

EXPLOSIVE SPIN COMPACTION OF CERAMIC NANOPARTICLES

by

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Abstract

A new technique for explosive compaction was introduced known as explosive spin compaction. Neodymium iron boron and Barium Titanate were studied before and after spin explosive compaction using SEM, XRD and vibrating sample magnetometer. The structural properties were studied before and after spin compaction of both bulk and milled nano particles of both materials. The compacts were prepared by spin compaction at different velocities for both the materials and compacts retrieved and studied. The compacts of bulk and milled magnetic materials were studied using a vibrating sample magnetometer and slight variations were seen when compared to compacts from traditional compaction. This may be due to achieving near theoretical density and different microstructures observed in the compacts. The variations seen in bulk and milled magnetic compacts were due to the compact's amorphous structure and contamination that may have occurred during the milling process.

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1 Introduction

1.1 Background

In the Powder Metallurgy industry, there is a constant demand for affordable, high-density, net shape parts. In commercial sectors such as the auto industry there is a need for almost theoretical density parts for high performance applications, such as gears. In these industries today, such parts are machined from wrought metal stock, which is expensive given the high machining costs. The need to strive to produce high quality parts at minimal costs has led to new research directions in the last decade – shock, magnetic and spin compaction, etc.

1.2 Motivation

Conventional and cost-effective techniques like press and sinter processing methods can produce parts of very low densities of 6.9- 7.4 g/cc. Throughput is low and it is expensive given the time taken and secondary processing involved in producing the required shape. Powder materials such as permanent magnetic powders for motor applications, thermo electrics for efficient heating and cooling devices are commercially processed by dynamic compaction. The need for preservation of inherent properties of the original materials, at the same time yielding high density and low cost was the motivation for investigating dynamic compaction.

1.3 Objective

This thesis is aimed at analysing the efficiency of explosive spin compaction on Magnequench Powders and Barium Titanate. The following are specific objectives of this research:

1. A detailed study of existing methods in compaction, processes involved and compatibility of such methods with material types
2. Investigation of a new explosive compaction method that adds shear stress and strain during the compaction.
3. To test the effect of shear stress and strain on the properties of M1- Magnequench Nd-Fe-B, M2- Magnequench Nd-Pr-Nb-Fe-B and B1- Barium titanate bulk and milled powder compacts.

1.4 Scope

The literature review covers important aspects of compaction methods and their compatibility with specific materials that can contribute to the goal of producing high quality components at minimal costs. This thesis focuses on the explosive spin compaction technique and its effectiveness to attain potentially more durable and reliable components.

1.5 Thesis Organization

Chapter 1 deals with the background and motivation for this study and recent developments in the compaction techniques.

Chapter 2 discusses previous work carried out on compaction techniques, properties of different materials and results, for materials that have been chosen for this research. Various applications of the materials studied and the impact of compaction are also discussed.

Chapter 3 deals with the experimental technique that was designed and executed on different materials to research the impact of spin compaction in modern day applications. It also discusses the methods of characterisation of samples.

Chapter 4 presents the results and discussions on the recovered samples obtained from the spin compaction process. Characterization of compacts was done using on XRD, SEM, Magnetic properties and density.

Chapter 5 concludes the study with the highlights of inferences. A discussion of the challenges faced and scope for future work are presented in Chapters 6 & 7, respectively.

2 Literature Survey

2.1 Literature Review

K.H. Chen et al [9] dealt with the formation of exchange coupled magnetic materials that consist of both hard and soft phases. This combination of different phases can lead to a higher energy product when compared to their individual counterparts. The materials studied and reported in this paper were $\text{Nd}_2 \text{Fe}_{14}\text{B}$ and $\text{Pr}_2 \text{Fe}_{14}\text{B}$ alloys. Both these materials were chosen because of very similar and practically indistinguishable structural, mechanical, and magnetic properties. The starting materials were prepared by pulverizing rapidly quenched and annealed ribbons of the two materials with Fe wt. 10% and 20% respectively. These samples were cold pressed into steel capsules at 65%-78% of the theoretical density. The shock-consolidation experiments were carried out using a three-capsule plate fixture mounted at the edge of an 80mm diameter single stage gas-gun barrel [10]. The velocities of the projectiles obtained were about 605, 880 and 950 m/s. This resulted in the propagation of shock waves through the capsules and compaction of the powders producing 12mm by 3mm thickness discs. The bonding between the particles was observed using SEM and the crystallinity was studied using the diffraction patterns obtained by XRD and TEM. The magnetic properties of the bonded pellets were studied using a superconducting quantum interference magnetometer.

The dynamic and heterogeneous nature of plastic deformation due to shock compaction of the materials led to localized deposition of shock energy at particle interfaces, which can result in melting and resolidification at the interparticle regions. This localized phenomenon was observed

in the present study of samples consolidated at 950 m/s. This presence of interparticle melting and resolidification have adverse effects on the material as they lead to unfavorable grain growth or decomposition of the magnetic phases. This can be avoided by optimization of shock parameters, for example, adjusting the initial packing density using flake particles of 20-100 μm in size and lowering the impact velocity. Powders pressed to initial packing density of 78% of the theoretical density and consolidated with an impact velocity of 605-800 m/s had a density of nearly 98% of the theoretical density and no localized melting or resolidification was observed in these samples. The only features seen were plastic deformation and fracture in the powder flakes. It was also seen that when the powders were compacted initially they tended to align in the direction of pressing with the flat surfaces perpendicular to the pressing direction. It was observed that they continued to possess that same orientation after shock consolidation. This was a valuable result as it can be useful in producing highly isotropic magnets by improving their magnetic properties. This orientation in the morphology was observed in both the samples. The Vicker's hardness test also showed the increase in hardness of the consolidated samples indicating a strong interparticle bonding in the compacts. The samples were studied using XRD and the peaks matched in both the starting ribbon and compacts consolidated at 880m/s. Both the ribbons and compacts were ground prior to XRD analysis.

The XRD peaks formed (Fig 2-1) showed both the hard and soft magnetic phases of the material. The shock consolidated samples show more broadening of peaks for the Fe phase than for the hard phase. This broadening of peaks maybe related to microstrain and a decrease in the crystallite size caused by shock-consolidation. Both of these can be studied using Williamson-Hall plots [12]. The grain size was also confirmed using TEM analysis. The fine grains were seen in the compacted

samples when compared to starting sample. This is due to the dynamic compaction which led to shearing and fragmentation of the grains during impact. The compacts were then studied using a magnetometer which proved that the magnetic properties of the starting material was preserved and also improved after shock compaction. The nano size of the grains and intimate interparticle contacts lead to effective interphase exchange coupling in the shock consolidated samples. Thus a nearly complete densification, retention, and refinement of nano- structure led to improved magnetic properties.

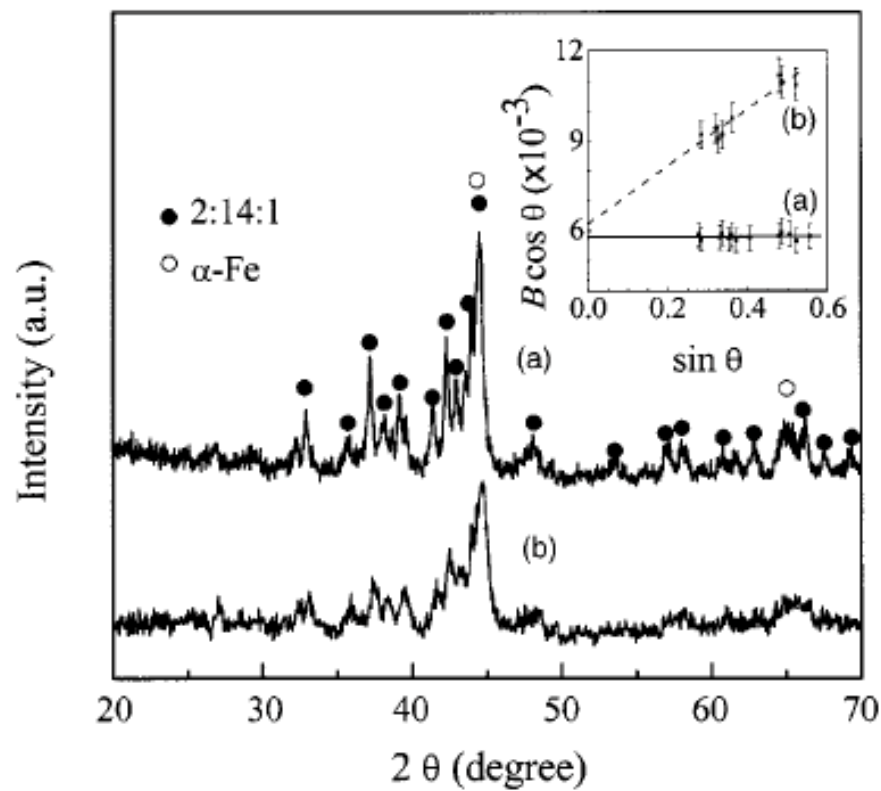


Fig 2-1: XRD patterns comparing the structure of (a) starting $\text{Pr}_2\text{Fe}_{14}\text{B/a-Fe}$ material and (b) recovered compact shock consolidated at 880 m/s [9]

V.M. Chakka et al [12] prepared nano magnetic powders using a surfactant assisted milling technique. Fe, Co, FeCo, SmCo, and NdFeB were some of the materials studied after milling using this technique. The starting powders had particle sizes ranging from 10 to 45 μm . Commercially available powders of Fe (98%) and Co (99.5%) were used and NdFeB (2:14:1) and FeCo alloy formed by arc melting and grinding were used. The milling and handling were done in oxygen-free controlled environments using inert argon gas in a glove box. For the milling process, the solvent used was heptane (99.8% purity) and surfactants used were oleic acid (90% purity) and oleyl amine (>98%). The instrument used was a high energy Spex 8000M mill with milling vial and the balls used were 440C hardened steel. The durations of milling were from 1- 50 hours and the ball to powder ratio was 5:1. The amount of surfactant used was about ~10%-12% that of the starting powder.

The magnetic measurement samples were prepared by embedding the nanoparticles in epoxy inside the glove box. The magnetic measurements were done using an alternating gradient magnetometer and 5K Superconducting Quantum Interference Device (SQUID). The structural and morphological changes were characterized using the TEM and XRD. The compositional characterizations were done using the Energy Dispersive X-Ray and inductively coupled plasma.

When surfactants were added to the nano powders and milled, sediment of colored slurry was observed at the bottom of the mill. The nano particles that were found dispersed in the slurry were of the order of ~30nm in size. When surfactants were not added, the slurry was colorless, as nanoparticles were not formed. TEM images of nanoparticles formed by milling Fe powders showed spherical or aspherical nanoparticles in sizes ranging from 3 to 9nm. TEM images of

nanoparticles prepared by milling FeCo powders showed ultra-fine spherical nanoparticles with a narrow particle distribution ranging from 1.7 to 4 nm for samples prepared using oleic acid. This narrow size distribution and fine particle sizes are advantageous over other techniques.

All of the materials studied were found to be compatible with the surfactants used except for Co. When Co was milled for 5 hours with oleyl amine, nano rods were formed. When milled for longer periods, larger concentrations of nano rods were formed. Similar nanorods were obtained when NdFeB and SmCo were ball milled as well and the percentages of nanoparticles also increased with an increase in milling time. The mechanism of ball milling is fairly complex and does not lend itself easily to rigorous theoretical analysis due to its dynamic nature. The nanorods could be produced by fracture along some preferred crystalline orientation during milling. The increase in temperature in the milling chamber may facilitate the growth.

Materials with hexagonal (SmCo₅, Sm₂Co₁₇, Co) and tetragonal (Nd₂Fe₁₄B) structures have a preferred orientation for fracture along the close-packed planes and these would result in the production of elongated nanoparticles.

In the case of nanoparticles prepared by milling Fe, Co, and FeCo, super paramagnetic behavior was observed at room temperature and using SQUID at 5K a combination of super paramagnetic and ferromagnetic behavior was observed. In the SmCo compounds, the remanence was found to increase with milling time. The cause for remanence enhancement could be due to the increasing percentage of elongated rod shapes and higher aspect ratios for longer milling times or the formation of most of the nanoparticles from the crystalline regions of the primary particles rather than the disordered boundary regions with increasing milling time.

The starting powders used in the study by M.Yue et al [13] were MQ-C and NdFeB ribbons from the Magnequench company. Heptane (~99.8% purity) was used as a solvent and the surfactants were oleic acid and oleyl amine. The powders were milled in a milling vial using stainless steel balls using SPEX 8000M high-energy ball milling machine and the ratio of the powders to balls was 1:10. The amount of solvent and surfactants used were about 50% and 10% of the weight of starting powders. After milling for 12 hours, the slurry was dispersed into heptane solvent by ultrasonic agitation and transferred to centrifugal tubes for size selection. The samples for magnetic measurements were prepared in epoxy in controlled environments in the presence of an argon atmosphere. The magnetic measurements were done using a Quantum Design Inc., magnetic properties measurement system magnetometer. The structural and morphological characterizations were done using XRD and TEM. The as-milled NFB slurry was ultrasonically agitated and allowed to settle in order to collect the smaller nanoparticles. To obtain larger nanoparticles the slurry was washed with heptane to remove smallest nanoparticles. The remaining part was once again centrifuged and the nanoparticles were collected after dispersion of 3 hours. The TEM images were studied and the particle size distribution was calculated using LPSA. The average sizes of the small and big nanoparticles were 10 and 100 nm respectively.

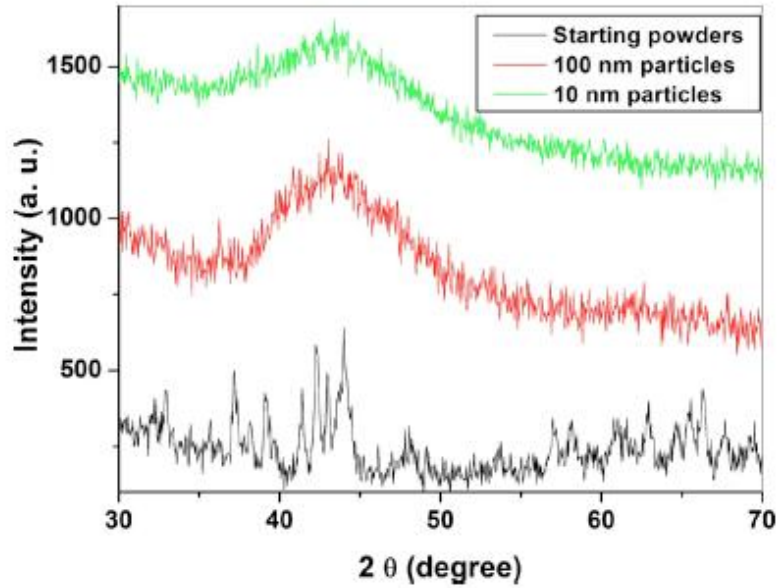


Fig 2-2: XRD patterns of small, big NdFeB nanoparticles & starting NdFeB ribbons [13]

The XRD analysis showed that both diffraction peaks of small and large nanoparticles broadened into big humps. Apart from the decrease in the grain size, the strains and amorphization formed due to the ball milling process also contributed to the broadening of the diffraction peaks. Thus it was harder to calculate the particle sizes from the width of the peaks. Also no diffraction peaks from rare earth oxides were found in both patterns for big and small nanoparticles although those peaks showed up in the graphs for oxidized samples, which were not prepared in controlled environments. This proved that the materials were protected from oxidation during the preparation and analysis processes.

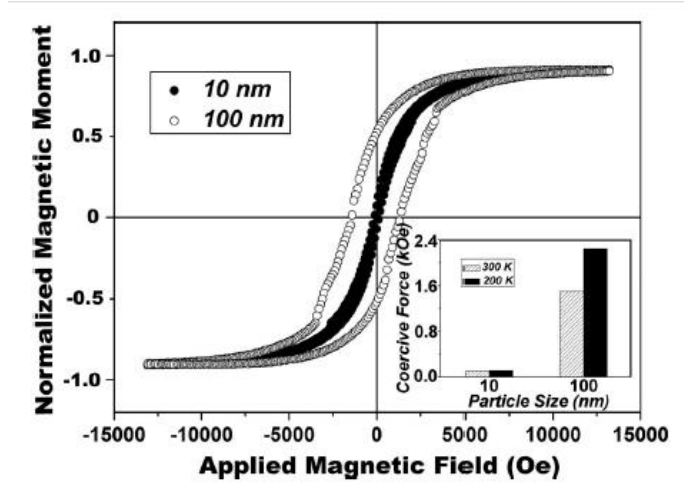


Fig 2-3: Magnetization hysteresis loop of both the small and big Nd-Fe-B nanoparticles at 300 K. Insets: intrinsic coercive force of the particles vs measuring temperature [12]

Magnetic properties when studied showed that coercive forces at room temperature are 0.1 and 1.5 kOe for small and large nanoparticles respectively. The coercive force of large nanoparticles increases by 50% as the temperature drops to 200K due to enhancement of magneto crystalline anisotropy of NdFeB phase in the particles. The reduction in coercive force maybe due to the amorphous state of nano particles and contaminations that may have occurred during the preparation.

Guruswamy et al [1] performed explosive compaction of Magnequench NdFeB having high and low rare earth contents into cylindrical magnets and studied them. The Magnequench NdFeB powders were prepared by melt spinning Nd-Fe-B alloy followed by comminution. Two isotropic powders were studied in this process MQP-A and MQP-B. The nominal compositions of MQP-A powder was Fe-30.5 wt % and 0.9 wt % of B and for the MQP-B , Fe-5 wt %, Co-27.5 wt % total

rare-earth -0.9 wt % of B. The rare earth component consisted of mainly Nd with a maximum 0.5 wt % of Pr and maximum 0.2 wt % of other rare-earths.

The experimental procedure consisted of detonation of the powders in a copper tube closed at one end with a steel cap and a tightly fitting steel rod on the other end. The packed density of powders was about 55% .The explosive (Dyno Nobel 207X), was prepared by mixing the two components, perchlorate-base liquid component A and an aluminum-based component B. The detonator fuse was fitted snugly as shown in Fig 2-4.

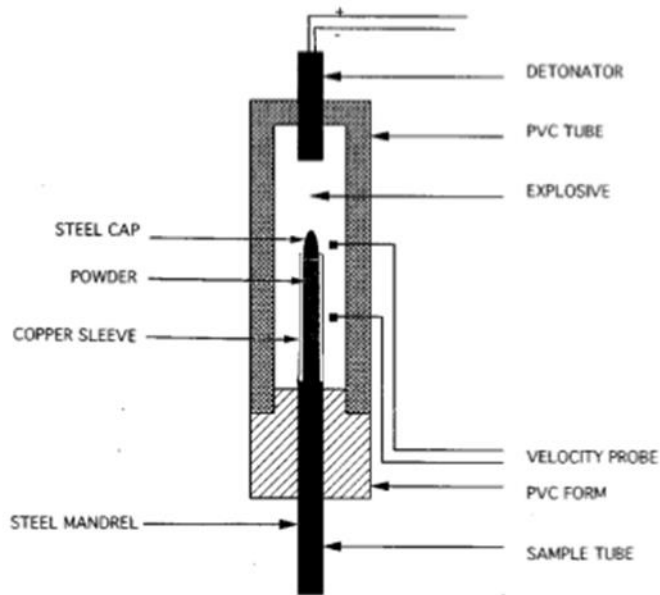


Fig 2-4: Explosive container assembly for explosive compaction of NdFeB powder [1]

The shock wave velocities observed was about ~4300 m/s. The detonation velocity was found to be related to the compaction pressure by the following equation [4]

$$DP= 2.325 \times 10^{-7} \rho (VOD)^2$$

where, VOD is the velocity of detonation in ft/s , DP is the detonation pressure in kbar, and ρ is the density of explosive mix in g/cc.

The compacted specimens were named EC-A and EC-B. The samples were studied using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), X-Ray Diffraction (XRD), Differential Thermal Analysis (DTA) and Vibrating Sample Magnetometer (VSM).

The crack formation and porosity were studied using the SEM. In the cross section of the sample a small hole was observed along the axis of the cylindrical sample. This hole was found to be due to the melting and ejection of material under the focused energy of shock waves formed in the center. The hole is due to excessive consolidation pressure. The hole can be eliminated by lowering the detonation pressure or by using an additional copper tube sleeve surrounding the stainless steel tube in which the sample is present. The cracks observed were also due to the same reason and can be minimized using the same techniques discussed above [2].

The XRD pattern (Fig 2-5) confirmed the presence of crystalline 2-14-1 phase. Demagnetization curves were obtained using the VSM in three orthogonal directions and it determined that the magnetic properties were isotropic in both EC-A and EC-B [6][8]. This proves that the inherent structure and properties of the starting material were preserved. The TEM micrograph showed the formation of equiaxed grains on the order of 20-25nm and structures similar to that seen in the original NdFeB ribbon. The equiaxed microstructure was observed to be consistent with the isotropic properties observed for both the specimens EC-A and EC-B. It was also determined that

the explosive compaction has preserved the rapidly solidified microstructure of the ribbon and thus does not have effects on grain growth.

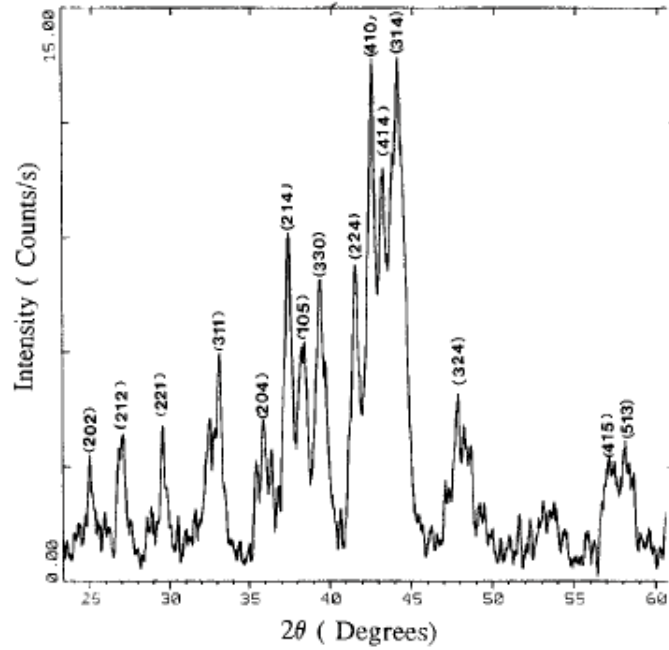


Fig 2-5: X-ray diffraction pattern of explosively compacted EC-B magnet [2]

A.K. Goswami [14] prepared polycrystalline Barium Titanate pellets close to 95% theoretical density and investigated the piezoelectric and dielectric properties of the pellets. Barium Titanate powder of ~99% was placed in a stainless steel tube and explosively compacted at a detonation velocity of about 4500m/s. It was seen that the material exhibited no Curie point nor hysteresis loop and thus no ferroelectric properties [15]. It was observed that the material behaved as a linear dielectric and the dissipation factor was 0.5% and a room temperature dielectric constant of ~250 and compacts poled at 20 kV/cm exhibited no piezoelectricity. It was seen that when the compacts were sintered piezoelectric, conventional dielectric behavior and ferroelectric properties

reappeared. It was reported that the absence of ferroelectricity was due to defects in surface layer and its crystallites.

Hence it is seen that explosive compaction is more effective than sintering and near theoretical densities were achieved. With this technique it was possible to achieve materials possessing unique microstructures as the compaction occurs in microseconds. Since the process occurs in such a short periods the heating is limited, and oxidation is reduced. The heterogeneous deposition of energy causes bonding within particles and the voids are eliminated leading to high-density compacts. The different microstructures and lack of oxidation lead to the improved magnetic and piezoelectric properties.

2.2 Takeaways

Explosive compaction (Fig 2-6) is a potentially important method for synthesis and processing of materials. It can be used on ceramic, metallic and composite materials to attain high-density compacts for various applications. Powders ranging from nano to micro size particles can be compacted using this technique. The mechanism of shock compaction is significantly different

from that encountered during densification via conventional powder compaction methods. The critical processes occurring during explosive compaction occur within a microsecond, involve a

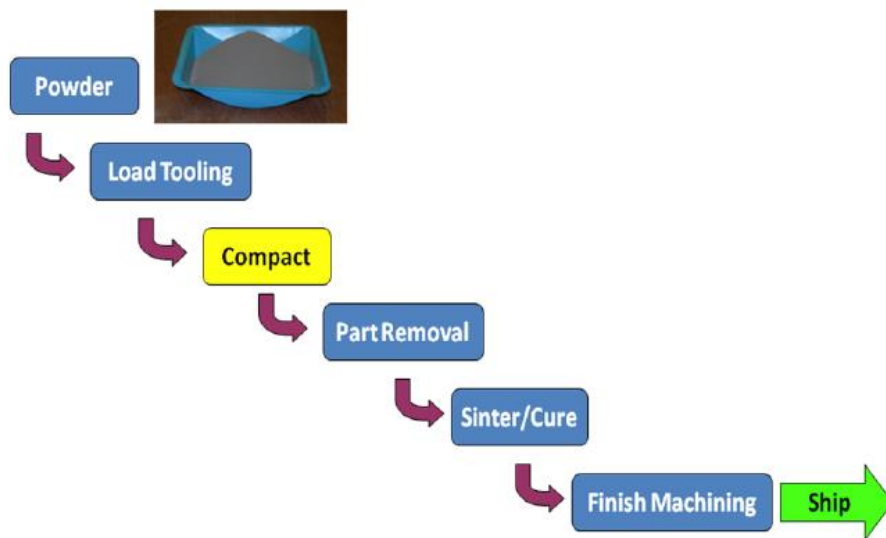


Fig 2-6: Steps involved in magnetic compaction process

heterogeneous deposition of shock energy that results in an inter particle bonding and changes in the particles due to obliteration of voids through plastic flow & dispersion of fragments. Hence, explosive compaction makes it possible to fabricate compacts close to theoretical density. The heating is limited as the actual compaction takes place in microseconds, no grain growth occurs and oxidation is limited. Due to the microstructural modifications occurring and high densities achieved, the resulting compacts have highly improved properties.

Traditional explosive compaction processes involve normal impact of a plate on a sleeve containing powder, surrounded by a detonator activated by ballistic velocities. In the traditional process there is a limitation on the shear component due to near normal impact. Spin compaction

being investigated in this research involves shear components of stress and strain. It was anticipated that the microstructures resulting from the spin compaction technique would be different from those obtained using traditional compaction techniques. It was also expected that there would be a change in the inherent properties of the material. The addition of a shear component whose amplitude can be made to exceed that of the normal component may enable new phases to be produced again, significantly altering the performance over that of traditionally explosively compacted and sintered materials.

One of the advantages of using explosive spin compaction was that the testing can be done with small projectiles and hence the amount of material to be compacted can be reduced. For traditional explosive compaction a large open space and large specimens are required. However, in the spin compaction technique, the material to be compacted can be contained in a small projectile that can be considered as the flyer plate and shot at a base plate. The properties of the recovered compacts can be studied. This is an inexpensive technique compared with traditional explosive compaction techniques.

Ceramic materials are defined as any inorganic crystalline materials that are compounds of a metal and a non-metal. They are solid, inert, hard and brittle. They have a high tolerance to high temperatures and corrosive environments. The properties of ceramics are a result of their crystalline structure and chemical composition. They also exhibit improved mechanical, electrical and optical properties. Some of the commonly used ceramics are silicon carbide, silicon nitride, Barium Titanate, earthenware, etc. My work is based on the study of properties of Barium Titanate and rare earth magnetic powders before and after spin explosive compaction.

Barium Titanate is an inorganic compound which is a white powder and is transparent when crystals are larger. It is a ferroelectric ceramic material that has photorefractive and piezoelectric properties. It can be used with a dielectric material in capacitors. It is also used a piezoelectric material for transducers and microphones. It has a positive temperature coefficient and therefore can be used for thermistors and other self-regulating temperature controlling systems.

The materials studied in this research are Neodymium iron boron and Barium Titanate. The neodymium iron boron material used was Magnequench MQPA, prepared by melt spinning ribbons of NdFeB followed by comminution. The process applied is spin compaction, which applies a shear component to the traditional explosive compaction process. It is anticipated that the materials produced by this technique will have different properties when compared to traditional compaction techniques. These new properties will be due to the shear component having higher amplitude than that of the traditional compaction technique, which may lead to the formation of new phases. One of the advantages of this technique is that it can be done on a smaller scale using a projectile containing the powder to be compacted. This projectile is analogous to the flyer plate and it can be shot at a base plate. The recovered samples can be analysed to study the changes in the microstructure. The experimental procedure to prepare the compacts is discussed in the next chapter.

3 Experimental Techniques

The powders compacted were Magnequench MQP-A-10179-070 (Nd-Fe-B), Magnequench MQP-13-14-20203-070 (Nd-Pr-Nb-Fe-B) and Barium Titanate (IV) (powder, < 2 μm , 99.5% trace metals basis, Sigma Aldrich).

3.1 Spin Compaction Process

The process of spin compaction is shown in the Fig 3-1. It consists of a gun mounted on a holder and a chronograph to measure the velocities of the projectiles. There is a steel plate, which holds the base plate against which the projectile is shot. There are heavy concrete blocks onto which the base plate is clamped for support. A capture box was used surrounding the base plate so as to prevent loss of projectiles. Protective glasses and earmuffs were worn when conducting the experiments. The distance between the base plate and rifle barrel was 30 feet.

3 types of Bushnell guns containing different twists were used for preparing the compacts. They were 1:8, 1:16 and smooth barrel containing no twist. The velocities investigated ranged from 300-490 m/s. The explosive gun powders used were H4227 and SR4256 and the cartridges were of 0.357-inch calibre. The experimental set up on the field is shown in Fig 3-2 and a capture box (Fig 3-3) was used to prevent projectiles from getting lost.

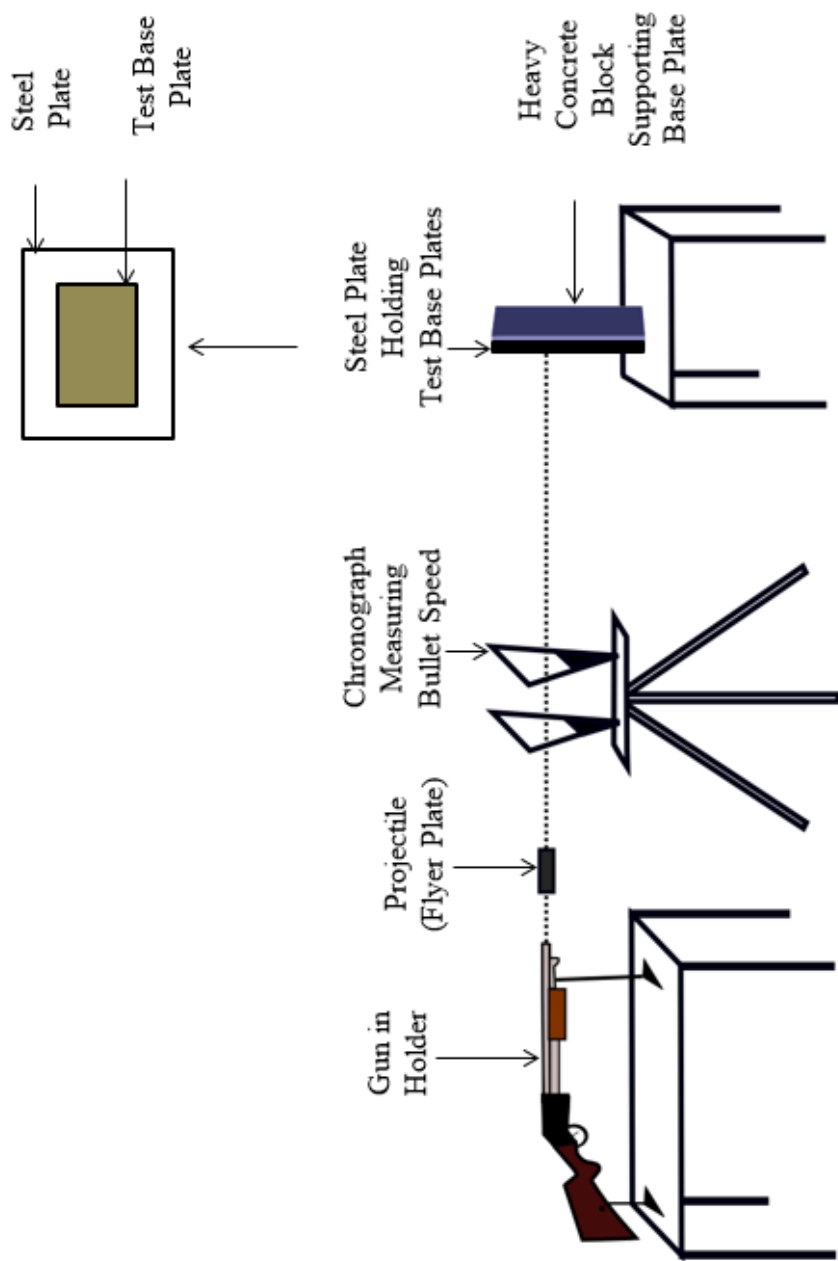


Fig 3-1: Schematic of experimental set up for explosive spin



Fig 3-2: Experimental set-up for explosive spin compaction



Fig 3-3: Capture box to prevent fly aways

The spin velocity of the projectile is dependent on the twist rate of the rifle barrel and the velocity of the projectile as it leaves the barrel. The spin is as shown in figure 3-4.

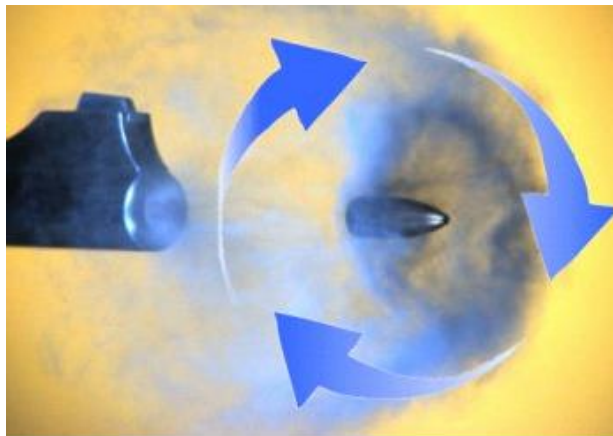


Fig 3-4: Twist in rifle

The bullet RPM is calculated by the formula,

$BRPM = V \times 720 / \text{Twist Rate (in inches)}$ where, V is the velocity. BRPM is obtained as follows:

Twist	Velocity (m/s)	BRPM
1:8	365	32850
	488	43920
1:16	365	16425
	488	21960

Table 3-1: RPM calculation for 1:8 and 1:16 twist

3.2 Preparation of Projectiles

3.2.1 Projectile design

The projectiles consisted of a stainless steel capsule enclosed with caps on both ends into which the powder to be compacted is filled. This is then placed in a copper jacket, crimped and held in place. The function of the stainless steel capsule is to prevent the material from coming into contact with the explosive powder. This entire capsule is then enclosed in the projectile and the explosive powder is loaded. The powders were loaded into the stainless steel capsules using a press and pressed to about 70% volume of the capsule.

The capsule consists of two plugs and the weights before and after filling with the powders were measured and tabulated. The outside diameter of the capsules was carefully controlled by forcing the capsules through dies of appropriate sizes so they do not bulge and cause problems when shooting.



Fig 3-5: Projectiles before loading powders

The powders were loaded by pressing up to approximately 1200 pounds per square inch. The load was measured using a load cell (Fig 3-6).

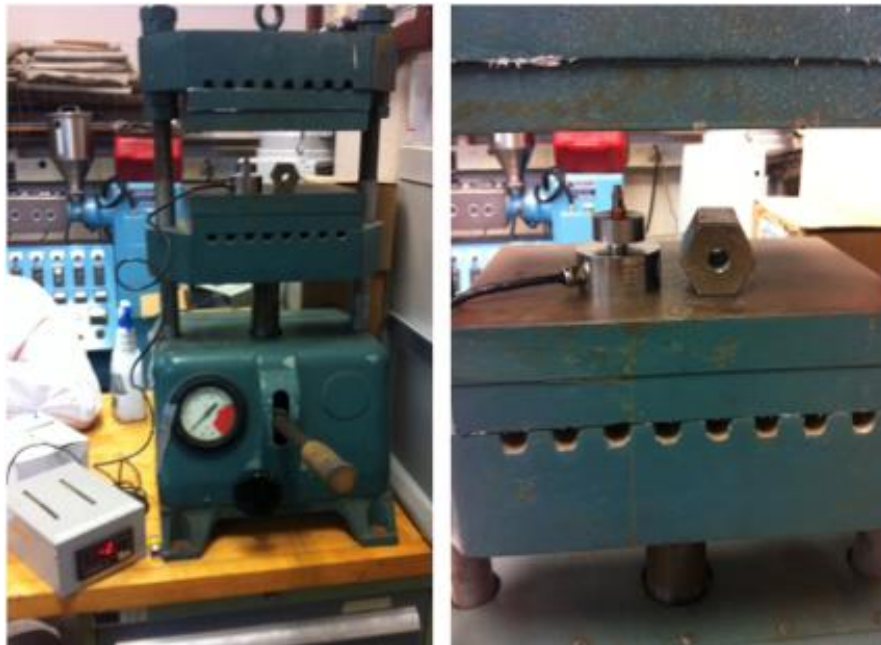


Fig 3-6: Powders compacted using press and load cell

The loaded stainless steel capsules were then pressed into the copper jacket and again forced through a die so that it will fit in the bullet casing. The copper projectiles weighed approximately 125 g. The copper projectiles were then loaded into bullets of .357 mm caliber (fig 3-7) with different powder loads to obtain different velocities during shooting.



Fig 3-7: Loaded projectile

3.2.2 Powder preparation

The initial tests were done with bulk powders of BaTiO₃, MQPA and MQP 13-14. The bulk powders were analysed before compaction using SEM, EDS, XRD and particle distribution analyzer, which will be discussed later. The latter experiments were done using milled powders.

3.2.3 Milling

The particles were milled first using a roller mill and it was seen that the process was not effective to achieve the required sizes. The mill that proved effective was the Glen mills pulverizing mill: model LM1.5-P. The pulverizing mill (figure 3-8) has a stainless steel canister and inside it are a

stainless steel ring and a solid puck that grinds the sample when agitated. In order to prevent oxidation the milling was done with a constant nitrogen flow during the milling process, an inlet and outlet was machined on the lid of the canister and the gas flow was monitored using a flow meter.



Fig 3-8: Parts of pulverizing mill

A flow rate of 1.0 SCFH (standard cubic feet per hour) was maintained and the material was milled. Since it is a high-energy milling machine it had to be done in intervals of 20 seconds to keep the powders below 60°C. The particle sizes were analysed using SEM every five minutes. It was seen that about 60 minutes was needed to obtain nano particles. SEM/EDS analysis was also done to monitor the composition and microstructure.

Some oxidation in initial samples was noted and hence the powders were degassed in a VWR 1410 vacuum oven at 100°C for 2 hours to remove absorbed moisture.

3.3 Analysis of prepared powders

3.3.1 XRD

The XRD analysis was done using the Bruker D8-Discover. The powders were placed on a glass slide and placed on the stage. The x-ray beam was focused onto the powder using the laser beam. The scan was done from 20-80 degrees and the peaks were indexed. It was done in different positions on the sample material and the data was collected using the Diffrac software. The data was then imported into an excel file that can be used to index the plots. The Barium Titanate particles were also analysed using the same method. The peaks were obtained for the powders from 20-65 degrees and studied.

3.3.2 SEM/EDS

The MQP13-14 and MQPA powders were placed on an aluminum stub with a double-sided adhesive carbon tape on it. The stub was then placed in a sample holder and mounted in the Jeol JSM-7000F SEM (Scanning Electron Microscope). The images were then captured starting at 100x to about 15000x and the different phases were observed. The working distance was set at about 10mm and voltage was 20kV. The particle sizes were measured. The EDS was also done for the samples and the composition was obtained. The Barium Titanate powders were mounted in a similar way on the stub. A thin film of gold was sputter deposited using a Pelco SC-6 sputter coater, on the material to make it conducting as it is an insulating material. The composition was

studied using EDS (Energy dispersive spectroscopy) analysis. Fig 3-9 shows an aluminum stub with adhesive carbon tape and powders.



Fig 3-9: Aluminum stub with powders for SEM analysis

The Barium Titanate powders were mounted in a similar way on the stub. A thin film of gold was sputter deposited on the material to make it conducting as it is an insulating material. The composition was studied using EDS analysis. The milled powders were also analysed similarly.

3.3.3 Particle distribution analysis

The particle sizes were analysed using a Microtrac s3500 Particle Distribution Analyzer (Fig 3-10). The principle of this instrument is that light scatters more intensely and at smaller angles off of larger particles than smaller particles. The powders were fed in at a set speed of 10 seconds by using the Turbotrac auto feed feature. The graphs for particle size vs percentage of passing were

plotted using the microtrac software. The information about the largest particle and the smallest particle detected was obtained from the data. The measurement capability for this instrument is from 0.02 to 2800 microns.



Fig 3-10: Microtrac S3500 particle analyzer

To measure particle sizes below this range a Zeta sizer was used. About 1 mg of the milled particle was dispersed in pure ethanol and particle size distribution was measured by laser diffraction. Particles of sizes ranging from 2nm to 20 microns were measured using this technique.

Another method is by using a Hosikawa Mikro Air Jet sieve (Fig 3-11). The particle size was studied by percentage of passing and weight loss when the material was sieved with the increasing mesh sizes. The meshes used were 20, 25, 32 and 53 microns in size.

3.3.4 Retrieving compacts

After spin compaction notes were made about the projectiles and pictures were taken with a handheld Dinolite microscope. The compacts were removed carefully from the opened and unopened projectiles using a Dremel tool.



Fig 3-11: Hosikawa Mikro Air Jet sieve

3.3.5 Capacitance measurement

The Barium Titanate compact was covered with a thin film of silver paint and the capacitance values were measured using a Craftsman 82400 multimeter.

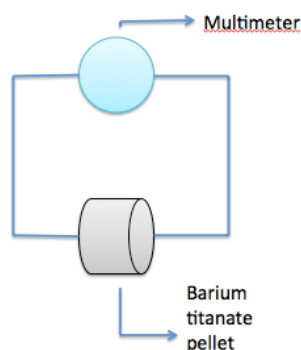


Figure 3-12 Capacitance measurement

3.3.6 Density measurement

Density measurements were done using liquid displacement method with methanol in a graduated cylinder. The mass of the compact was measured in a weighing scale. The initial level of methanol in the 5cc graduated cylinder was noted and the compact was dropped into the cylinder. The final level is noted. The liquid displacement was measured by the difference between final level and initial level. If air bubbles were seen, they were removed by gently tapping against the wall of the cylinder. The density is calculated by the following formula:

$$\text{Density} = \text{Mass of compact} / \text{volume of displaced liquid}$$

3.3.7 Magnetic measurement

The B-H curve was plotted with the data obtained by analyzing the samples with a Quantum Design VersaLab Vibrating Sample Magnetometer (VSM).

4 Results & Discussion

4.1 Before Compaction

The bulk powder of Magnequench NFB and Barium Titanate were characterized before compaction process using Particle Distribution Analysis, Scanning Electron Microscopy and X-Ray Diffraction.

4.1.1 Magnequench NFB

The bulk particles, as purchased from the manufacturer, were analysed using the Microtrac S3000 unit which employs a laser diffraction process. Three trials were carried out and the results obtained were identical - the particle sizes were confirmed to be in the range of 100-700 microns with majority falling between 300-500 microns (Fig 4-1). The particles were then analysed using SEM (Fig 4-2)

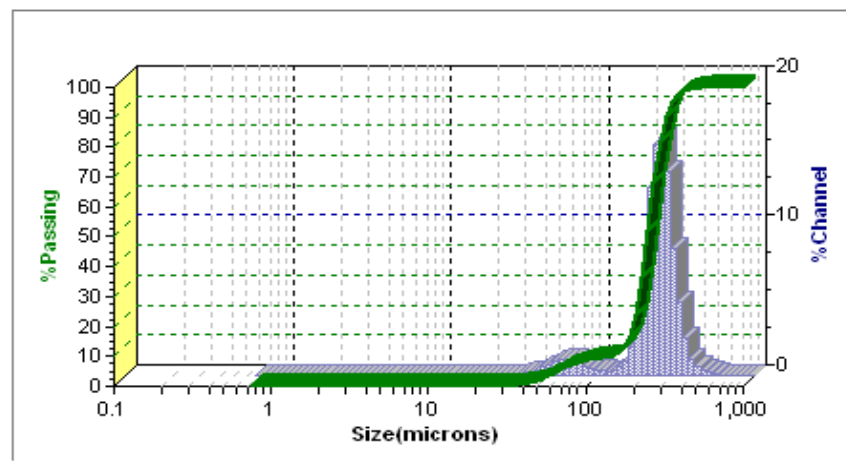


Fig 4-1: Particle size distribution for bulk MQPA powder (Microtrac S3000)

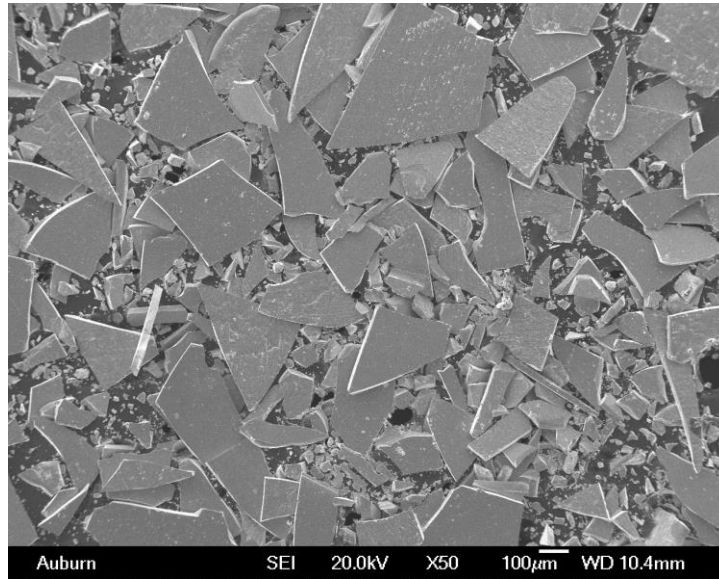


Fig 4-2: SEM image for MQPA13-14 before compaction

The XRD pattern (Fig 4-3) was obtained for MQPA-bulk material using the XRD (Bruker).

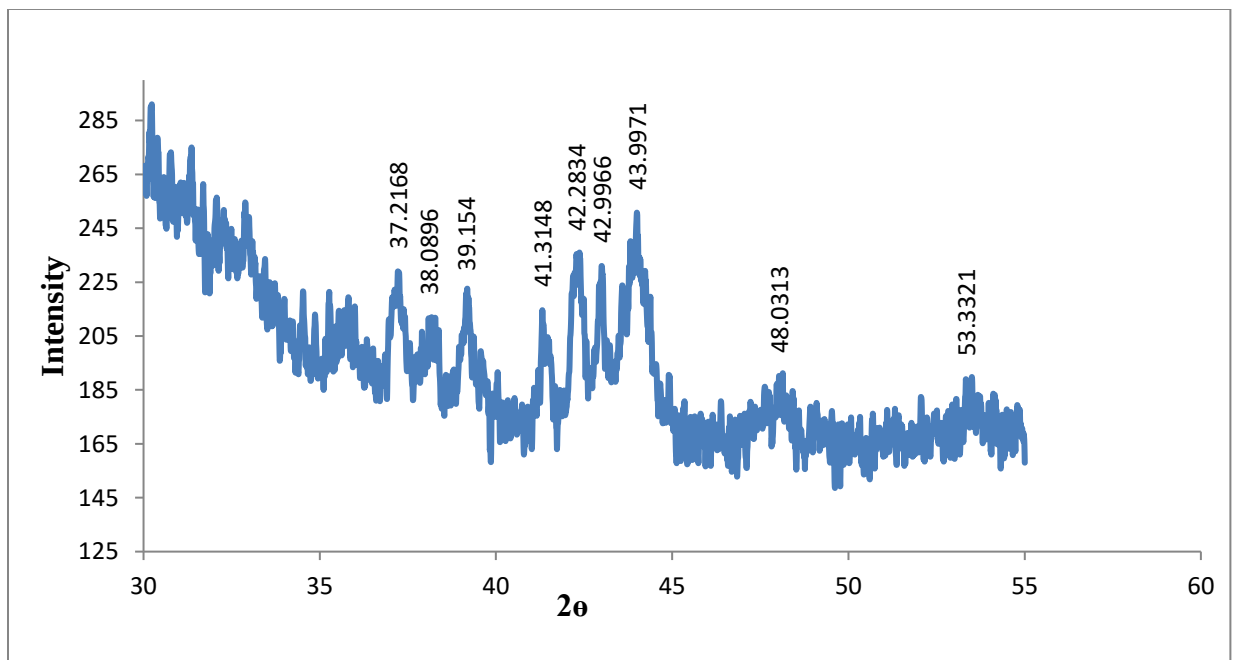


Fig 4-3: XRD pattern for bulk MQPA 13-14

All the major peaks were evident (Fig 4-3). The same scan was carried out after milling the material for 60 minutes. The peak with the highest intensity was 43.997 degrees and is in accordance with data.

The material was then milled using the Glen Mills pulverizing mill and subsequently analysed using Microtrac S3000 Particle distribution analyzer, Zeta sizer, SEM and XRD. The following image (Fig 4-4) shows the distribution as analysed by Turbotrac feature in Microtrac S3000.

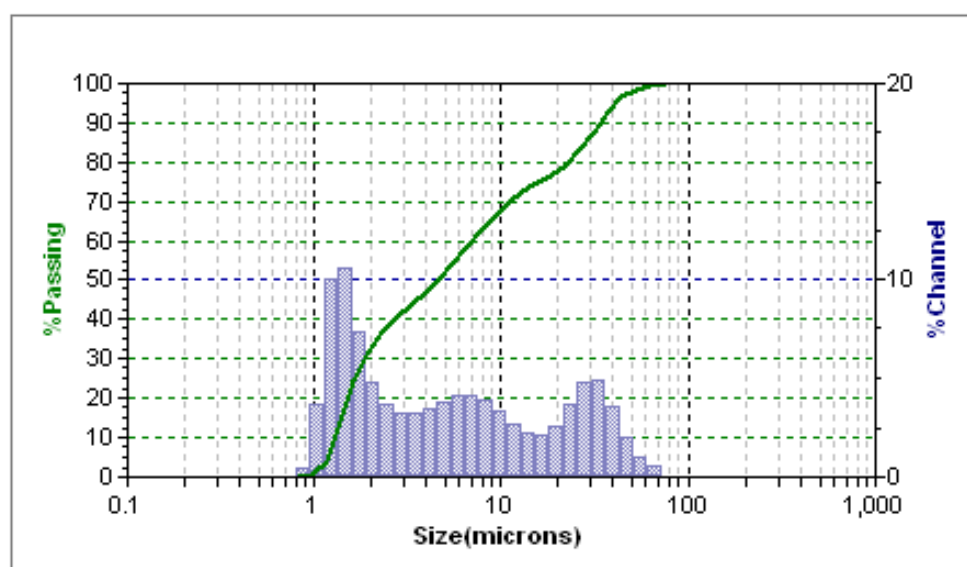


Fig 4-4 Particle size distribution for milled MQPA 13-14 powder (Microtrac S3000)

It was seen that the smallest particles detected were about 1 micron, which is the lowest size that can be determined by this instrument. The test was repeated 3 times and the same results were obtained. The particles were then passed through the zeta sizer.

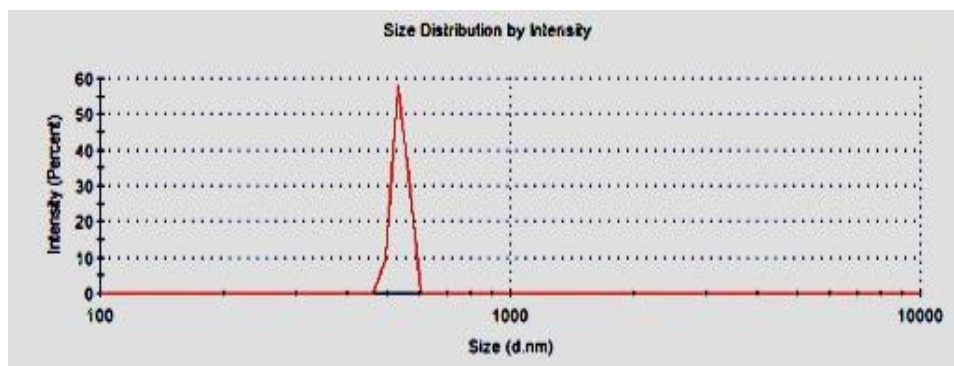


Fig 4-5: Particle size distribution for milled MQPA 13-14 powder (Zeta sizer)

However the same material, when dispersed in ethanol and analysed by Zetasizer showed a distribution where majority of the particles were in the range of 700nm. The particles were then analysed using the SEM (Jeol).

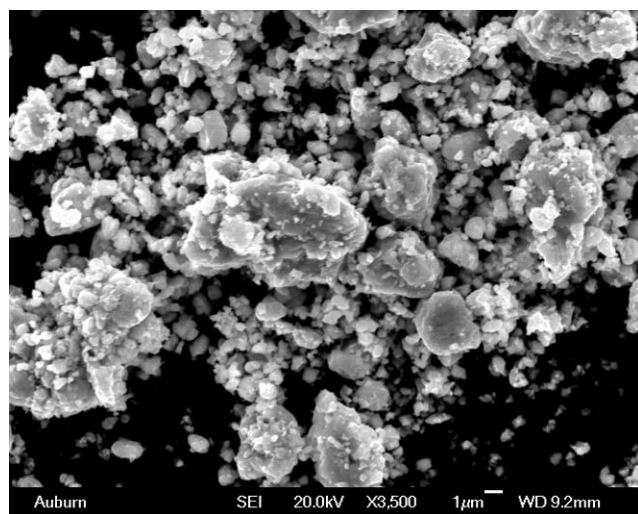


Fig 4-6: SEM image for milled MQPA 13-14 (x3,500)

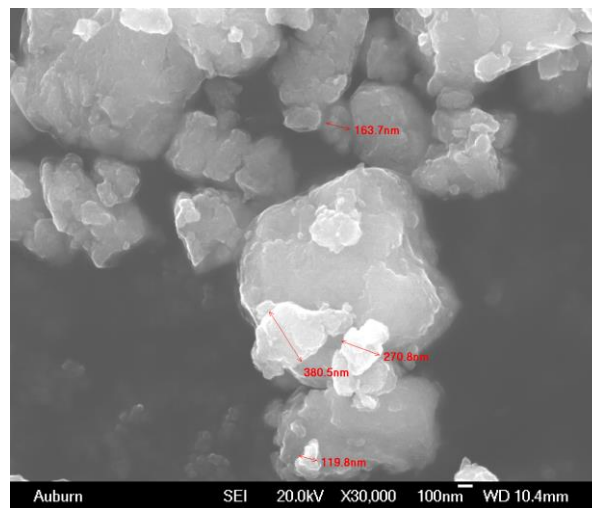


Fig 4-7: SEM image for milled MQPA 13-14 (x30,000)

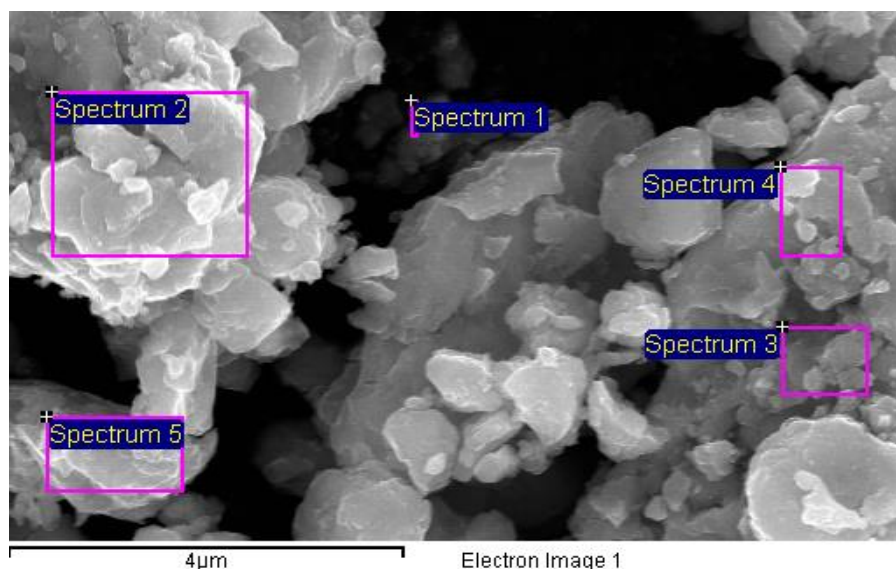


Fig 4-8: EDS information at different locations on the sample

It was seen that the smallest particles were about 100 nm and the largest were in micron scale.

Spectrum	In stats.	O	Fe	Zn	Nd	Total
Spectrum 1	Yes		100.00			100.00
Spectrum 2	Yes	14.14	62.59	0.00	23.28	100.00
Spectrum 3	Yes	13.12	60.93		25.95	100.00
Spectrum 4	Yes	16.55	61.44		22.01	100.00
Spectrum 5	Yes	18.32	62.68		18.99	100.00
Max		18.32	100		25.95	
Min		13.12	60.93		18.99	

Fig 4-9: Composition of the material in weight percentage

Oxide formation was seen in the milled particles from the EDS data obtained and the particles were then analysed using XRD (Fig 4-9). The peak's broadening and change in intensity that was observed is due to significant change in size of the particles. The XRD analysis showed that the diffraction peaks of milled particles broadened into big humps. Apart from the decrease in the

grain size, the strains and amorphization formed due to the milling process also contributed to the broadening of the diffraction peaks. The same pattern (Fig 4-10) was obtained after pre-compaction, i.e., green compact formed.

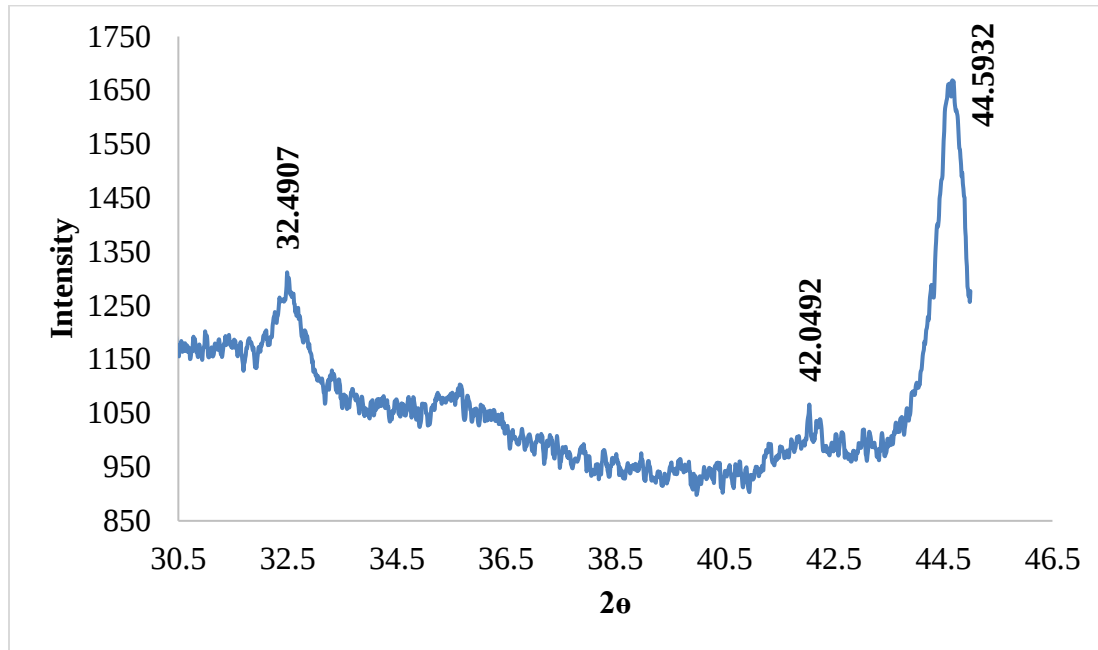


Fig 4-10: XRD pattern for milled MQPA

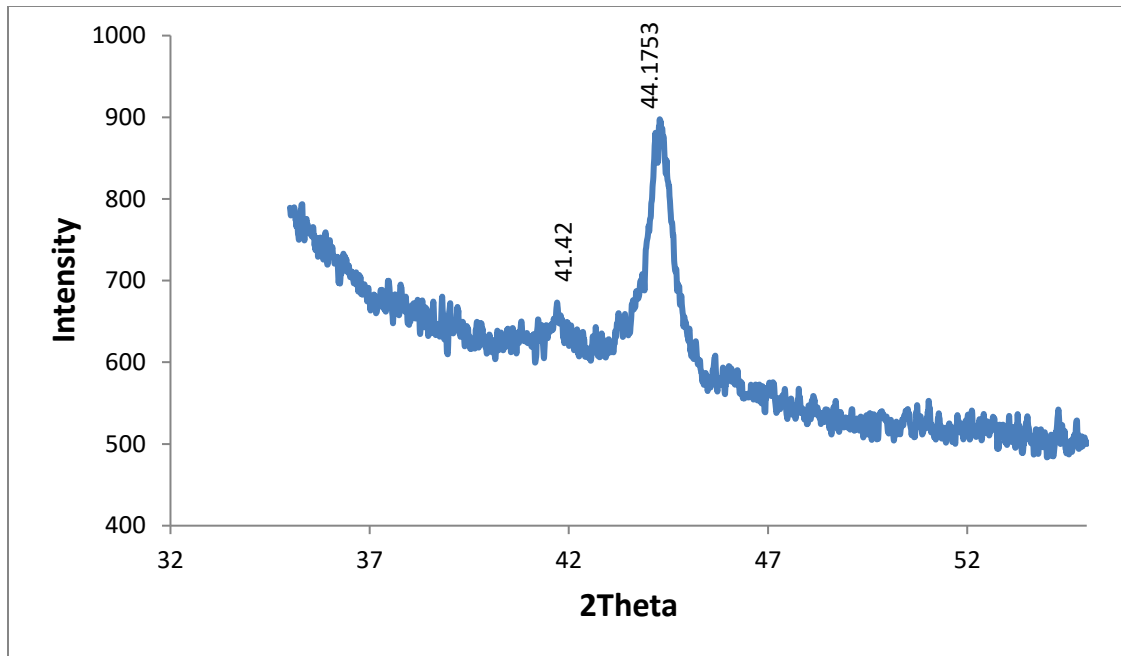


Fig 4-11: XRD pattern for pre compacted milled MQPA 13-14

Only two major peaks were seen at 41.42 and 44.18 (Fig 4-11). The peak broadening was seen when the particle sizes were smaller and in nano scale. This is in accordance with the Scherrer equation,

$$D = \frac{K\lambda}{\beta \cos \theta} = \frac{K\lambda}{D \cos \theta}$$

where, D = Grain diameter

β = peak width in radians at FWHM

k = 0.9 to 1.0 depending upon grain shape.

λ = wavelength of x-ray.

4.1.2 Barium Titanate

Barium Titanate particles were studied in a similar method followed for Magnequench NFB beginning with particle size distribution (Fig 4-12).

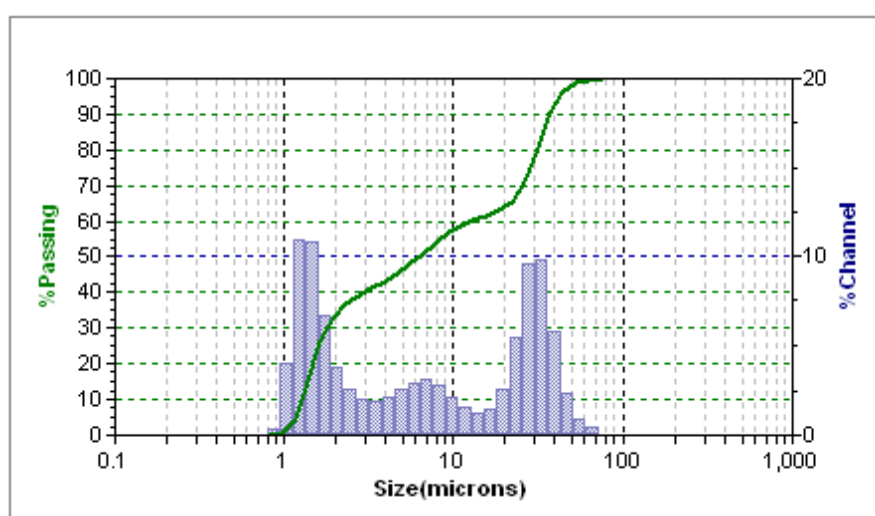


Fig 4-12: Particle size distribution of bulk Barium Titanate powder (Microtrac S3000)

The following is the data obtained for bulk Barium Titanate for XRD and SEM analysis:

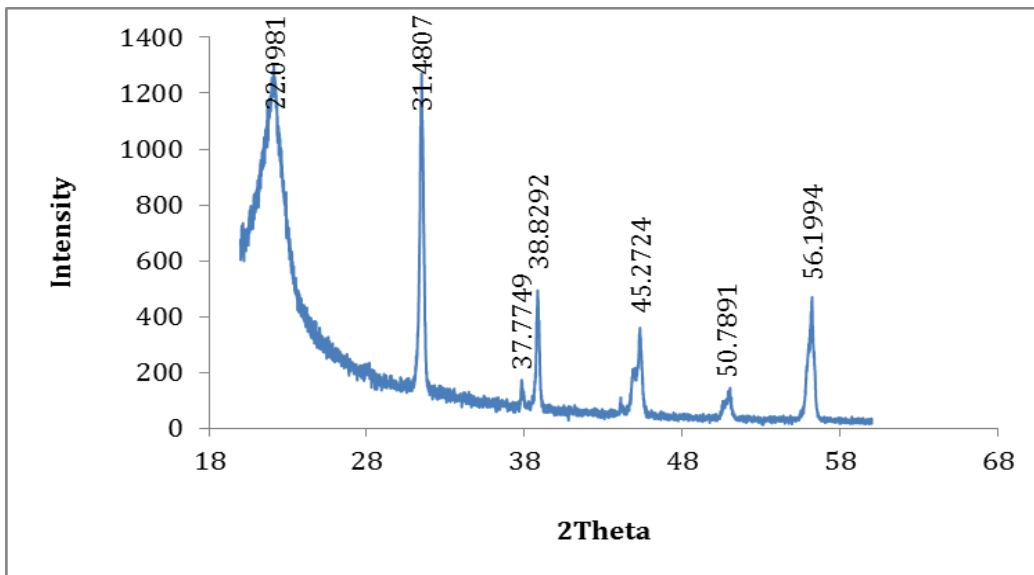


Fig 4-13: XRD pattern for bulk Barium Titanate

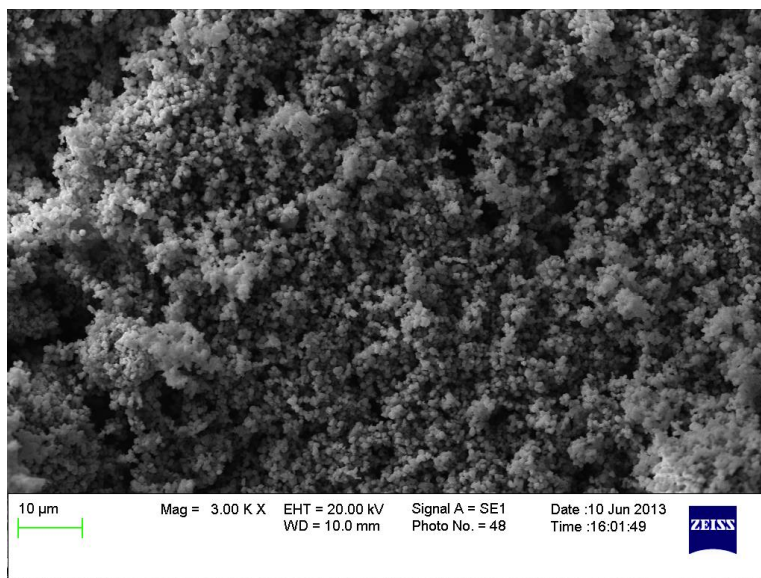


Fig 4-14: SEM image for bulk Barium Titanate

The Barium Titanate powder were then milled using the Glen mills pulverizing mill for about 30 minutes and passed through the Microtrac S3000. The particle distribution graph is as follows.

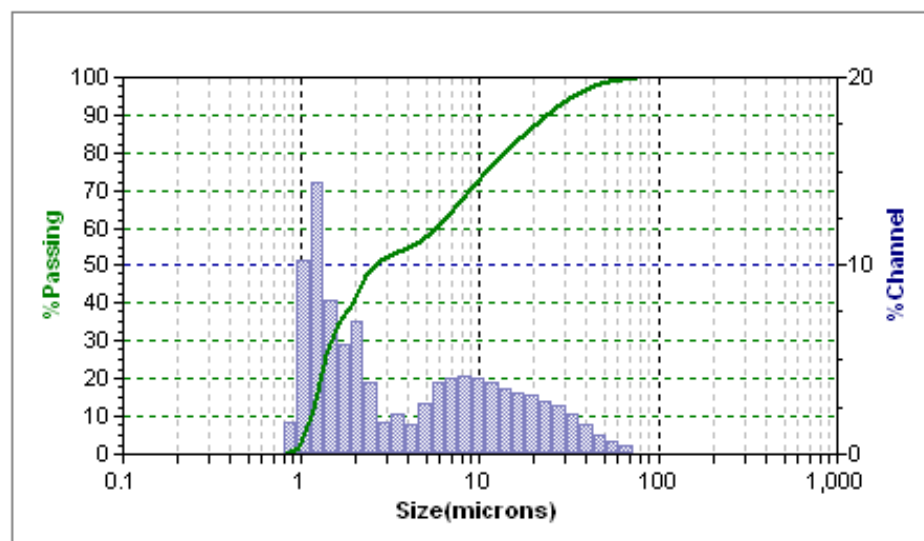


Fig 4-15: Particle size distribution of milled Barium Titanate (Microtrac S3000)

It was seen that after milling most of the particles were less than 5 microns in size, the particles clumped together and so the distribution showed large particle sizes.

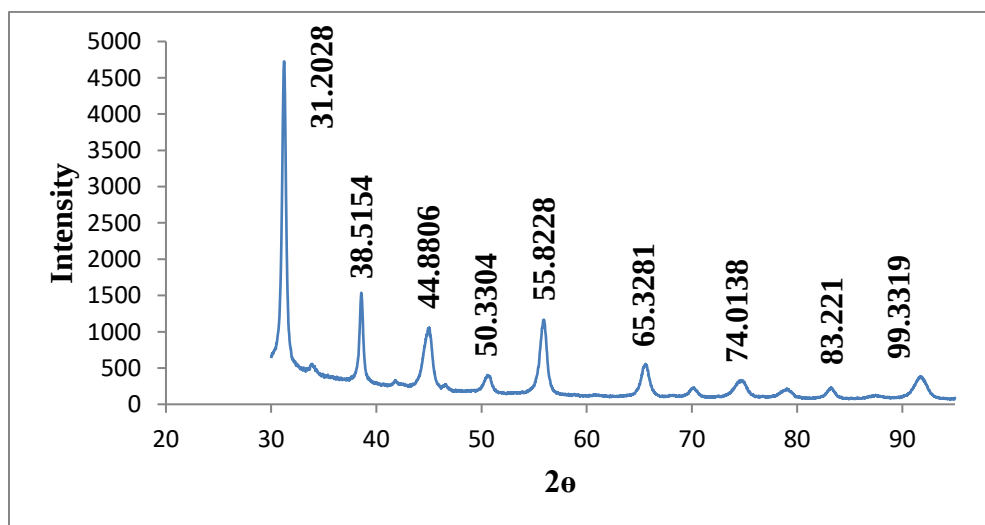


Fig-4-16: XRD pattern for milled Barium Titanate

The XRD peaks for milled Barium titanate particles showed higher intensity than that of bulk particles. The pre-compacted Barium Titanate was also analysed using XRD.

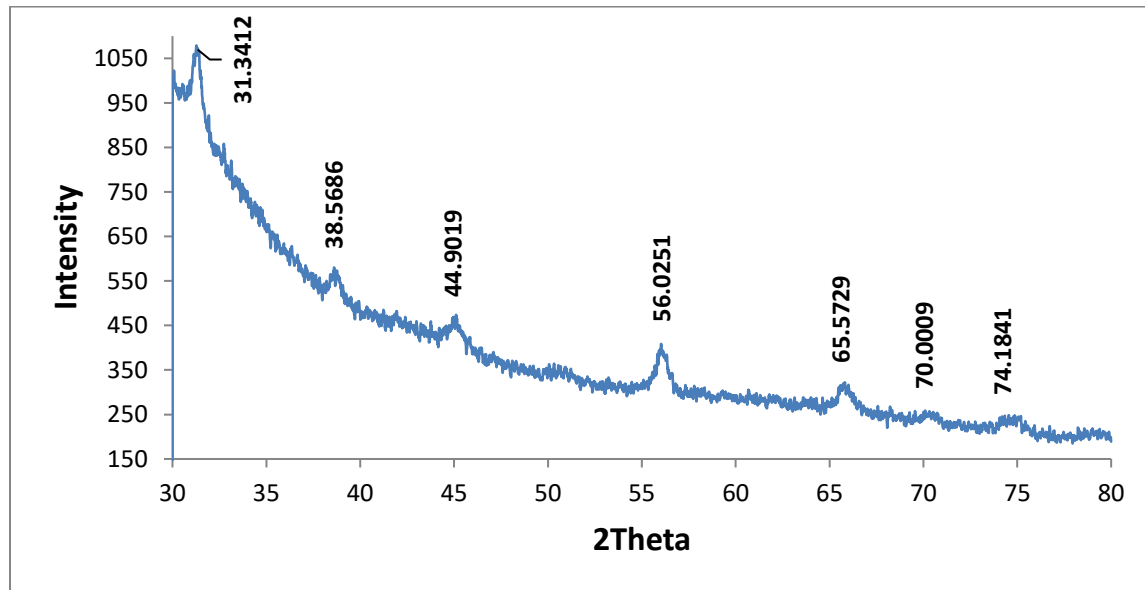


Fig 4-17: XRD pattern for pre compacted Barium Titanate

4.2 After Spin Compaction

Spin compaction process was carried out, as illustrated in the previous chapter, on the following and the results were tabulated as below::

1. M1 – Magnequench Nd-Fe-B
2. M2 – Magnequench Nd-Pr-Nb-Fe-B and
3. B1 – Barium Titanate

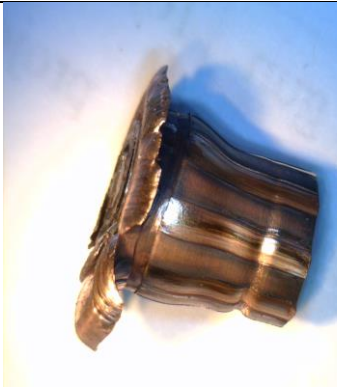
No.	Material Composition	Sample	Twist (inches)	Velocity (m/s)	Rotational speed (/s)	Compact
1	M1 (Bulk)	A1	1:16	539.19	9468.479703	No
2	M1 (Bulk)	A2	1:16	463.6	8141.076783	No
3	M1 (Bulk)	A3	1:16	413.4	7259.536544	No
4	M1 (Bulk)	A4	1:16	264.96	4652.846644	Yes
5	M2 (Bulk)	A5	1:16	535.22	9398.76427	No
6	M2 (Bulk)	A6	1:16	445.92	7830.606037	No
7	M2 (Bulk)	A7	1:16	384.25	6747.646147	Yes
8	M2 (Bulk)	A8	1:16	266.27	4675.850981	Yes
9	B1 (Bulk)	A9	1:16	553.51	9719.946959	No
10	B1 (Bulk)	A10	1:16	482.19	8467.52764	No
11	B1 (Bulk)	A11	1:16	439.82	7723.486606	Yes
12	B1 (Bulk)	A12	1:16	310.89	5459.403281	No
13	M1 (Bulk)	B1	1:16	357.8	6283.169269	Yes
14	M1 (Bulk)	B2	1:16	382.2	6711.646994	No
15	M1 (Bulk)	B3	1:16	Error	NA	No
16	M1 (Bulk)	B4	1:16	Error	NA	No
17	M2 (Bulk)	B5	1:16	392.5	6892.520788	Yes
18	M2 (Bulk)	B6	1:16	376.1	6604.527563	No
19	M2 (Bulk)	B7	1:16	352.7	6193.6104	No

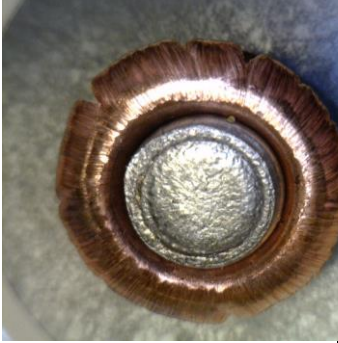
20	M2 (Bulk)	B8	1:16	413	7252.512319	No
21	B1 (Bulk)	B9	1:16	385.26	6765.382315	No
22	B1 (Bulk)	B10	1:16	386.79	6792.249976	No
23	B1 (Bulk)	B11	1:16	365.94	6426.112247	No
24	B1 (Bulk)	B12	1:16	425.19	7466.575577	No
25	M1 (Milled)	C1	1:16	366.67	6438.931458	No
26	M1 (Milled)	C2	1:8	342.59	6016.073113	Yes
27	M1 (Milled)	C3	1:16	344.78	6054.530745	Yes
28	M1 (Milled)	C4	Smooth	323.39	5678.910312	No
29	M1 (Milled)	C5	1:16	343.81	6037.496999	Yes
30	M1 (Milled)	C6	1:8	301.44	5293.455965	Yes
31	M2 (Milled)	D1	1:8	256.32	4501.123384	No
32	M2 (Milled)	D2	1:16	262.15	4603.501464	Yes
33	M2 (Milled)	D3	Smooth	311.81	5475.558998	No
34	M2 (Milled)	D4	1:16	323.39	5678.910312	Yes
35	M2 (Milled)	D5	1:8	285.87	5020.038007	Yes
36	M2 (Milled)	D6	Smooth	355.39	6240.848313	No
37	M2 (Milled)	D7	1:16	285.87	5020.038007	No
38	M2 (Milled)	D8	1:8	358.44	6294.408029	Yes
39	M2 (Milled)	D9	1:8	373.38	6556.762833	No
40	B1 (Milled)	D10	1:16	261	4583.306817	No

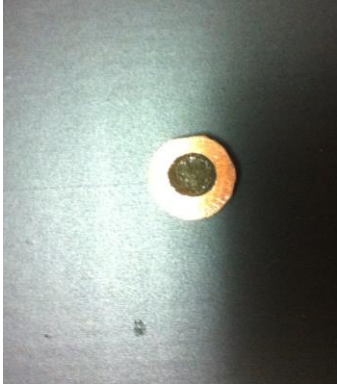
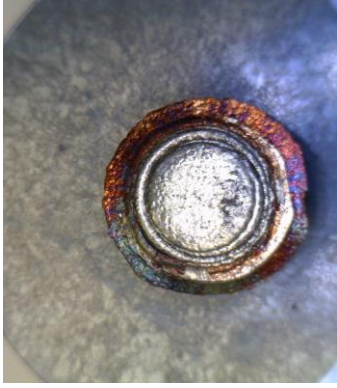
41	B1 (Milled)	D11	1:16	336.4	5907.373231	Yes
42	B1 (Milled)	D12	1:16	289.7	5087.294961	Yes
43	B1 (Milled)	D13	1:8	177.7	3120.511959	No
44	B1 (Milled)	D14	1:8	Stuck in barrel	NA	No
45	B1 (Milled)	D15	1:8	Stuck in barrel	NA	No
46	B1 (Milled)	E1	1:8	Stuck in barrel	NA	No
47	B1 (Milled)	E2	1:16	Stuck in barrel	NA	No
48	B1 (Milled)	E3	1:16	500	8780.281258	Yes
49	B1 (Milled)	E4	1:8	Stuck in barrel	NA	No
50	B1 (Milled)	E5	1:8	617.7	10847.15947	No
51	B1 (Milled)	E6	1:8	788.3	13842.99143	No
52	B1 (Milled)	E7	1:8	990.5	17393.73717	No
53	B1 (Milled)	E8	1:16	878.1	15419.92995	No
54	B1 (Milled)	E9	1:16	Stuck in barrel	NA	No
55	B1 (Milled)	E10	Smooth	1114	19562.46664	No

Table 4-1: List of all samples

No	Sample	Spin (inches)	Velocity (m/s)	Picture	SEM	XRD	Density
1	A4	1:16	264.96		Yes	Yes	Yes
2	A7	1:16	384.25		Yes	Yes	Yes
3	A8	1:16	266.27		Yes	Yes	Yes

4	A11	1:16	439.82		Yes	Yes	Yes
5	B1	1:16	357.8		Yes	Yes	No
6	B5	1:16	392.5		Yes	Yes	No

7	C2	1:16	342.59		Yes	No	Yes
8	C3	1:8	343.81		Yes	No	Yes
9	C5	1:16	344.78		No	No	No

10	D4	1:8	262.15		Yes	No	Yes
11	D5	1:8	285.87		Yes	No	No
12	D8	1:8	358.44		Yes	No	No

13	D11	1:16	336.4		No	Yes	Yes
14	D12	1:16	289.7		No	Yes	No

Table 4-2: List of successful samples

4.2.1 Characterisation of recovered compacts

1. Compact: A4

Material: M1 material bulk

Twist: 1:16

Velocity: 264.96 m/s

Rotational Speed: 4653 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density: 6.044 g/cc

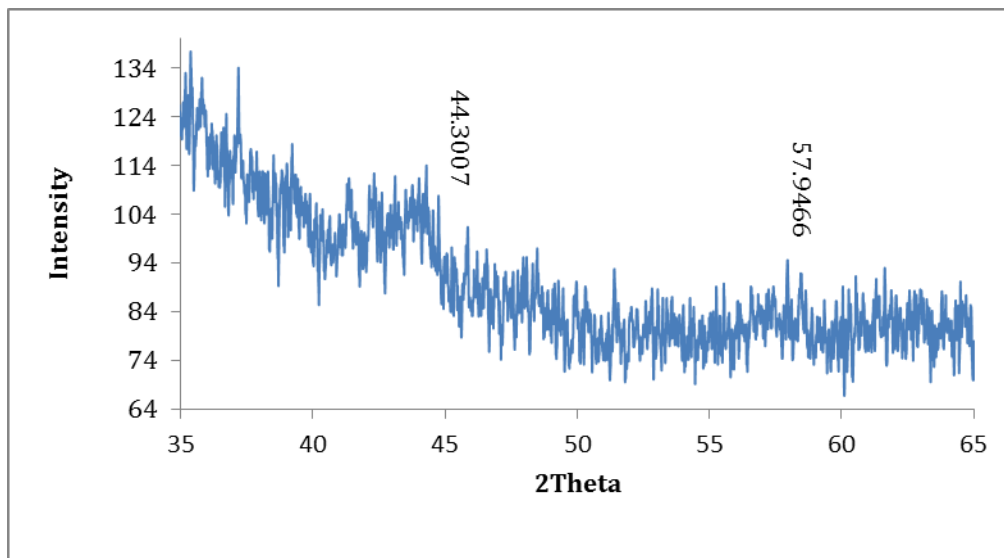


Fig 4-18: XRD pattern for A4 compact

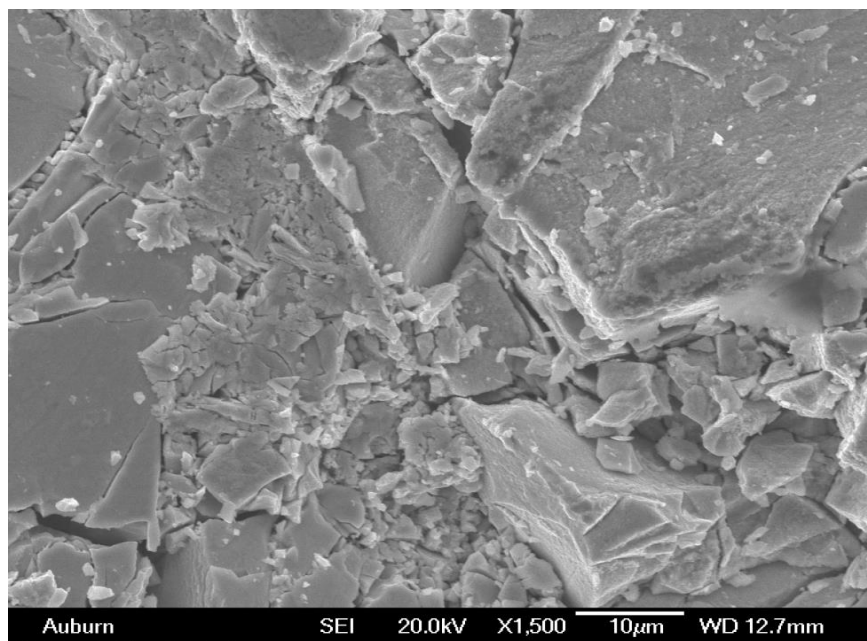


Fig 4-19: SEM image of A4 compact

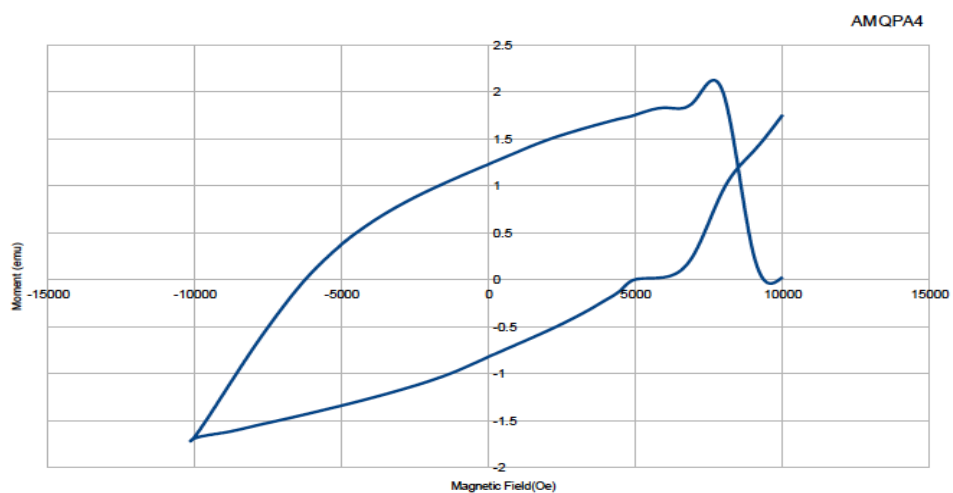


Fig 4-20: Magnetization curve for A4 compact

2. Compact: A7

Material: M2 material bulk

Twist: 1:16

Velocity: 384.35 m/s

Rotational Speed: 6749.4 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.90 g/cc

Compacted density: 7.32 g/cc

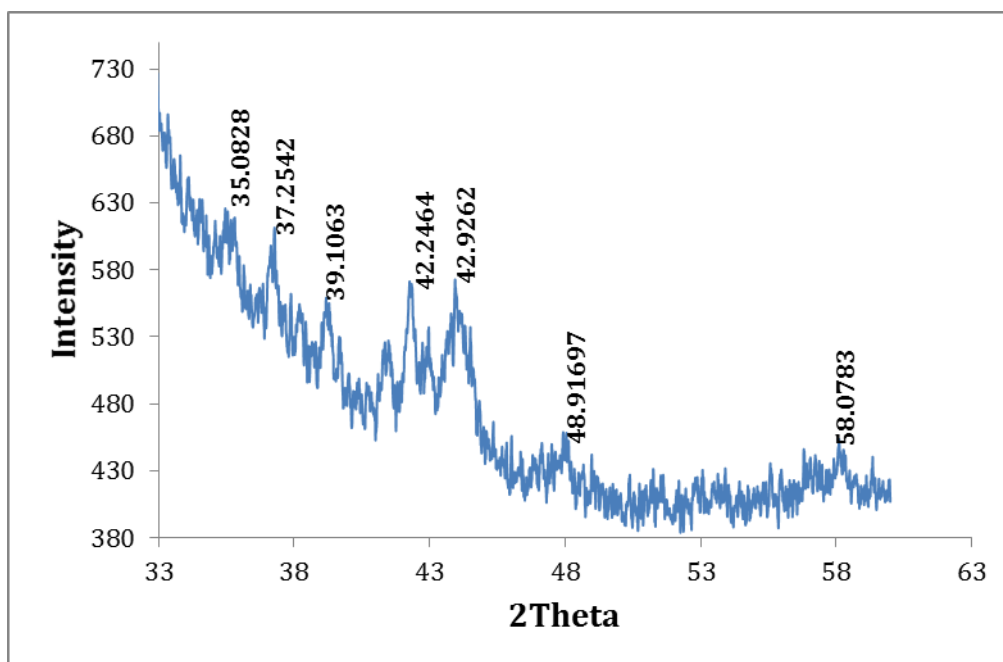


Fig 4-21: XRD pattern for A7 compact

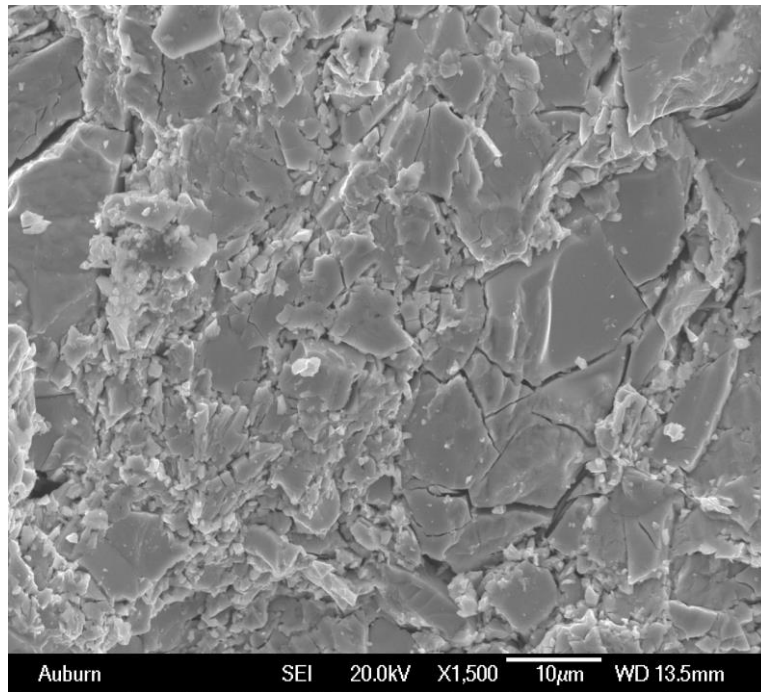


Fig 4-22 SEM image of A7 compact

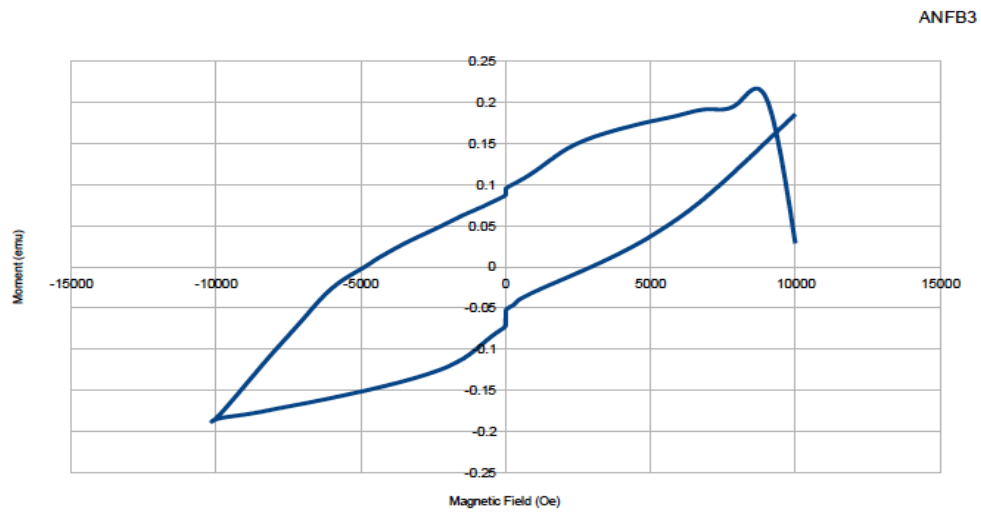


Fig 4-23: Magnetization curve for A7 compact

3. Compact A8

Material: M2 material bulk

Twist: 1:16

Velocity: 266.27 m/s

Rotational Speed: 4675.85 /s

Theoretical Density: 5.853 g/cc

Apparent Density: 2.7 g/cc

Green Density: 3.790 g/cc

Compacted density: 5.853 g/cc

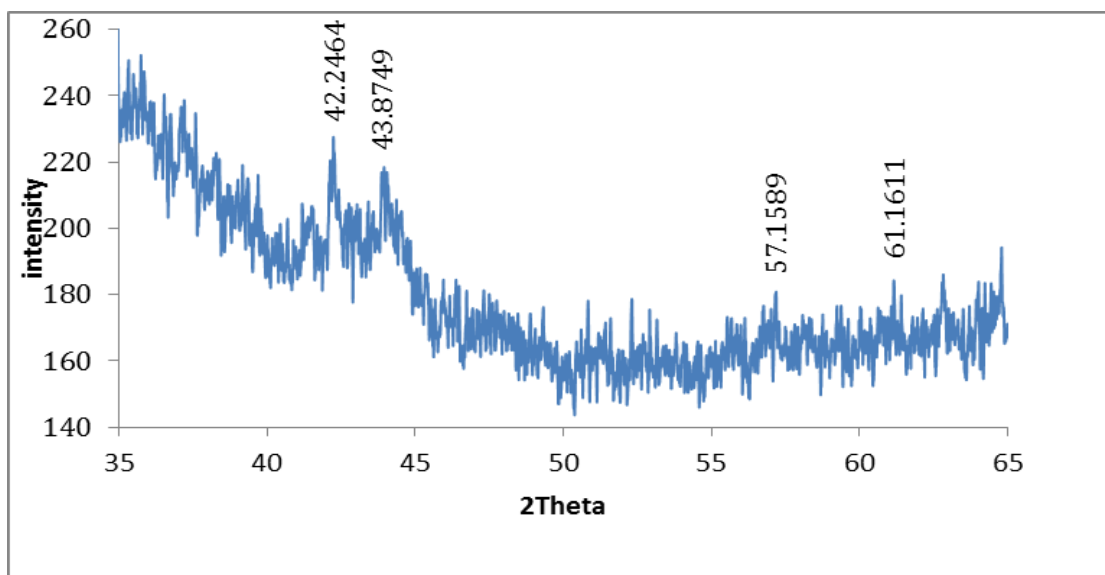


Fig 4-24: XRD pattern for A8 compact

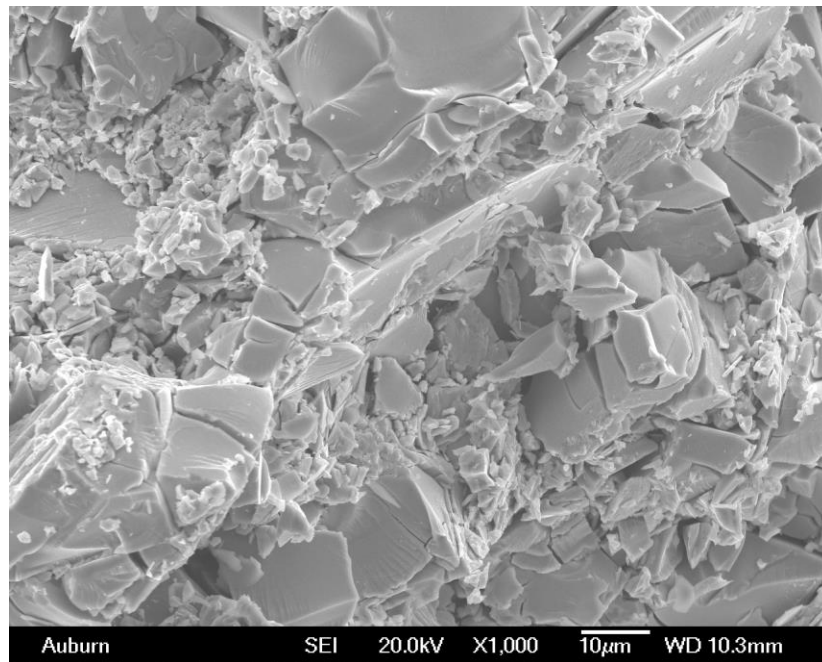


Fig 4-25: SEM image for A8 compact

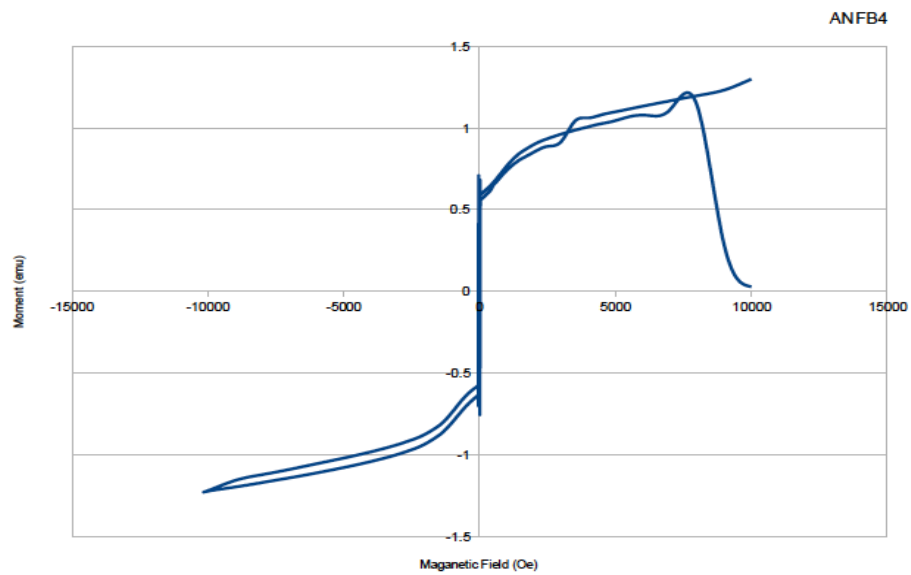


Fig 4-26: Magnetization curve for A8 compact

4. Compact: B1

Material: M1 material bulk

Twist: 1:16

Velocity: 264.96 m/s

Rotational Speed: 4653 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density: 6.044 g/cc

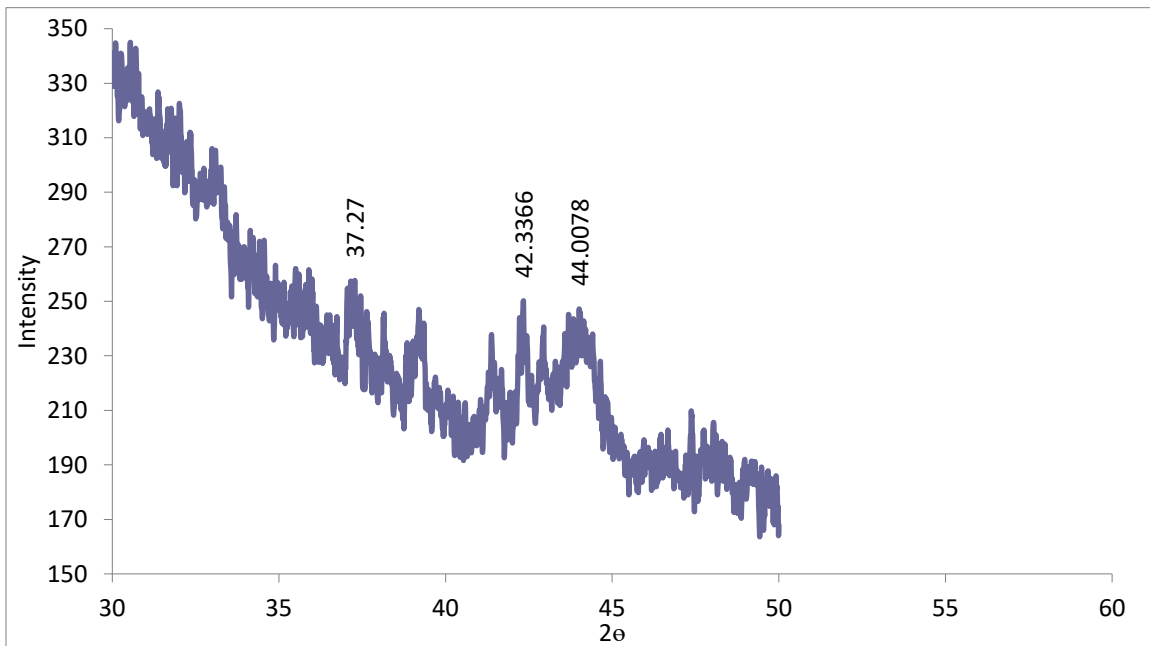


Fig 4-27: XRD pattern for B1 compact

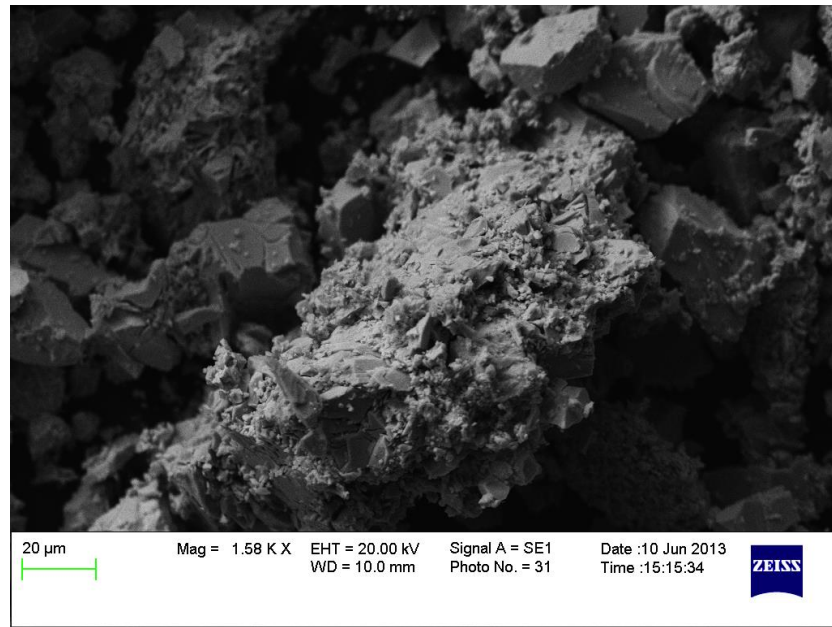


Fig 4-28: SEM image for B1 compact

5. Compact: B5

Material: M2 material bulk

Twist: 1:16

Velocity: 392.5 m/s

Rotational Speed: 6892.52 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density could not be estimated due to the size of the compact obtained; a measurable displacement of fluid could not be seen.

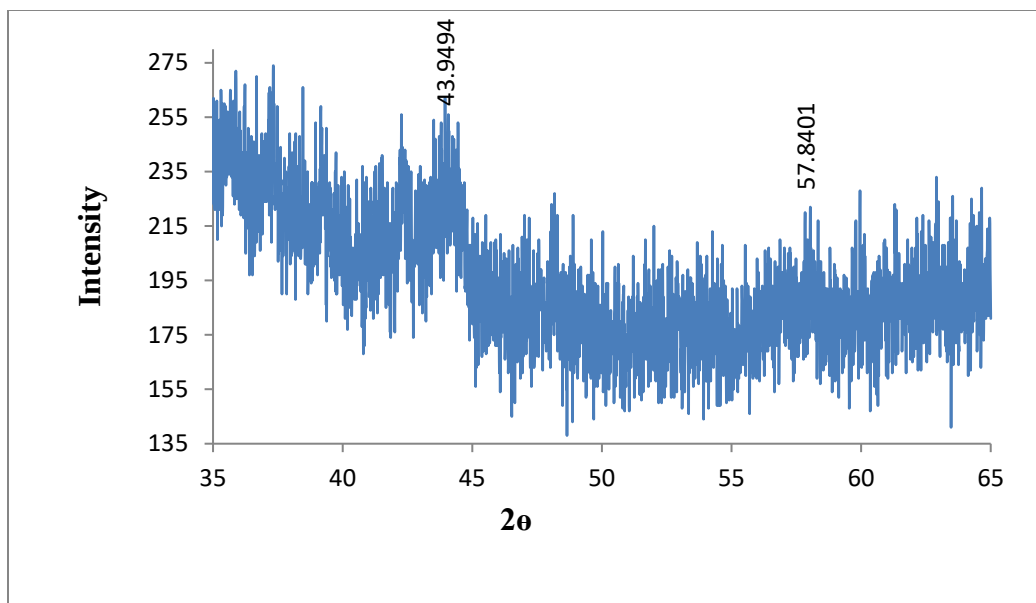


Fig 4-29: XRD pattern for B5 compact

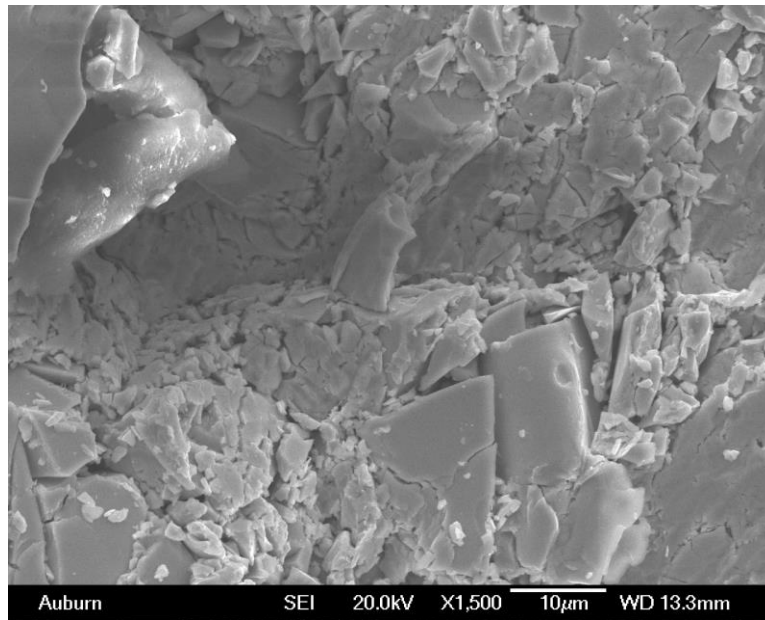


Fig 4-30: SEM image for B5 compact

6. Compact: C2

Material: M1 material milled

Twist: 1:8

Velocity: 366.67 m/s

Rotational Speed: 5678.91 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 5.3 g/cc

Compacted density: 5.32 g/cc

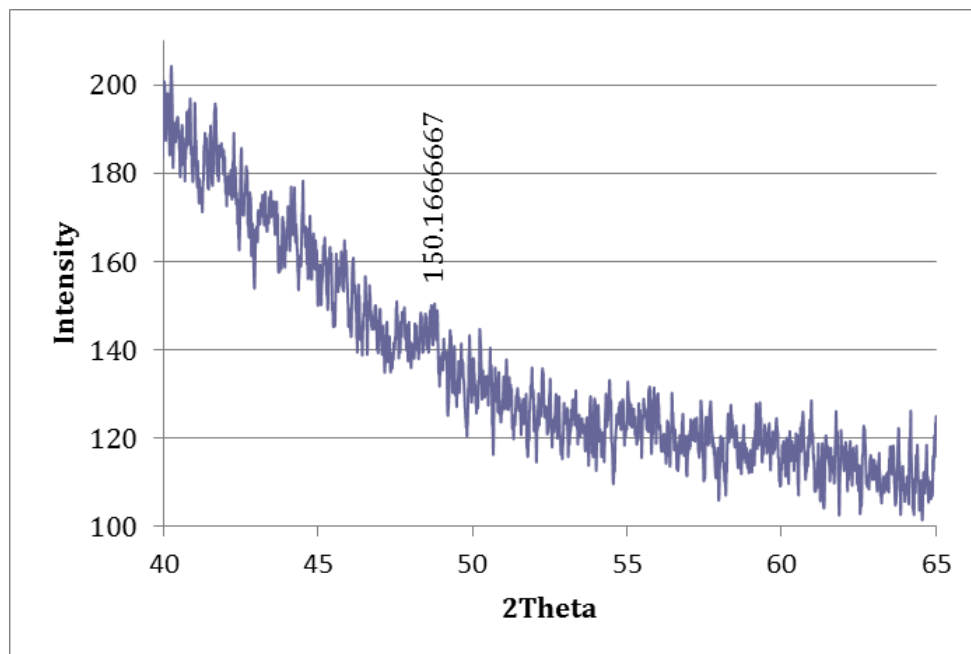


Fig 4-31: XRD pattern for C2 compact

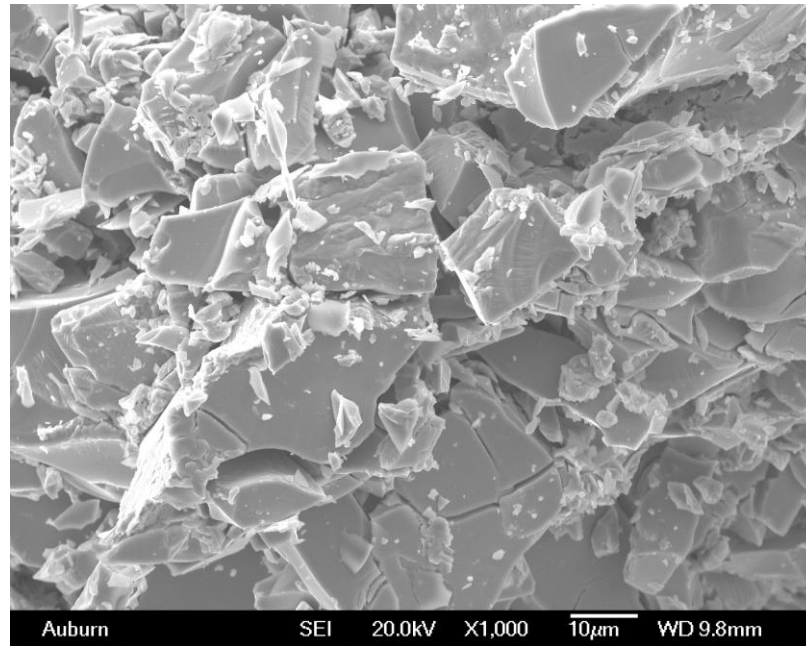


Fig 4-32: SEM image for C2 compact

Compact: C6

Material: M1 material milled

Twist: 1:8

Velocity: 301.44 m/s

Rotational Speed: 5293.45 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density: 6.023 g/cc

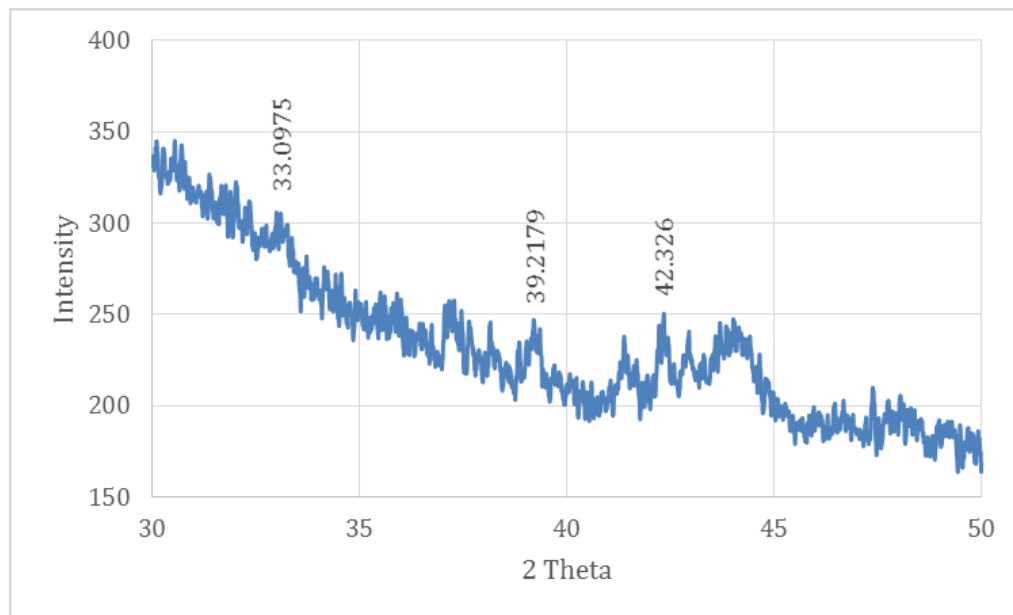


Fig 4-33: XRD pattern for C6 compact

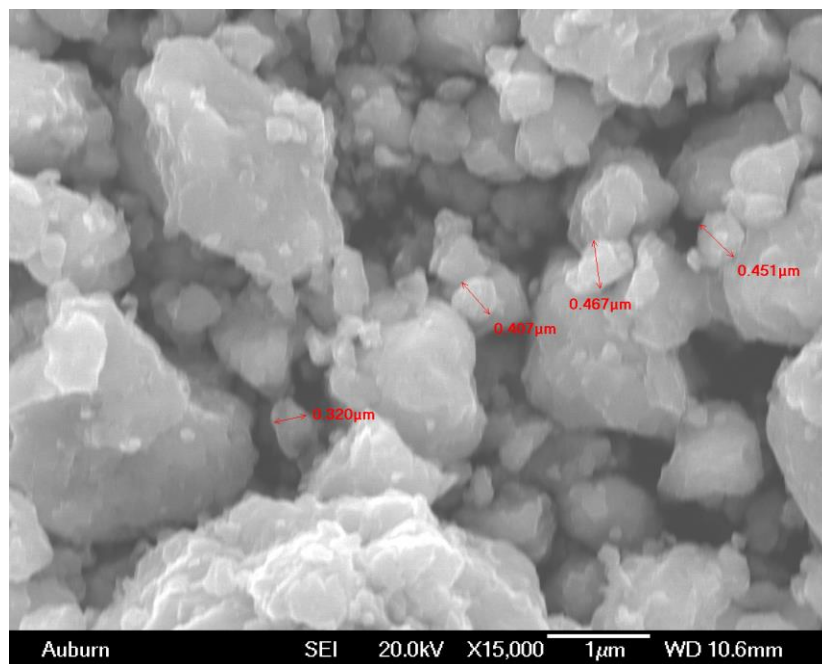


Fig 4-36: SEM image for C6 compact

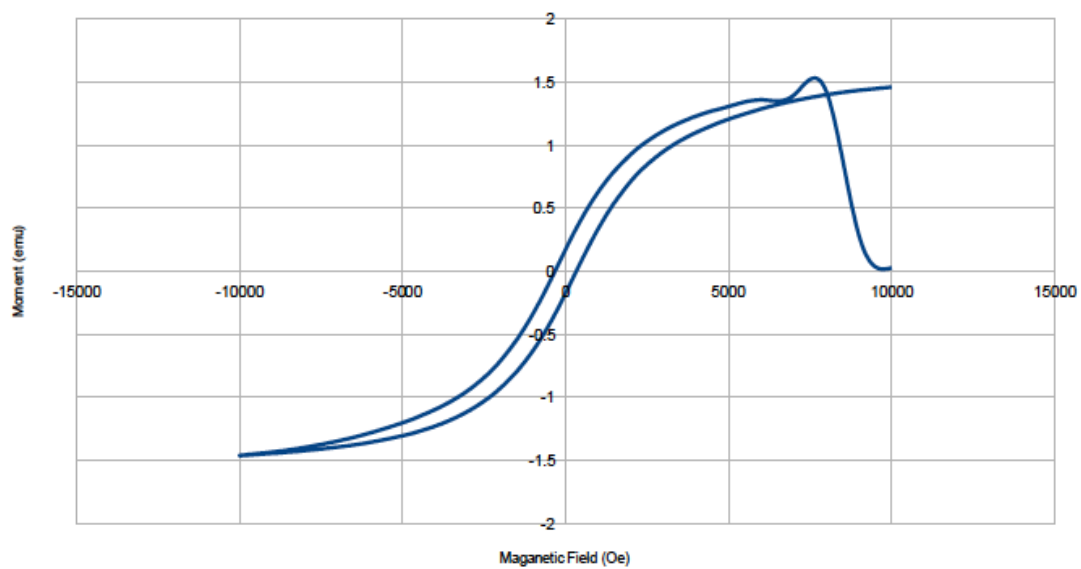


Fig 4-37: Magnetization curve for C6 compact

7. Compact: D5

Material: M2 material milled

Twist: 1:8

Velocity: 262.15 m/s

Rotational Speed: 4603.50 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density: 7.309 g/cc

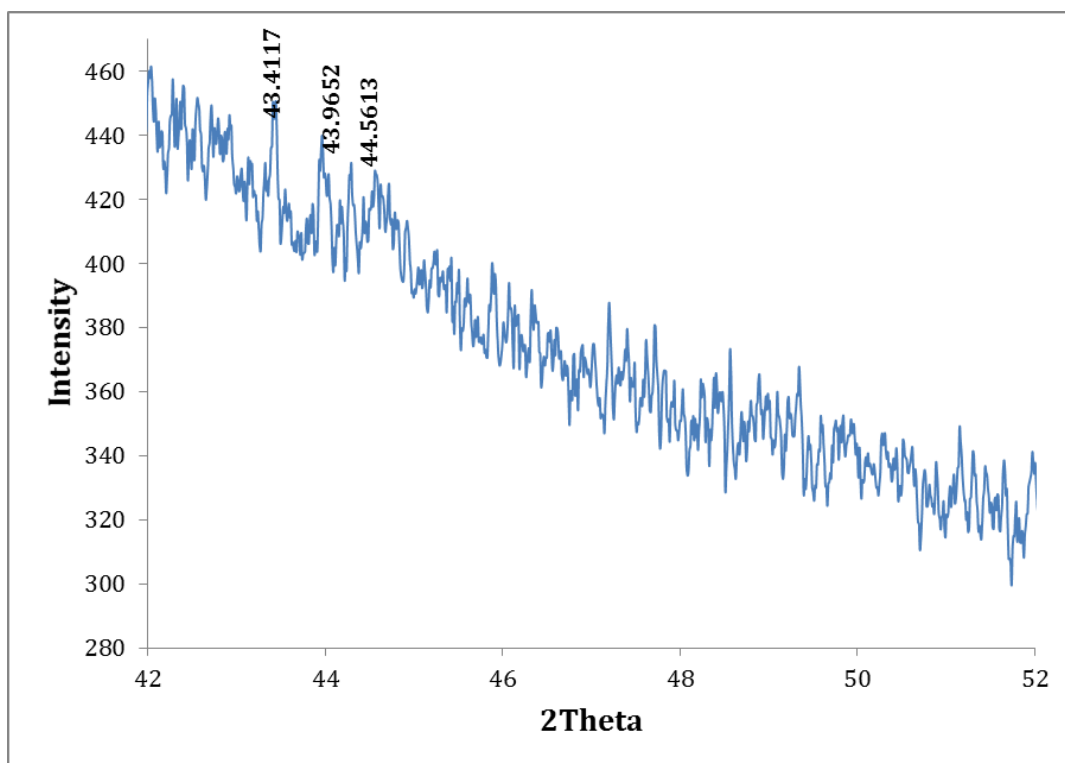


Fig 4-38: XRD pattern for D5 compact

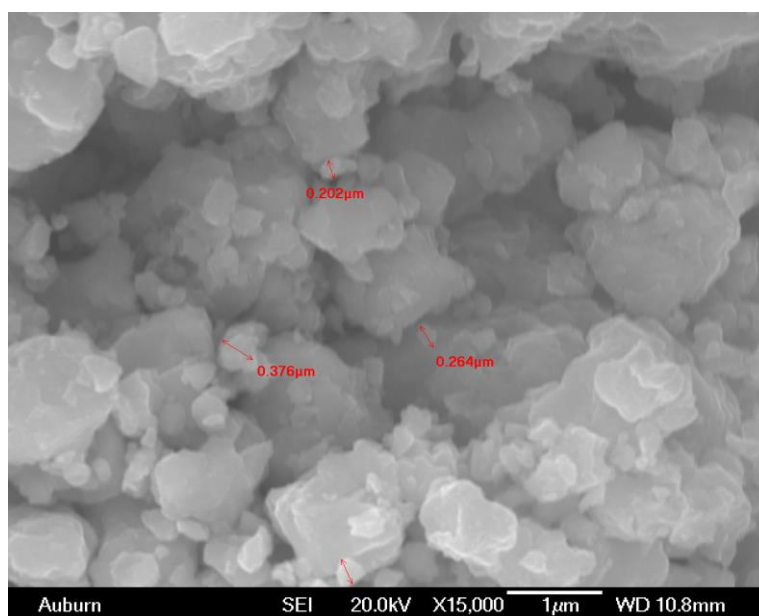


Fig 4-39: SEM image for D5 compact

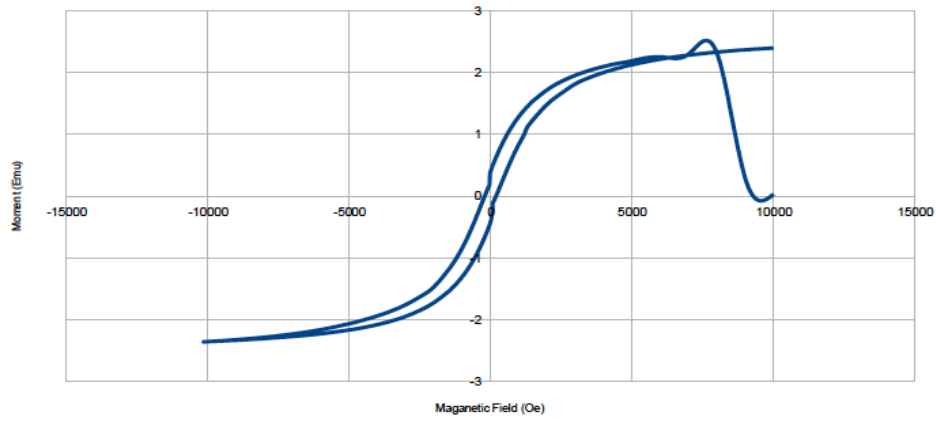


Fig 4-40: Magnetization curve for D5 compact

8. Compact: D8

Material: M2 material milled

Twist: 1:8

Velocity: 358.44 m/s

Rotational Speed: 6294.40 /s

Theoretical Density: 7.60 g/cc

Apparent Density: 2.7 g/cc

Green Density: 4.790 g/cc

Compacted density: - g/cc (Could not be measure due to size and weight)

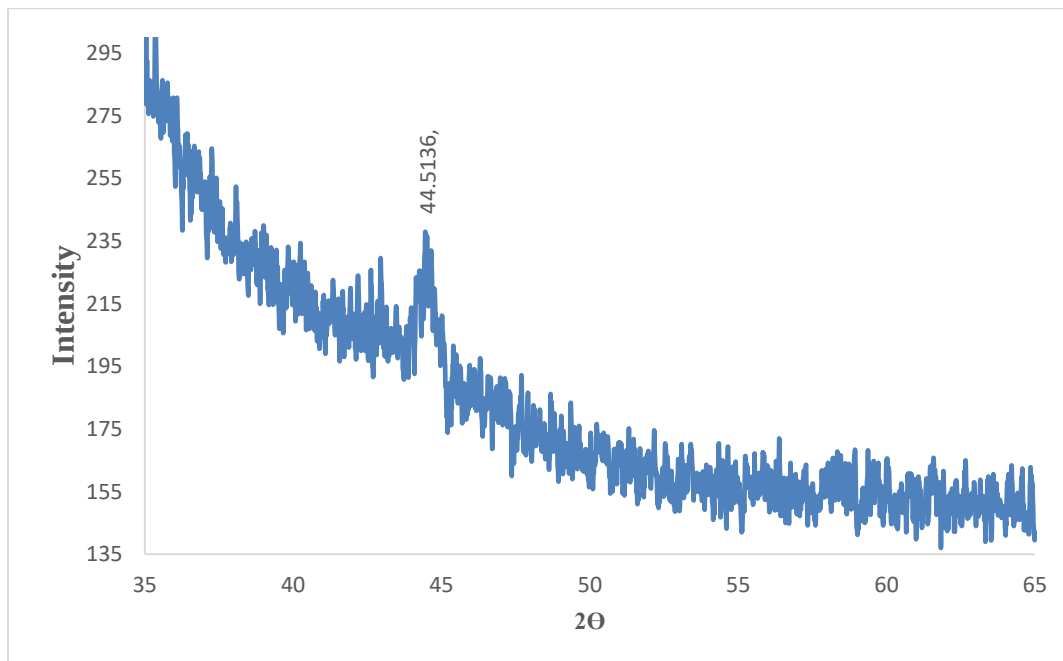


Fig 4-41: XRD pattern for D8 compact

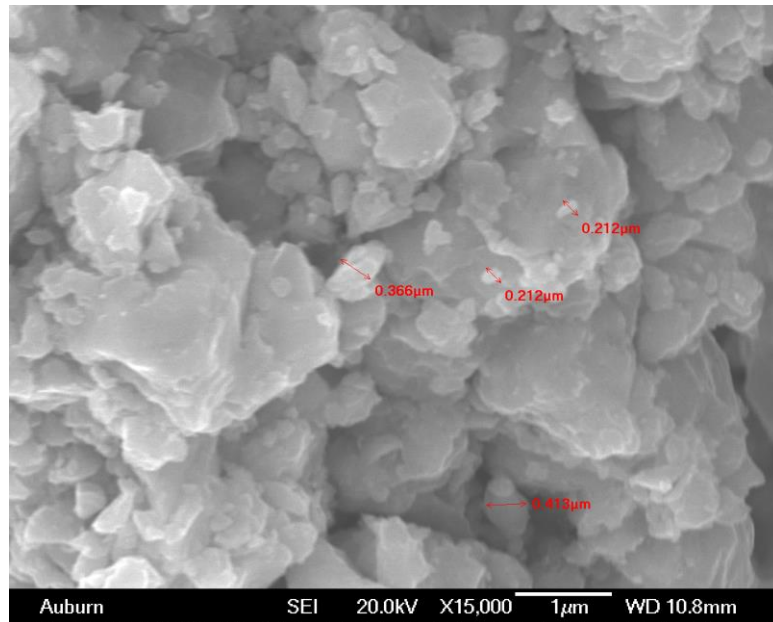


Fig 4-42: SEM image for D8 compact

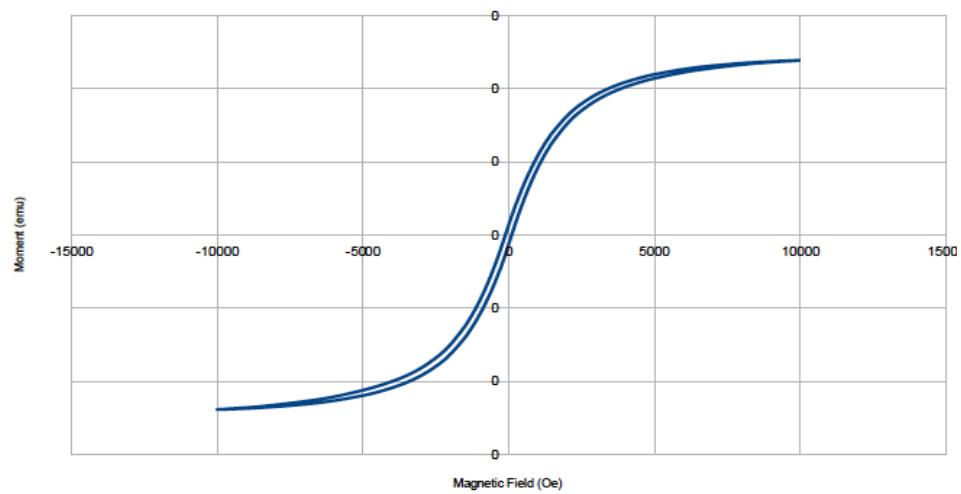


Fig 4-43: Magnetization curve for D8

Summary of M1 and M2 test results:

A. Successful tests

For several tests, the casing didn't hold together, material loaded was lost. This was due to excessive shock waves from compaction process. Better compacts could be obtained by eliminating radial cracks which propagate from centre of the compact outward, i.e. along longitudinal axis of a cylinder. For a few samples, compact may have formed but couldn't be retrieved due to set up. Sometimes capsules held the powder in place but compact was not formed and the loaded powders were lost when retrieved capsules were cut open. Sometimes capsules bulged during the loading process and ended up getting stuck in the barrel during the shooting process.

A successful compact is one that could be retrieved where at least a small amount of material formed a compact that could be used for analysis. It was also seen that, on average more samples were obtained when using lower spin of 1:16 than with 1:8. This was because of lower velocities and lower rotational speed. It was impossible to produce compacts without spin, using a smooth barrel rifle, because the bullets tend to "tumble" and could not withstand impact. Human error when loading capsules and explosive powders also added to inconsistent results.

A1. Summary of M1 and M2 Tests

Material Composition	Sample	Twist (inches)	Tests Done			
			SEM	XRD	Density	BH curve
M1 (Bulk)	A4	1:16	Yes	Yes	Yes	Yes
M2 (Bulk)	A7	1:16	Yes	Yes	Yes	Yes
M2 (Bulk)	A8	1:16	Yes	Yes	Yes	Yes
M1 (Bulk)	B1	1:16	Yes	Yes	No	No
M2 (Bulk)	B5	1:16	Yes	Yes	No	No
M1 (Milled)	C2	1:08	Yes	No	Yes	No
M1(Milled)	C3	1:08	Yes	No	No	No
M1(Milled)	C5	1:16	No	No	No	No
M1 (Milled)	C6	1:08	Yes	Yes	Yes	No
M2 (Milled)	D5	1:08	Yes	Yes	Yes	Yes
M2 (Milled)	D8	1:08	Yes	Yes	No	Yes

Table 4-3: Summary of M1 and M2 Tests

A2. Effect of milling on the properties of the compacts of material M1:

C5 compact could not be retrieved from the capsule as the material did not consolidate and disintegrated when the sample was cut open. Comparing samples A4 and A7 with C3, it was seen from A4 and A7 that the particles sizes on the compact was larger as expected, than the milled material. However, plastic deformation and fracture that occurred on the

flakes of material in the compact was observed in the SEM images. In the C3 compact, a range of particle sizes were seen and they were preserved in the compact. From the XRD pattern it was seen that the compacts formed from the bulk material showed more of a crystalline structure when compared to those of the milled material. The magnetic properties indicate that the compacts of bulk material act as hard magnetic material as opposed to those of the milled materials. For the former ones the material cannot be easily magnetized and the permeability is low. For the latter ones the coercivity is low and permeability is high as it can be easily magnetized.

A significant decrease in coercivity was seen in compacts prepared from milled magnetic powders. This may be due to amorphous structure of nano particles and some possible contaminations during the preparation process. It was seen when compared to the literature [13] that in traditional explosive compaction there was an increase in coercivity for the compacts made with bulk magnetic powders and decrease with the compacts made from milled powders in the case of spin explosive compaction. This shows that the high densities obtained using explosive spin compaction produced different microstructures that in turn produce slightly different magnetic properties from traditional compaction techniques that can be used for appropriate applications as required.

A3. Effect of milling on the properties of the compacts of material M2:

M1 and M2 material are very similar in composition as follows.

M1 - Magnequench MQP-A-10179-070 (Nd-Fe-B)

M2 - Magnequench MQP-13-14-20203-070 (Nd-Pr-Nb-Fe-B)

So, the bulk and milled M2 materials behave similar to bulk and milled M1 material when it comes to the structural and magnetic properties as it can be seen.

A4. Effect of spin velocity on compacts retrieved:

The only difference seen when comparing the samples of two different spins was in the density. It was seen that higher rotational speed (higher spin) produced slightly denser samples when compared to lower rotational speed (lower spin). However, when compared with traditional explosive compaction, the compacts produced with a spin component had slightly different microstructures and much higher densities. The spin compaction technique can this be applied to other materials where density would play a major role, for example, applications where materials of high strength is needed. It was impossible to obtain compacts with the smooth barrel gun available to compare as part of this study because of the type of rifle and inconsistency of the results. The issues faced will be discussed in Chapter 6.

4.2.2 Characterisation of recovered compacts (Continued.)

9. Compact: A11

Material: B1 material bulk

Twist: 1:16

Velocity: 439.82 m/s

Rotational Speed: 7723.48 /s

Theoretical Density: 6.08 g/cc

Green Density: 4.4 g/cc

Compacted density: 7.39 g/cc

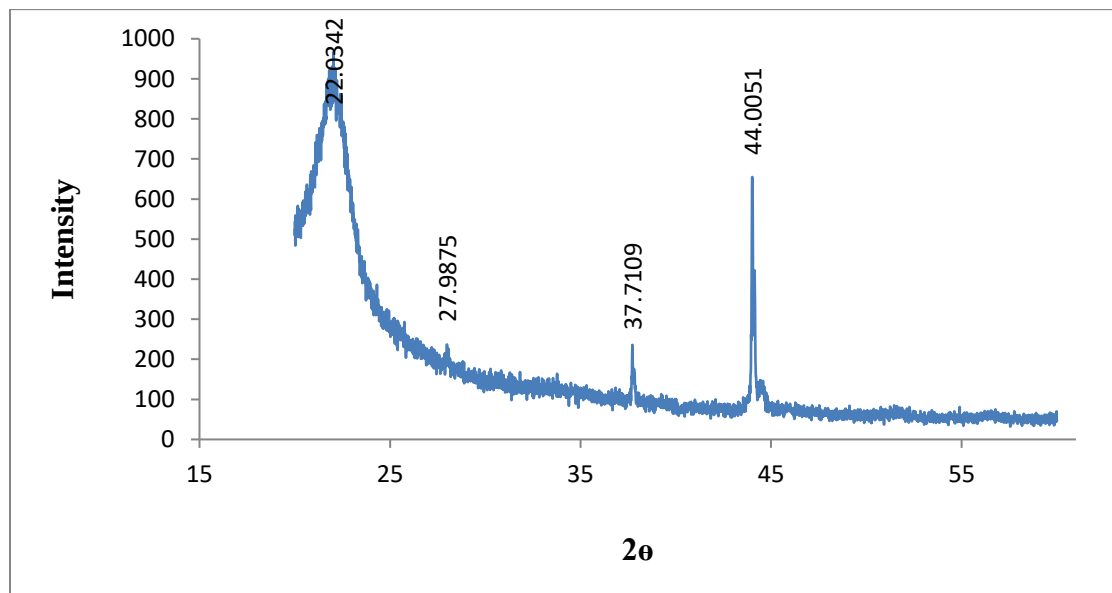


Fig 4-44: XRD pattern for A11 compact

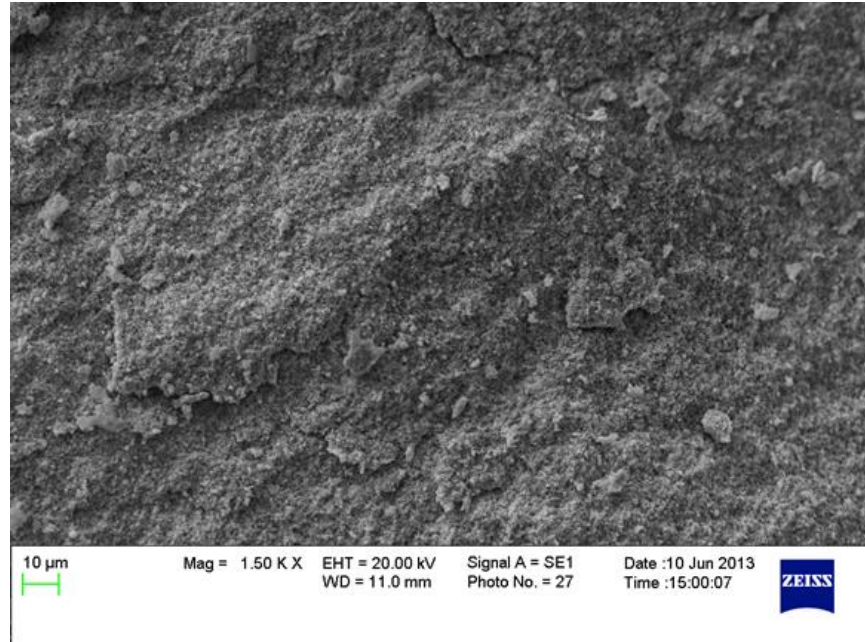


Fig 4-45: SEM image for A11 compact

10. Compact: D11

Material: B1 material milled

Twist: 1:16

Velocity: 261 m/s

Rotational Speed: 4583.3 /s

Theoretical Density: 6.08 g/cc

Green Density: 4.4 g/cc

Compacted density: 5.32 g/cc

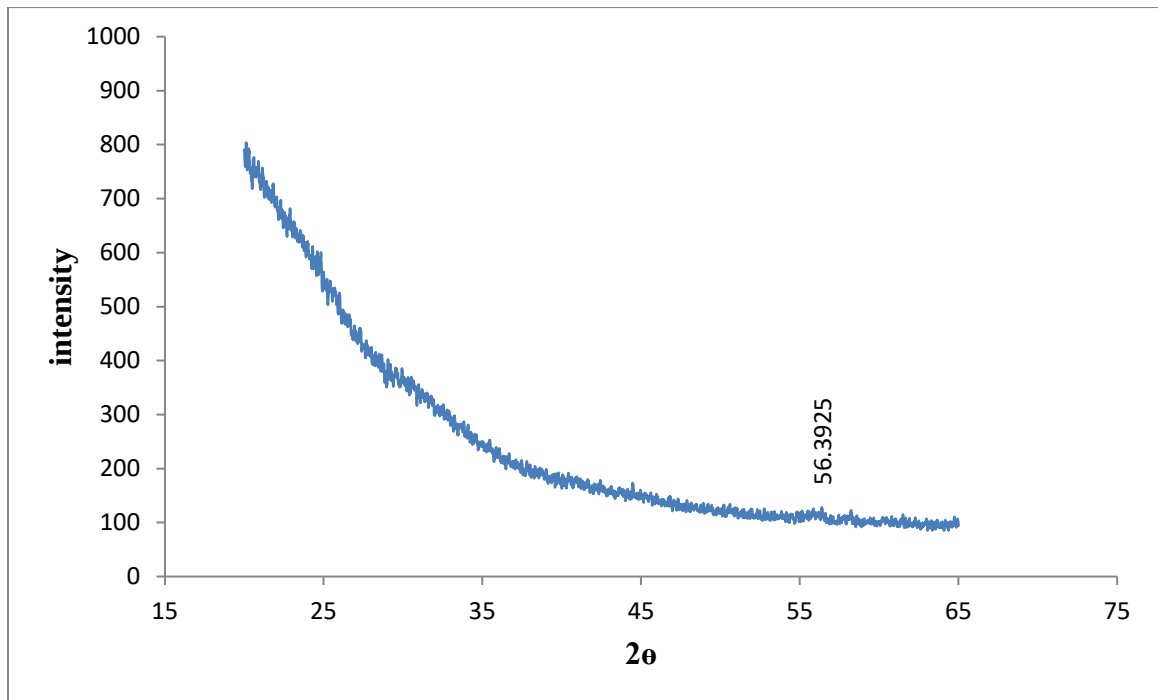


Fig 4-46: XRD pattern for D11 compact

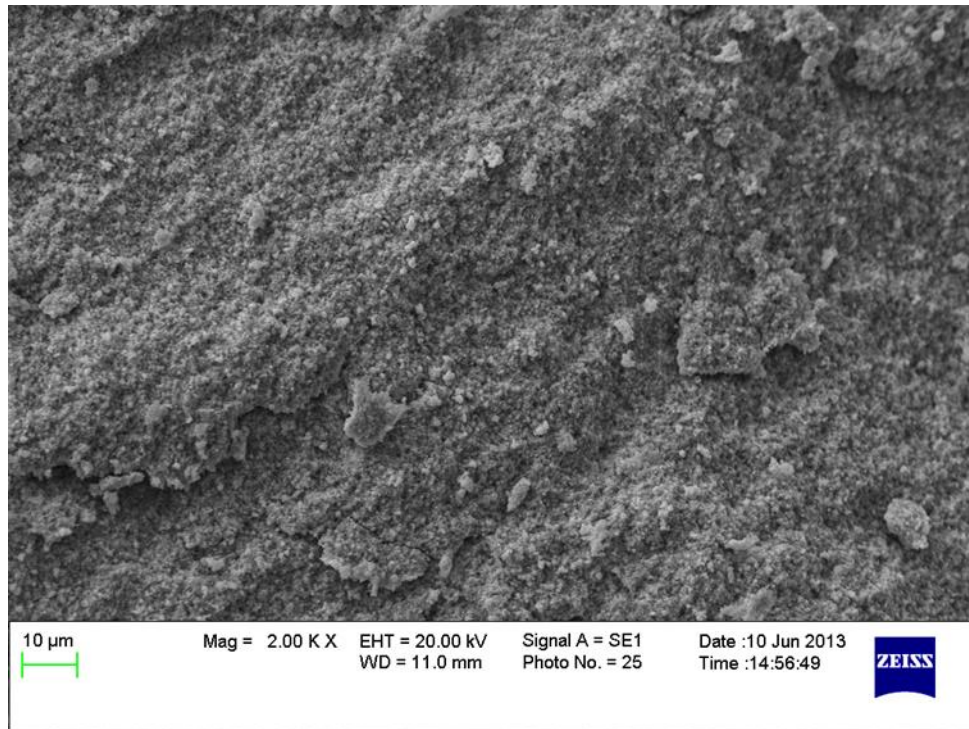


Fig 4-47: SEM image for D11 compact

11. Compact: D12

Material: B1 material milled

Twist: 1:16

Velocity: 289.7 m/s

Rotational Speed: 5087.3 /s

Theoretical Density: 6.08 g/cc

Green Density: 4.4 g/cc

Compacted density: 5.39 g/cc

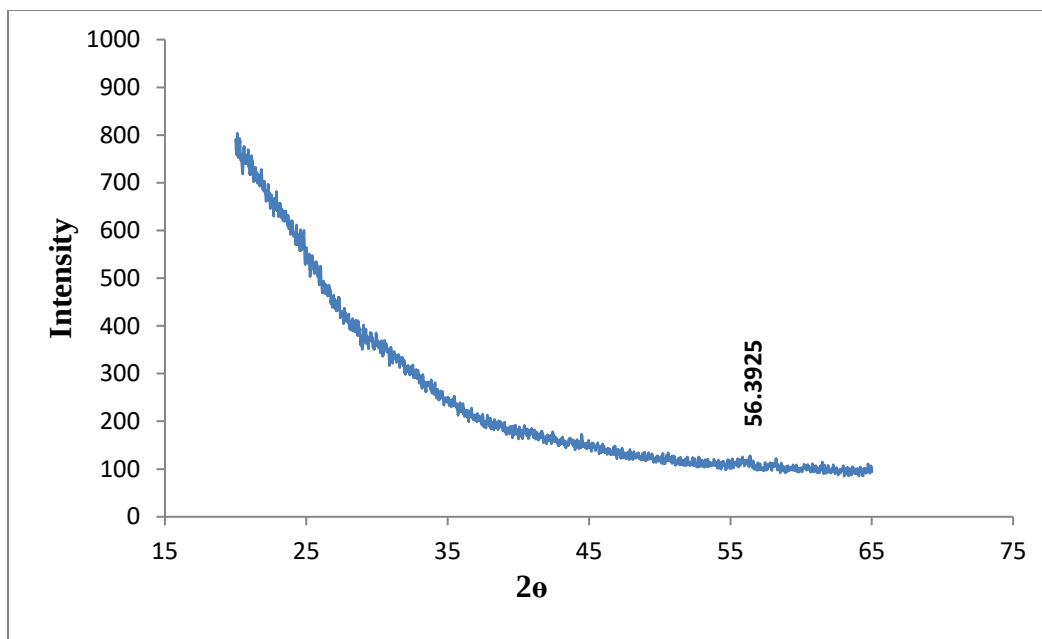


Fig 4-48: XRD pattern for D12 compact

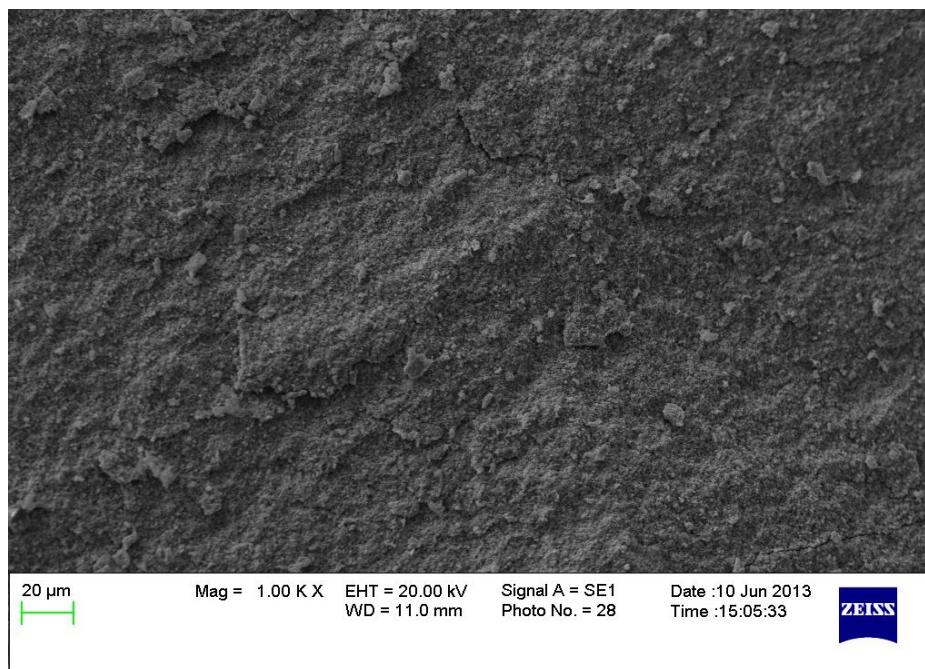


Fig 4-49: SEM image for D12 compact

Capacitance measurement

The capacitance was measured for barium titanate pellets are in the range of 1.11nF. Since the compacts obtained were not uniform in size there were changes in values. The area of these samples could not be measured due to irregular size of the compacts.

Summary:

Material Composition	Sample	Twist	Tests Done			
			SEM	XRD	Density	Capacitance
B1 (Bulk)	A11	1:16	Yes	Yes	Yes	Yes
B1 (Milled)	D11	1:16	Yes	Yes	Yes	No
B1 (Milled)	D12	1:16	Yes	Yes	Yes	Yes

Table 4-4: Summary of B1 Tests

Effect of milling:

Comparing samples A11 to D11 and D12, it was seen that the bulk material was crystalline and as the particle sizes reduced the material was more amorphous and only the major peak at 56.2° was seen. Since the compacts obtained were irregularly shaped, it was not possible to measure the surface area. The capacitance value obtained was very close for all three compacts obtained.

5 Conclusions

The different materials were purchased and compacted by spin compaction method. Two different rifles containing twist 1:8 and 1:16 were used and compacts were retrieved and studied using XRD, SEM, EDS and Vibrating Sample Magnetometer. The bulk materials of both barium titanate and neodymium iron boron were loaded into capsules and spin compacted. The compacts were then analysed using the above-mentioned techniques. The powders were then milled to particle sizes in nano-scale and compacted by spin compaction. Different velocities were used for compaction process and the compacts were analysed. The densities of compacts obtained were measured and were seen to be close to theoretical density.

It was also seen that higher rotational velocities produced compacts of higher densities and their microstructures were studied. The NeFeB compacts were then studied using vibrating sample magnetometer and the magnetization curves were obtained.

The capacitances of BaTiO₃ compacts were measured. It was seen from the results that the spin component does not have any effect on the properties of the material. Several challenges were faced during this study that led to inconsistent results. They are discussed in Chapter 6. Future work is discussed in Chapter 7.

Effect of milling on properties of M1 and M2 compacts:

It was seen that the flakes in compacts of bulk materials showed more plastic deformation and failure. It was also seen that the reduction in particle sizes broadened the peaks in the XRD pattern that proved that the material was more amorphous than crystalline. It was also seen that magnetic

curves obtained showed that the bulk material compacts acted as hard magnets and milled material compacts acted as soft magnets. Hard magnetic materials have higher coercivity than soft magnetic materials. The particle size thus has a drastic effect of the magnetic properties.

Effect of Spin on the Properties of M1 and M2 Compacts:

The effect of spin was studied on both the magnetic materials. No changes were seen in the magnetic properties; however the densities were impacted by the rotational velocities. At higher rotational speeds, denser samples were obtained than those produced by traditional explosive compaction discussed in the literature. This shows that the spin technique is successful in producing compacts of near theoretical densities and can be used to compact materials for applications where high strength is a desirable property.

Effect of Milling on Properties of Barium Titanate Compacts:

It was seen that the milling process changed the crystalline nature of the bulk material and made it more amorphous. It was proved by the broadening of peaks in the XRD. It was also seen that only one major peak was seen when the compacts of the milled material was analysed which confirms the phase change occurring in it due to the milling process. Milling did not have an effect on the dielectric properties.

Effect of Spin on Properties of Barium Titanate Compacts:

It was seen that the rotational velocity did not play a role in altering any properties other than the density of the compacts. The density produced was close to theoretical density.

6 Challenges Faced

Some of the challenges faced in these experiments are:

- Oxidation of magnetic powders during milling due to overheating and burning



Fig 6-1: Powder burnt during high energy milling process

- Several the samples were lost during shooting
- Many the projectiles did not hold together during compaction and so the compacts could not be recovered
- Some of the projectiles bulged while the powders were loaded causing it to get stuck in the barrel

7 Future Work

The major problem encountered was oxidation of particles during preparation of finer particles. If the milling of magnetic particles was done in an inert atmosphere this can be prevented and the magnetic properties can be significantly improved. This can be achieved using a high-energy planetary ball mill where the milling process can be accomplished at a steady rate in an inert atmosphere. This can also ensure that the particles do not overheat during the milling process. Another problem that was encountered was that the capsules did not hold together during shooting. This can be improved by machining the capsules from a different steel of higher ultimate tensile stress.

The inconsistent results during shooting could also be due to human error when loading the explosive powders in the bullet or while loading the actual powders in the capsules that caused the capsules to bulge and get jammed in the barrel of the rifles. A better system to capture the capsules after shooting and prevent fly-aways will help one recover more samples to characterize in order to obtain more accurate information about the process.

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