Magnetic Properties and Corrosion Study on Bonded NdFeB Magnet Using Wet and Dry Binders

by

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Dissertation submitted in partial fulfilment of
the requirements for the
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or person.

(Syahidatulmazidah binti Ismail)

ABSTRACT

Bonded NdFeB magnet is made from a mixture of NdFeB powder and polymer binder which undergo compression to obtain the shape required. With the advantages of outstanding mechanical strength, high accuracy in dimension and easy manufacturing process, these magnets have good resistant to shock and usually further machining is not required. They also possess excellent stability and evenness of magnetic flux density as well as much higher corrosion resistance in comparison with sintered NdFeB. As an extension to this proclamation, a study need to be done to determine the properties of bonded NdFeB magnets achieved when different types of binder are being used. This paper investigates the magnetic properties and corrosion behaviour of bonded NdFeB magnet when different types of binders were used during the fabrication process. Two types of polymer binders were used which are wet binder- liquid epoxy and dry binder – phenolic powder. The magnetic properties of the magnets were measured using vibrating sample magnetometer (VSM) at applied magnetic field up to 30 kOe while corrosion behaviour analysis was done in salt water environment using weight loss method. The obtained results reveal that the magnetic property of epoxy-bonded magnet is higher than phenolic-bonded magnet. On the other for corrosion behaviour analysis, it was revealed that phenolic-bonded magnet have better corrosion resistance than epoxy-bonded magnet.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

NdFeB, namely Neodymium (Nd) – Iron (Fe) – Boron (B) is the new generation of rare-earth permanent magnets and the strongest permanent magnets to date, has not only the excellent qualities of high remanent magnetization [8] , high coercive force [6] and high magnetic energy product but also the advantages of being easy to process and a relatively high performance to cost ratio. Since the alloy is made from Neodymium, one of the most plentiful rare-earth elements and inexpensive iron, NdFeB magnets offer the best value in cost and performance [14].

Polymer bonded NdFeB magnet is produced through the process of forming and solidification [15] of mixed magnetic NdFeB powder and polymer adhesive or binder. In this study, compression bonded is adopted to fabricate the magnet. Although the highest magnetic property is offered by sintered NdFeB which is between 33 MGOe to 51 MGOe [14], bonded NdFeB magnet can still offer good energy property which is between 4 MGOe to 12 MGOe [12]. With the advantages of outstanding mechanical strength and high accuracy in dimension obtained from the fabrication process, they compliment the magnetic properties of bonded NdFeB magnet and subsequently lead to the increase in demand of this type of magnet.

In order to fulfil the high demands of bonded NdFeB magnet, a thorough effort needs to be done in terms of simplifying its fabrication process. One of the ways to do this is to replace the polymer binder used to make the magnet. Currently, wet polymer binder is used as a binder for NdFeB powder. However as the material technology is expanding, more types of polymer binder had been introduced in the market which give more options to fabricator in choosing more reliable and manageable material.

1.2 PROBLEM STATEMENT

Since the fabrication process of bonded NdFeB magnet using wet binder is more tedious because of the requirement to use acetone during the mixing process and wait for the mixture to vaporise and become paste-like form, there had been some idea of replacing the wet binder with the dry one. This is one of the way to reduce the production cycle time which directly may lead to increase in the magnets production rate. However, the idea of using dry binder is still in consideration because the unknown effect on the properties of bonded NdFeB magnet. Although the usage of dry binder is favourable because of the easiness to handle dry material as compared to the wet one, it is necessary to determine if there will be any negative effect on the magnet properties prior to the change in binder.

1.3 OBJECTIVES AND SCOPE OF STUDY

The main objective of this study is to determine the magnetic properties and corrosion behaviour of bonded NdFeB when different types of polymer material are used as binder. To be specific the target of this study is to determine the effect of using wet and dry binder on the magnetic property and corrosion resistance of the magnet so that at the end of day this study would be able to give clarification to the manufacturer on the advantage and disadvantages of using wet and dry binders on magnet properties.

The scope of study will be focusing on obtaining the properties of bonded NdFeB magnet using liquid epoxy and phenolic powder as the wet and dry binder. Besides that, the scope of study will be guided by parameters that had been specified in the methodology part.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 MAGNET BACKGROUND

Magnet is a material that can exert a noticeable force on other materials without actually contacting them. This force is known as a magnetic force and may either attract or repel [28] the material that they act onto. All material has different magnetic forces and materials that having much larger magnetic force are referred to as magnets.

2.1.1 Types of Magnets

Permanent Magnets

This kind of magnet exerts a force on objects without any outside influence. The iron ore magnetite, also known as lodestone, is a natural permanent magnet. Other permanent magnets can be made by subjecting certain materials to a magnetic force. When the force is removed, these materials retain their own magnetic properties. Although the magnetic properties may change over time or at elevated temperatures, these materials are generally considered to be permanently magnetized [28].

Electromagnets

Electromagnets are made by surrounding certain materials with a coil of wire. When an electric current is passed through the coil, these materials exert a magnetic force. When the current is shut off, the magnetic force of these materials drops to nearly zero. Electromagnet materials retain little, if any, magnetic properties without a flow of electric current in the coil [28].

2.2 PERMANENT MAGNETS

The permanent magnet family consists of non-rare earth permanent magnets like Alnico (Aluminum-Nickel-Cobalt) magnets and Ceramic (Strontium and Barium Ferrite) magnets as well as rare earth magnets like SmCo (Samarium-Cobalt) magnets and NdFeB magnets.

2.2.1 Alnico Magnets

Alnico magnetic material is an alloy of aluminum-nickel-cobalt which possesses an excellent temperature stability and high residual induction. However, its low coercive force limits its applications in many cases. Casting and sintering are two major processes used to manufacture the Alnico magnets. Alnico magnets with complex shapes may be manufactured by casting. However, once the Alnico magnets are formed, it is difficult to machine or drill them due to the hard and brittle mechanical properties of Alnico [26].

2.2.2 Ceramic Magnets

Ceramic permanent magnets are made from barium ferrite or strontium ferrite formed under heat and pressure. Their magnetic strength is enhanced by aligning the powder particles with a strong magnetic field during forming. Ceramic magnets are comparable to Alnico magnets in terms of magnetic force and have the advantage of being able to be pressed into various shapes without significant machining [28].

2.2.3 Flexible Magnets

Flexible permanent magnets are made from powdered barium ferrite or strontium ferrite mixed in a binding material like rubber or a flexible plastic like polyvinyl chloride [23]. Flexible magnets can be either isotropic or anisotropic. The anisotropic flexible magnets are made by extrusion or injection molding.

2.2.4 Rare-Earth Magnets

Rare-earth magnets are strong, permanent magnets made from alloys of rare earth elements. They are substantially stronger than Ferrite or Alnico magnets. The magnetic field typically produced can be in excess of 1.2 teslas compared to ferrite or ceramic magnets typically exhibit fields of 50 to 100 milliteslas [14]. Two types of material from rare-earth group are SmCo and NdFeB.

Samarium-Cobalt

SmCo magnets are less common than Neodymium magnets because they are more expensive to produce and create a weaker magnetic field than Neodymium magnets. However, Samarium-cobalt magnets have a relatively high Curie point, which makes them suitable for high-temperature environments. [14].

Neodymium-Iron-Boron

Neodymium magnets are the strongest and most affordable type of rare-earth magnet. Neodymium magnets are made of neodymium, iron and boron. They have the highest magnetic field strength, but are inferior to SmCo in resistance to oxidation and temperature [14].

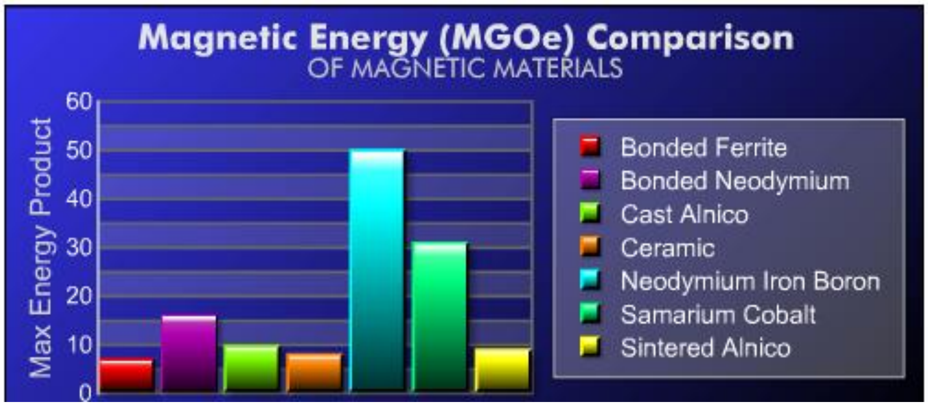


Figure 2.1: Comparison of Magnetic Material [21]

2.3 NEODYMIUM-IRON-BORON MAGNET

An NdFeB magnet is a powerful magnet and very strong in comparison to their mass. Like other ferromagnetic materials, neodymium magnets lose their magnetism above a temperature known as the Curie point. But the most powerful grades lose their magnetism at a relatively low temperature: 80°C (176°F) and above. High-temperature grades will operate at up to 200 and even 230°C but their strength is only marginally greater than that of samarium-cobalt magnets [20].

2.3.1 Range of Strength

Neodymium magnets are graded in strength from N24 to the strongest, N54. The theoretical limit for neodymium magnets is grade N64. The number after the N represents the magnetic energy product, in megagauss-oersteds (MGOe) (1 MGOe = 7,958 T·A/m = 7,958 J/m^3). N48 has a remnant static magnetic field of 1.38 teslas and magnetic field intensity, H of 13,000 oersteds (1.0 MA/m). By volume one requires about 18 times as much ceramic magnet material for the equivalent magnet lifting strength, and about 3 to 5 times as much for the equivalent dipole moment [20].

2.3.2 Forms of NdFeB Magnets

NdFeB magnets are available in sintered as well as bonded forms. The bonded form of the material can be produced with close tolerances off tool, with little or no finishing required. The energy product of the bonded form is much lower than that of the sintered form which can go up to 12 MGOe [12]. The sintered form usually requires some finishing operations in order to hold close mechanical tolerances.

2.3.3 Magnet Fabrication Process

Sintering

In sintering process, fine NdFeB powder is compacted in a die and then sintered, fusing the powder into a solid material. There are two forms of pressing which are die pressing (it involves a hard die into which the powder is placed and then pressed), and isostatic pressing (it involves a special "rubber" die into which powder is placed and then pressed with equal force in all directions on the powder). Although the magnetic properties of isostatically pressed parts are higher, the uniformity of magnetic characteristics is usually lower than that of die pressed parts. Sintered parts normally need some finish machining in order to meet final tolerances [17].

Compression Moulding

This is a technique whereby a special form of NdFeB powder is blended with a plastic carrier material, die pressed and then heated. Parts made in this way can be of complex shapes and come off the tool with close tolerances, requiring no further finish machining [17]. They have lower energy products than sintered materials where falls in the range of 10 to 12 MGOe [12]. The Bonded NdFeB materials are isotropic thus they can be magnetized in any direction.

Injection Moulding

For injection molded type of magnet, NdFeB powder is blended with a plastic material and injection molded. The resulting parts usually shall have energy products in the range of 5 MGOe. One advantage of this process is the magnets can be made with extremely intricate shapes [17].

Extrusion

Though not popular at present, NdFeB based flexible magnets can be made by binding NdFeB powder with a carrier material and extruding the material in sheet or strip form. Energy products up to about 6 MGOe have been achieved in this way [17].

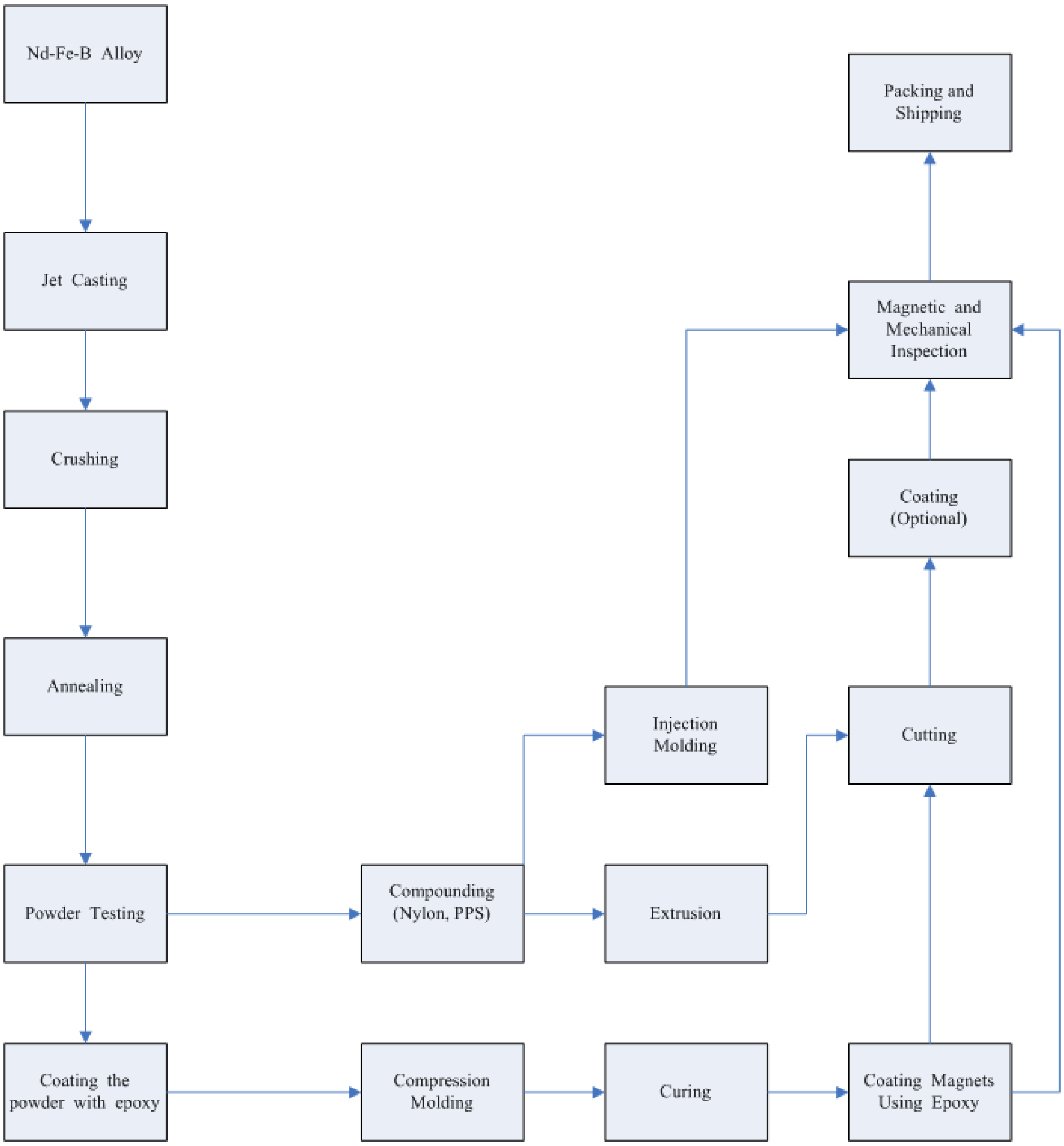


Figure 2.2: Typical Manufacturing Process of Bonded NdFeB Magnet [16].

2.4 BONDED ND-FE-B MAGNET

2.4.1 Magnetic Property

As mentioned in previous section, bonded NdFeB promise a reliable magnetic property to the customer. However, there are slight differences for the properties between bonded magnets that undergo different manufacturing process. Table 2.1 and Table 2.2 below will highlight the properties of polymer bonded NdFeB using compression moulding as well as injection moulding fabrication process. These property are taken from *Stanford Magnets Company* [26], a magnet manufacturer that has been involved in research, development and manufacturing of rare earth permanent magnets since the middle of 1980.

Table 2.1: Properties of Polymer Bonded NdFeB Magnets by Compression Bonded

Grade	B_r (KGs)	H_c (KOe)	H_{ci} (KOe)	$(BH)_{max}$ (MGOe)	Density (g/cm^3)	Recoil Perm.	Temp. Coeff. of Br (%/°C).	Max. Op. Temp(°C)
BNP-6	5.2-6.0	3.8-4.5	8.0-10	5-7	5.3-5.8	1.15	-0.13	140
BNP-8	6.0-6.5	4.5-5.5	8.0-12	7-9	5.6-6.0	1.15	-0.13	140
BNP-10	6.5-7.0	4.5-5.8	8.0-12	9-10	5.8-6.1	1.22	-0.07 ~ -0.105	120
BNP-12	7.0-7.6	5.3-6.0	8.0-11	10-12	6.0-6.2	1.22	-0.13	130
BNP-8H	5.5-6.2	5.0-6.0	12-16	6-9	5.6-6.0	1.15	-0.07 ~ -0.105	120

Table 2.2: Properties of Polymer Bonded NdFeB Magnets by Injection Molding

Grade	B_r (KGs)	H_c (KOe)	H_{ci} (KOe)	$(BH)_{max}$ (MGOe)	Density (g/cm^3)	Recoil Perm.	Temp. Coeff. of Br (%/°C).
BNI-2	2.0-4.0	1.5-3.0	7.0-9.0	0.8-3.0	3.5-4.0	1.25	-0.13
BNI-4	4.0-4.9	3.1-3.9	7.2-9.2	3.5-4.5	4.0-5.0	1.20	-0.10
BNI-6	4.9-5.7	3.9-4.8	8.0-10.0	5.2-7.0	5.0-5.5	1.20	-0.10
BNI-8	5.7-6.3	4.8-5.4	8.5-10.5	7.4-8.4	5.0-5.5	1.20	-0.10
BNI-8H	4.8-5.6	4.2-5.0	13-17	5.0-6.5	5.0-5.5	1.13	-0.15

Isotropic and Anisotropic Magnets

During data gathering process, there are several times that the author has come into across with these terminologies. For clarification purpose, some reading had been done and the findings are; isotropic tells that the properties of a material are identical in all directions [25] while for anisotropic, the properties of a material depend on the direction [25]. To relate it with the study, isotropic magnets is a magnet material which magnetic properties are the same in any direction, and therefore be magnetized in any direction without loss of magnetic characteristics while anisotropic magnet is a magnet having a preferred direction of magnetic orientation, so that the magnetic characteristics are optimum in that direction [26].

2.4.2 Compression Bonded NdFeB Magnets

While ferrite still dominates the market, bonded rare earth magnets are becoming more and more important due to their superior magnetic properties. Besides that there are also some other factors that lead to increasing demand for bonded NdFeB magnet in the industry. The following are some advantages and disadvantages of this compression polymer-bonded magnet.

Advantages

From the manufacturing perspective, a major advantage of bonded magnets is they can be made by net-shape or near net-shape process. Thus, tight tolerances can be held without secondary or finish machining [8] which significantly reduces the production cost allowing for competitive pricing [22] Although making compression bonded NdFeB requires higher loading than injection molded but it is still lower than sintering process, which create a compromise in energy product. Besides that, thin wall cylinder magnet can be manufactured using compression bonding where they are not practical with sintering process due to warpage during sintering and breakage during grinding.

From the material perspective, NdFeB is relatively abundant resource [8] with large proven reserves and its refining give the manufacturer moderate to low cost process. It also promises the highest magnetic output of all commercially available permanent magnet material. The second important material for bonded NdFeB is definitely polymer binder. A broad selection of polymer binders and polymer additives in the market has gives the flexibility for production and meets the requirements of various applications [22].

Another advantage is, isotropic bonded magnets can be easily magnetized into various and complex magnetization patterns [8, 22]. Fully automated production processes lead to more uniform magnetic properties and lower cost.

Disadvantages

On the down side, the manufacturing process of compression bonded NdFeB magnets is limited to simple geometries like rectangle, cylinders and arc. The magnets produced also have lower magnetic properties compared to their sintered counterparts due to polymer dilution effect [8, 22]. The maximum operating temperature is limited, to some extent, by the temperature characteristics of polymer binders. One more disadvantage is NdFeB have a high tendency to corrode when compared with samarium cobalt or ferrite based magnet. Thus, it is imperative to obtain material from quality manufacturer and specify coating that can reduce corrosion risk in any application.

2.4.3 Application of Bonded NdFeB Magnets

Bonded NdFeB magnets have been incorporated into numerous end market applications. The normal applications of these magnets range from below -40°C and beyond 165°C [24]. In 2005, the total bonded NdFeB magnet market volume was approximately 4000 tons [26].

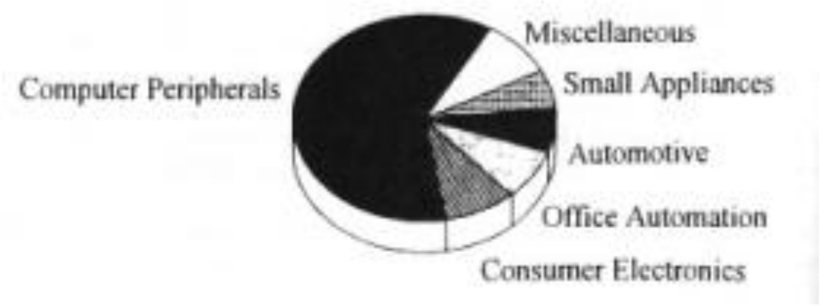


Figure 2.3: Breakdown of applications of bonded NdFeB [12]

High performance bonded magnets are used in various electronic devices like personal video recorders & MP3 music players, office automation equipment like fax, photocopy machine and printer, automotive components like instrument panel motors and air bag sensors, home appliances like hard disk drives [8], scanners, CDs, DVDs, sensors, transducers, magnetic bearings, optical disk drive motors, step motors, servo motors, horologe motors and other motor or motion related applications [15, 27].

Bonded NdFeB has also been used in the applications that require higher performance, smaller size, greater efficiencies and reduced costs than which was achieved with ferrite magnets. These "ferrite replacement" applications include air conditioner fan motor, power tool motors, automotive seat motors and many others [26].

Figure 2.4: Application of Bonded NdFeB

2.5 THEORY OF MAGNETISM AND MAGNETIC PROPERTY ANALYSIS

2.5.1 The B-H Curve

The basis of magnet design is the B-H curve, or hysteresis loop, which characterizes each magnet material. This curve as seen in Figure 2.5 describes the cycling of a magnet in a closed circuit as it is brought to saturation, demagnetized, saturated in the opposite direction, and then demagnetized again under the influence of an external magnetic field [18].

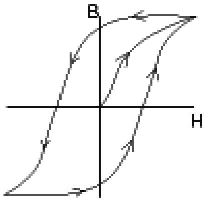


Figure 2.5 Typical B-H Curve

The second quadrant of the B-H curve, commonly referred to as the "Demagnetization Curve", describes the conditions under which permanent magnets are used in practice. A permanent magnet will have a unique, static operating point if air-gap dimensions are fixed and if any adjacent fields are held constant. Otherwise, the operating point will move about the demagnetization curve, the manner of which must be accounted for in the design of the device [18].

The three most important characteristics of the B-H curve are the points at which it intersects the B and H axes (at B_r - the residual induction - and H_c - the coercive force - respectively), and the point at which the product of B and H are at a maximum (BH_{max} - the maximum energy product). B_r represents the maximum flux the magnet is able to produce under closed circuit conditions. In actual useful operation permanent magnets can only approach this point. H_C represents the point at which the magnet becomes demagnetized under the influence of an externally applied magnetic field. BH_{max} represents the point at which the product of B and H, and the energy density of the magnetic field into the air gap surrounding the magnet, is at a maximum. The higher this product, the smaller need be the volume of the magnet.

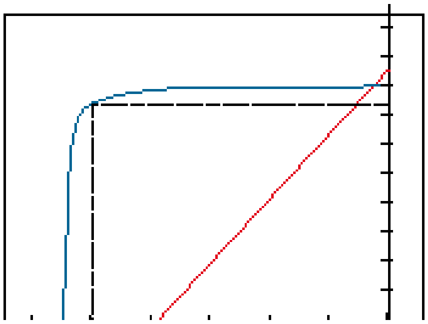


Figure 2.6 B-H Curve in First Quadrant

When plotting a B-H curve as in Figure 2.6, the value of B is obtained by measuring the total flux in the magnet (ø), dividing this by the magnet pole area (A) to obtain the flux density (B=ø/A). The total flux is composed of the flux produced in the magnet by the magnetizing field (H), and the intrinsic ability of the magnet material to produce more flux due to the orientation of the domains. The flux density of the magnet is therefore composed of two components, one equal to the applied H, and the other created by the intrinsic ability of ferromagnetic materials to produce flux.

The intrinsic flux density is given the symbol B_i where total flux B = H + BI, or, BI = B - H. In normal operating conditions, no external magnetizing field is present, and the magnet operates in the second quadrant, where H has a negative value. Although strictly negative, H is usually referred to as a positive number, and therefore, in normal practice, BI = B + H. It is possible to plot an intrinsic as well as a normal B-H curve.

The point at which the intrinsic curve crosses the H axis is the intrinsic coercive force, and is given the symbol H_{ci}. High H_{ci} values are an indicator of inherent stability of the magnet material. The normal curve can be derived from the intrinsic curve and vice versa. In practice, if a magnet is operated in a static manner with no external fields present, the normal curve is sufficient for design purposes. When external fields are present, the normal and intrinsic curves are used to determine the changes in the intrinsic properties of the material [18].

2.5.2 Magnetic Property Analysis

From the research done, there are several methods that had been used by other researchers to come out with the magnetic property NdFeB Magnet.

Method I

The density of the sample was measured by the Archimedes method. The magnetic properties were measured using a B–H tracer after premagnetization with a 90 kOe field. The microstructural evolution was analyzed using a field emission scanning electron microscope [1].

Method II

Magnetic properties were measured at room temperature by using a vibrating sample magnetometer at applied magnetic fields up to 15 kOe. The microstructure was examined by X-ray diffraction and by transmission electron microscopy (TEM) [2].

Method III

The magnetic properties were determined from the demagnetization curves measured on a permeameter. The microstructure of the magnets were examined and characterized on a Jeol 6300 SEM with Energy Dispersive X-ray (EDX) analysis [3].

Method IV

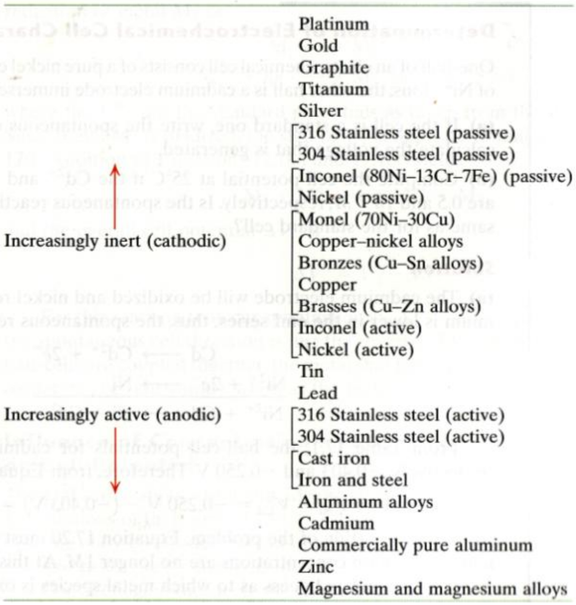
The microstructure of the samples was characterized by an X-ray diffractometer (XRD) using Cu Kα radiation and a transmission electron microscopy (TEM). The samples used for recording the demagnetization curves were magnetized to saturation in a pulse field of 6 T, prior to measurement. A VSM was used to measure the magnetic properties [4].

2.6 THEORY ON CORROSION AND CORROSION BEHAVIOR ANALYSIS

2.6.1 Corrosion of Metal

Corrosion is defined as the destructive and unintentional attack of a metal; it is electrochemical and ordinarily begins at the surface. The problem of metallic corrosion is one of significant proportions; in economic terms, it has been estimated that approximately 5% of an industrialized nation's income is spent on corrosion prevention and the maintenance as a result of corrosion reaction. For metallic materials, the corrosion process is normally electrochemical, that is, a chemical reaction in which there is transfer of electrons from one chemical species to another. Metal atoms characteristically lose or give up electrons in what is called an oxidation reaction. The electron generated will be transferred to and become part of another chemical species in what is termed as reduction reaction. Table 2.3 below represents the relative reactivities of number of metals and commercial alloys in seawater. The alloys near the top are cathodic and unreactive whereas those at the bottom are most anodic or prone to corrosion in seawater environment.

Table 2.3 Galvanic Series. Source: M.G. Fontana, Corrosion Engineering, 3rd Edition, Copyright 1986 by McGraw-Hill Book Company.



Increasingly inert (cathodic)

Increasingly active (anodic)

Platinum
Gold
Graphite
Titanium
Silver
316 Stainless steel (passive)
304 Stainless steel (passive)
Inconel (80Ni–13Cr–7Fe) (passive)
Nickel (passive)
Monel (70Ni–30Cu)
Copper–nickel alloys
Bronzes (Cu–Sn alloys)
Copper
Brasses (Cu–Zn alloys)
Inconel (active)
Nickel (active)
Tin
Lead
316 Stainless steel (active)
304 Stainless steel (active)
Cast iron
Iron and steel
Aluminum alloys
Cadmium
Commercially pure aluminum
Zinc
Magnesium and magnesium alloys

2.6.2 Corrosion Behaviour

Polymer bonded magnets are one of the fastest growing segments of the permanent magnet market, due to lower cost and good shape-forming ability. The development of bonded magnets has not only eliminated the need for a sintering step in the process, but has also allowed the introduction of rapid solidification techniques for producing new NdFeB alloys. However, corrosion resistance remains as major problem, and most previous work dealing with corrosion has been performed on sintered NdFeB magnets rather than bonded.

Some authors have suggested that the poor corrosion resistance of NdFeB materials is due to the reaction of an Nd-rich material with humid air or hydrogen, either as a pure gas or as a by product of the decomposition of water. Alloying sintered magnets with elements such as CO, V, Cr or Ni improves corrosion resistance but may also degrade magnetic properties. Recent investigations have shown that melt spun NdFeB materials containing TiC yields finer grain sizes by controlling nucleation, smaller amounts of Nd-rich material, and results in little or no degradation of magnetic properties. TiC also enhances the glass-forming ability of the alloy and allows optimum cooling rates to be achieved at lower wheel speeds. A similar finding has also been reported for NdFeB powders produced by gas atomization [10].

Other method that also used to enhance the corrosion resistance is to apply effective coating material such as nickel plating, zinc plating and organic coating. More recently, works have been performed on phosphating of NdFeB magnets and its effect on their corrosion resistance. Chromating of this type of magnet has also been used for corrosion protection but this treatment generates toxic and carcinogenic products and the other types of environmentally friendly conversion coating layers have been studied in order to replace the chromate coatings due to the toxicity of Cr^{+6} [7].

2.6.3 Corrosion Behavior Analysis

In this study we will determine the corrosion behavior of bonded NdFeB magnets when different types of polymer are used as binder. A few methods had been used by previous researchers to obtain the corrosion behavior of their matter of study.

Method I

The electrochemical tests were conducted using a three electrodes cell with a platinum counter electrode and saturated calomel reference electrode (SCE mV). The working electrode was a disc electrode with rotation speed of 720 rpm. Potentiodynamic polarization measurements were performed in 0.1 M sulfuric acid (pH 1), in phthalate buffer (pH 5), and in 0.1 M sodium hydroxide (pH 13) using a scan rate of 0.5 mV/s [6].

Method II

A saturated calomel electrode (SCE) was used as reference electrode and all potentials are referred to it. The auxiliary electrode was Pt gauze. The experiments were carried out under naturally aerated conditions at 25 °C. The electrochemical measurements were performed using a potentiostat coupled to a frequency response analyzer. All measurements were performed in potentiostatic mode at the open circuit potential, OCP. The OCP after potential stabilization is referred in this work as the corrosion potential, E_{corr}. The corrosion resistance of all magnets was evaluated in 0.1 mol L−1 Na_2SO_4 and 0.01 mol L−1Na_2SO_4 solutions [7].

Method III - Autoclave Test

Accelerated corrosion test is conducted by placing carefully weighted and sized specimens in an autoclave partially filled with deionized and distilled water. The autoclave was first pressurized to a steam pressure of 10–15 psi for 5 minutes, released to expel residual air in the chamber, and repressurized to 12 psi (100% RH) for 100 hours. Samples were then removed, dried in air, and weighed again after carefully removing corrosion products with a brush. Bulk corrosion rates in mg/cm were determined from the ratio of weight loss to the initial external surface area [5].

Method IV

An EG&G Model 273 potentiostat and computer were used to perform anodic potentiodynamic polarization curves (20 mV/s sweep rate) in ambient, deaerated 0.9 M Na2S04 at two pH levels, 4.4 and 6.5. Corrosion rates were also determined by measuring iron dissolution versus time during potentiostatic (constant potential) experiments at -600 mV vs. SCE. Galvanostatic charge/discharge experiments were carried out on selected samples in 6M KOH by charging with hydrogen for 30 min (-1 mA cathodic current) then discharging (+1 mA anodic current). Accurate measurement of the active surface area, necessary for determining current density, was accomplished by means of a Nikon Epiphot-TME metallograph and Cue-2 Image Analyzer software. Corrosion potential **(**E_{corr} vs. SCE) and corrosion current (i_{corr}) were determined using EG&G M352 software [10].

2.6.4 Corrosion Rates

The corrosion rate, or rate of material removal as a consequence of the chemical reaction, is an important corrosion parameter. This may be expressed as the corrosion penetration rate (CPR), or thickness lost of material per unit time. The formula for this calculation is

$$CPR = \frac{KW}{\rho At}$$

where W is the weight loss after exposure time t; ρ and A represent the density and exposed specimen area, respectively, and K is a constant, its magnitude depending on the system of units used. The CPR is conveniently expressed in terms of either mil per year (mpy) of millimeter per year (mm/yr). In the first case, K=534 to give CPR in mpy (where 1 mil = 0,001 in.), and W, ρ, A and t are specified in units of milligrams, gram per cubic centimeter, square inches, and hours respectively. In the second case, K= 87.6 for mm/yr, and units for the other parameters are the same as mils per year, except that A is given in square centimeters. For most applications, a corrosion penetration rate less than about 20 mpy or 0.5 mm/yr is acceptable

Inasmuch as there is an electric current associated with electrochemical corrosion reactions, we can also express corrosion rate in terms of this current or more specifically current density - that is, the current per unit surface area or material corroding- which is designated *i*. the rate r, in units of ol/m2-s is determined using the expression

$$r = \frac{i}{nF}$$

where, again, n is the number of electrons associated with the ionization of each metal atom, and F is 96500 C/mol.

CHAPTER 3

METHODOLOGY / PROJECT WORK

At the beginning of the project several literatures were collected to gain main ideas and information about NdFeB, its fabrication process, characteristics possessed as well as variables affecting its properties. From the revision that had been done, experimental procedures were then identified which covers the preparation of bonded NdFeB magnet samples as well as performing analysis which will be focusing on the magnetic properties and corrosion resistance of the magnets. This is the most crucial part of the study where the procedures will determine the experiment results obtained. Finally the result had be compiled and analyzed to conclude the study.

3.1 PLANNING

Planning work was done during the early week of the first semester. Within the period, several process are involved like identification of supervisor and project title, submission of project title, confirmation of project title by the coordinator and work plan for that semester. For the second semester, the works for this project were focusing on the practical work like sample preparation and analysis. A meeting had been done with the supervisor to plan some activities that should be done to complete the project within the specified time range.

3.2 STUDY AND RESEARCH

A lot of revisions were involved during study and research work. The subject matter for the study and research varies from the background of magnet, the different types of magnet available, the current market trend, the characteristic of bonded NdFeB, the magnetic properties and corrosion behaviour of the magnet, the advantages and disadvantages of bonded NdFeB as well as to the application of NdFeB magnet. All the information was gathered from journals, online articles, books and manufacturers/ suppliers websites.

3.3 EQUIPEMENT AND TOOL FAMILIRIZATION

During the project execution, several tools had been used to aid the project progress. For the time being, tools that had involved are mixer, balance max (500 g), auto pressed machine, automatic mounted machine, optical microscope and scanning electron microscope (SEM). For the project purpose a training session was conducted by the supervisor in order to familiarize on how to use the equipment and setting the variable value for each process.

3.4 SAMPLE PREPARATION

The sample of Bonded NdFeB magnets were prepared by using compression bonded process. To produce two kinds of magnets, NdFeB powder had been mixed with liquid epoxy and phenolic powder which will act as wet and dry polymer binder respectively. The mixtures were then compressed to form cylindrical-shaped magnet sample. The detailed experimental procedures to fabricate both types of magnet are as follow:

Procedures of Wet Sample Preparation

1. 210µm of NdFeB powder was weighed for 95g
2. 5g of epoxy binder was weighed
3. Both powder and binder are mixed together with 3g hardener and acetone. The mixture was then stirred until the acetone evaporated and left the mixture in paste form.
4. The mixture was then divided by several fractions which every fraction is 3g each.

5. The fraction was then compressed in a die using Auto Pressed machine. The compression load used was 5 tonnes.
6. The sample prepared were next cured in an oven for 4 hours at 65°C
7. The dimension and weight of each sample was measured to calculate the density of the samples

After preparing the wet sample, the author moved towards preparing the magnet samples using dry binder. The material that was used as dry binder is phenolic powder which is usually used to mount sample for sample metallography analysis purposes. A few discussions had also been done to identify process variable and parameter. As a result it had been decided to fabricate the magnet by using hot mounting machine.

Procedures of Dry Sample Preparation

1. Phenolic powder was first sieved to gain a fine powder size
2. 210µm of NdFeB powder was weighed for 95g
3. 75µm of phenolic powder was weighed for 5g
4. Both powder and binder are mixed together until homogeneous mixture is achieved
5. The mixture was then divided by several fractions which every fraction is 10g each.
6. The fraction was then compressed using hot mounting machine. The compression load used was 4000 psi and simultaneously, heat is applied at 350°F.
7. The dimension and weight of each sample was measured to calculate the density of the samples.

3.5 SAMPLE ANALYSIS

In this project, two properties had been focused on which are magnetic properties and corrosion behaviour of epoxy-bonded magnet and phenolic-bonded magnet.

The first step of the analysis was to calculate the density of both types of magnet samples. For this purpose, 6 samples of epoxy-bonded magnet and 6 samples of phenolic-bonded magnet had been prepared. Each sample is then measured to get the thickness, diameter and weight. The densities were then calculated by dividing the sample volume with its weight.

Then four types of samples were taken out from the whole batch of sample- two samples of epoxy-bonded magnet and two sample of phenolic-bonded magnet. The details of analysis for each sample is as follow

Table 3.1: Sample Details

Sample Name	Sample Detail
Sample A	Non-corroded epoxy-bonded magnet
Sample B	Non-corroded phenolic-bonded magnet
Sample C	corroded epoxy-bonded magnet
Sample D	corroded phenolic-bonded magnet

3.5.1 Magnetic Property Analysis

The samples magnetic properties analysis was carried out at SIRIM Berhad in Kulim using the VSM equipment. Each magnet sample was magnetized at applied magnetic field up to 30 kOe.

3.5.2 Corrosion behaviour analysis

The corrosion rate for Sample C and Sample D were performed by electrochemical method. The type of environment chosen was salt water. The salt water was prepared by dissolving 3.5 g of sodium hydroxide powder into 96.5 g distilled water. Then the samples were dipped into different beaker filled with the salt water. A droplet of 0.1 M sulphuric acid used to initial the corrosion process. The samples were left for two weeks in the specific environment. After two week, the samples were then removed from the salt water, dried in an oven using heated air at 130°C for 10 minutes, and weighed again after carefully removing corrosion products with a brush.

CHAPTER 4

RESULT AND DISCUSSION

This chapter will discuss about the results achieved from the magnetic properties and corrosion behaviour analysis which had been done on the epoxy-bonded and phenolic-bonded magnet samples.

4.1 MAGNETIC PROPERTY ANALYSIS

During the magnetic property analysis, there were a few activities involved which were to determine the density of each type of magnets, to get the magnets' microstructure, as well as to magnetize the magnets using VSM.

4.1.1 Density

Density is one of the important variables to get the magnetic properties of a magnet sample. It contributes to the value of magnetic induction, B_r and maximum energy product, BH_{max} for the magnets that had been fabricated. To get the density of epoxy-bonded and phenolic-bonded magnet, the density for each sample had been calculated by and the average density had been determined. The results of density analysis for both types of magnets are shown in table 4.1 and 4.2:

From the data tabulated below, it had been determined that the averages of epoxy-bonded and phenolic-bonded magnets are 5.362g/cm^3 and 4.390g/cm^3 respectively. Thus, this value will be used to determine the magnetic properties for both types of magnets.

Table 4.1: Density of Epoxy-Bonded Magnet

Sample no.	Diameter (cm)	Thickness (cm)	Volume (cm3)	Weight (g)	Density (g/cm3)	Sample Density (g/cm3)	Average Density (g/cm3)
1	1.028 1.029 1.028	0.528 0.530 0.526	0.44 0.44 0.44	2.453 2.452 2.454	5.597 5.562 5.620	5.593	5.362
2	1.016 1.020 1.014	0.622 0.623 0.622	0.50 0.51 0.50	2.842 2.843 2.843	5.635 5.584 5.659	5.626	
3	1.006 1.008 1.008	0.744 0.760 0.742	0.59 0.61 0.59	3.027 3.028 3.028	5.118 4.992 5.113	5.074	
4	1.006 1.004 1.004	0.740 0.741 0.740	0.59 0.59 0.59	3.152 3.153 3.152	5.358 5.374 5.379	5.371	
5	1.004 1.006 1.006	0.774 0.772 0.773	0.61 0.61 0.61	3.088 3.089 3.088	5.039 5.033 5.025	5.032	
6	1.002 1.003 1.002	0.688 0.700 0.680	0.54 0.55 0.54	2.977 2.977 2.977	5.487 5.382 5.551	5.473	

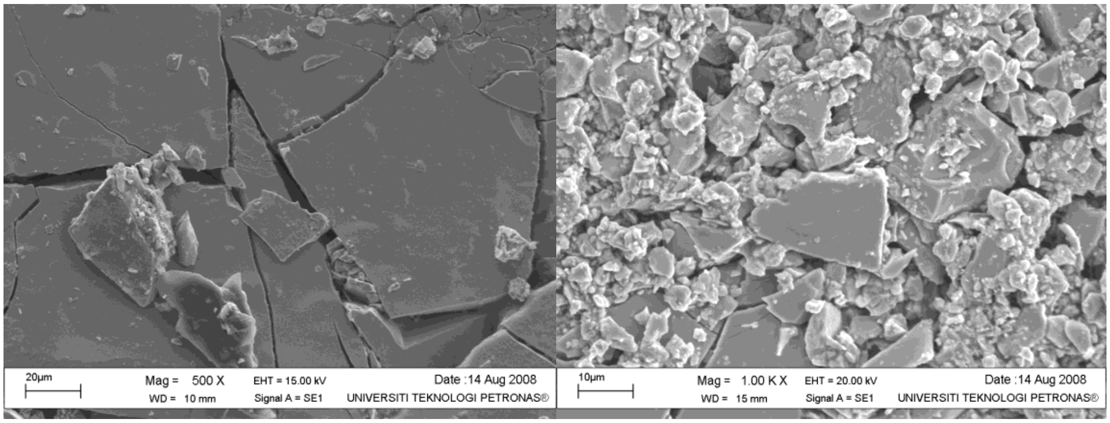
Table 4.2: Density of Phenolic-Bonded Magnet

Sample no.	Diameter (cm)	Thickness (cm)	Volume (cm3)	Weight (g)	Density (g/cm3)	Sample Density (g/cm3)	Average Density (g/cm3)
1	2.504 2.504 2.504	0.460 0.462 0.460	2.27 2.28 2.27	9.932 9.933 9.933	4.384 4.365 4.384	4.378	4.390
2	2.504 2.504 2.504	0.462 0.462 0.461	2.28 2.28 2.27	9.920 9.919 9.920	4.359 4.359 4.369	4.363	
3	2.504 2.504 2.504	0.450 0.452 0.452	2.22 2.23 2.23	10.000 10.000 10.000	4.512 4.492 4.492	4.499	
4	2.504 2.504 2.504	0.470 0.470 0.470	2.31 2.31 2.31	9.986 9.986 9.986	4.314 4.314 4.314	4.314	
5	2.504 2.504 2.504	0.469 0.468 0.468	2.31 2.30 2.30	9.969 9.969 9.969	4.316 4.325 4.325	4.322	
6	2.504 2.504 2.504	0.452 0.452 0.452	2.23 2.23 2.23	9.944 9.944 9.944	4.467 4.467 4.467	4.467	

4.1.2 Magnet Sample Microstructure

This chapter will continue with the discussion about the microstructure analysis performed on epoxy-bonded and phenolic-bonded magnets. In this section, the microstructure analysis for epoxy-bonded magnet will be discussed.

The sample microstructure was analyzed using SEM equipment. The microstructure can be seen by referring to Figure 4.1 that shows a closer view of the sample using at the magnification of 500x and 1000x respectively.



(a) **(b)**

Figure 4.1: Epoxy-bonded microstructure magnet microstructure after curing at 65°C for 1 hour (a) at magnification 500X and (b) at magnification 1000X

From the figure it can be seen that there are cracks present in the particulates of the sample. This might happened due to the uneven stress during compression loading or heat-shock during curing process of the magnet samples.

In the micrograph, pores existence had also been observed. This may result in the change of physical properties of the magnet samples. Figure 4.2 shows the sample microstructure analysis using SEM with Quadrant Back Scattered Diffraction (QBSD).

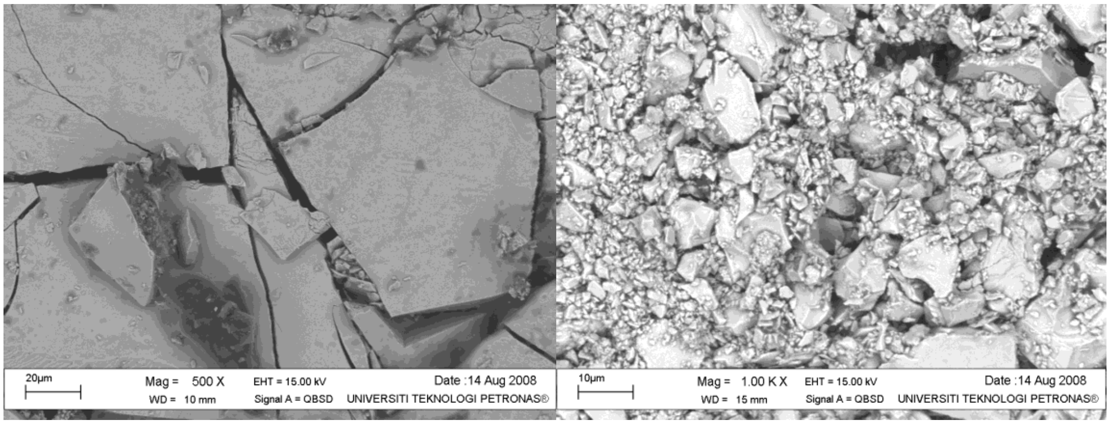


Figure 4.2: Sample analysis using QBSD

In addition, EDAX analysis had also been performed on the samples. Figure 4.3 and Figure 4.4 show the result of elements percentage in the sample and the distribution of element throughout the sample.

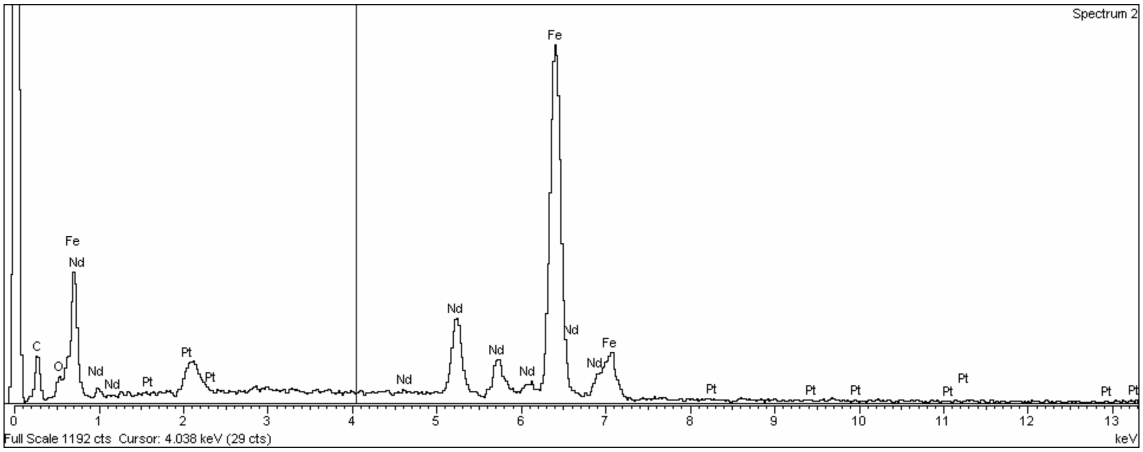


Figure 4.3: Qualitative analysis of bonded NdFeB

The fundamental elements like neodymium and iron were found, however boron presence within the magnet sample cannot be picked up during the analysis as it only constitute a low weight percent of the NdFeB alloy. On the other hand, EDX Mapping showsthe elements are distributed quite evenly throughout the sample.

Table 4.3: Element percentage in bonded NdFeB magnet sample

Element	Weight%	Atomic%
C K	15.43	47.77
O K	3.49	8.12
Fe K	57.54	38.31
Nd L	19.56	5.04
Pt M	3.98	0.76
Totals	100.00	

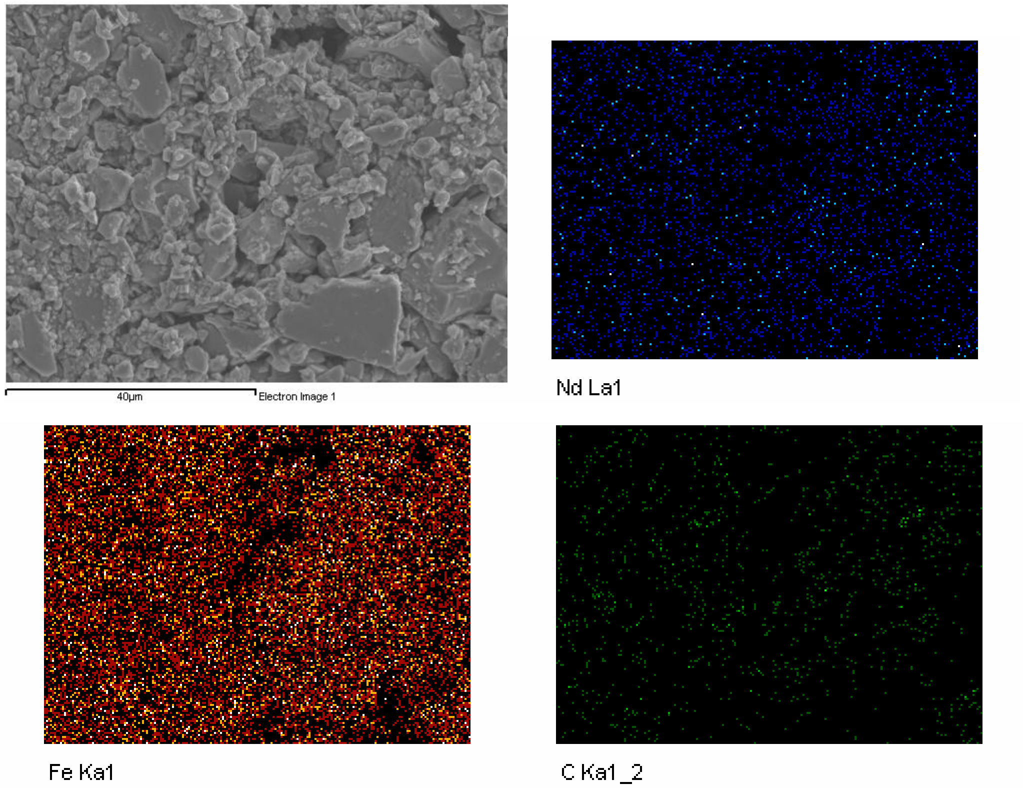


Figure 4.4: EDX mapping shows the element distribution in the sample

The microstructure analysis for phenolic-bonded magnet had not been carried out since the SEM equipment is not available after the time forward. However, it can still be assumed that there will be existence of pores in the sample and the elements of the magnet will be the same as the epoxy-bonded one.

4.1. 3 Magnetic Properties

Figure 4.5 below shows the magnetic induction, B versus energy product, BH for Sample A (epoxy-bonded magnet before corrosion), Sample B (phenolic-bonded magnet before corrosion), Sample C (epoxy-bonded magnet after corrosion) and Sample D (phenolic-bonded magnet after corrosion). It show that Sample A is having the highest BH_{max} value which is 6.26 MGOe, followed by Sample B and Sample C which is 3.85 MGOe and 4.94 MGOe respectively. From the figure also, we can see that Sample D is having the least BH_{max} which is 2.99 MGOe.

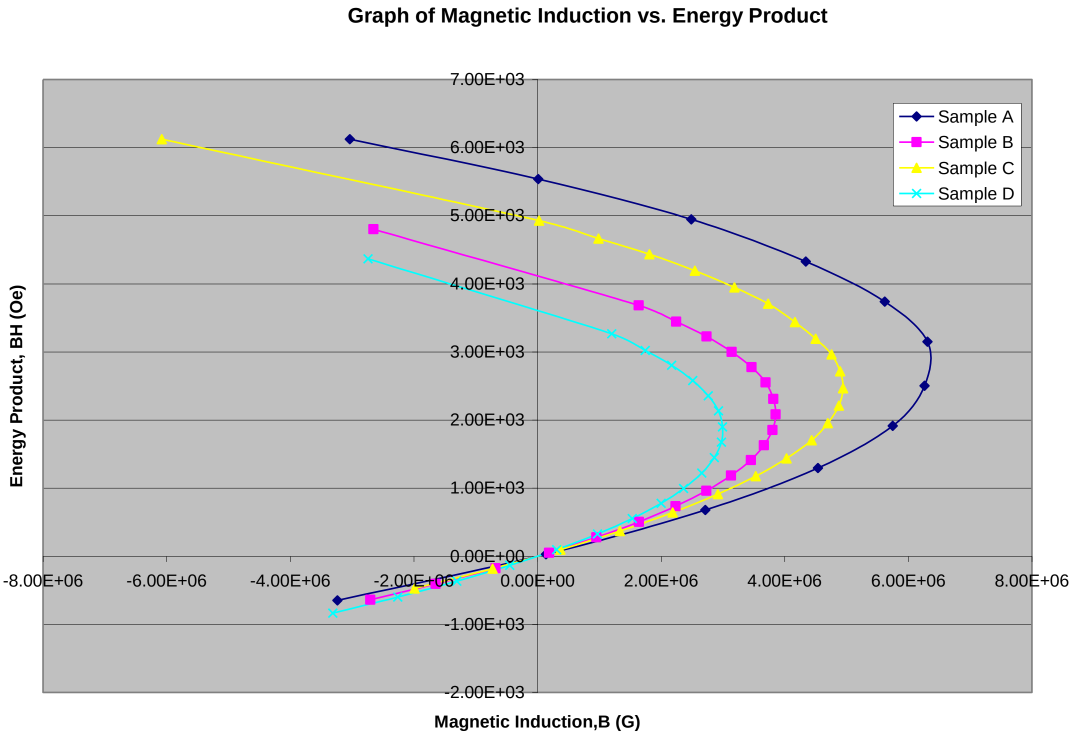


Figure 4.5: Graph of B versus BH for Sample A, B, C, and D

From the result achieve, it is proven that epoxy-bonded magnet is having the higher maximum energy product than phenolic-bonded magnet. On the other hand, we also had proved the effect of corrosion on the magnet energy product here we can see that there were reduction of magnetic properties by about 21% for epoxy bonded magnet and 22% for phenolic-bonded.

4.2 Corrosion Behavior Analysis

As what we can refer back to the percentage of elements in bonded NdFeB magnet, it shows that more than 50% of the NdFeB element is made of iron. For the galvanic series, Iron is an element that is categorized as anodic and quite susceptible to corrosion. An analysis had been done to determine the corrosion rate of magnet when different types of binders are used.

The corrosion analysis was conducted on Sample C and Sample D by immersing the samples in salt water. The corrosion rate was calculated using weight loss method. The results for this analysis are shown in Table 4.2.

Table 4.4: Calculated corrosion rate for Sample C and Sample D

Sample No.	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)	K (mm/yr)	Density (g/cm3)	Exposed Area (cm2)	Time (hours)	Corrosion Rate (mm/yr)
3	2.453	2.447	6	87.6	5.362	2.535	336	0.115083035
4	9.9578	9.9438	14	87.6	4.39	8.543	336	0.097323549

From the result above, it shows that the corrosion rate for Sample 3 is higher than Sample 4 which is 0.115 mm/yr compared to 0.097 mm/yr. Something that can be highlighted from the results is; the difference of corrosion rate for both type of magnet is not so big. Maybe what cause the difference between them is improper mixing of the magnet powder and polymer binder. As we know from the sample prepared, we can see that the surface of magnet using dry binder is covered with phenolic which may hinder the corrosion from happening and that result in more corrosion inhibition while for the epoxy-bonded magnet, the corrosion might be higher because of loss in epoxy binder during fabrication process- during compression process, the epoxy run out from the die through the space between the die and plunger.

When comparing the results with current industrial standard, the corrosion rate for both materials are still within the industrial limit (for most application a corrosion penetration rate less than about 0.5 mm/yr is acceptable).

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The study on magnetic properties and corrosion behaviour of bonded NdFeB magnet using wet and dry binder shows that there is a slight difference in the results from both analysis especially for the magnets magnetic properties.

From the study it shows that the magnet samples using epoxy binder have higher magnetic properties than one that use phenolic binder. However it is the other way round for corrosion rate, where it was founded that the magnet using phenolic binder has a lower corrosion rate than the one using epoxy binder although there is no big difference between the corrosion penetration rate values.

Conclusion can be deduced that the usage of different binders material for fabrication of magnets will lead to different magnet properties and each of the binder have their own advantages and disadvantages. Thus, as a good fabricator or user, one needs to identify what is the type or performance required and the usage of the magnet needs to be suited with the environment itself. If the environment is not very prone to corrosion, the usage of wet polymer bonded magnet is recommended. On the other hand, if the magnet application is exposed to a more volatile environment, the magnet which is made from dry binder might be more suitable.

On the other hand, if we are concerning about the fabrication process, dry polymer bonded magnet is recommended since the process is simpler and if automation is used, then the production rate can be increase and we can meet the customers' demand and reduce the cost of production for one batch of supply since time is saved.

The study on bonded NdFeB magnet is important since it is widely used in the current market. Thus, further research and development on its manufacturing and process will help us to understand the magnet properties and corrosion behaviour and enhance it to suit with current technology and advancement. The initiation of this project will provide new perspective and idea to manufacturers as well as consumers on how the improvement on bonded magnets can be done and hopefully from the result gained during the analysis, these group will be benefited from it.

For future work, there author would like to recommend further study on this project, since this is the first time that such project were done. So it is good if there will be further work done so that the results are proven to be right. In terms of project work it is recommended that the project should be more detailed. All parameters and variables need to be specified so that the results can be more accurate. For example, to specify the size of NdFeB powder that is more suitable to be used. Then for the sample preparation, maybe another method of preparing wet polymer bonded magnet should be used since the current process is very tedious and it is good to use other simpler method if there is any. Different types of binder might also be used for future work in order to vary the process option. For the analysis part, it needs to be done more carefully so that error can be avoided. For corrosion behaviour analysis, another method can also be used to get more accurate results (for example: using automated corrosion meter equipment). Hopefully from this recommendation it can help the next worker to achieve better results and prove the findings that had been conducted during this study.

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APPENDICES

APPENDIX 1: PROJECT SHEDULE

First Semester

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	■	■						Mid-semester break							
2	Preliminary Research Work		■	■	■											
3	Submission of Preliminary Report				●											
4	Project Work					■	■	■								
5	Submission of Progress Report									●						
6	Project Seminar										■	■	■	■		
7	Project work continues									■	■	■	■	■		
8	Submission of Interim Report Final Draft														●	
9	Oral Presentation															●

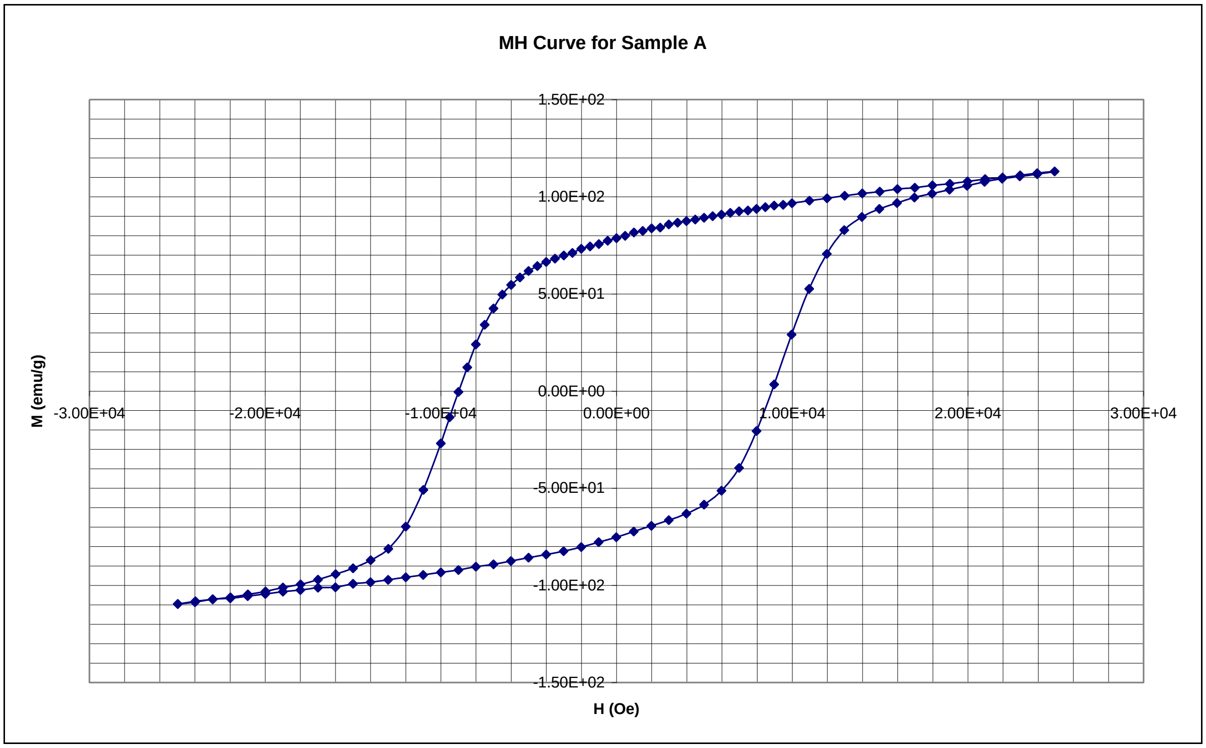
● Project milestone
■ Process

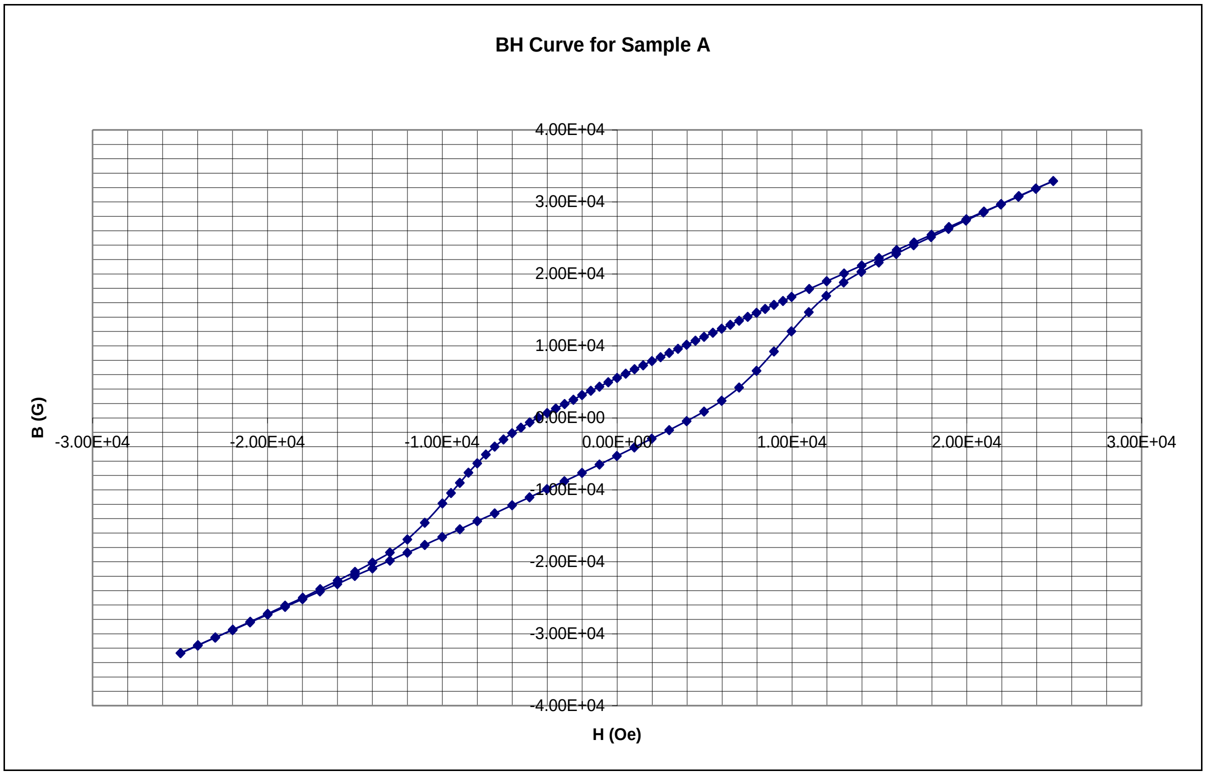
Second Semester

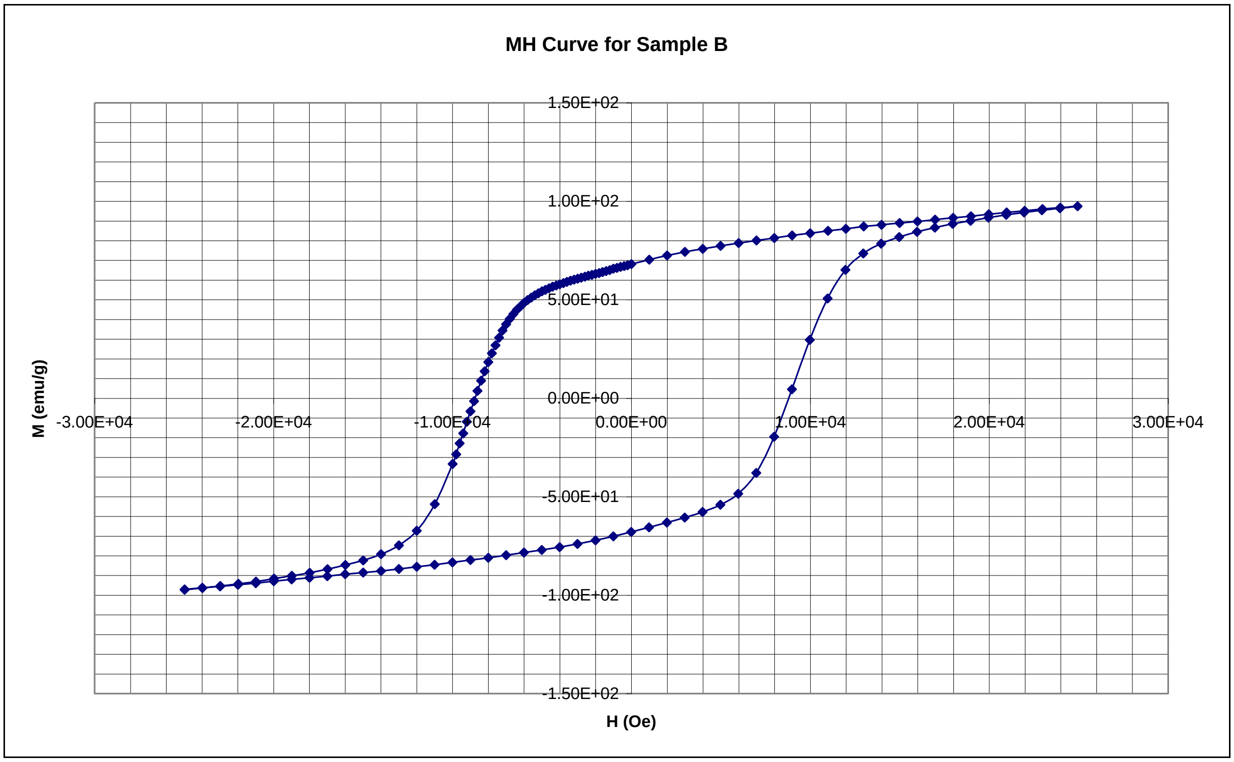
No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10		11	12	13	14	Exam Week
1	Project Work Continue - Epoxy-bonded magnet sample preparation	■	■	■								Mid-Semester Break					
2	Submission of Progress Report 1				●												
3	Project Work Continue - Epoxy- bonded magnet sample analysis (density and microstructure)				■	■	■	■									
4	Submission of Progress Report 2								●								
5	Seminar (compulsory)									■	■		■				
5	Project work continue - phenolic-bonded sample preparation								■	■	■		■	■			
	- corrosion analysis														■	■	■
	- magnetic properties analysis																■
6	Poster Exhibition																
7	Submission of Dissertation (soft bound)																●
8	Oral Presentation																●
9	Submission of Project Dissertation (Hard Bound)																●

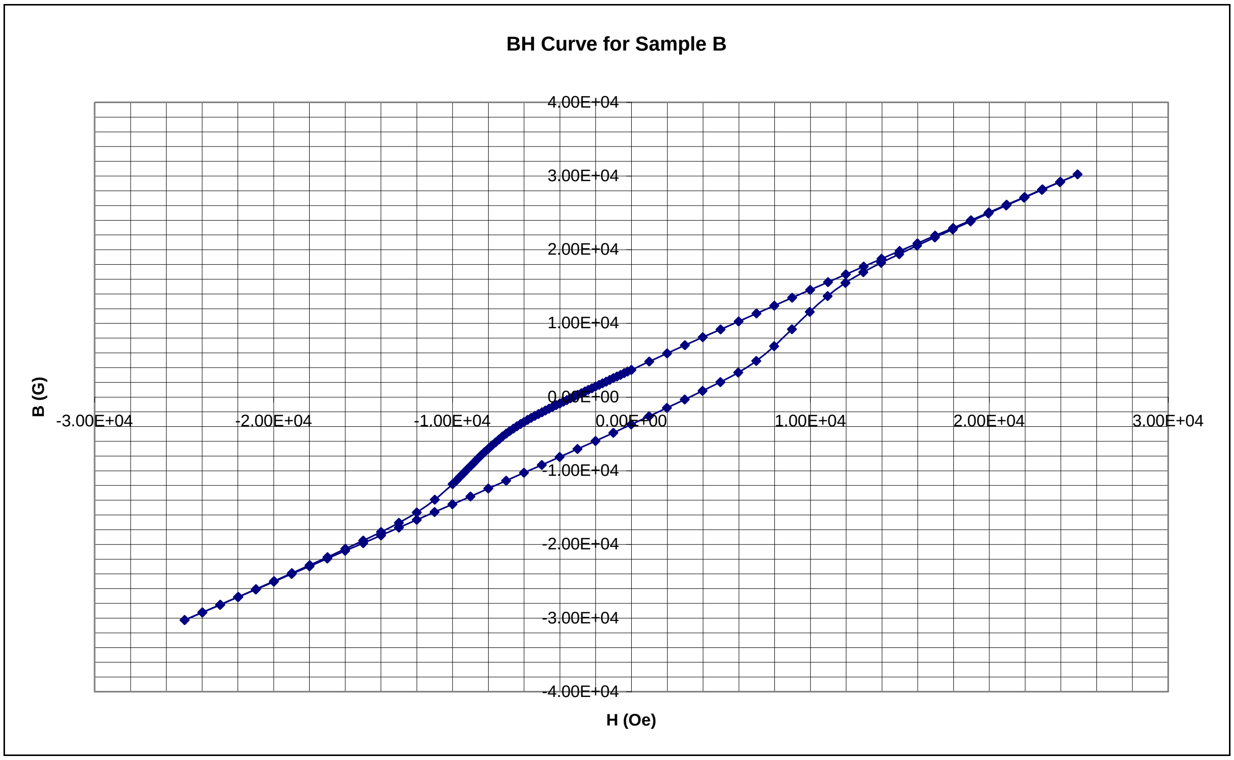
APPENDIX 2

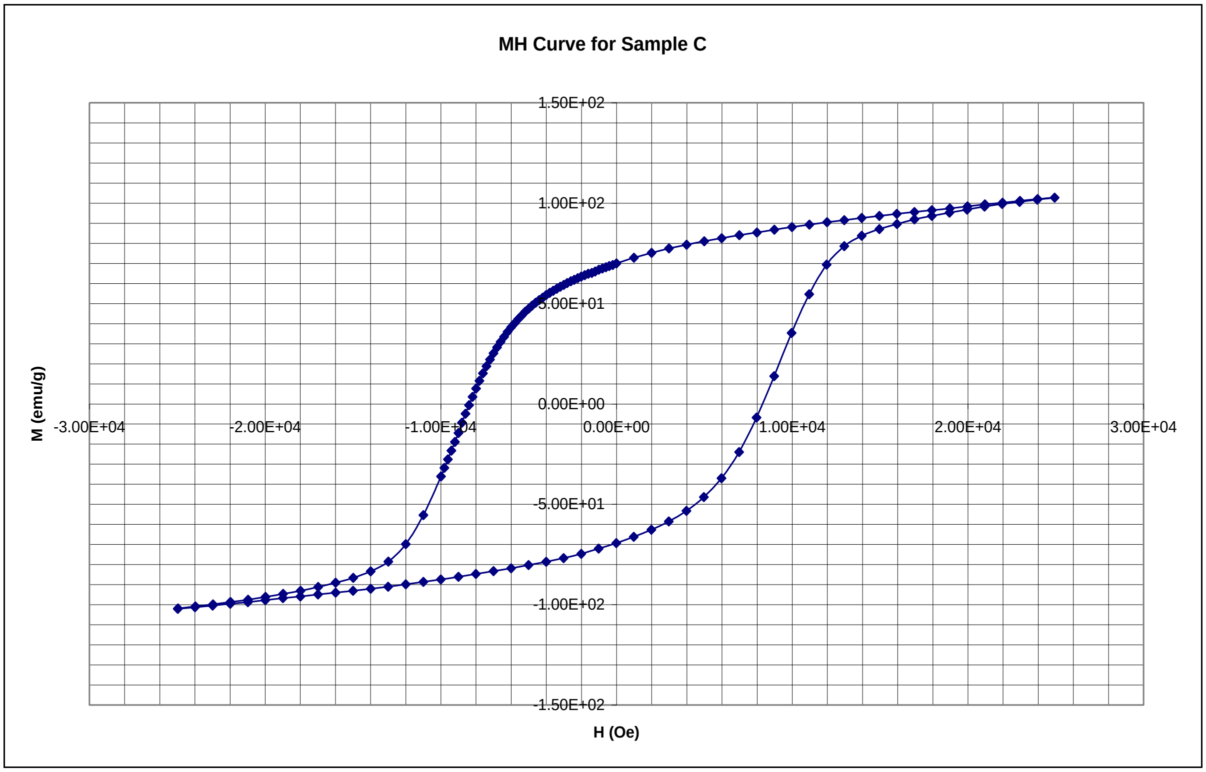
Magnetic Properties Graphs

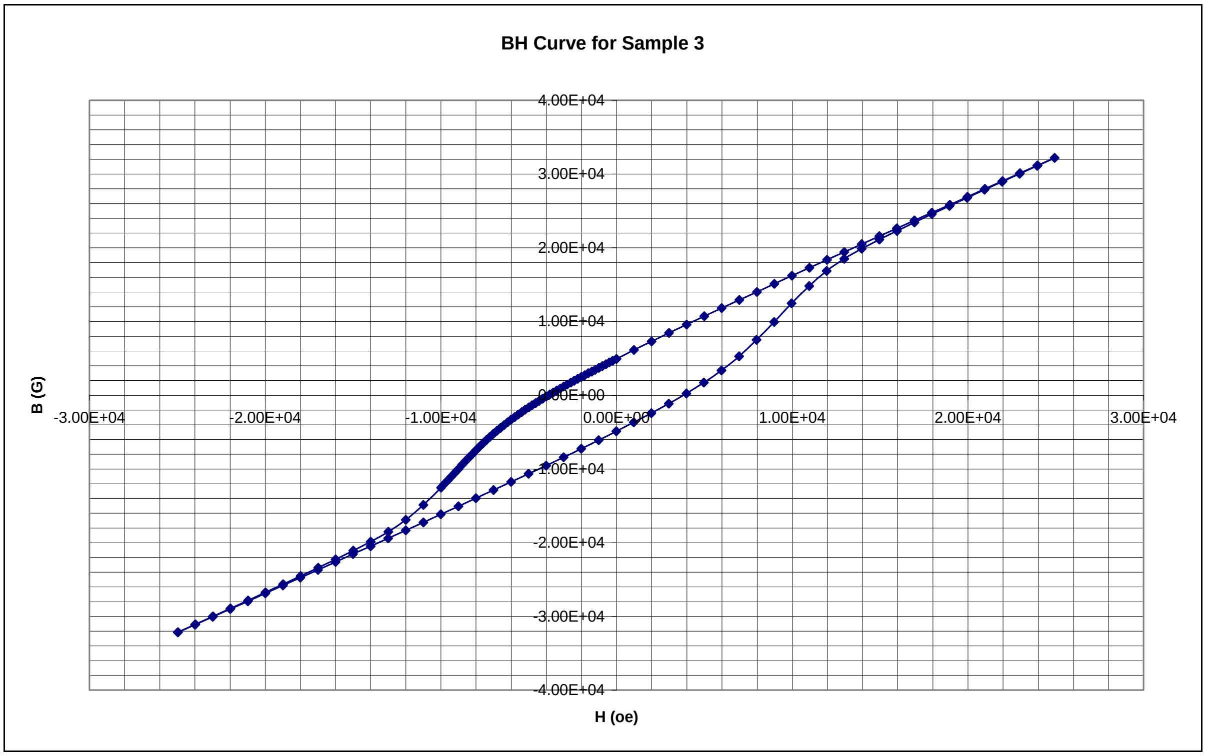


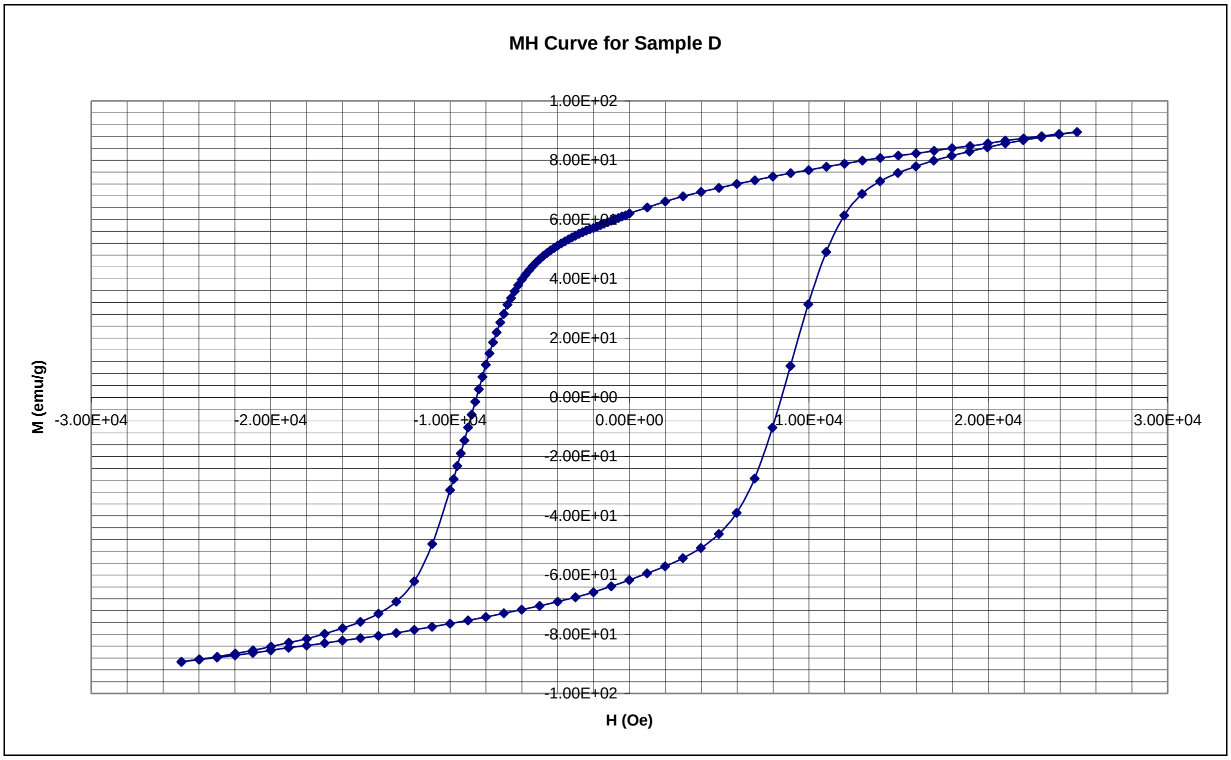


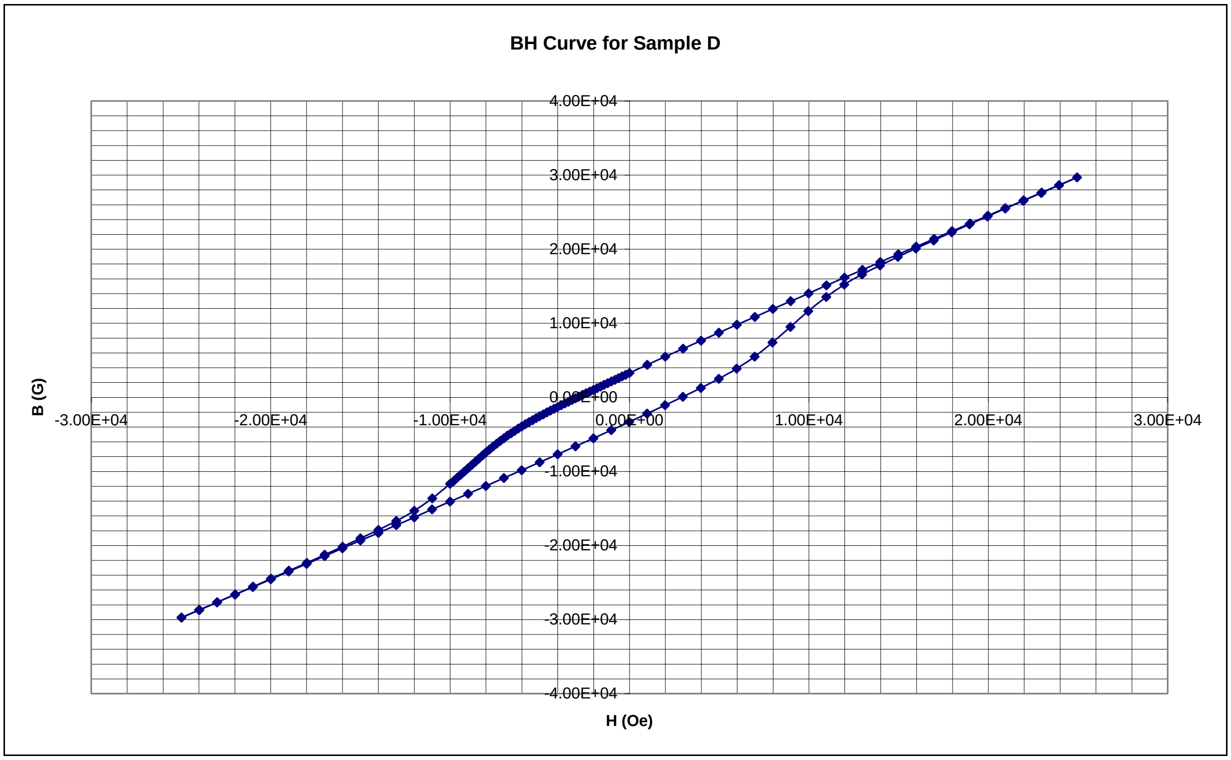

MH Curve for Sample B
M (emu/g)
H (Oe)
1.50E+02
1.00E+02
5.00E+01
0.00E+00
-5.00E+01
-1.00E+02
-1.50E+02
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04


BH Curve for Sample B
B (G)
H (Oe)
4.00E+04
3.00E+04
2.00E+04
1.00E+04
0.00E+00
-1.00E+04
-2.00E+04
-3.00E+04
-4.00E+04
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04


MH Curve for Sample C
1.50E+02
1.00E+02
5.00E+01
0.00E+00
-5.00E+01
-1.00E+02
-1.50E+02
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04
M (emu/g)
H (Oe)


BH Curve for Sample 3
4.00E+04
3.00E+04
2.00E+04
1.00E+04
0.00E+00
-1.00E+04
-2.00E+04
-3.00E+04
-4.00E+04
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04
B (G)
H (oe)

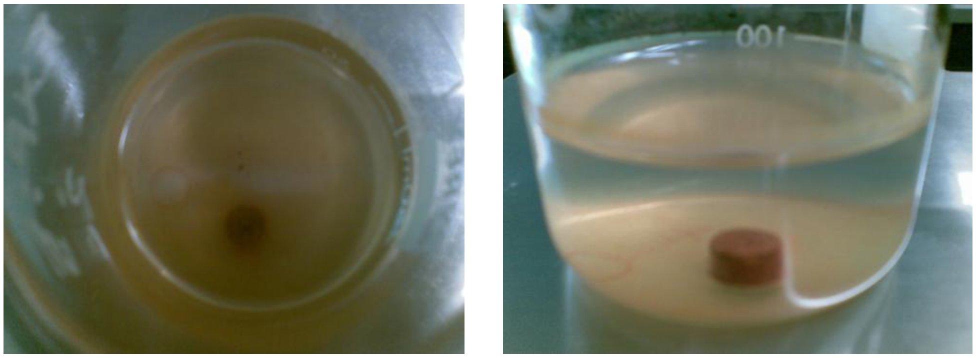

MH Curve for Sample D
M (emu/g)
H (Oe)
1.00E+02
8.00E+01
6.00E+01
4.00E+01
2.00E+01
0.00E+00
-2.00E+01
-4.00E+01
-6.00E+01
-8.00E+01
-1.00E+02
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04


BH Curve for Sample D
B (G)
H (Oe)
4.00E+04
3.00E+04
2.00E+04
1.00E+04
0.00E+00
-1.00E+04
-2.00E+04
-3.00E+04
-4.00E+04
-3.00E+04
-2.00E+04
-1.00E+04
0.00E+00
1.00E+04
2.00E+04
3.00E+04

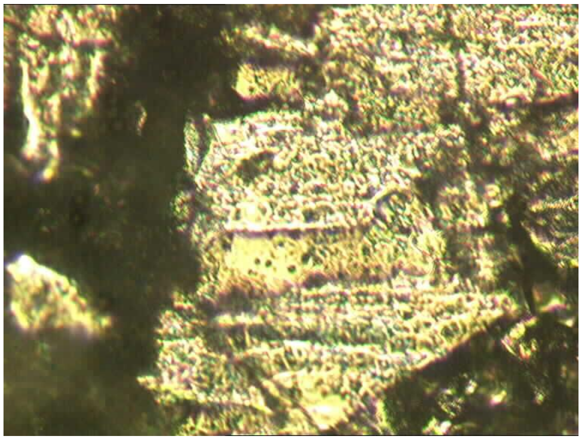
APPENDIX 3

Corrosion Behavior Analysis

Sample C



Sample C – Before Corrosion

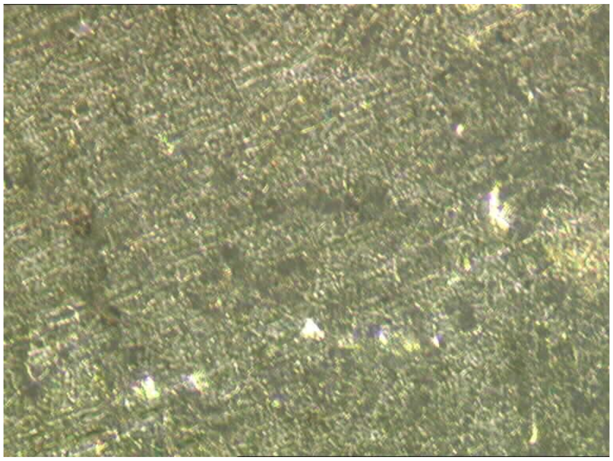


Sample C – After Corrosion

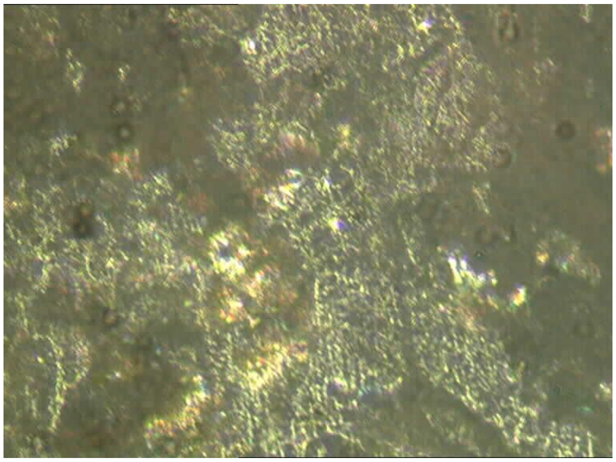
*The micrograph picture is taken by using optical microscope at magnification 20X

Sample D

Sample D – Before Corrosion



Sample D – After Corrosion



*The micrograph picture is taken by using optical microscope at magnification 20X