Υ stands for shear rate. The parameter $\tau(\mathrm{H})$ can be calculated by the following formula introduced by Lord Corporation [32].

$$\tau(\mathrm{H}) = \mathrm{C} \times 271.7 \times \varphi^{1.5239} \times \tanh(6.33 \times 10^{-6} \times \mathrm{H}) \qquad (3.11)$$

In this formula, φ is the volume fraction of iron particles, H is the magnetic field strength and C is a constant depending on the chosen carrier liquid.

3.8 Amplifier

Amplifier is to increase signal amplitude or power of the device, which is an important component of automation technology tools in signal processing. The Amplification of an amplifier is controlled by the input signal, the amplification provided by the energy required power consumption. Here we will introduce a very useful amplifier, which is called operational amplifier.

3.8.1 Operational Amplifier

Operational amplifier is also called op-amps that are the basic building block for analogue electronic circuits. A typical operational amplifier has three terminals consists of two inputs with high impedance, one is called the inverting input (marked with a negative sign '-') and the other is non-inverting input (marked with a positive sign '+'), the third terminal of the operational amplifier is the output which can be a sink and a voltage or current source.

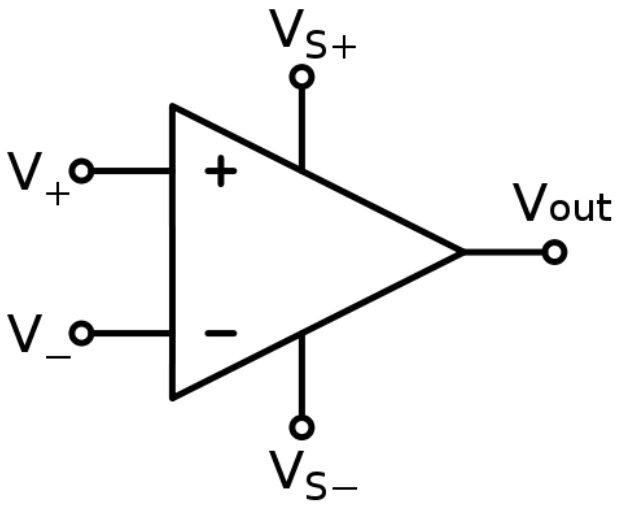

Figure 19: Symbol of Operational Amplifier

Image courtesy of: http://www.ustudy.in/node/728

3.8.1.1 Inverting Amplifier

In some case of an amplifier, to achieve a very high gain, this signal will be accompanied by a lot of instability and it is hard to control if the input is small, just a few microvolt would be enough to cause the output voltage to saturate and swing towards one or the other of the voltage supply rails loosing complete control. To overcome this a resistor crossing the amplifier from the output terminal back to the inverting input terminal will both decrease and control the overall gain of the amplifier, this produces an effect called negative feedback and the amplifier is called inverting amplifier.

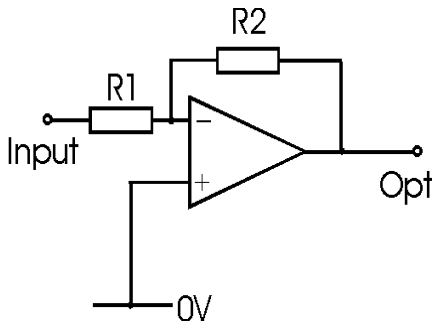

Figure 20: Inverting Amplifier

Image courtesy of:
http://www.electronics-radio.com/articles/analogue_circuits/operational-amplifier-op-amp/inverting-amplifier.php

The basic circuit for the inverting operational amplifier circuit is quite straightforward and only needs a few components in addition to the operational amplifier integrated circuit. The circuit consists of a resistor from the input terminal to the inverting input of the circuit, and another resistor connected from the output to the inverting input of the op-amp. The non-inverting input is connected to ground.

Figure 20 is the diagram of an inverting amplifier, and the voltage gain of an inverting amplifier is given as:

$$V_{output} = -\frac{R_2}{R_1} \times V_{input} \qquad (3.12)$$

3.8.1.2 Non-Inverting Amplifier

Non-inverting amplifier is to take the input signal which in this case is put directly onto the non-inverting input of the operational amplifier and output an amplifier version of that input signal with the same polarity. The amplification of this circuit can be controlled using a voltage divider from two resistors as a negative feedback closed loop system, this closed loop system produces a non-inverting amplifier circuit with very good stability and very high input impedance as no current flows into the positive input port.

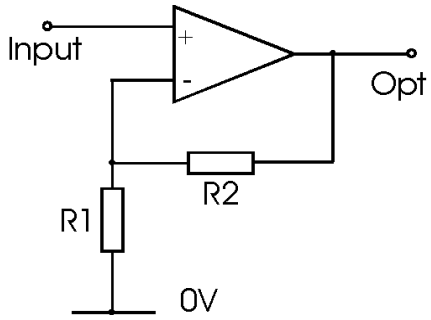

Figure 21: Non-Inverting Amplifier

Image courtesy of:
http://www.electronics-radio.com/articles/analogue_circuits/operational-amplifier-op-amp/inverting-amplifier.php

As we can see form the above figure, the signal is applied to the non-inverting input of the operational amplifier. However the feedback is taken from the output of the op-amp by a resistor to the inverting input of the operational amplifier where another resistor is taken to ground. It is the value of these two resistors that govern the gain of the operational amplifier circuit.

Figure 21 is the diagram of a non-inverting amplifier, and the voltage gain of non-inverting amplifier is given as:

$$V_{output} = 1 + \frac{R_2}{R_1} \qquad (3.13)$$

3.9 Pressure Sensor

A pressure sensor measures pressure, typically of gases. Pressure is an expression of the force required to stop a fluid from expanding, and is usually stated in terms of force per unit area. A pressure sensor usually acts as a transducer; it generates a signal as a function of the pressure imposed.

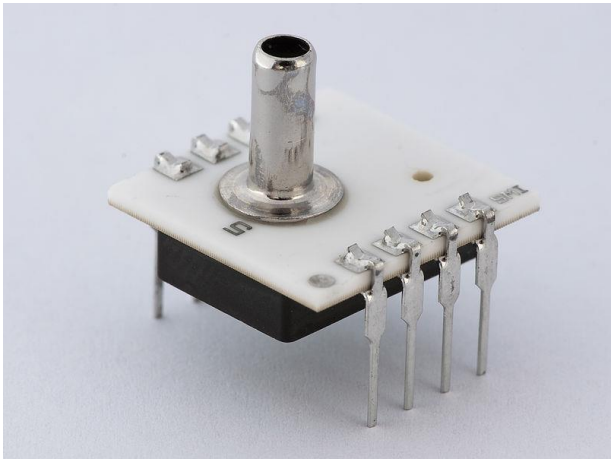

Figure 22: Pressure Sensor

Image courtesy of: Wikipedia Pressure sensor

The purpose of introducing a pressure sensor is to monitor the pressure created by the syringe when we give force to MR fluid surrounded by the magnetic field to study its yield stress. We can't know how much force we applied, so we use a pressure sensor to get know the quantity of the force indirectly.

3.10 Measurement techniques

Measurement is an important step during the research process, which involves using some special instruments to further understanding the properties of the products, when we study magnetic particles and their applications, we need to know particle size, shape, distribution, composition, magnetic property, etc. So here we introduce some apparatus needed in the experiments.

3.10.1 Optical Microscope

The optical microscope is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. Basic optical microscopes can be very simple, although there are many complex designs that aim to improve resolution and sample contrast. The simplest microscopes consist of single convex lenses. These can provide magnification factors of up to 10× or 20×. In the laboratory, an instrument called the compound microscope is preferred because it allows for much greater magnification.

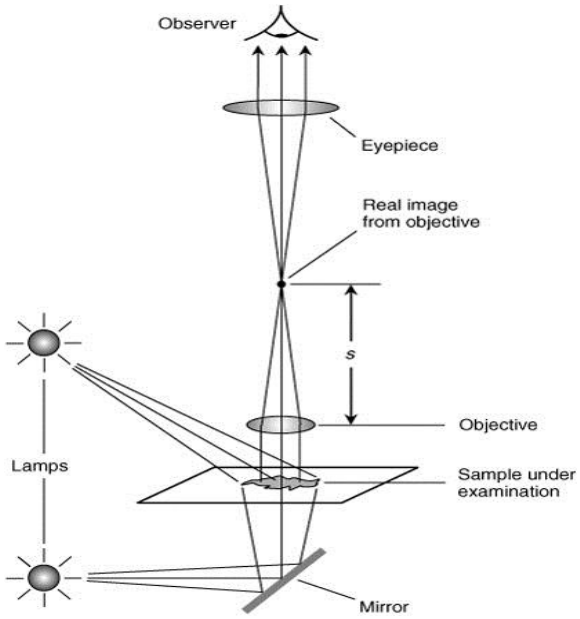

Figure 23: Analysis Diagram of Microscope

Image courtesy of:
http://www.education.com/study-help/article/physics-help-compound-microscope/

As we can see from the above figure, the light passes through a series of lenses, the sample is illuminated with visible light and it creates a magnified image of the sample under observation, and of course this captured image can be stored in a computer.

Laboratory-grade compound microscopes have two or more objectives, which can be selected by rotating a wheel to the object is attached. This provides several different levels of magnification for a given eyepiece. In general, as the focal length of the object becomes shorter, the magnification of the microscope increases, in another words, the resolution is inversely proportional to the wavelength of the light used. Some compound microscopes can magnify images up to about 2,500 times.

3.10.2 Electron Beam Interaction

An advanced form of imaging technique is with bombardment of the specimen with high-energy electrons instead of visible lights which produces lots of different effects on the sample and its surroundings. By Electrons beam emitting onto a material and interactions with the atoms of the target material, those accelerated electrons can pass through the sample without interaction, undergo elastic scattering and can be inelastically scattered. Elastic and inelastic scattering result in a number of signals that are used for imaging, quantitative and semi-quantitative information of the target sample and generation of an X-ray source.

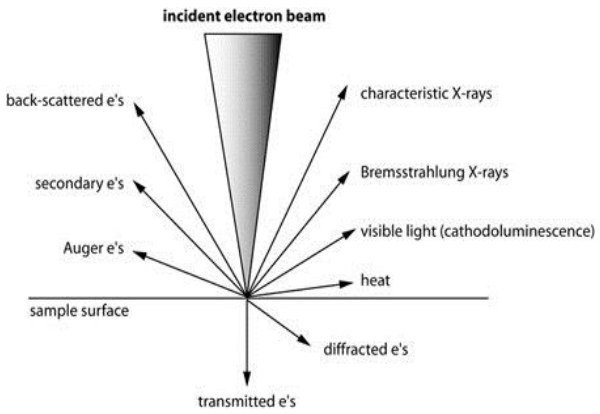

Figure 24: interactions between electrons and a sample

Image courtesy of:
http://serc.carleton.edu/research_education/geochemsheets/electroninteractions.html
By using this technology, a scanning electron microscope is created.

3.10.3 Scanning Electron Microscope

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external texture, chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-D image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected points on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions, crystalline structure, and crystal orientations.

SEM has many advantages compare to the traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified to a very high level. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

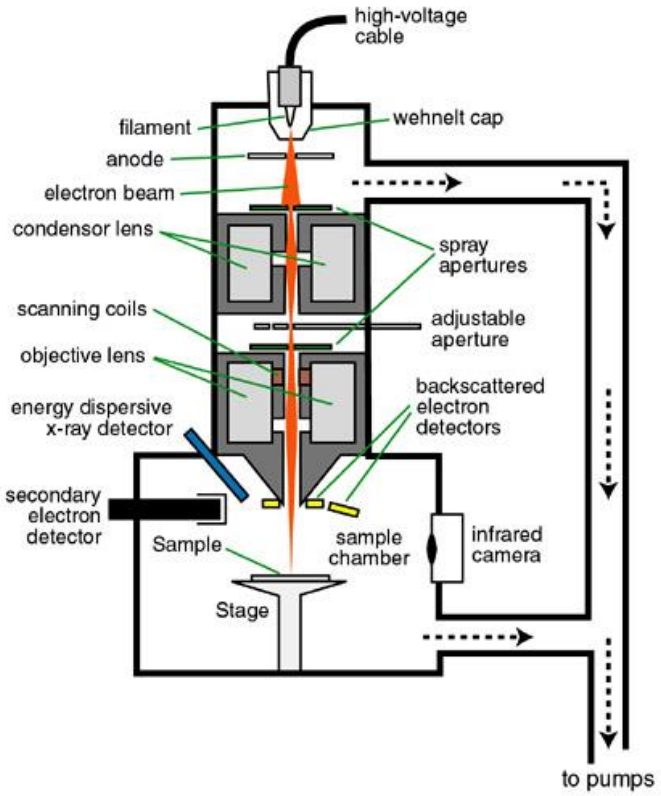

Figure 25: Schematic of SEM

Image courtesy of:

http://www4.nau.edu/microanalysis/Microprobe-SEM/Instrumentation.html

The high voltage cable create a electron gun which generating and firing electrons, the sample will be illuminated with E-beam instead of a light source, this occurs because the wavelength of electrons bombarding the sample is much smaller than the wavelength of light. Then the image is formed when other kinds of electrons which are an outcome of bombardment on the sample are scanned and observed through a series of magnetic lenses. SEM mainly uses secondary electrons which has been explained above (generated by electron beam interaction) to form a magnified image of the sample.

3.10.4 Energy-dispersive X-ray Spectroscopy

Energy-dispersive X-ray spectroscopy (EDX) is a very useful analytical technique used for chemical characterization of a material, EDX makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. It works on the principle of interaction of X-ray excitation on the material, each element can be determined because each element in the material sample has a unique atomic structure allowing unique set of peaks on its x-ray spectrum.

Electron beam excitation is used in electron microscopes such as scanning electron microscopes discussed previously, and scanning transmission electron microscopes. X-ray beam excitation is used in X-ray fluorescence (XRF) spectrometers. A detector is used to convert X-ray energy into voltage signals; this information is sent to a pulse processor, which measures the signals and passes them onto an analyzer for data display and analysis.

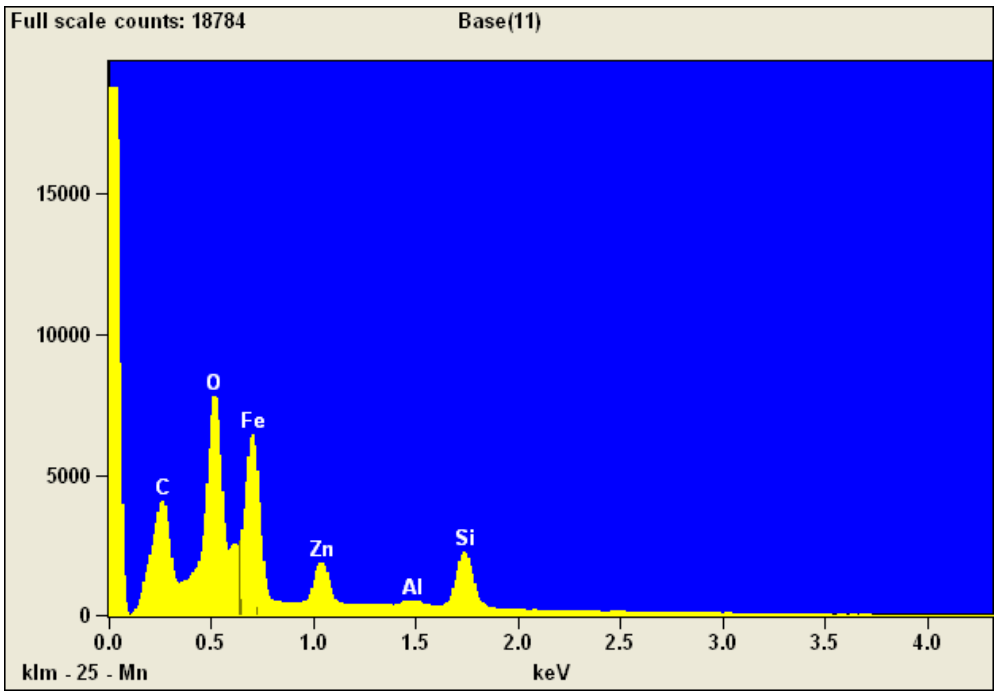

Figure 26: EDX analysis results example

As we can see from the above figure, we can get the information of the samples like: element type, the element volume, etc.

3.10.5 Transmission Electron Microscopy

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen and interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen, TEMs are capable of imaging at a significantly higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This enables the instrument's user to examine fine detail, even as small as a single column of atoms, which is thousands of times smaller than the smallest resolvable object in a light microscope. TEM forms a major analysis method in a range of scientific fields, in both physical and biological sciences.

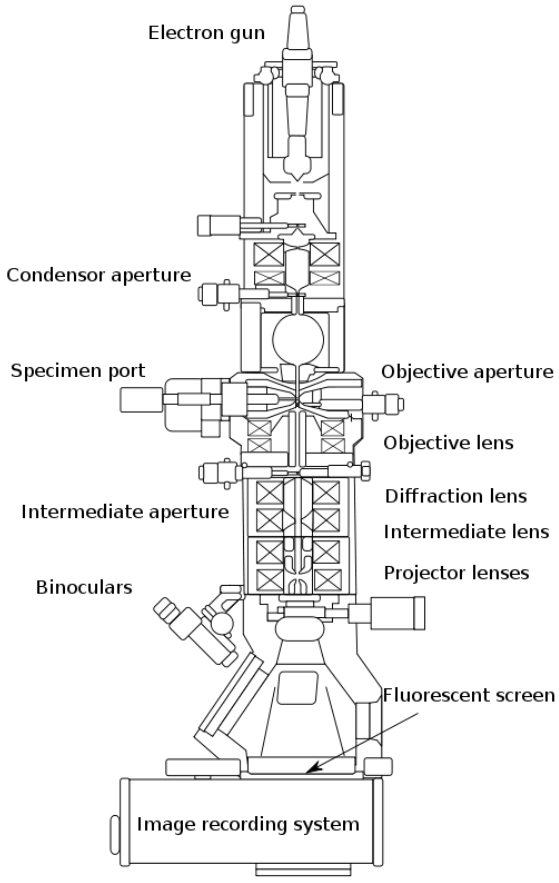

Figure 27: Schematic of Transmission Electron Microscopy

Image courtesy of: http://fgamedia.org/faculty/rdcormia/NANO53/TEM.htm

A TEM uses much higher energy beams than the SEM, thus penetrating the sample under observation much deeper, which means the image produced is of much larger magnifications than any other type of microscopy and also when observing the diffraction pattern of a sample, it is analyzed that each element under observation has a fixed diffraction pattern, this fixed diffraction pattern can be compared with the diffraction pattern observed from the TEM, then giving the information about the element, by this method an analysis can be made on the chemical structure or lattice structure of the material.

But the TEM sample preparation is generally requires more time and experience than for most other characterization techniques. A TEM specimen must be approximately 1000 Å or less in thickness in the area of interest. The entire specimen must fit into a 3mm diameter cup and be less than about 100 microns in thickness. A thin, disc shaped sample with a hole in the middle, the edges of the hole being thin enough for TEM viewing, is typical. The initial disk is usually formed by cutting and grinding from bulk or thin substrate material, and the final thinning done by ion milling.

3.10.6 Vibrating Sample Magnetometer

A vibrating sample magnetometer (VSM) is an instrument that measures magnetic properties by mechanically vibrating the samples inside of an inductive pickup coil. Induced current or changing flux in the coil is measured.

The principle behind the VSM is based on Faradays laws of Induction. When imposing an electric field, the sample in the system gives a current which will generate a magnetic field.

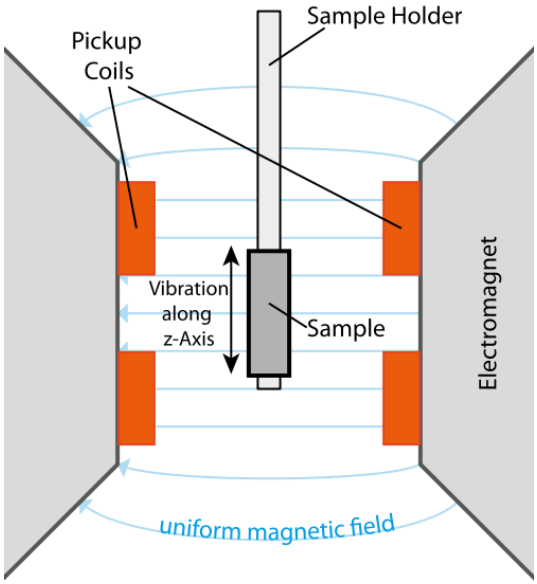

Figure 28: Schematic of vibrating sample magnetometer

Image courtesy of: Wikipedia VSM

As we can see from the above figure, the sample is placed in a constant H-field, and will be slowly magnetized. All the magnetic domains of the sample will be aligned with the applied field. The stronger the applied field the large will be the value of the magnetization. The sample will vibrate (in y direction and the field is applied in the x direction) which will change the magnetic stray field as a function of time which is sensed by the pick-up coils.

The alternating H-filed will cause an electric filed be created in the coils according to faraday's law and this current will be proportional to the magnetization of the sample. This induced current is amplified and the output signal is feedback to the computer to plot hysteresis loops.

4. Experiments

This part involves the experiments performed at University of York. Each experiment has been explained in separate section and the results from these experiments have been detailed with a short conclusion after each subsequent section.

There are a lot of methods to prepare magnetic nano particles, all methods have their own advantages or disadvantages, here we introduce some common methods that have been taken for magnetic nano particles preparation, and they are:

- *Ball Milling:*

This is the initial method to prepare nano particles for ferrofluid. It was introduced by S.S.Papell in 1966 [33]. The principle of this method is to add the rough magnetic particles (Fe_3O_4) and the surfactant to a carrier liquid, after a long time milling, usually takes almost 1000 hours in a ball milling machine. This method seems very simple but the raw material usage is very low, and also hard to get the particles diameter less than 300nm. Moreover, it will cause serious damage to the ball milling system, and create a lot of impurities. Though Reimers and Khalafalla improved this method [34], but it still didn't solve the problems like high cost and particles unevenly dispersing.

- *Thermal Decomposition:*

By using raw magnetic material like $Fe(CO)_5$, dissolved it into an organic solvent [35], then using thermal decomposition to get Free-State metal, after that adding the dispersant into the solution to separate, finally by dissolving the obtained particles into the carrier to get the magnetic fluid. The disadvantage of this method is produced the carbon monoxide gas which will pollute the environment and this method is not suitable for mass production.

- *Discharge method:*

This was introduced by Berkowitz in 1983. In a container full of carrier liquid, by putting the magnetic materials between two electrodes, adding the pulse voltage spark discharge and etching. During this process it will form the tiny magnetic

particles, but method like this can't prepare the well dispersed and stable ferrofluid [36].

- *Co-precipitation Method:*

By mixing two or more salt solutions together and adding alkali to form the magnetic sedimentation [37] [38], the co-precipitation method is the main way to produce nano particles at the present time. It's kind of titration hydrolysis and this method is based on the chemical equation:

$$Fe^{2+} + 2Fe^{3+} + 8OH^{-} \xrightarrow{Heat} Fe_3O_4 \downarrow + 4H_2O$$

The co-precipitation method can obtain uniform products, the preparation efficiency is also very high and the whole process is not so complicated that can realize the automatic production. This method has good prospects for industrial applications

In this project, we used the co-precipitation method, because the needed material and environment are easily achieve, and this method is pretty mature, normally can get fine particles.

4.1 Preparation of Magnetic Nano Particles (magnetite)

This experiment aimed to prepare fine magnetic nano particles. The nano particle is the core part of a good quality ferrofluid. The whole experiment is conducted in clean room, University of York.

Requirements List (Including Reagents and Instruments):

- Ferrous Sulfate Heptahydrate ($FeO_4S \cdot 7H_2O$) provided by Sigma Aldrich Company, the Molar Mass is 278.01g/mol.
- Iron (III) Sulfate Hydrate ($Fe_2O_{12}S_3 \cdot xH_2O$) provided by Sigma Aldrich Company, the Molar Mass is 399.88g/mol.
- Ammonia (NH_4OH) Molar Mass is 17.031 g/mol.
- Hydrochloric acid (HCl) Molar Mass is 36.46094 g/mol.
- Deionised water (DI water), Ethanol (CH_3CH_2OH) for dissolving and particles washing.
- Electric mixer
- Water bathing container
- Electronic scale
- Beaker, Glass rod, Graduate, etc.
- PH meter
- Tripod
- Permanent magnets

Experimental Procedures:

We used the co-precipitation method to prepare the nano Fe_3O_4 particles. First we mixed ferrous salt and ferric salt evenly. The ratio of the salt should be Fe^{2+} : Fe^{3+} = 1:1. To prevent the ferrous salt from being oxidized, we can add some dilute hydrochloric acid to enhance antioxidant capacity of the mixing solution. Then we used the water bath to heat the reaction solution. Finally we poured the ammonia to adjust PH level to 10. The whole reaction time lasts 120 minutes.

The Details of Experiment:

According to the chemical equation we mentioned above:

$$Fe^{2+} + 2Fe^{3+} + 8OH^{-} \xrightarrow{Heat} Fe_3O_4 \downarrow + 4H_2O$$

We can calculate the amount of Ferrous Sulfate Heptahydrate and Iron (III) Sulfate Hydrate needed in the experiment.

- Get 5.198g $Fe_2O_{12}S_3 \cdot xH_2O$ dissolved into Xml (X is a variable) DI water to get the ferric slat solution, the concentration of solution will be 1.2mol/L, 0.7mol/L, and 0.15mol/L respectively depending on the volume of water for the experiment.

Figure 29: Ferrous Sulfate Heptahydrate

- Get 3.9g $FeO_4S \cdot 7H_2O$ dissolved into Xml (X is a variable) DI water to get the ferrous salt solution, the concentration of solution will be 1mol/L, 0.5mol/L, and 0.1mol/L respectively depending water volume for the experiment.

Figure 30: Iron(III) Sulfate Hydrate

You may notice that the molar ratio of Fe^{2+}, Fe^{3+} is not 1:1, because the Fe^{2+} is very easy to be oxidized in the air, so we added a litter more ferrous salt or added less ferric salt in case of the actual molar ration of those two iron salts was not right. As Figure 30 shows, the foil on the top of the beaker is also for precaution (Prevent from oxidization).

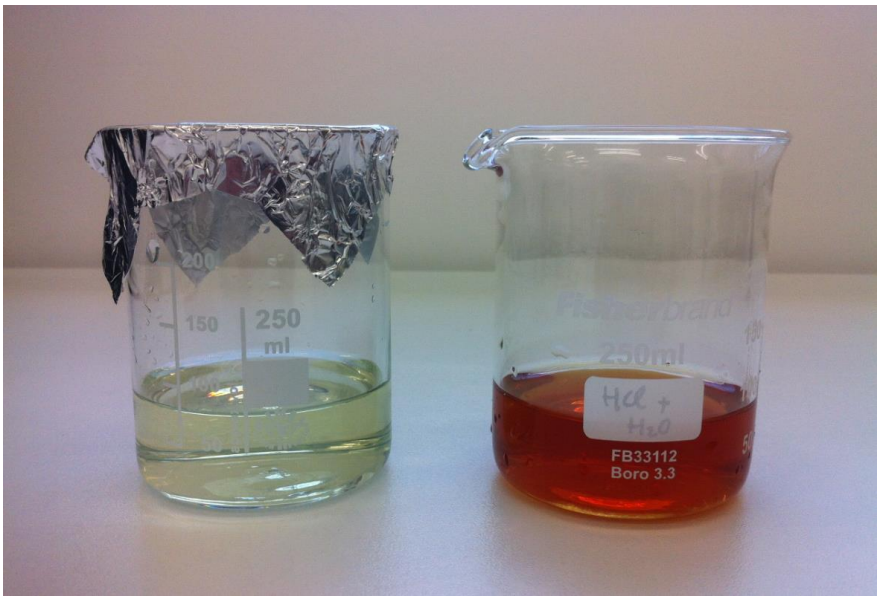

Figure 31: Ferrous Sulfate Heptahydrate and Iron (III) Sulfate Hydrate (left to right)

- Mix those two salt solutions together and keep stirring, then place the mixed solution in a water bath container, heat it at temperature (20℃, 40℃, 70℃, 90℃,) respectively.

Figure 32: Reaction Setup

- Add ammonia to adjust PH level to 10 by using dropper, the Nitrogen gas applied by a Nitrogen gun in the clean room is for preventing ferrous ions of the mixed solution from being oxidized.

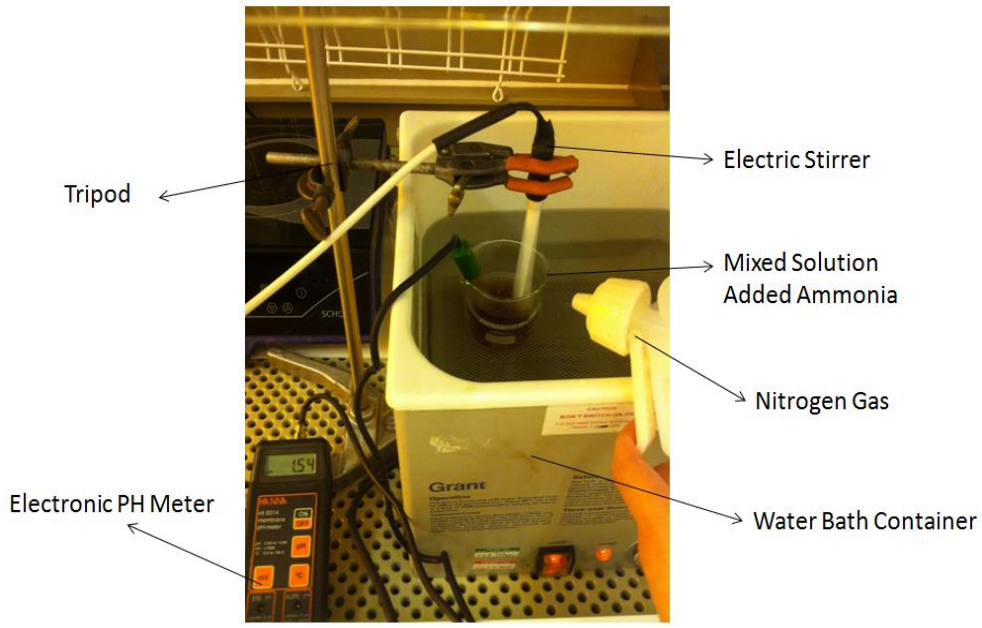

Figure 33: Experiment setup when preparing

- Keep stirring the solution for 100 minutes and get it from the water bath container, use DI water and ethanol wash at least 5 times to wash out the impurity and unwanted ions. During the washing process, we need to use a glass rod to keep stirring the obtained nano particles, and the stirring is to make the ion-state unwanted particles dissolving into the solvent, then pour out. Finally put a permanent magnet under the beaker to attract the magnetic particles to the bottom of the beaker, and then pour out the DI water and ethanol.

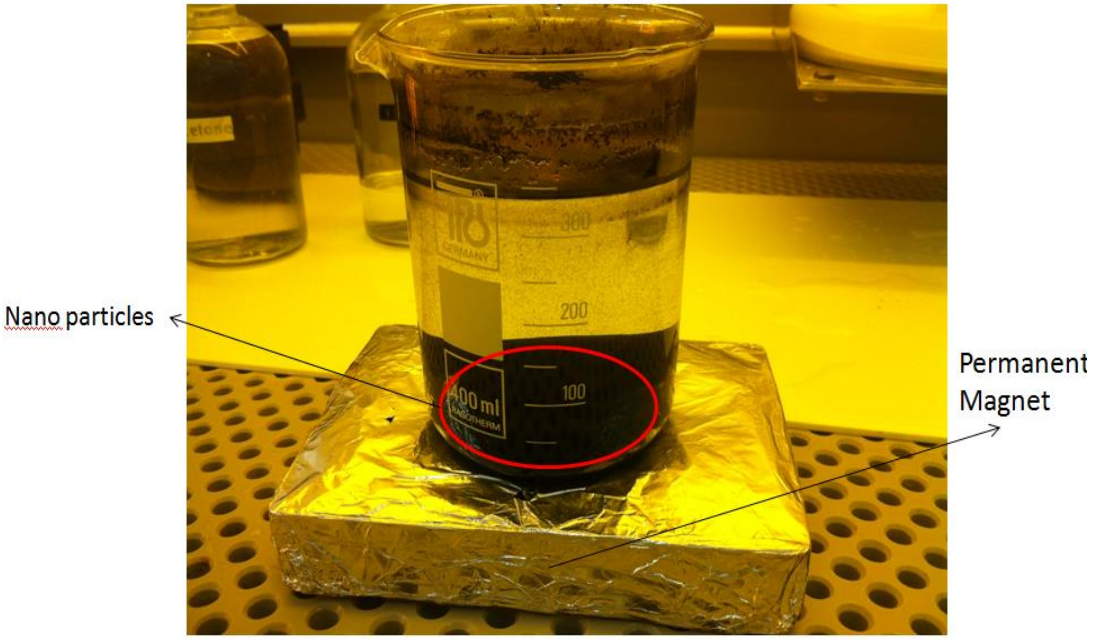

Figure 34: Obtained Nano magnetic particles attracted by a magnet

As we can see from Figure 34, those magnetic particles have settled down because the attraction of the magnets.

4.1.1 Preparation of Magnetic Nano Particles Experiment Result

Temperature Influence on Results:

From the experiments we prepared 4 magnetic nano particles samples, try best to control other variables not changing (it is very difficult to ensure that all the variables stable) but giving 4 different reaction temperature to observe the products. The result can be showed in the following table:

Temperature of Reaction	Products Description
20℃	The reaction happened very slowly. Huge amount of particles settled down at the bottom of the beaker when the reaction was over, but the particles were not attracted by the magnet well
40℃	The reaction also happened not as quickly as we expected. Huge amounts of black particles be created when the reaction was finished, particles can be attracted by the magnet partly, some particles still didn't react to the magnetic field
70℃	Vast black precipitation had been created after the reaction finished, and those black precipitation react to the magnet very well
90℃	Black precipitant created with some yellow unidentified precipitant, majority particles not react to magnet

Table 2: Temperature's Influence on Production

From Table 2, we can conclude that when we preparing the nano particle, the reaction temperature are an important factor that will influence the

results of the particles, when reaction temperature is below 50℃, the reaction happens very slowly and the result is not so satisfied. When the reaction temperature is around 70℃, reaction is going smoothly and the final product is satisfied. The particles generated will react to the magnetic field quickly. But once the reaction temperature reached 90℃, the created nano particles were not attracted easily by the magnet again.

Concentration Influence on Results:

From the experiments, we prepared another 3 samples. This time, by controlling reaction temperature at around 70℃, but changing the concentration of mixed solution to study its influence on the final products.

The results are showed in the following table:

Ferrous salt Concentration	Ferric salt Concentration	Result Description
1.2mol/L	1mol/L	Reaction speed was pretty quick and lots of black precipitant created, particles react to magnetic field well, but there was some cluster being generated
0.7mol/L	0.5mol/L	Reaction speed was quick and particles react to magnetic field pretty well and almost no aggregation
0.15mol/L	0.1mol/L	Though there were black precipitant generated, but most of nano particles cannot react to the magnetic field

Table 3: Concentration of reaction reagent influence on production

From this table we can conclude that the concentration of the reaction solution does influence the final products. If the solution is too low like 0.1mol/L, it will lead the products not reacting to the magnetic field well. It may be because the

concentration cannot reach the required conditions that the co-precipitation needs. Concentration around 0.5mol/L or higher is pretty good for the whole reaction. But if the concentration is too high more than 1mol/L that will causes the reaction speed too quickly and form huge amounts of Fe_3O_4 instantly and trigger the particles not to disperse well and get aggregated finally.

4.2 Coating Experiment

Last experiment we prepared some qualified nano magnetic particles, and as we know that ferrofluid must get the magnetic particles coated with surfactant, otherwise particles will settle to the bottom quickly and get aggregated. It is necessary to do the particles coating to enhance the stability of ferrofluid.

Requirements List (Including Reagents and Instruments):

- Fe_3O_4 Nano particles
- Sodium Oleate from Sigma Aldrich
- DI water
- Electric mixer
- Ultrasonic cleaning
- Electronic scale
- Beaker, Glass rod, Graduate, etc.
- PH meter
- Tripod

Experimental Procedure:

Get the prepared Fe_3O_4 particles dissolved into the liquid with the surfactant (here we choose sodium oleate as the surfactant). Adjust PH level several times and heat to 80℃, the amount of sodium oleate was depended on how much nano particles we used. After a 30 minutes reaction, we adjusted the solution to PH neutral level, then we used ethanol washing several times and dried it, the last procedure is adding the coated magnetic particles disperse into water and using ultrasonic bath to make it disperse well and obtain the ferrofluid.

The Details of Experiment:

1. Prepare the surfactant solution, get sodium oleate dissolved in DI water creating the aqueous solution, the concentration is10g/L.
2. Adjust PH level and using Magnetic Stirrer with heating function to heat the solution to 80℃.
3. Keep stirring for more than 30 minutes and adjust PH level to neutral.

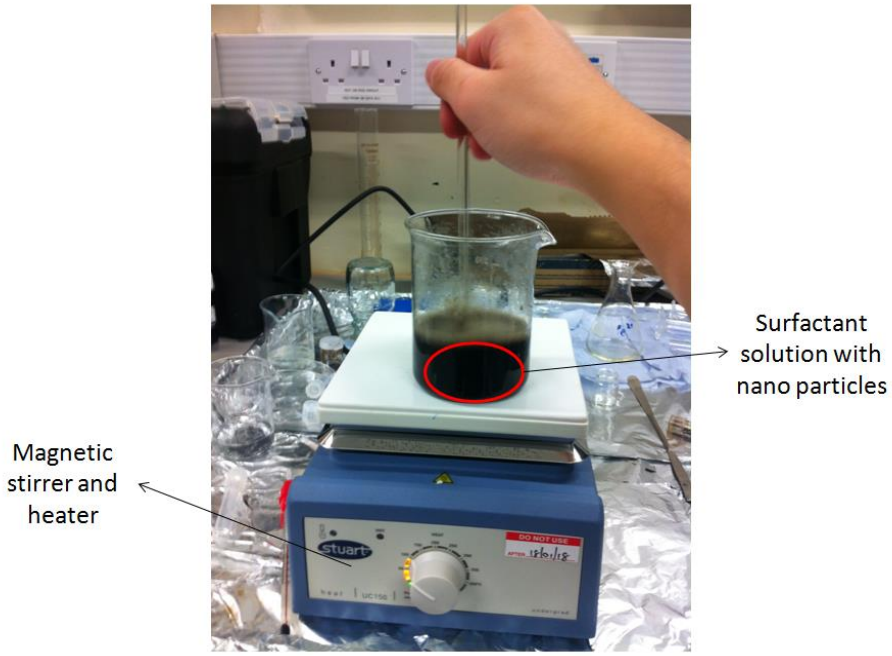

Figure 35: Heating by the magnetic stirrer and keep stirring

4. Separate the coated magnetic particles and use ethanol to wash more than 2 times to remove the excess sodium oleate and dry the final products.

Figure 36: Final product coated magnetic particles in a sealed container to prevent oxidizing.

4.2.1 Coating Experiment Results and Analysis

The chemical equilibriums existing in coating process are:

A: $Fe_3O_4 + OH^- \leftrightarrow [Fe_3O_4 - OH]^-$

B: $R - COOH \leftrightarrow R - COO^- + H^+$

C: $[Fe_3O_4 - OH]^- + R - COOH \leftrightarrow [R - COO - Fe_3O_4]^- + H_2O$

As we can see from the equilibrium, when the PH level of the solution is too low that will lead equilibrium A move to the left part of the equation, then the created $[Fe_3O_4 - OH]^-$ ions will be less than required, thus the stability of ferrofluid will be affected. In the acidic environment, Fe_3O_4 particles carrying positive charges, but in the alkaline environment, carrying negative charges, the anionic surfactant like sodium oleate is easier to attract and adsorb the Fe_3O_4 particles carrying positive charge.

But according to Kristy.M.Ainslie and Gaurav Sharma's theory discussed in their paper Attenuation of Protein Adsorption on Static and Oscillating Magnetostrictive Nanowires in 2005 [39], bilayer structure particles like Fe_3O_4 will repel the oleic which will have no sufficient driving force to do the attract and adsorb. Overall, acidulous reaction environment is better for particles coating.

4.3 Synthesis of Magnetorheological Fluid

MR fluid is composed by three parts, which are micro size magnetic particles, carrier liquid (oil or organic solvent) and surfactant, MR fluids' main different with ferrofluid is particle size and this has been introduced in the previous content. The most important part of MR fluid is particles too, a good quality of particles will determine the fine characteristics of MR fluid.

The carbonyl iron powder (CIP) used in the experiments was obtained from BASF Company in Germany, the CIP produced by BASF is well behaved in commercial applications. Carbonyl iron powder is obtained by decomposition of pentacarbonyl, during the process of decomposition, carbon monoxide will be generated and this reaction needs high pressure and high temperature, which is full of risk, so we didn't prepare carbonyl iron powder by our own.

The carrier liquid we chose in synthesis experiment was the hydraulic oil obtained from Shell Advanced Fork Oil for shock absorber, which served this projects purpose well,

Safety:

Be sure all dry particles will only be conducted when all persons in the vicinity have been told and all persons have respiratory safety equipment. When handling dry particles, we should move to the ventilation chamber in the student workshop. Protective gloves will be worn at all times when handling dry particles and fluids.

Experiment Details

The experiments were cooperated with James Romaine, the procedures will be:

1. Weigh the desired amount of carrier fluid in a beaker
2. Reset scales and weigh out CIP directly into the hydraulic fluid
3. Reset the scale again and add other additives
4. Place the sample in the ultra-sonic bath for 3-5minutes
5. Finally, stir the sample hardly to make the CIP disperse sufficiently

We produced eight samples totally, the difference between those samples are CIP ratio in the MR fluid and different type of additives like lithium grease and crashed glass. The samples were:

A. 50% MR Fluid (5g hydraulic fluid, 2.5g coated CIP)
B. 70% MR Fluid (5g hydraulic fluid, 2.5g coated CIP)
C. 50% MR Fluid with crashed glass (3g hydraulic fluid, 1.5g coated CIP and small glass particles mixture, where 25% is CIP and 25% is small glass particles)
D. 80% MR Fluid (5g hydraulic fluid, 4g coated CIP)
E. 70%MR Fluid mixed with ferrofluid (3g hydraulic fluid, 2.1g 70% coated CIP, 0.9g 30% crashed glass particles)
F. MR Fluid mixed with ferrofluid (2g hydraulic fluid, 1.4g 70% coated CIP, 0.6g 30% ferrofluid)
G. MR Fluid with 20% Lithium Grease (3.1g hydraulic fluid, 2.17g coated CIP and 0.62g lithium grease)
H. MR Fluid with 5% Lithium Grease (3.1g hydraulic fluid, 2.17g coated CIP and 0.155g lithium grease)

4.3.1 Particles Observing in SEM

The SEM been used in this experiment was from York JOEL nano centre, the results can be seen as following figures:

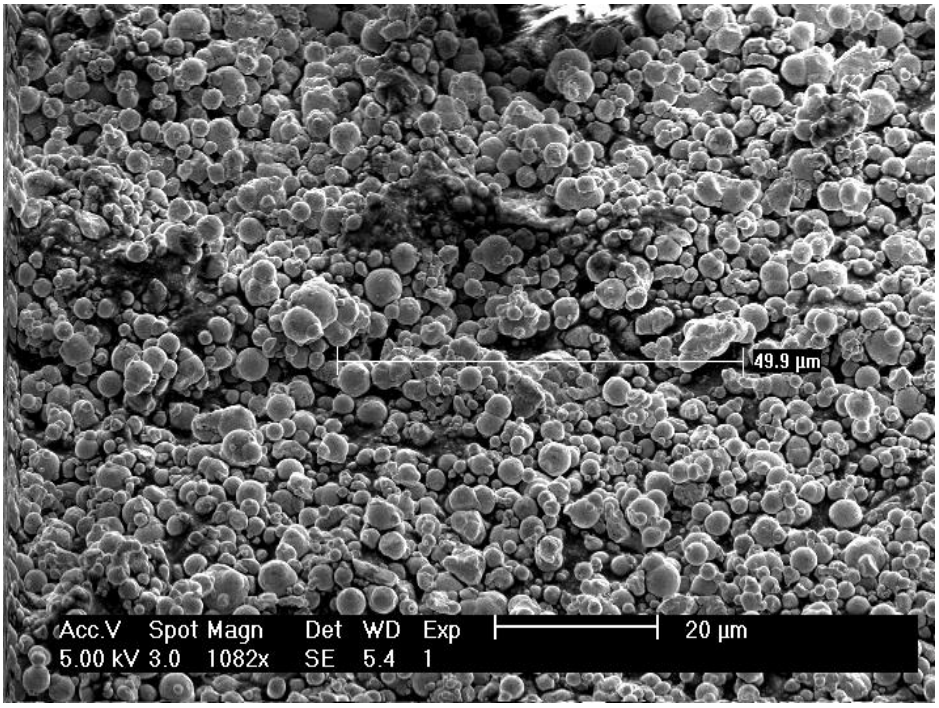

Figure 37: Carbonyl Iron Powder (CIP) general view in a SEM

The magnification of this image was up to 1082×, from this image we can see the CIP particles were extremely tiny micro size which cannot be distinguished from a naked eye.

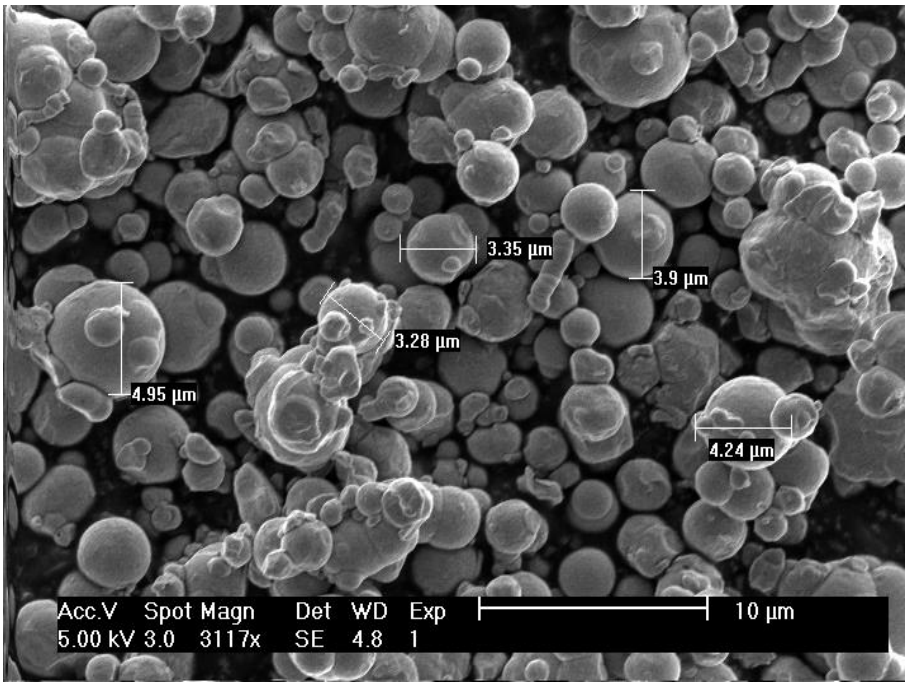

Figure 38: Carbonyl Iron Powder Magnified

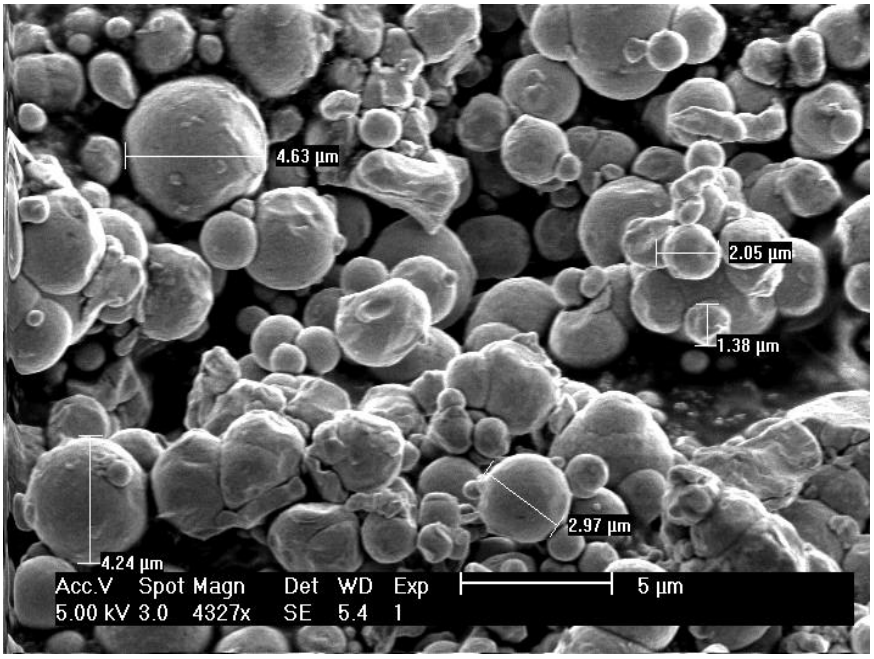

Figure 39: Carbonyl Iron Powder Magnified with a higher resolution

As Figure 38 and Figure 39 showed, after adjusting the magnification of the image to 3117×, the details of the CIP particles can be seen clearly, those particles' shape is pretty uniform and almost represented as a sphere. The diameter of the particle is around 4µm and it is much bigger compare to the nano particles in ferrofluid.

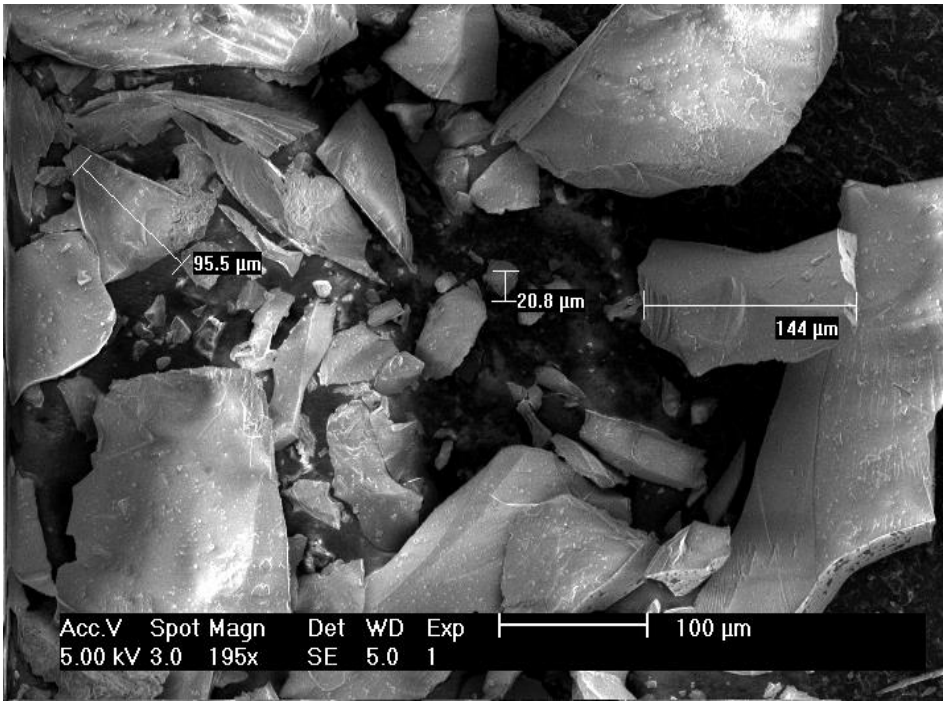

Figure 40: Crashed glass particles

The crashed glass was used in the following experiment as a kind of additive in the MR fluid, We conducted the yield stress tests to see if those small glass particles can have the same effects as CIP or not? From the Figure 40 above we can see, though those crashed glass seems like a powder, which are very small also cannot distinguish from a naked eye, but compare to the CIP particles, it is quite big that reached 20 µm to 100+ µm, the shape of those particles is so irregular, and the size is not uniformed too. We all know that glass is a kind of material shows yield stress, and here we used crashed glass as additive will definitely change the interaction inside the particles, the analysis why this happened was explained in the following Yield Stress tests.

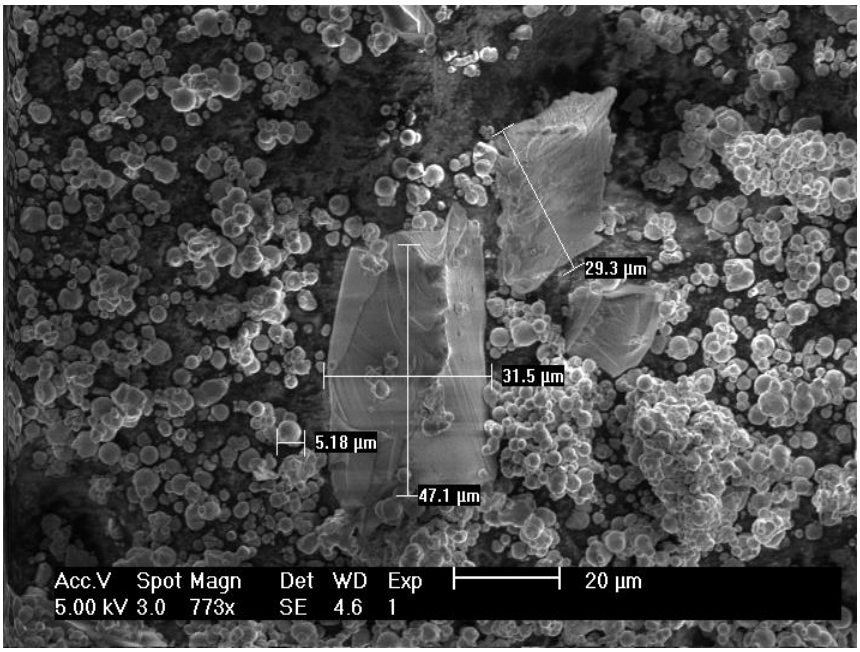

Figure 41: Crashed glass particles with CIP

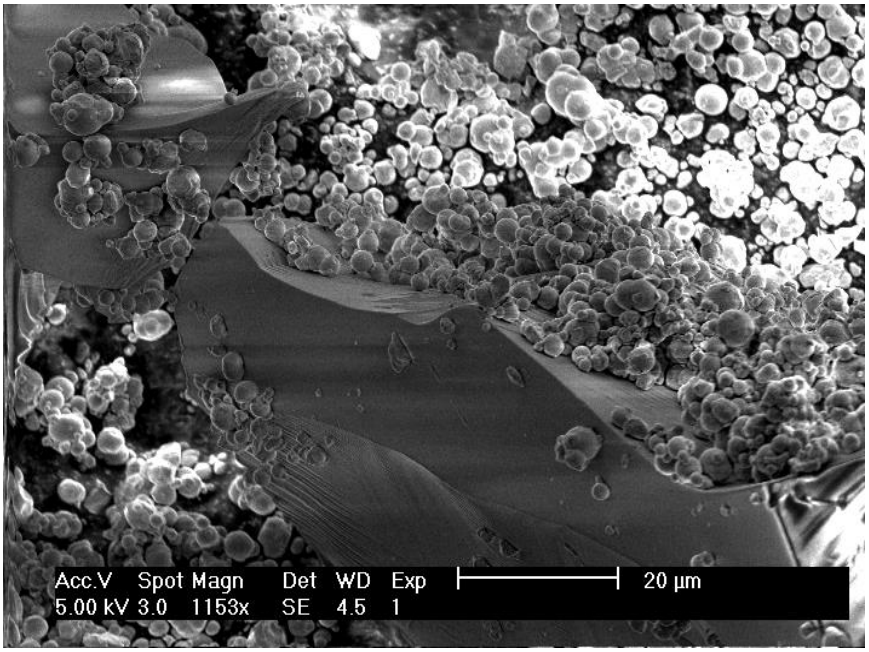

Figure 42: A higher Magnification of Comparison Image

Figure 41 and Figure 42 give a comparison of CIP particles and the crashed glass particles. Some CIP attached to the tiny particles but we can see from this image that the glass particles are much bigger than the CIP particles, where the diameter of CIP is around 5µm and the diameter of glass particles is 30µm or more. And the shape of CIP is quite uniform compared to the crashed glass particles.

4.4 EDX

EDX was performed to get the chemical composition of carbonyl iron powder with a small amount crashed glass, Figure 43 below shows the sample SEM image that will be imported to the EDX system connected to the SEM. By using the point and shoot method of analysis, the two most representative parts (CIP and Crashed Glass) of the sample were selected for the analysis.

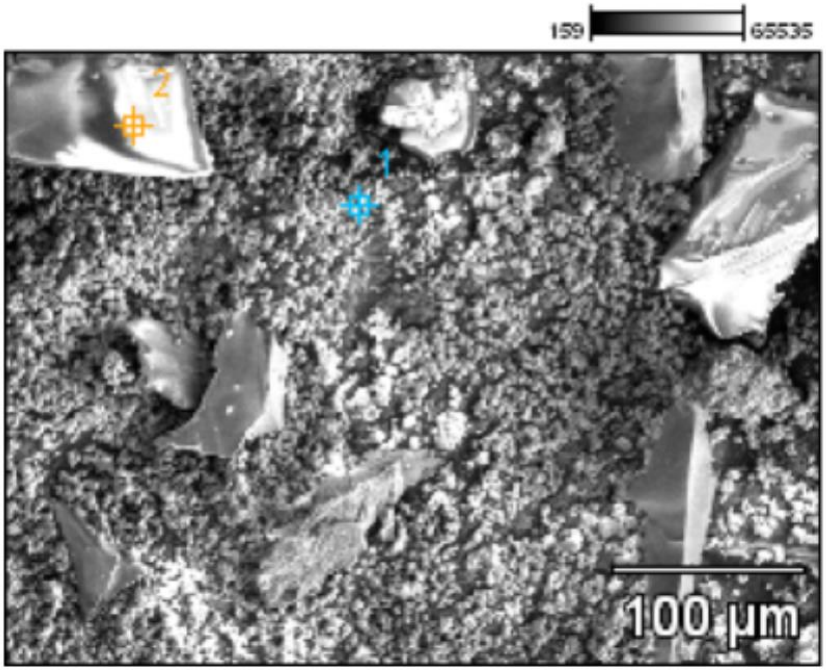

Figure 43: EDX image of the selected sample

From EDX system, two graphs were generated, and the target elements had been presented by highlighting as blue dot and orange dot, which can be seen from Figure 43.

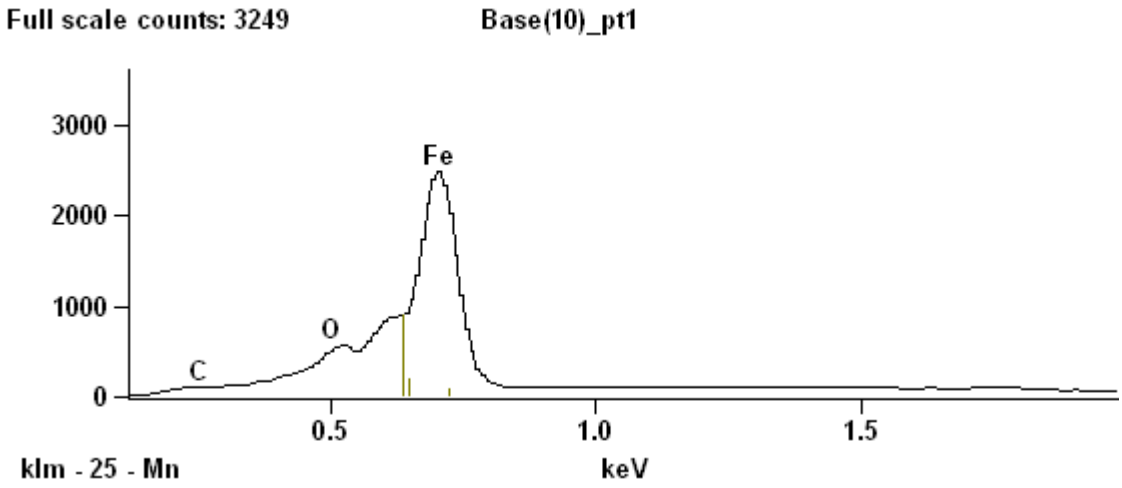

Figure 44: EDX graph analysis of CIP area

We can see from Figure 44 that Iron is the main element been detected, and this is pretty reasonable after considering the MR particles are the composed of carbonyl iron. The second detected element is Oxygen. We considered that is because small parts of Iron element have been oxidized after exposed in the air for a long while. Also some Carbon can be seen from the graph, carbon monoxide as one raw material to produce pentacarbonyl iron cause the residual of carbon element existing in the sample is not surprising.

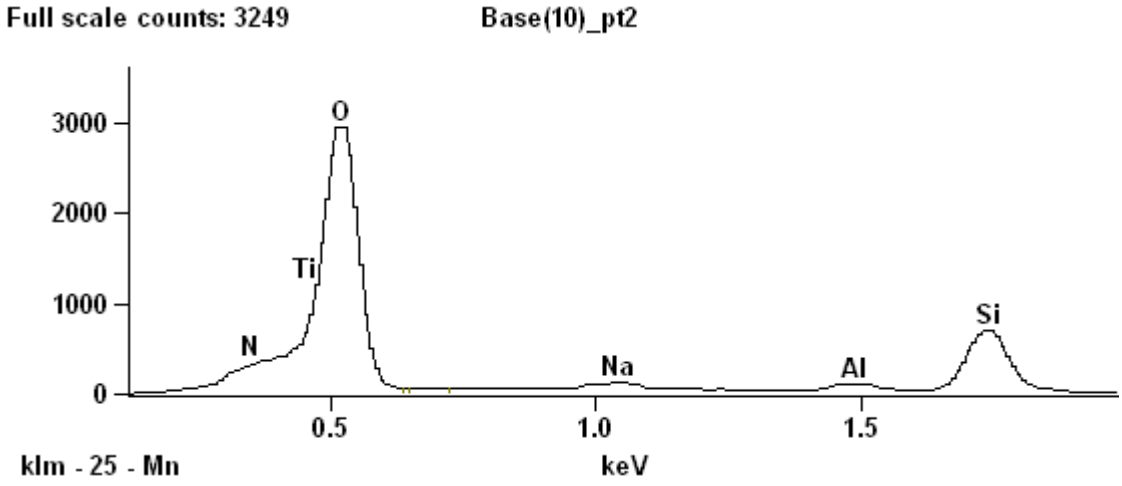

Figure 45: EDX graph analysis of glass particles

We can see from above Figure 45 clearly that the main elements detected in this area are Silicon, Oxygen, Aluminum, Titanium, Sodium, Nitrogen and etc. For the

main composition of glass is silicon dioxide (SiO_2), it is reasonable and that Oxygen (O) and Silica (Si) is much higher than other elements.

The following tables provide the detailed information about the EDX analysis:

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	524		2554					26410
Crashed Glass		3025	21311	649	684	6517	0	

Table 4: Net Counts

Table 4 shows the number of x-ray detected and at what energy level K or L. From this table we can see that for the MR particles the most amount of X-rays detected were iron, the most common element in glass is Oxygen followed by Silica.

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	17.467		85.133					880.33
Crashed Glass		100.83	710.37	21.633	22.800	217.23	0.000	

Table 5: Intensity

Table 5 shows the strength of the x-rays detected.

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	2.067		5.233					8.87
Crashed Glass		7.17	6.77	1.267	1.333	3.37	5.567	

Table 6: Intensity Error (+/- 1 Sigma)

The intensity error table is the amount of variable error possibly present in the intensity, the higher the count and intensity the bigger the error.

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	0.011 C		0.048 C					0.941
Crashed Glass		0.173 C	0.585 C	0.012	0.017	0.213	0.000	

Table 7: K-Ratio

K-ratio is the ration of the number of counts detected in the sample to the standard number assigned to that element.

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	+/- 0.001		+/- 0.003					+/- 0.009
Crashed Glass		+/- 0.012	+/- 0.006	+/- 0.001	+/- 0.001	+/- 0.003	+/- 0.000	

Table 8: K-Ratio Error (+/- 1 Sigma)

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	1.27		4.00					94.73
Crashed Glass		16.69	60.19	1.26	1.66	20.20	0.00	

Table 9: Normalized Wt. %

Normalized weight is the percentage of an element in the selected area by the weight.

	C-K	*N-K*	*O-K*	*Na-K*	*Al-K*	*Si-K*	*Ti-L*	*Fe-L*
CIP	5.16		12.19					82.65
Crashed Glass		20.58	64.99	0.95	1.06	12.42	0.00	

Table 10: Atom %

Table 10 is the percentage of an element in the selected area by atomic weight.

The EDX analysis system also gave us the distribution of elements detected on the sample by highlighting the area with different colours.

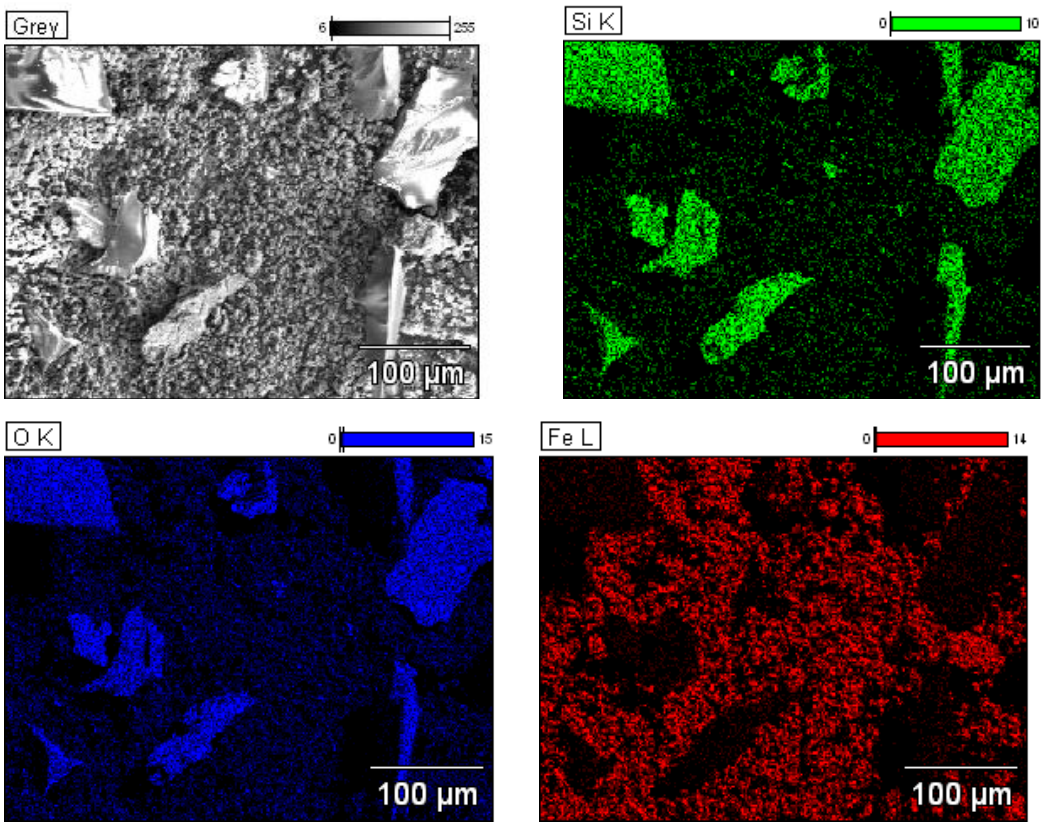

Figure 46: Elements distribution highlight by different colours

From Figure 46 we can read the distribution of elements on the sample intuitively, where the grey is the sample for analysis, blue is Oxygen distribution on the sample, green stand for Silica, and red is represent the position of Iron. Compare to the original grey one, the main distribution of Silica and Oxygen (blue and green) is exactly the position of glass (silicon dioxide SiO_2), and the red area is where the CIP (iron) locates.

4.5 Yield Stress

Yield stress is an important property of MR fluid that makes MR fluid widely used in engineering field. The yield stress experiments were designed to know how the particle ratio and additives or the densities affect the yield point of MR fluid.

4.5.1 Principle of Yield Stress Test

We used a circular dropper tube (Figure 48) to hold the sample of MR fluid. An adjustable electromagnet was placed around this dropper to apply an external field to the fluid and let the magnetic flux interact with the sample. A syringe used to apply pressure to the sample under a given H-field by plastic pipe, the pressure obtained from the sample every second was read by a pressure sensor and displayed to a laptop by a programmed microchip interface.

Because the yield point is the maximum amount of stress applied to the sample before the material getting breaking or deformation, which is given as:

$$\tau = \frac{F}{A} \qquad (4.1)$$

And the amount of pressure on the sample is given as:

$$P = \frac{F}{A} \qquad (4.2)$$

We can get know the yield point by measuring the amount of pressure indirectly.

4.5.2 Experiment Setup

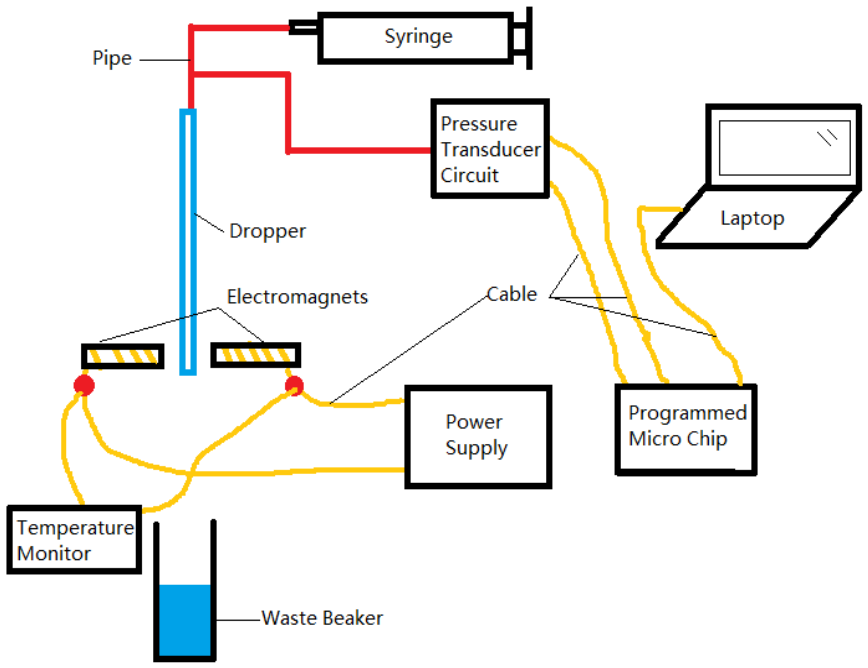

Figure 47: Diagram of the experiment setup

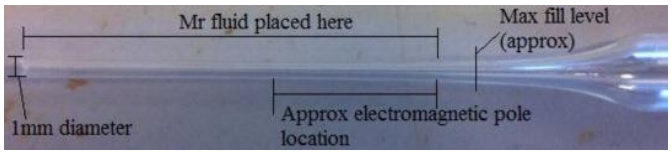

Figure 48: Details of the dropper for sample

The whole experiment components were fixed on a back plate, which thanks to Dr. Iain Will's help.

The electromagnet used in the experiment was a circular shaped electromagnet from the spintronics laboratory. The gap of the circle was approximately 3cm, which was perfect for the experiments. The copper wire on the circular iron was connected to an adjustable DC power supply to control the magnetic field flexibly. The magnetic field strength created by the electromagnet can be read form the magnetometer (Figure 89).

For the safety reason, a temperature gauge was attached to the copper wire for electromagnet to monitor the temperature of the wire.

The current generated by a power supply creates the magnetic field, here gives reference table of the relationship between current and strength of magnetic field.

Current/Amps	Magnetic Field Strength/mT	Temperature/℃	Voltage/Volts
0.4	19	21.5	7
0.8	37	21.8	13
1.2	54	23.8	17.5
1.6	70	26.7	22.5
2.0	82	30.6	30

Table 11: Reference table of the relationship between all parameter

4.5.3 Experiment Details and Results

For safety reasons, the protective gloves were to be worn at all times during the experiment by any participating member and all people conducting the experiment should wear the goggles for eye protection. Any spillage of fluid should be cleaned immediately and care the temperature of electromagnet at any time, between tests the electromagnet current will be set to zero to let the temperature down.

The yield stress experiment will be conducted over the 5 set currents (0.4A, 0.8A, 1.2A, 1.6A, 2.0A), and will be conducted on the sample (A-H) prepared before.

Steps:

1. Take a fluid sample, put the sample into a plastic test tube, and stir hardly for at least 10 seconds and draw the sample into the dropper by using the syringe till the required amount of sample is collected.
2. Slowly increase current to approximately 0.4A, let the MR fluid sample exposed in the magnetic field, this step will prevent the settling of particles and prevent the absorbed sample from falling the tube under the influence of gravity force.

3. Slowly increase the current to a desired value (0.8A 1.2A 1.6A 2.0A).
4. Open the programmed microchip terminal console and set to log data in a word document (.doc).
5. Press the Y key on the computer keyboard to start the yield stress test.
6. Apply pressure to the sample by the syringe until a significant flow is established or the pressure significantly drops (yield point).
7. Slowly decrease the current to 0A.
8. Empty the remaining fluid in the dropper.
9. Reset the syringe positions ready for the following test.
10. Repeat above steps 8 times to get the results.

Collected data was placed into Mac OS-X Numbers and plotted as the function of pressure and magnetic field.

Results:

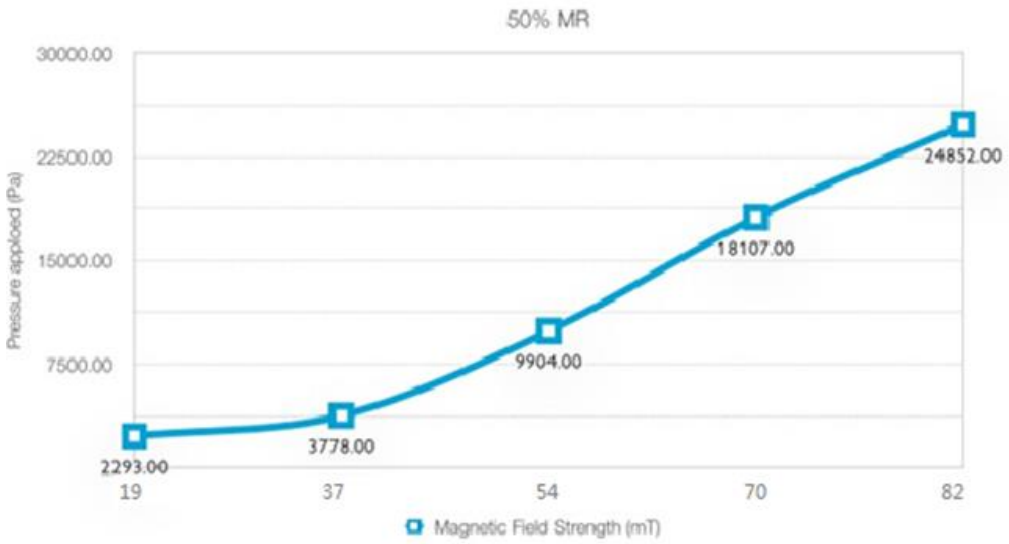

Figure 49: 50% MR Fluid (5g hydraulic fluid, 2.5g coated CIP)

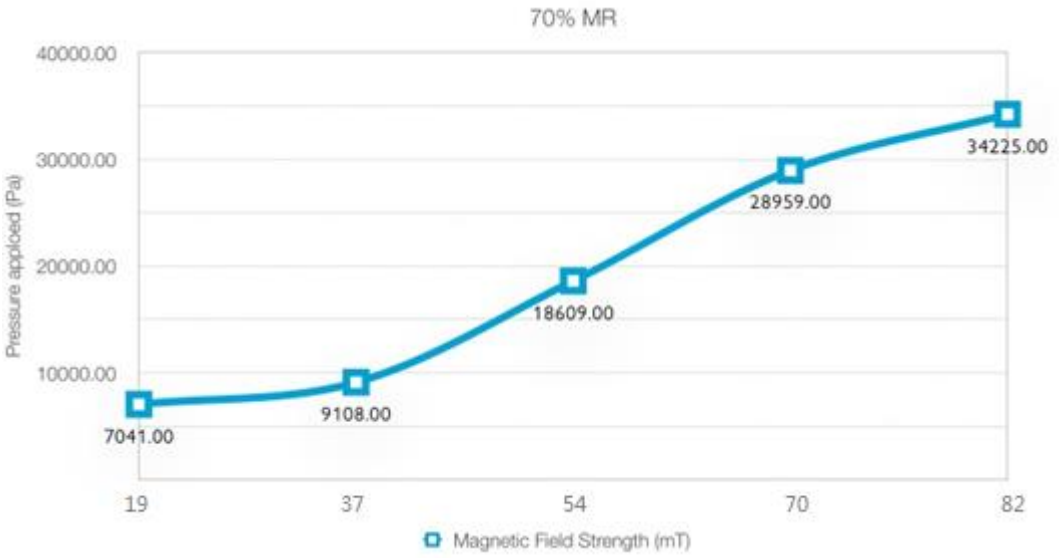

Figure 50: 70% MR Fluid (5g hydraulic fluid, 2.5g coated CIP)

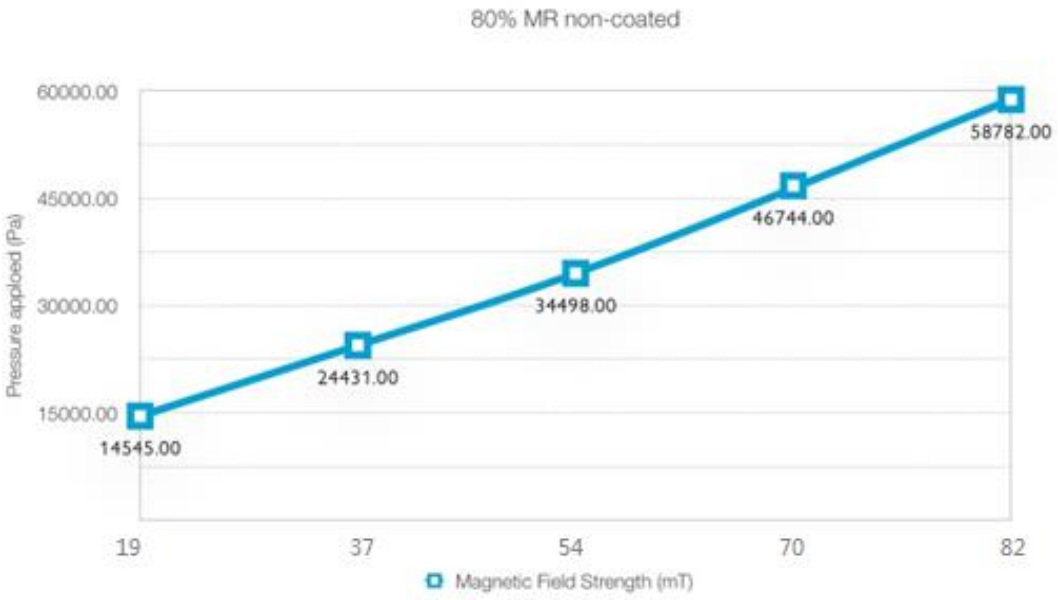

Figure 51: 80% MR Fluid (5g hydraulic fluid, 4g coated CIP)

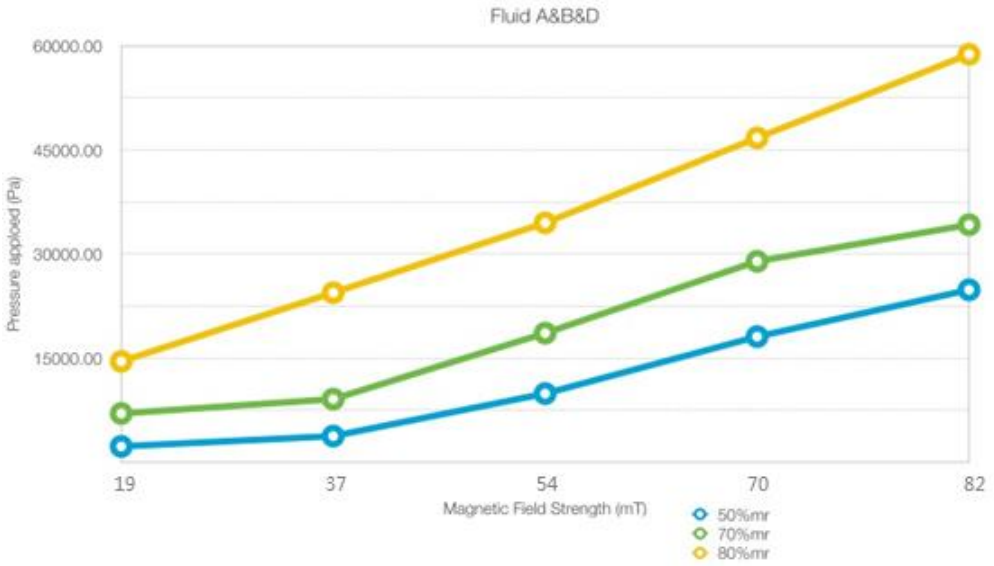

Figure 52: Comparison of Sample A&B&D

We can clearly see from Figure 50 the comparison of the three samples with different concentration of the CIP in a MR fluid, which prove concentration affects on the yield point of MR fluid surrounded by a magnetic field greatly, as the concentration increased, the yield point of MR fluid will increase too when the same value of magnetic field is applied, and the graphs do approximately follow the predicted y=kx+c, linear equation graph.

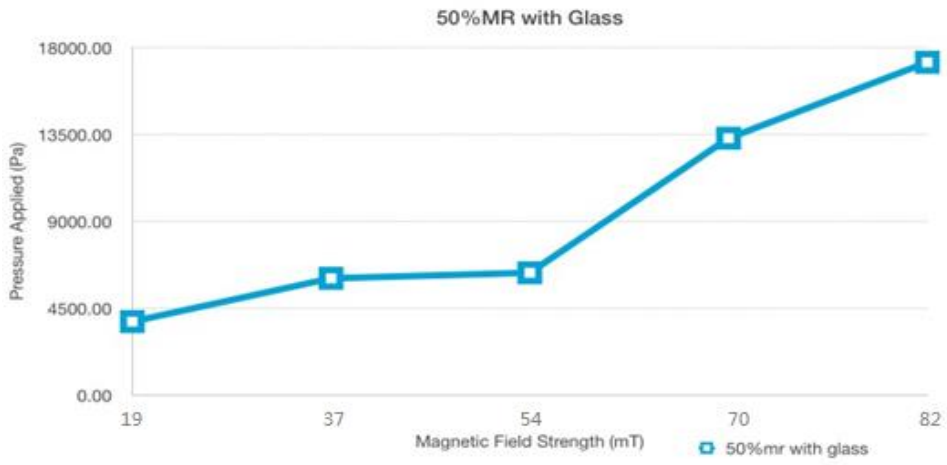

Figure 53: 50% MR Fluid with crashed glass

3g hydraulic fluid, 1.5g coated CIP and small glass particles mixture, where 25% is CIP and 25% is small glass particles.

Figure 54: 70% MR Fluid mixed with Glass

3g hydraulic fluid, 2.1g 70% coated CIP, 0.9g 30% crashed glass particles.

Figure 55: MR Fluid mixed with Ferrofluid

2g hydraulic fluid, 1.4g 70% coated CIP, 0.6g 30% ferrofluid.

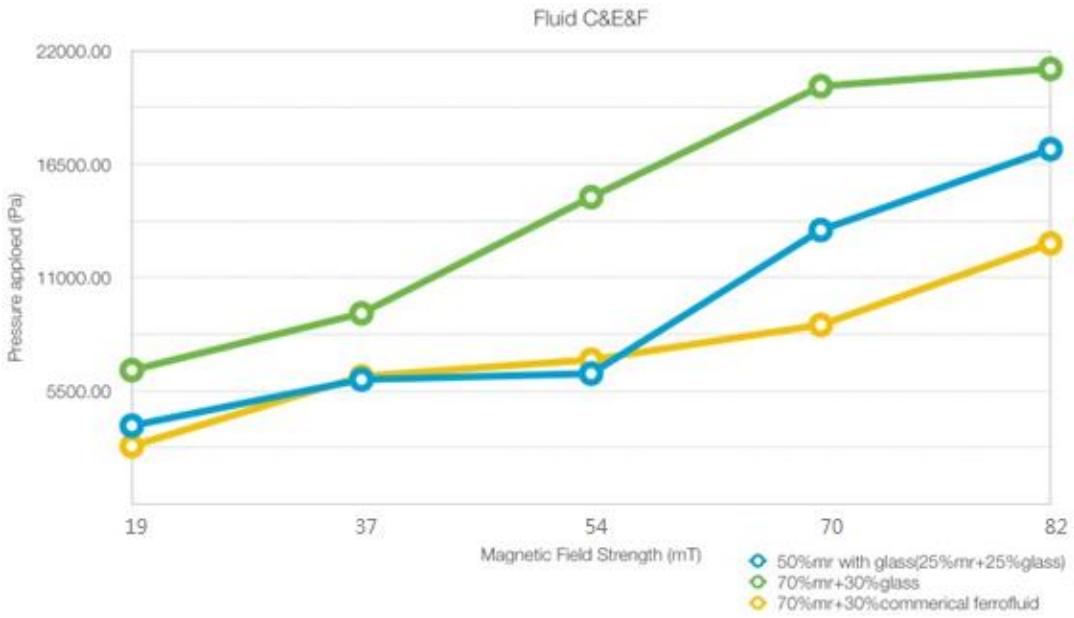

Figure 56: Comparison of Sample C&E&F

From this comparison Figure 56 we can see that the crashed glass added into the MR fluid can have the same effect as CIP particles in the MR fluid to a certain extent. I think it is because the magnetic field leads the CIP particles aligned them as a line and at the same time trigger the crashed glass tiny particles aligned as a chain structure between them. We know that glass has its own yield point, so glass particles can substitute CIP particles inside the fluid in a way. But since the glass particles cannot be magnetized, which creating weak points in the chain structures, even though the glass is being pulled into position, but according to its non-magnetic property, it weakens the bond between the neighboring magnetic MR particles. This is possibly the reason why at higher magnetic field strength the pure CIP particles fluid is far superior in yield point than using small amount of glass particles as replacement. Comparing the green curve and the yellow curve in Figure 56, we can conclude that the crashed glass that has a yield point can change the yield point of MR fluid, but the ferrofluid that has not yield point will barely affect the yield point of MR fluid.

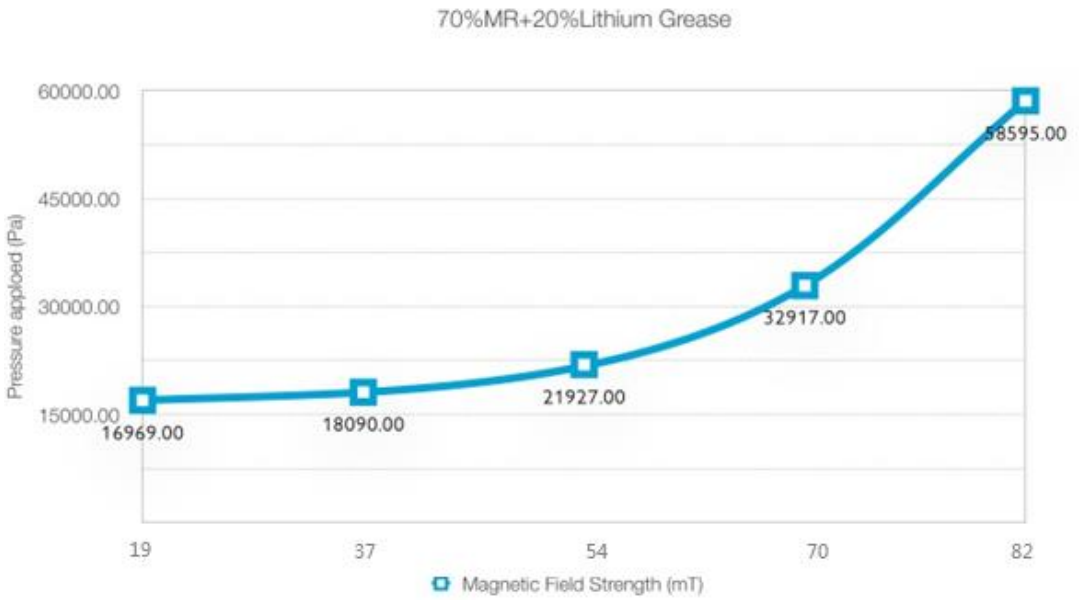

Figure 57: MR Fluid with 20% Lithium Grease

3.1g hydraulic fluid, 2.17g coated CIP and 0.62g lithium grease.

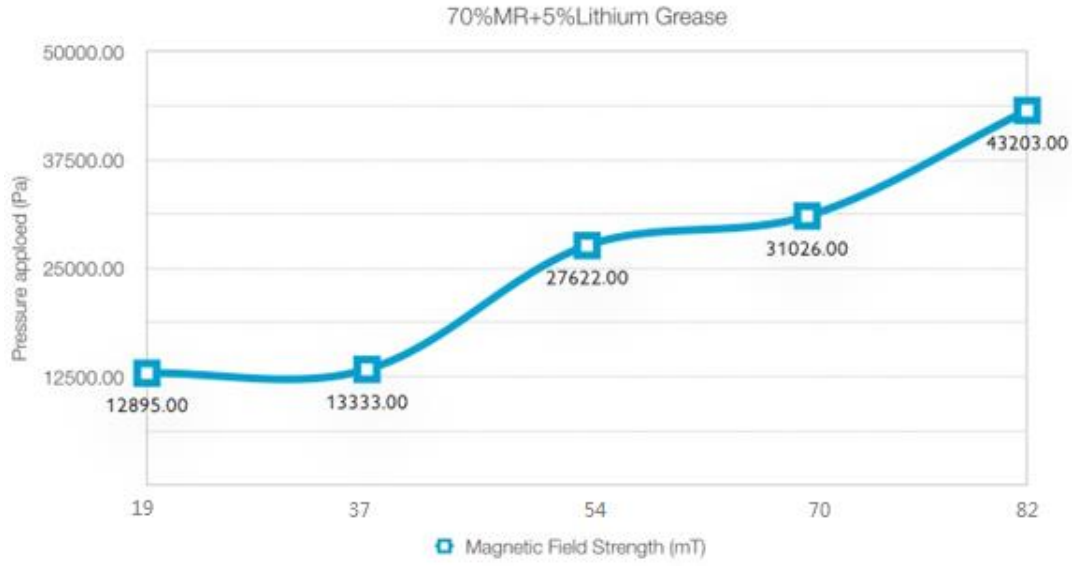

Figure 58: MR Fluid with 5% Lithium Grease

3.1g hydraulic fluid, 2.17g coated CIP and 0.155g lithium grease.

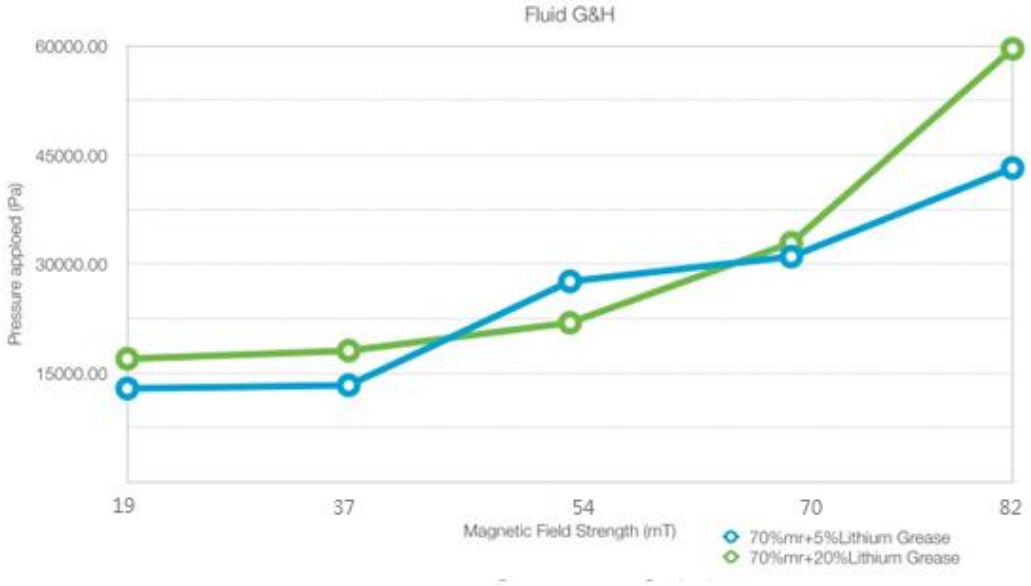

Figure 59：Comparison of Sample G&H

Lithium grease is clotted cream like high viscosity semi solid, when added into the fluid, it will increase the density of fluid instantly, from Figure 59 we can see that density does influence on the yield point of MR fluid, and as the density get higher, the yield point of the MR fluid in a magnetic field will get higher too, but the difference is not so obvious.

4.6 VSM

The VSM measurements were taken to produce a hysteresis loop for the samples. In following hysteresis loops, the x-axis has no unit value. It is because the VSM used in the experiment needed calibrating by a gauss meter. For the purpose of this experiment only the basic characteristics need to be identified and this can be done without x-axis calibration. So the following given point along the x-axis will simply be referred to as a percentage proportional to the maximum magnetic field intensity.

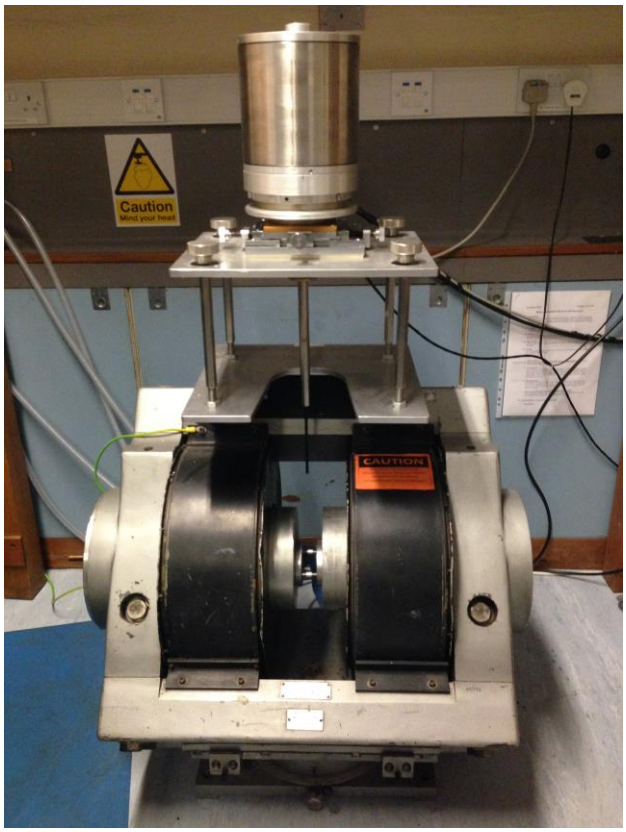

Figure 60: VSM been used in the experiment form Spintronics Lab

The VSM Results (where the Y-axis is the flux density, and X-axis is the magnetic field) are as follows:

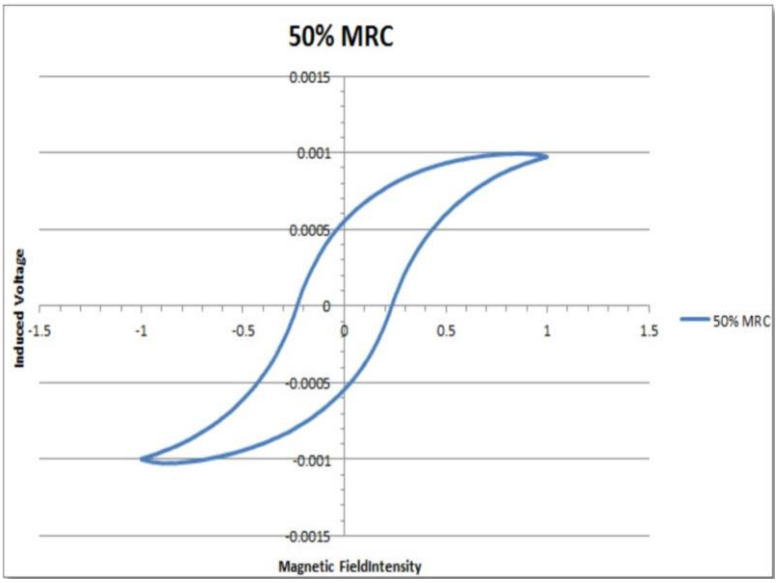

Figure 61: Hysteresis loop for 50% MR Fluid

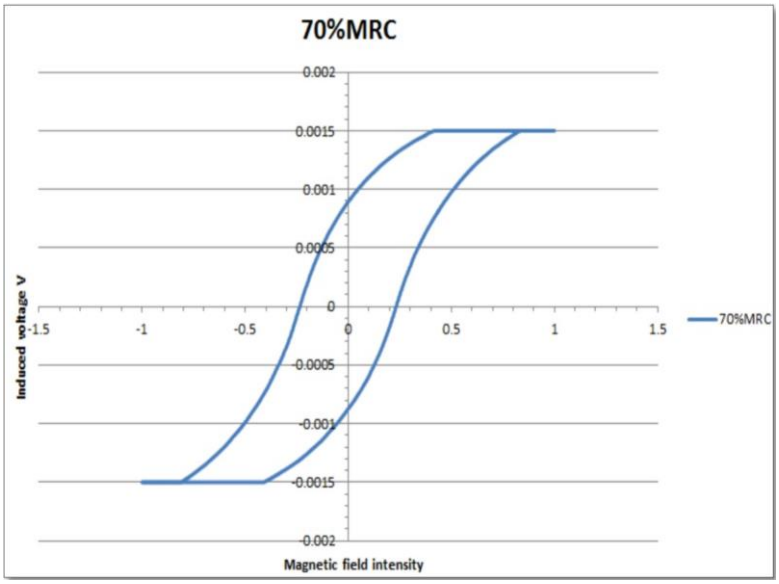

Figure 62: Hysteresis loop for 70% MR Fluid

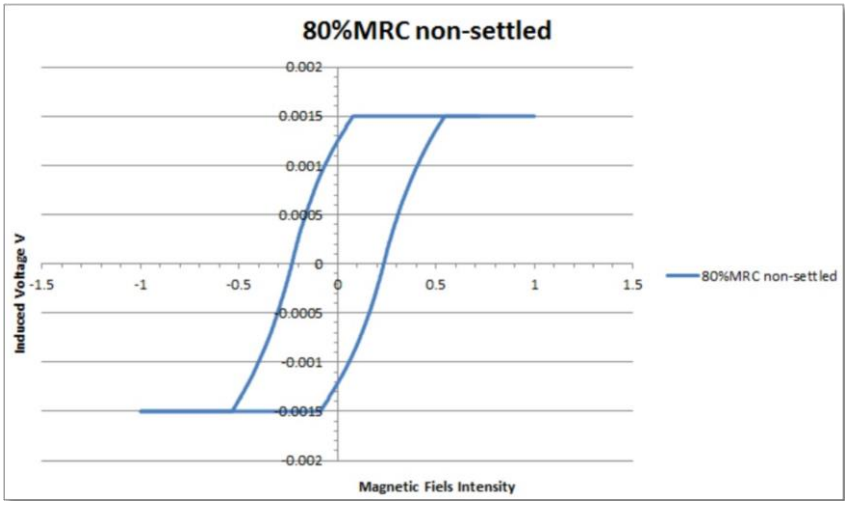

Figure 63: Hysteresis loop for 80% MR Fluid

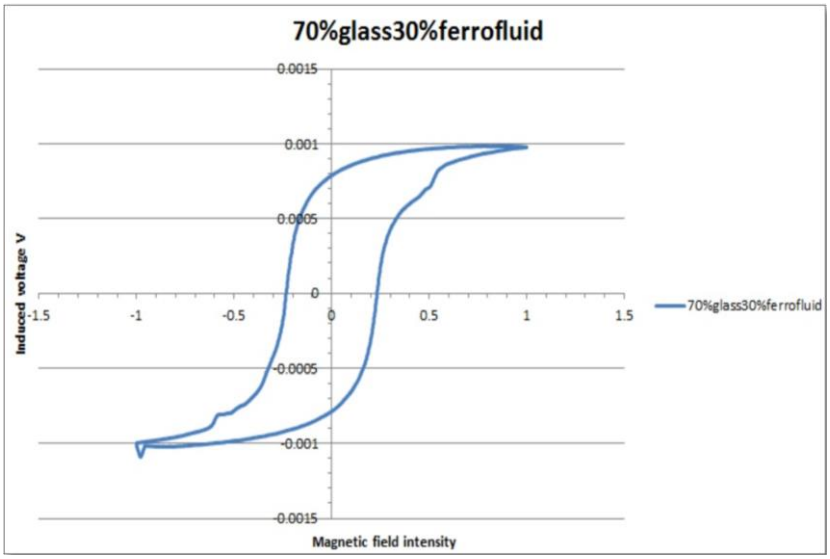

Figure 64: Hysteresis loop for 70%glass and 30% commercial ferrofluid

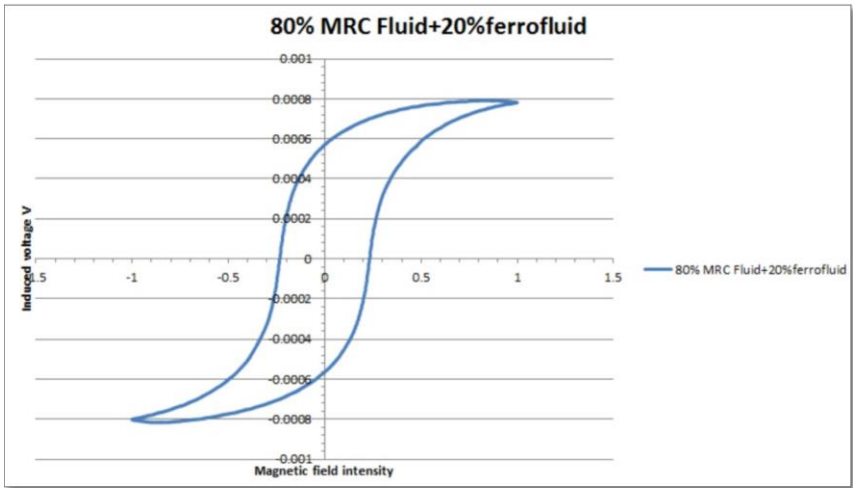

Figure 65: 80% MR Fluid with 20% ferrofluid

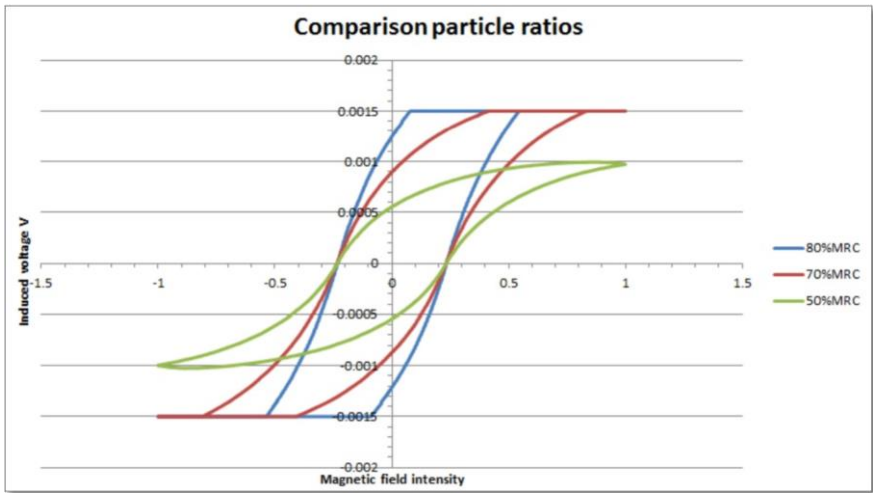

Figure 66: Comparison of three different CIP concentrations MR fluid

The results were generally satisfying as expected. These particles were too large to behave as superparamagnetic nano particles' hysteresis loop. The VSM graphs can clearly show the saturation, retentivity, coercivity of a magnetic material. what should be explained here is Figure 62 70% MR fluid and Figure 63 80% MR fluid hysteresis loop, it seems reached the maximum measurement point of the VSM, but actually I think it is because the VMS hasn't been calibrated or the sample should lose some magnetization.

Figure 64 gives the hysteresis loop of 70% glass with 30% ferrofluid, the deformities of the figure could be due to the glass particles disrupting the alignment of the nano particles inside the ferrofluid.

As we can see from the comparison picture Figure 66, three different concentrations samples, the 80% and 70% MR fluid (high concentration) possess a higher saturation than that of the 50% MR fluid, we can conclude that the ratio of particles present in the fluid greatly affect the value of magnetization that MR fluid can reach. The particle retentivity is also affected by ratio, from the comparison picture it shows the higher concentration of CIP, the higher the retentivity.

For the purpose of apply MR fluid in a commercial shock absorber, the two main characteristics: fast saturation and low retentivity should be required, the fast saturation means an indication of good response time, CIP in the fluid can quickly aligns them in a magnetic field. The low retentivity means when the electromagnetic field decreased, it will return back to liquid state which is desirable for the MR fluids magnetization to decrease proportionally, but if the MR fluid shows a high retentivity, it will trigger even the H-field (magnetic field) reach to zero, the MR fluid will still present yield stress.

4.7 Particle Settling Experiments

The particles inside the fluid are in micro scale, so it is destiny to settling down finally. We have known that the viscosity, density of a carrier liquid, surfactant, will influence the settling time of the particles in MR Fluid. But in order to know whether or not the particle ratio, magnetizing condition and the ultrasonic bath will affect the suspending duration, positively or negatively, we introduced a designed infrared device to study the influence of above parameters, and also applied a microcontroller to record the data which helped the results analysis much easier.

4.7.1 Experiment Principle

As we know, after the most suspended particles settling down to the bottom of a test tube, the top carrier liquid will turn to supernatant, this will make the test tube much more transparent and can let the beam pass through. So here we used an infrared device to detect how much beam had passed through the tube, to get when all particles had settled down indirectly.

Here we use an infrared diode to emit the infrared light passing through a sample. Then the signal will be collected by a photo receiving diode. The receiving diode converts the amount of light to a proportional voltage. The voltage readings will be stored automatically and the memory function was achieved by a programmed microcontroller. Once the testing magnetic particle sample is mixed and placed into the sample holder, and the computer interface starts. The interface will log the voltage, time and date every 5 seconds. After the voltage is stabilized, the programme will stop recording the data.

The reason that this instrument can be practical is when the fluid sample has put into the position, the voltage will drop significantly due to the grey fluid blocking the infrared beam. But over a long time the voltage will increase back towards a stable value, because most of the particles had settled to the bottom of the test tube and the top of the plastic tube will be supernatant which is much easier for light signal to pass through.

4.7.2 Experiment Design

A container was used to hold the sample and the infrared diode and the photodiode put in an complete dark location as not to be influenced by any light signals. The structure of the set-up can be seen from Figure 67.

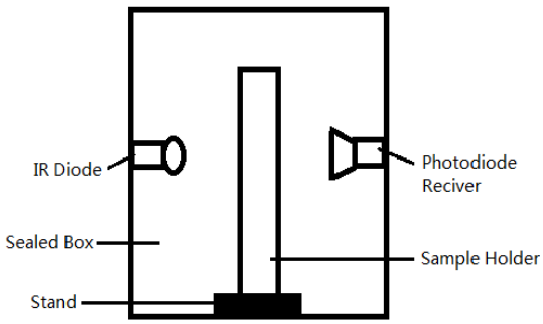

Figure 67: Sample Container

The circuit was composed by three main parts: voltage regulation, infrared sending circuit and the infrared receiving circuit. The voltage regulation section of the settling experiment circuit can be seen from Figure 68.

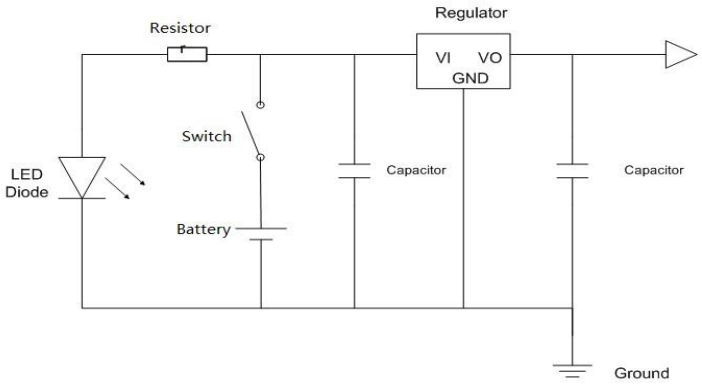

Figure 68: Power regulation and ON/OFF indicator

The infrared sending circuit was extremely simple and consisted of a current limiting resistor and an infrared diode connected directly to the 9V battery input. The infrared diode used in this section is the VISHAY-TSUS5202-IR EMITTER (Figure 90). This will emit infrared light at a peak wavelength of 950nm. This diode has a forward voltage drop of approximately 1.3V, so a current limiting resistor will be chosen as:

$$R = \frac{9-1.3}{0.1} = 77\Omega \quad (4.3)$$

This part of the circuit can be seen from Figure 69:

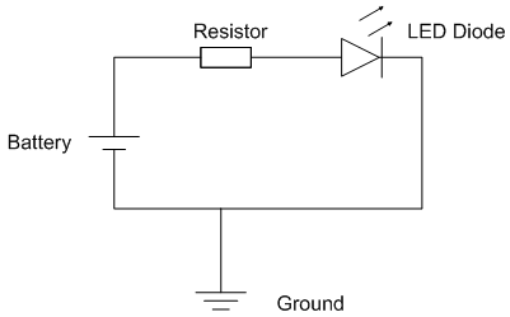

Figure 69: Infrared sending section of the settling experiment circuit

The final section of the circuit and it was used to receive the infrared signal and amplify it to a move readable value. The diode used to capture the emitted infrared signal was a VISHAY-BP104. This photodiode was specifically designed to capture 950nm wavelengths, which makes it the perfect receiver this part of circuit can be seen from Figure 70.

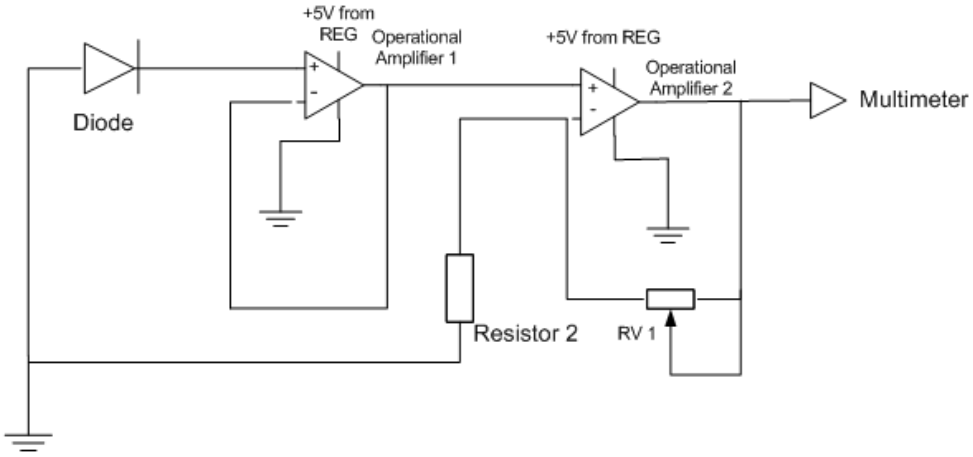

Figure 70: IR receiving section of the settling experiment circuit

The operational amplifiers applied in the circuit were an OPA2336A DUAL COMS from Texas Instruments. The microcontroller used in this experiment was FRDM-KL25Z board from FreeScale (Figure 91). The advantage of using this microcontroller to record data is that it can achieve log the data of voltage changes every set-time and plot a graph of percentage settled against time to be made

through programming. This would give us the answers of the questions such as at which point does the settling occur the fastest? Is the settling of particles linear?

4.7.3 Experiments and Results

We prepared three basic samples for testing, they were 10% 27% 47% MR Fluid respectively. The percentage is based on the quantity of the carbonyl iron powder in the hydraulic oil, the CIP (carbonyl iron powder) for this experiment was also from BASF and the hydraulic oil from Shell for shock absorber specially. Do the ultrasonic baths to those three samples, then use a strong permanent, the NdFeB magnet to magnetize 47% MR Fluid sample to study the influence of particles settling triggered by magnetization. The ultrasonic bath procedure is mainly used in material cleaning area, here the reason why we introduced ultrasonic bath is to see if the ultrasonic energy can separate the aggregated CIP particles or not, then to study its affect on the CIP particles settling.

The volume of the test tube was $4.4\,\mathrm{mm}^3$, the weight of the 4.4ml Shell suspension oil was 3.68g, and so the density ρ will be:

$$\rho = \frac{M}{V} = 0.836\,\mathrm{g/ml} \qquad (4.4)$$

The Ingredients of three testing samples are:

- 10% 3.778g oil + 0.443g CIP
- 27% 3.638g oil + 1.350g CIP
- 47% 3.388g oil + 3.065g CIP

The results of tests are showed in following figures. Y-Voltage (Volts), X-Time (Seconds).

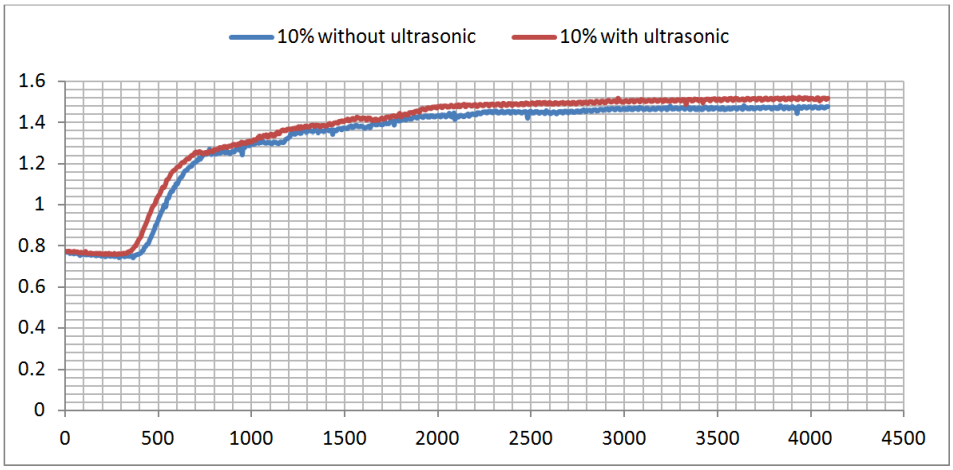

Figure 71: 10% MR Fluid without and with ultrasonic bath

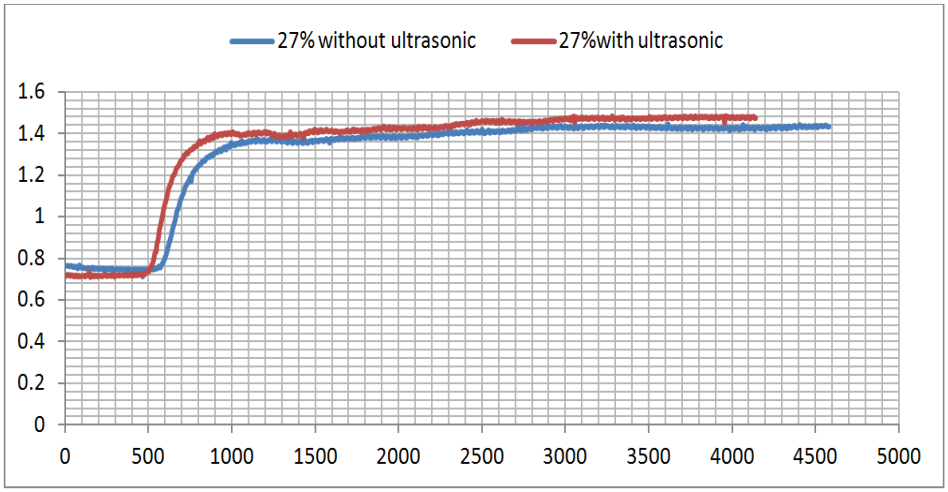

Figure 72: 27% MR Fluid without and with ultrasonic bath

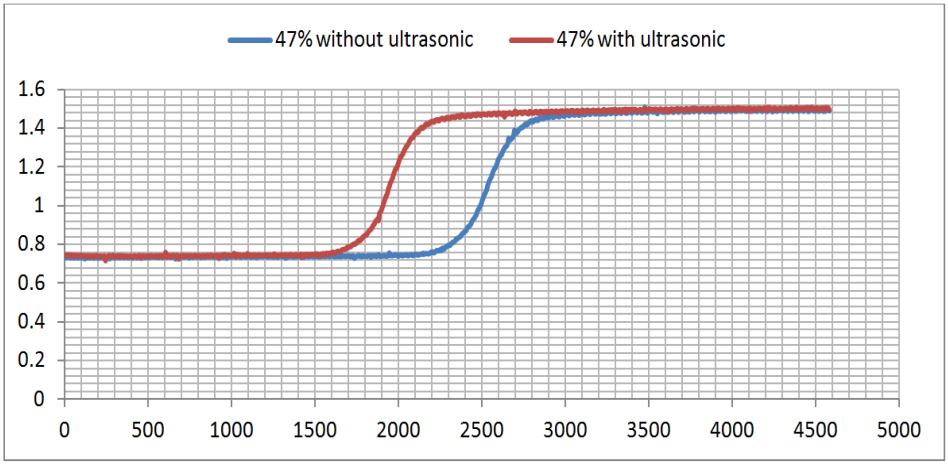

Figure 73: 47% MR Fluid without and with ultrasonic bath

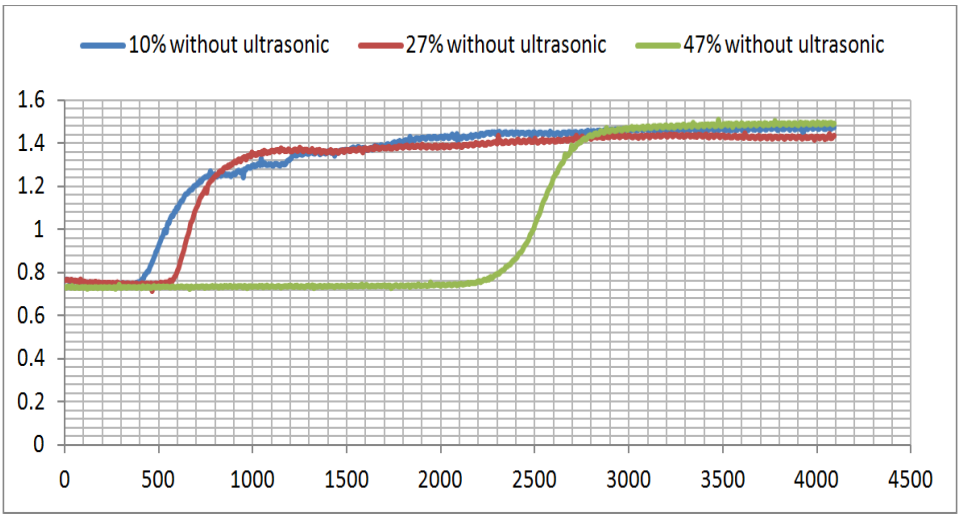

Figure 74: Comparison of 10% 27% 47% without ultrasonic

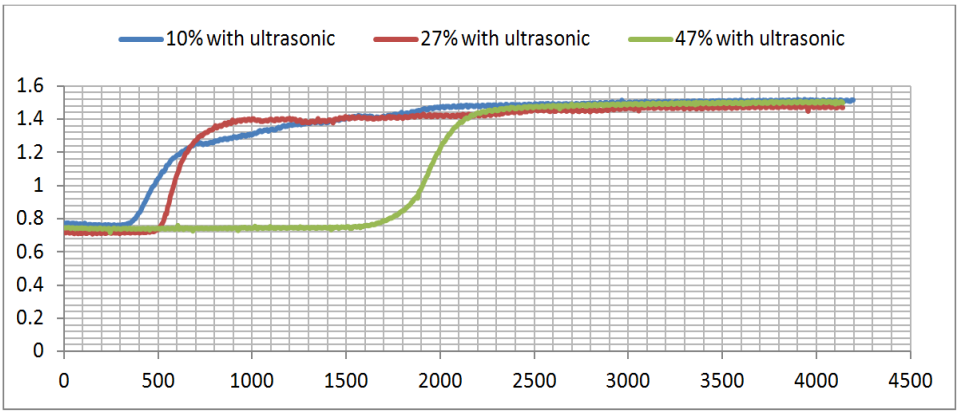

Figure 75: Comparison of 10% 27% 47% with ultrasonic

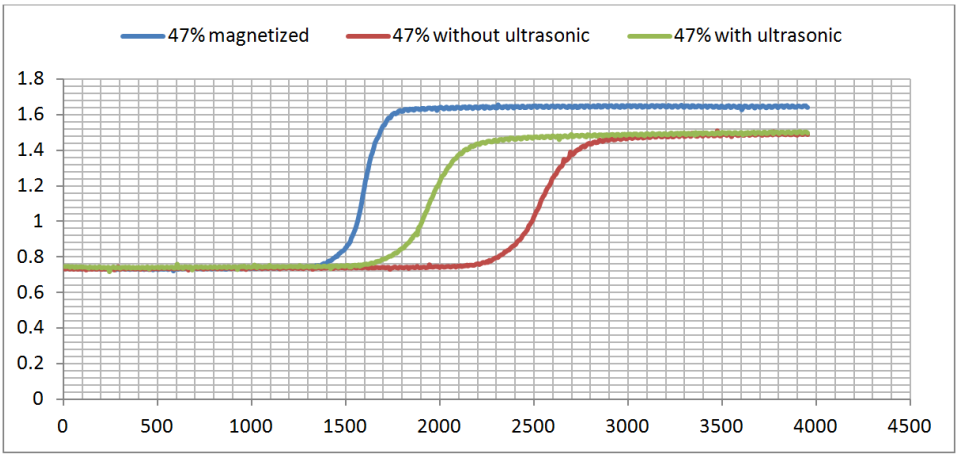

Figure 76: Comparison of 47% MR Fluid been magnetized, with and without ultrasonic bath

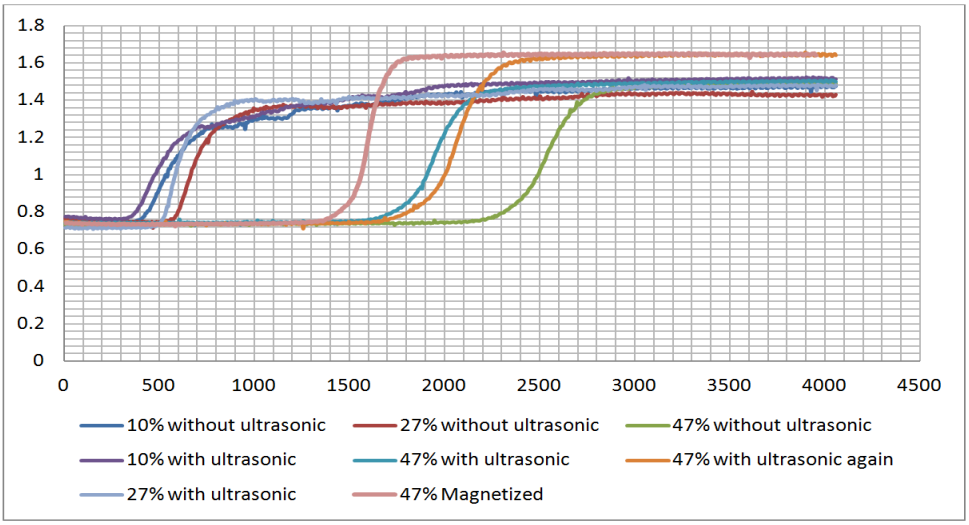

Figure 77: Comparison with all samples

After lasting the ultrasonic bath for 180 seconds, from Figure 71, Figure 72, Figure 73 we can see ultrasonic bath does influence the settling rate, but the effect was not so obvious, the samples been through ultrasonic were settling a little bit fast than the samples hadn't been through the ultrasonic bath.

From Figure 74 and Figure 75 we can say that particle ratio affected the settling time greatly, high ratio particle samples begin to settle slower compare to the low ratio particle samples, the low ratio samples began to finish settling quickly but much more gently compare with the high ratio one.

From Figure 76, the graph also clearly showed no matter with ultrasonic bath or without ultrasonic bath, the voltage can reach the same level finally, but it reached a different voltage level after the magnetized. Particles been magnetized were beginning to settle much quicker, and very dramatically. It can be explained as once particles are magnetized, most of the particles get agglomerating together and form a clump and much easier to be affected by the gravity. There must be some remnant magnetization inside the particles, and the micro scale particles are easier attracted together in the carrier liquid make the top carrier liquid more clear, then the light signal can pass through the sample holder. The light signal been received will be higher compare to samples haven't been magnetized.

And Figure 77 gives us an intuitive expression, the samples through different process will bring us a different particles settling time.

4.7.4 Brief Analysis of the Reasons Cause Particles Settling

The existence of gravity field will speed the sediment forming in the MR fluid, lead the distribution of particles concentration shows as the top is dilute and bottom is more concentrated. In the gravity field, the difference of concentration cause the particles spread from high to low. So in the gravity field, particles distribution was balanced by g-force and spread force. According to Lai Qiong Zheng's article [40], the spread force undertaken by each particle will be:

$$f' = \frac{kT}{n}\frac{dn}{dz} \tag{4.5}$$

where the f' is the spread force, k is Boltzmann constant， T is the absolute temperature, n is particle number in unit volume and z is the height.

The sedimentation force of each particle will be:

$$f = \frac{\pi}{6} = d^3(\rho_1 - \rho_2)g \tag{4.6}$$

where f is the sedimentation force, d is the diameter of particle, ρ_1 is the density of particles and ρ_2 is the density of carrier liquid, g is the acceleration of gravity.

For finally, after the settling speed get lower and lower, the spread force and sedimentation force should verge to equilibrium, so can get:

$$\frac{kT}{n}\frac{dn}{dz} = \frac{\pi}{6}d^3(\rho_1 - \rho_2)g \tag{4.7}$$

From above equations we can conclude that the settling of particles will be influenced by diameter of particles and density, even the density of carrier liquid. This had been proved in those experiments. By decreasing the particles' diameter and choose high density carrier liquid may prolong the settling time of CIP in MR fluid.

Also the settling speed equation [41] of CIP in MR fluids:

$$\mathrm{v} = \frac{[\rho_1 - \rho_1]gd^2}{18\eta} \tag{4.8}$$

Where v is relative velocity of CIP and carrier liquid, ρ_1 is the density of CIP, ρ_2 is the density of carrier liquid, and d is the diameter of particles and η is the dynamic viscosity of carrier liquid. From this equation we can know that, the smaller the diameter of CIP, lower the speed of settling will be.

5. Self-Design MR damper Piston

As we have introduced in the previous chapters, put it into another simple words, a MR damper replaces the hydraulic oil in the traditional damper with the controllable (by magnetic field) MR fluid. We tried to modify the structure of a typical shock absorber to achieve the MR fluid based shock absorber. We drew the 3D graph for the MR damper piston, by using Autodesk Inventor.

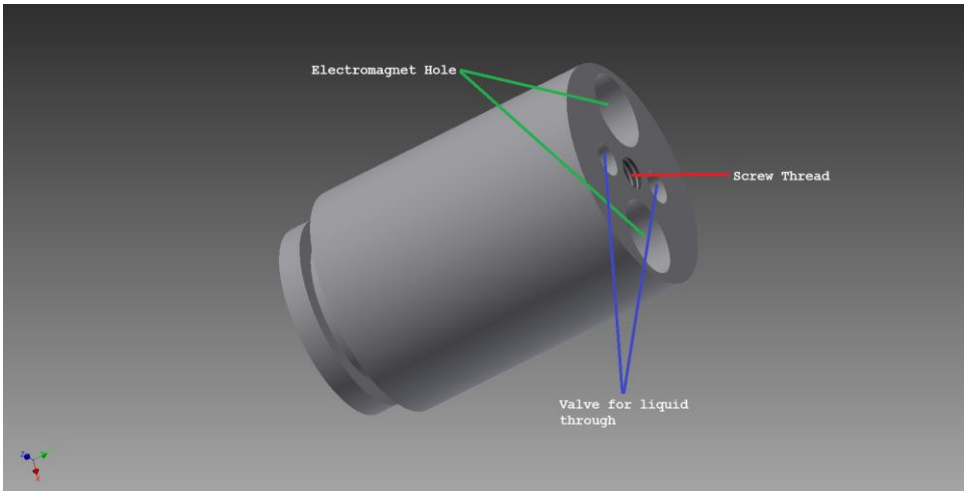

Figure 78: The general view of a bottom piston

As we can see from Figure 78, the two electromagnet holes are for insert electromagnets to create a controllable magnetic field. The two valve holes are for MR fluid to come through, and the screw thread is for the conjunction of piston and shaft (Figure 81).

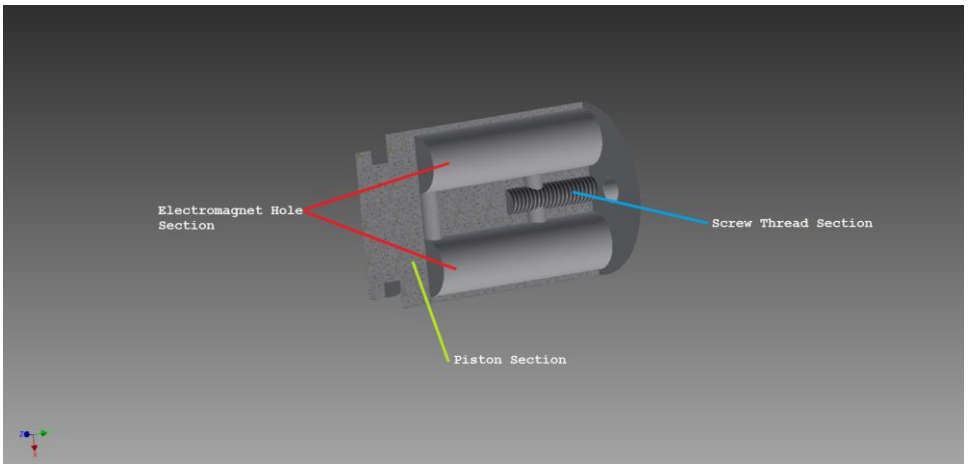

Figure 79: Cross section of piston (electromagnet holes part)

Figure 79 gives the inner view of the piston, we can see the inside structure of the piston clearly. The electromagnet holes should not run through the piston, they must be sealed in the chamber to prevent the MR fluid from sticking on the electromagnets.

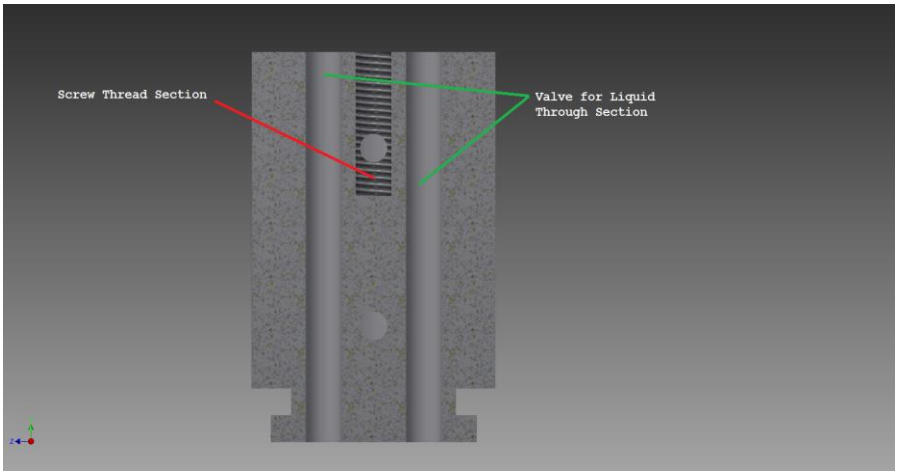

Figure 80: Cross section of piston (valve holes part)

From Figure 80, we can see the valve holes are totally through the piston, this design was to allow different states MR fluid through the piston, to adjust the mechanical properties of the shock absorber.

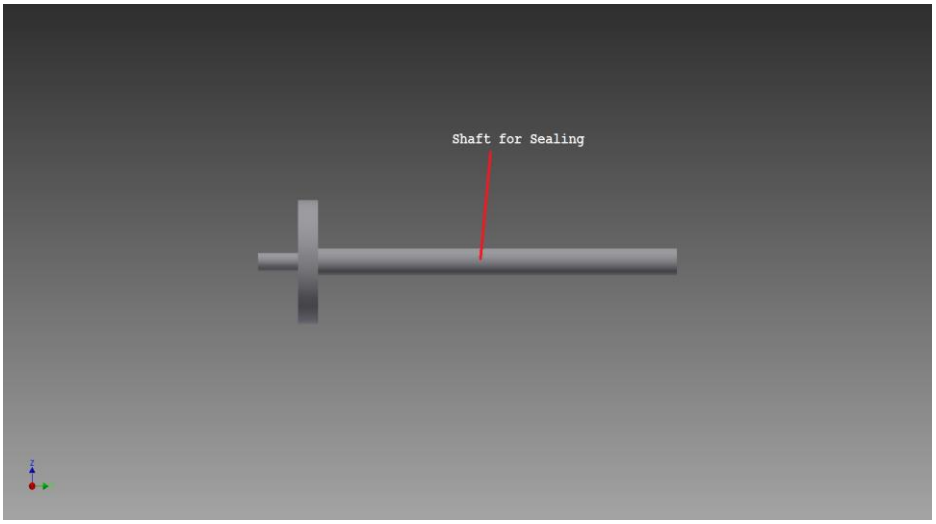

Figure 81: General view of the shaft

The cap on the left part of the shaft is for sealing the electromagnet inside the piston chamber, and the shaft is for the conjunction of piston to the outer mechanical components.

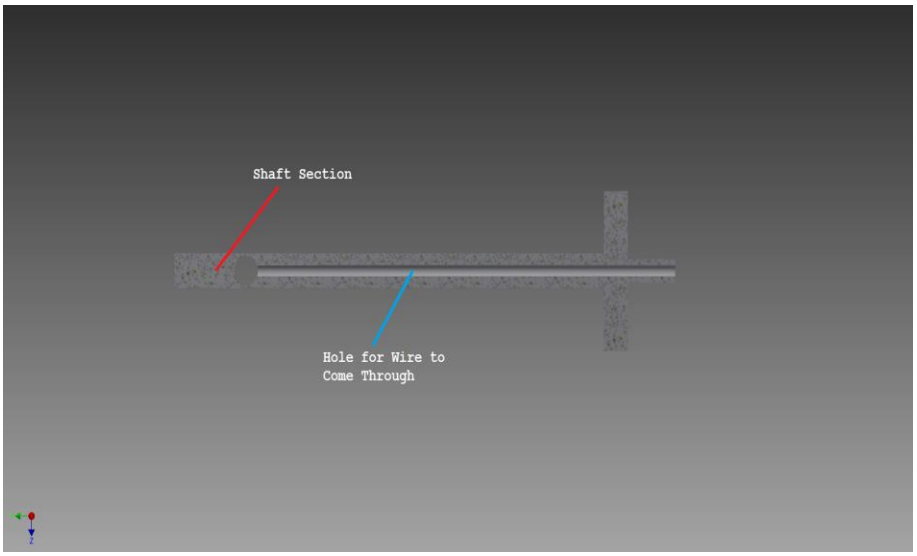

Figure 82: Cross section of the shaft

As we can see clearly from Figure 82, the inside part of the shaft is a perforation, and this is for the electromagnets' wire to come through it and lead it to the outside microcontroller.

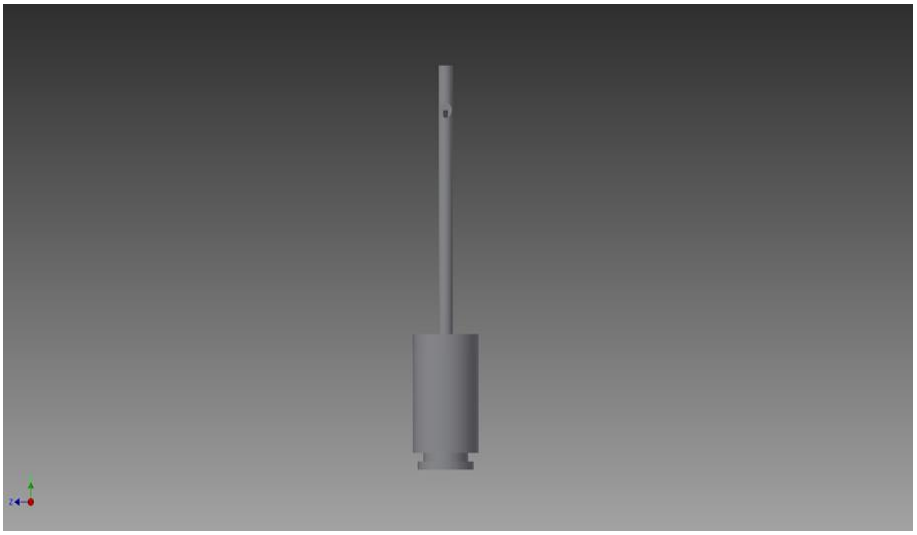

Figure 83: After assemble the piston head and the shaft together

Figure 83 is the illustration of the piston and the shaft after assembling.

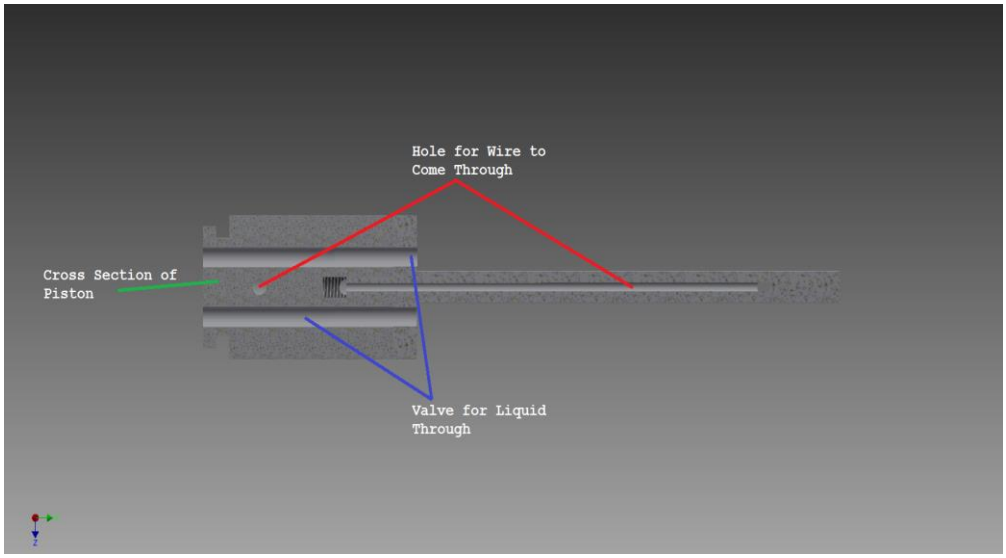

Figure 84: Cross section of the whole piston (valve holes part)

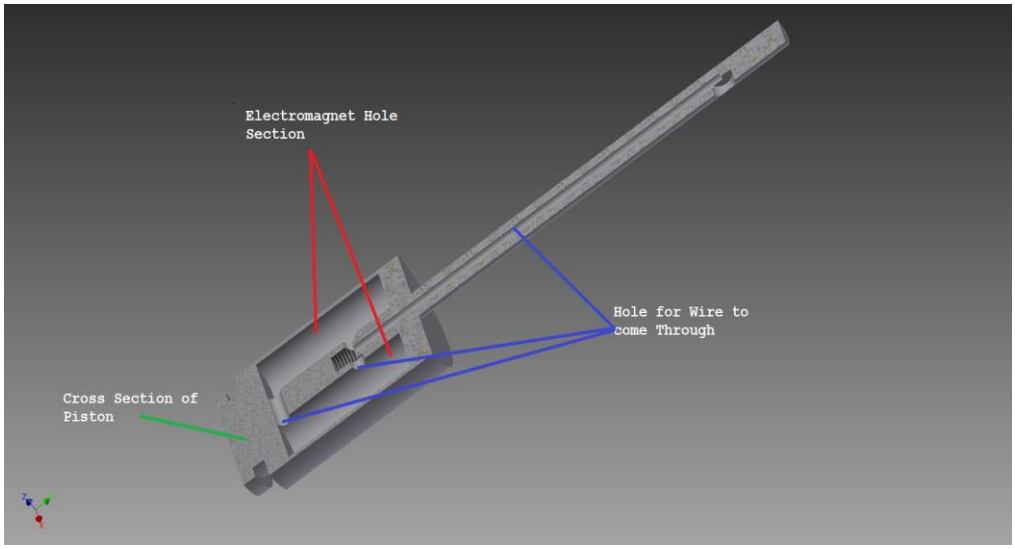

Figure 85: Cross section of the whole piston (electromagnets part)

Figure 84 and Figure 85 show the inner structure after assembling. The basic structure of MR damper piston is quite simple. It just needs slots for electromagnet and through holes for inside fluid to pass through. We may notice that there is a slot at the bottom part of the piston (Figure 85, green arrow). This is for locking the rubber there to seal the gas in the chamber and the gas is for repelling the piston back to its original position in the chamber.

To achieve a good quality MR damper, we need the assistance of the microchip controller, a good sensor system to feedback road condition to the microchip, and make the electromagnet reacts reasonably.

6. Conclusion

This research was aimed to study the properties of two commonly used magnetic fluids: ferrofluid and Magnetorheological fluid, and the application of MR fluid in modern auto shock absorber area. Before doing all the experimental work, the background research had been carried out to provide the essential and fundamental knowledge for this project including the basic theories about magnetism and magnetic particles.

The main research activities carried out during this work were:

- Preparation of magnetic nano particles
- Coating magnetic nano particle with surfactant and produce water based ferrofluid
- Synthesis of MR fluid
- Characterisation of MR fluid
- SEM, EDX, VSM, yield stress test, particles settling test of ferrofluid and MR fluid
- Design a MR fluid shock absorber

Regarding the preparation of the ferrofluid, though the properties of the product doesn't have the quality as the commercial one, the ferrofluid developed in this project does show essential characters of the ferrofluid including well defined magnetization and clear spikes in the applied magnetic field. They are also capable of being demagnetized quickly after removing the external magnetic field. From the experiments conducted during the research work, we may conclude that temperature and the concentration of reaction reagents will affect the magnetic nano particle production and properties. If the temperature of reaction is too low, it will cause the reaction to happen very slowly, and the particles been created will not be attracted by the magnet easily. During the preparation of magnetic nano particles by using co-precipitation method, the best reaction temperature will be around 70℃. Also the concentration of reaction reagents will greatly affect the final products too, the best concentration of ferrous salt solution and ferric salt solution has been found to be around 0.5mol/L, and the concentration of ferrous salt should be higher than the ferric salt to prevent small parts of ferrous salt being oxidized when exposed in the atmosphere.

When coating the particles with surfactant, it is essential to keep the temperature around 80℃ and stir the fluid continually. After the coating process had finished, the PH level of the solution should be adjusted to neutral.

The synthesis of MR fluid is relatively easier than the preparation of ferrofluid, 8 samples under various conditions had been made during the research work. From the yield stress test experiments, we can see the ratio of particles influence the yield stress greatly, as the ratio of CIP (carbonyl iron powder) in the MR fluid increased, the yield stress increased too. When the ratio of CIP reaches to 80%, the pressure that MR fluid can undertake will reach to 58782 Pa, which is quite high and can be used for engineering purpose. Also additive like crashed glass can replace a small part of CIP in the MR fluid somehow, though the samples with crashed glass showed the yield stress during the tests, but it doesn't mean that the crashed glass has the same function as CIP does in the MR fluid.

By using lithium grease to change the density of the samples, we found that as the density of MR fluid increased, the yield stress of MR fluid in a fixed magnetic field will increased either. But this change was not so obviously compared to the dramatically change when added more CIP into the MR fluid.

SEM, EDX and VSM measurements were massively time consuming. The SEM images showed that the CIP used in the experiment had uniform shape with the size around 3-5 μm, though the crashed glass particles used in the experiment cannot be distinguished by naked eyes, but it is much bigger than CIP, as showed by the SEM image, it reached to 30 μm or more. The EDX analysis showed that there was almost no impurity existing in the samples.

The particles settling experiments are important to improve the stability of MR fluid. We successfully designed an infrared monitor system and this design performed well during the study of the factors influencing the particles settling speed. The results of the experiments showed that the CIP ratio in the MR fluid, with or without ultrasonic and the states of magnetization will affect the settling speed. The low CIP ratio MR fluid started to settle earlier than the high CIP ratio MR fluid. And if the samples had been magnetized, it will trigger the particles begin to settle more quick than the samples hadn't been magnetized. We also

found that those samples after ultrasonic bath start to settle earlier than the samples haven't been through ultrasonic bath.

Finally, we designed a basic structure of MR damper by using the Autodesk Inventor, the storage format of the blue printer graph can import to 3D printer, which can illustrate the basic principle of the MRF shock absorber.

Because of the time limitation, the further work of this research has not been carried out yet, therefore, some problems still needed to be clarified and investigated, the best recipe of nano particles' preparation needed to be discovered and also the coating, synthesis method of ferrofluid should be optimized either.

7. Further Work

For current project:

To maximize the test apparatus potential, here is a list of future modifications and tests that could be conducted

- Use a digital pressure sensor instead of analogue, this would provide higher accuracy due to no analogue to digital conversion.
- Test fluids using different compound particles such as nickel or cobalt to see if they will lead a much higher yield point.
- Connect an LCD display to the microcontroller. This may eliminate the need for a computer interface, which is especially useful for the settling experiment requires a long period of time.

For further research:

Carbon Nano Tubes (CNT) is very frontier subject to study, we consider to combine CNT with nano size magnetic particles together to see if the nano magnetic particles can be created inside the CNT. As we all know the magnetic particles can react to the magnetic field, so when CNT is full of controllable magnetic particles, we can use a controllable magnetic field to manipulate CNT. This may have some good applications in the electronics field, because the CNT has excellent usage in electric conductivity. Theoretically, the metallic nanotubes can carry an electric current density of $4 \times 10^9 A/cm^2$, it is more than 1000 times greater than those of metals such as copper. When the diameter of CNT is less than 6 nanometers, it will show great conductivity and if diameter is less than 0.7 nanometer, it will show super conductivity (no resistance) [42].

According to the literature, the mass density of super-growth CNT is about $0.037 g/cm^2$, it is much lower than that of conventional CNT powders, which is $1.34 g/cm^2$, maybe because the later one contains metals and amorphous carbons [43].

The catalyst of produce CNT is nickel, etc. There must have some impurities like nickel, cobalt inside the products of CNT, so the first experiment we plan to do is CNT etching. The initial procedures could be as follows:

- Using both nitrate acid and hydrochloric acid to etch CNT respectively.
- Compare before etching and after etching, can the magnets attract the CNT sample or not, if there is nickel and cobalt in the sample, it will be attracted by the magnets. But after the etching, the metals turn to salt solution, which cannot be attracted by the magnets.
- Do EDX and SEM to analysis the presence of Nickel and other metal elements.

When etching experiment is done, we prepare the magnetic particles by co-precipitation method and during the process we add the CNT inside it, the specific details will be further deliberated.

After the combination, we do some measurements to the samples including the VSM, SEM, EDX and the most important one is TEM image, this will see if the nano particles have been put into the CNT successfully or not.

8. Appendices

Figure 86： The SEM used in the experiment

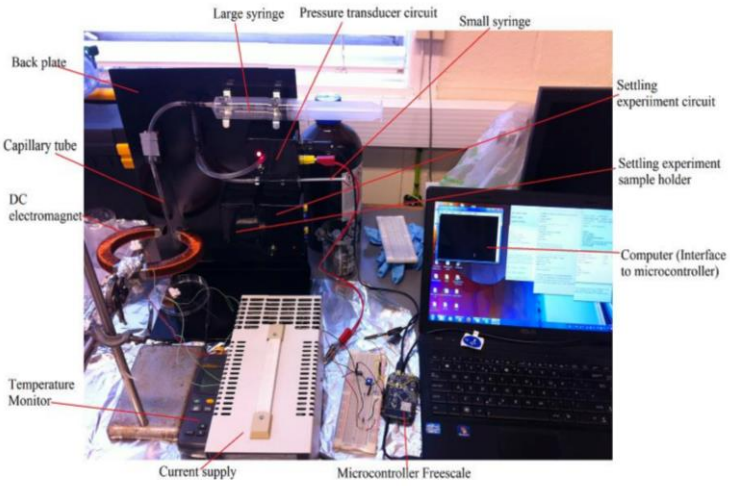

Figure 87: The yield stress test experiment setup

Figure 88: Circular Electromagnet of spintronics lab

Figure 89: Magnetometer

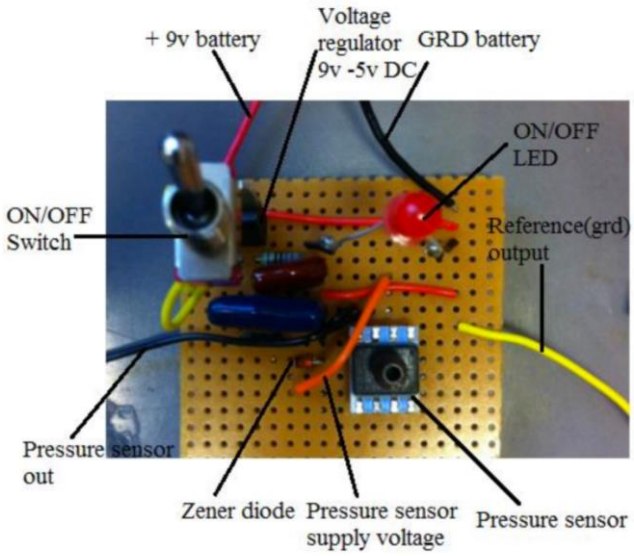

Figure 90: Circuit board of pressure sensor

This circuit was designed by James Romaine.

Figure 91: FRDM-KL25Z board from Freescale.

In the experiment the FRDM-KL25Z board from Freescale was used. This microcontroller uses an ARM cortex A0 processor is purposely designed to allow for rapid prototyping applications. The FRDM-KL25Z, which can be seen in Figure 91below is situated on one single board and is powered by a micro USB cable.

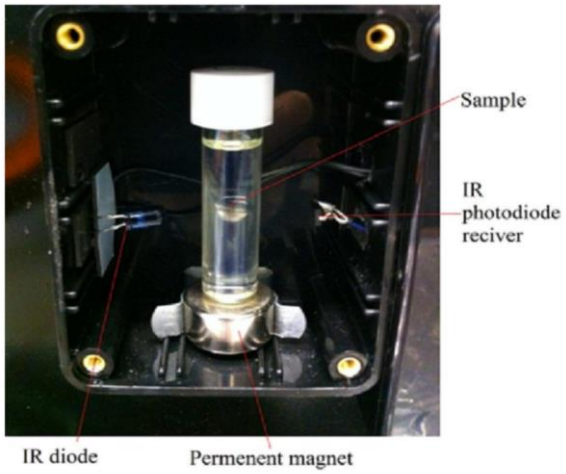

Figure 92: The particles settling experiment setup

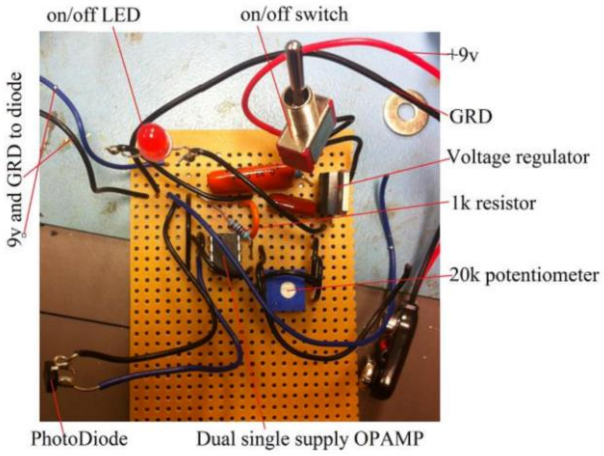

Figure 93: The circuit board of settling experiment

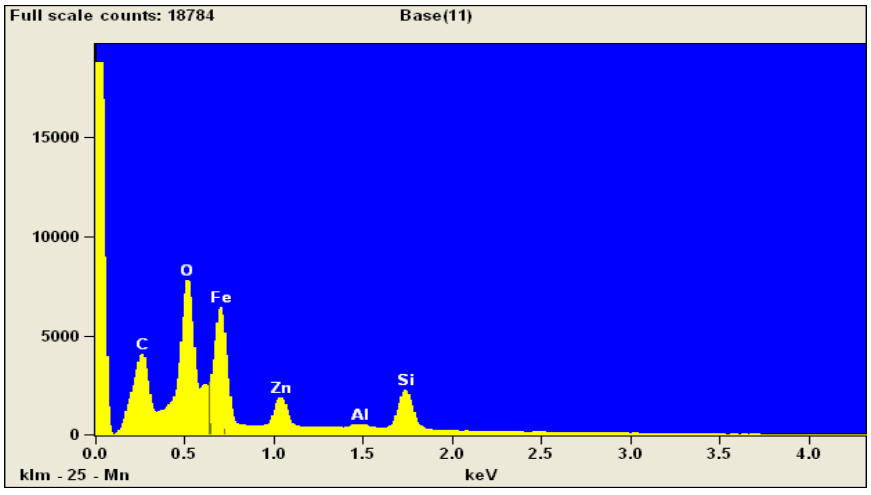

Figure 94: The EDX graph of the testing sample (Glass and CIP)

9. Definitions:

D.I Water: De-ionised water

SEM: Scanning Electron Microscopy

TEM: Transmission Electron Microscopy

VSM: Vibrating Sample Magnetometer

EDX: Energy-Dispersive X-ray Spectroscopy

MRI: Magnetic Resonance Imaging

MRF: Magnetorheological Fluid

FF: Ferrofluid

CIP: Carbonyl Iron Powder

CNT: Carbon Nanotubes

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