MAGNETIC PROPERTIES EVALUATION OF POLYAMIDE 4.6 BONDED MAGNETIC COMPOSITE

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ABSTRACT

Bonded magnetic composites combine the cost-effectiveness, low density, and manufacturing flexibility of conventional polymer binders with the unique magnetic characteristics of magnetic powders/fillers to form multifunctional magneto polymeric composites that offer superior properties to conventional sintered magnets. In this study, a co-rotating twin screw extruder was used to fabricate 20 and 40 wt.% strontium ferrite/polyamide 4.6 bonded magnetic composites viable for fused filament fabrication 3D printing. The characterization conducted on the bonded magnetic composites was scanning electron microscopy, simultaneous differential thermogravimetry, and vibrating sample magnetometry. The microstructure of the bonded composite exhibited a uniform platelet morphology of the strontium ferrite magnetic particles. There was no observable depreciation in the melting transitions, which suggests a thermally resistant magnetic composite. An appreciable increment in % crystallinity of 13 and 20% for 20wt.% and 40wt.% strontium ferrites bonded magnets were observed. This is attributable to the heterogeneous nucleation phenomenon, where the metal powders act as nucleation sites for increased crystalline domains. The bonded composite exhibited significant magnetic anisotropy, with the remanence (Mr), which is the most important property for magnetic application significantly increasing to 49.8% along the easy direction in comparison to the hard axis. This suggests the viability of the fabricated bonded composites in viable in producing anisotropic bonded magnetic devices, which are considered to exhibit stronger magnetic properties.

Keywords: *Strontium ferrites, Polyamide 4.6, Bonded Magnet, Twin screw technology, Fused filament fabrication, Materials characterization.*

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1. INTRODUCTION

Bonded magnets are a technologically advanced class of composite materials capable of achieving a good combination of material properties. By exploiting the benefits of polymeric materials such as affordability, manufacturability, and lightweight advantages, suitable engineering thermoplastics or thermosets can serve as a matrix or binder, for magnetic particles to fabricate a bonded magnetic composite. Polyamides, also known as nylons, have been widely considered suitable engineering thermoplastics for developing bonded magnetic composites. Based on the number of carbon atoms in their repeating monomer chains, polyamides can be categorized into polyamide 12, polyamide 11, polyamide 6, polyamide 6,6, and polyamide 4.6 [1]. Generally, they are semi-crystalline in nature and possess a good balance of material properties and as a result, they have been widely utilized in electronics, automotive, and sports-related applications [2]. The presence of the amide bonds present in their polymer chain makes them susceptible to moisture. This absorbed moisture could act as a plasticizer and consequently improve their flexibility, impact, and fatigue resistance, thereby improving mechanical properties when used as a binder in bonded magnetic composites.

Magnetic particles such as rare earth and ferrite powders can be used as fillers to develop permanent bonded magnetic composites. Despite the superior magnetic properties exhibited by rare earth magnetic materials such as samarium and neodymium-based powders, they are classified as critical materials due to a shortage in supply and challenges in mining and refining them [3]. This makes ferrite powders cost-effective alternatives in developing permanent magnetic composite materials. Strontium ferrites (SFO), a ceramic hard ferrite powder, have attracted global attention due to their moderate magnetic strength, cost-performance ratio, and chemical and oxidation resistance. As illustrated in **Figure 1a**, SFO exhibits a hexagonal crystal structure defined by two lattice constants, where a is the width of the unit cell in the hexagonal plane, and c, is the height of the unit cell. For the hard ferrites, the preferred orientation of the magnetic moment, also known as the easy axis, goes parallel to the c-axis. This makes the basal plane a hard direction for the magnetic dipole moment. Such anisotropy is known as magnetic crystal anisotropy which enables strontium ferrite magnetic powders to be viable for fabricating anisotropic bonded magnets. Unlike isotropic bonded magnetic composite, it is challenging to demagnetize anisotropic bonded magnets, thereby maximizing their intrinsic magnetic in certain directions and creating a stronger magnetic composite. The intrinsic magnetic properties of permanent bonded magnetic composite can be summarized on a typical magnetic hysteresis curve presented in **Figure 1b**. As illustrated in this figure, the maximum induced magnetization (symbolized as A) by an applied magnetic field is known as saturation magnetization *(M_s)*. This induced magnetization remaining (symbolized as B on the loop) after the removal of the applied field is referred to as remanence (M_r). The magnetic field required to revert magnetization to zero (symbolized as C and F on the loop) is known as coercivity or coercive field *(H_c)* [4]. In order to optimize and enhance the above intrinsic magnetic properties in bonded magnetic composites, researchers are employing different innovative polymer manufacturing processes.

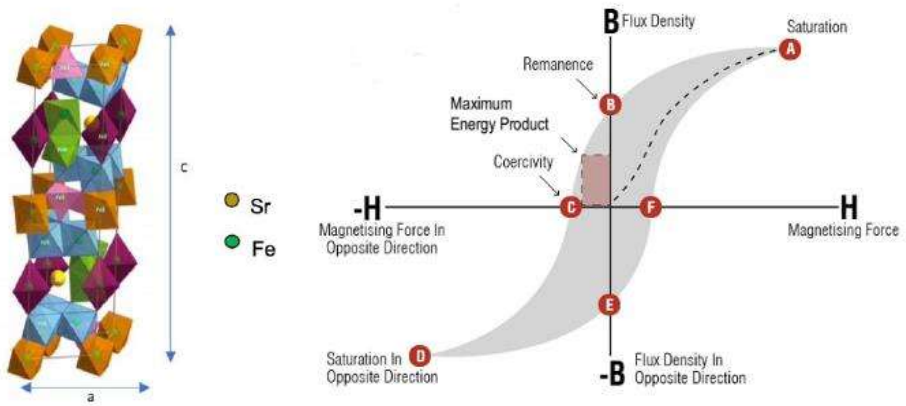


Figure 1. (a) Hexagonal crystal structure of strontium ferrites [5] (b) magnetic hysteresis loop [6].

Garrell et al. [7] utilized a high-performance thermoplastic polyphenylene-sulfide (PPS) to develop 60vol.% NdFeB bonded magnetic composite via the injection molding process. The injection molded polyphenylene-sulfide (PPS) bonded magnets exhibit low moisture absorption characteristics, good corrosion resistance, high dimensional stability, lightweight for metal replacement viable for many automotive and high-temperature applications. The polyphenylene-sulfide (PPS) bonded magnets exhibited an overall higher strength than nylon bonded magnets, particularly at elevated temperatures. In the work of S.H. Lee and W.Y. Jeung [8], powder injection molding (PIM) processing, which combines both sintering and injection molding processes was used to fabricate magnetic bonded magnets using a feedstock of 55vol% strontium ferrite magnetic powders and a binder composition system of 30vol% PP, 60vol% PEG-20000, and 10vol% PEG-4000. They utilized a solvent extraction debonding technique (solvent extraction), where the major binder components PEGs were removed and the minor binder component, PP, was subsequently burned out in the air. The obtained bonded magnetic composite was relatively dense due to the sintering debinding action and near-shape complex parts due to the injection molding technique. Despite the injection molding process being reported as a competitive process for the fabrication of ferrite magnetic products, limitations such as expensive custom tools that will increase the bonded magnets' fabrication cost have deemed them, not an economic option. This has called for a more flexible and sustainable manufacturing approach such as additive manufacturing, also known as 3D printing [9]. Huber et. al [10] was one of the pioneers in examining the feasibility and benefits of utilizing 3D printing technology to create polymer-bonded magnets. Pigliaru et.al [11] fabricated bonded 15, 34, and 41vol% NdFeB permanent bonded magnets via fused filament fabrication (FFF) 3D printing technology by using a polyether ether ketone (PEEK) binder. They were able to achieve promising magnetic properties with coercivity values between 672 and 684 kA/m and magnetic remanence values between 218 and 226 mT. Additionally, the fabricated bonded magnets were chemically resistant, thereby resolving the corrosion susceptibility of the NdFeB magnet. The high thermal resistance reported for the developed PEEK/NdFeB bonded magnetic composite makes it a valid choice for elevated operating conditions such as space applications [11].

Fabricating bonded magnets is driven by the need to replace or serve as an alternative option to the costly and commercially available sintered magnets. Unlike sintered magnets, several advantages offered by bonded magnets are competitive magnetic properties, exceptional mechanical properties and structural integrity, intricate shapes formability, light weight advantages, chemically resistance to harsh operating conditions and cost-effectiveness. Additionally, when a high thermal resistance binder is utilized such as high temperature thermoplastics (PEEK-polyether ether ketone, PPS-polyphenylene sulfide and HPPA-high performance polyamide), the fabricated bonded magnetic composites can be used in manufacturing high performance magnetic devices such as small generators, electric motors used in electric vehicles and 3D printed Halbach cylinder used in magnetic resonance imaging (MRI) scanners. In this study, we have reported the fabrication and magnetic performance characterization of strontium ferrite bonded magnetic composite, which would be viable for the fused filament fabrication (FFF) additive manufacturing process.

2. EXPERIMENTATION

2.1 Material System

(a) Strontium ferrite powders

The filler or reinforcement used in developing the bonded magnets is a strontium ferrite magnetic powder obtained from DOWA electronics. This magnetic powder is a ceramic hexaferrite magnetic powder developed suitably for magnetic orientation and it is branded as "OP 56". Strontium ferrite OP56 is an anisotropic magnetic powder viable for developing anisotropic bonded magnetic composites with a good combination of permanent magnetic properties such as coercivity, remanence, and magnetic saturation. As presented in the SEM image in **Figure 2**, strontium ferrite exhibits a platelet morphology with an average particle size of 0.96 nm.

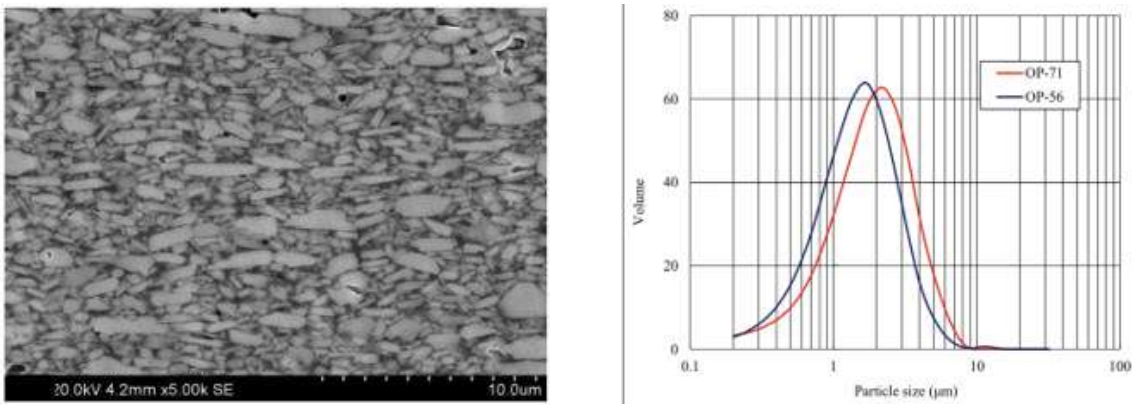


Figure 2. (a) Platelet morphology of the as-received strontium ferrite (left) (b) particle size Distribution (right) [12]

(b) Polyamide 4.6 binder

The binder chosen in developing bonded magnetic composite in this research is a polyamide 4.6 thermoplastic supplied in pellet form by PREMIER PLASTICS. Polyamide 4.6 (PA 4.6), also known as nylon 4.6 was chosen for its exceptional thermal properties, including resistance to thermal deformation and thermostability, alongside robust mechanical strengths such as high mechanical strength and stiffness. PA 4.6 is one of the most hygroscopic polyamides, and its water absorption at 23 °C is 9.5 Due to its water absorption properties, the PA 4.6. pellets were dried using an air dryer (as shown in **Figure 3**) for 24 hrs. at 180F before the twin screw extrusion processing.

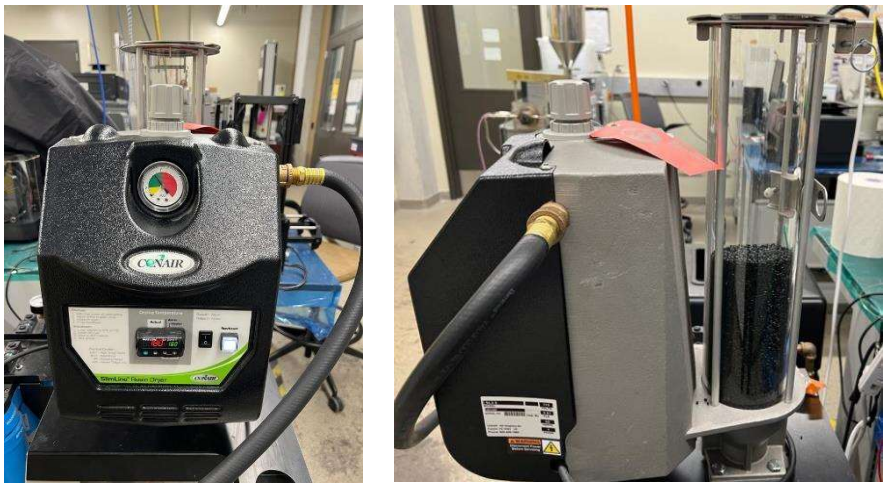


Figure 3. CONAIR resin air dryer

2.2 Twin Screw Extrusion

The bonded magnetic composite was fabricated via an extrusion process known as twin screw compounding. 20 and 40 wt.% packing fraction of strontium ferrite magnetic powders was compounded with polyamide 4.6 binders using a Thermo Scientific twin screw extruder (Process 11) as shown in **Figure 4** to fabricate the bonded magnetic composites into monofilaments with an average diameter of 1.75 ± 0.05 mm viable for FFF 3D printing. The process 11 twin screw extruder is equipped with 3 multifunctional barrel ports used for feeding and venting purposes, and 8 heating zone to control the extrusion temperature process. The extrusion process parameters optimized for fabricating the bonded magnetic monofilament composite were the screw speed, feed rate, torque percentage, and extrusion temperature. The extrusion temperature along the extruder barrel used in the twin screw compounding of the bonded magnetic composite is in the range of 290 – 330 ^{o} C.

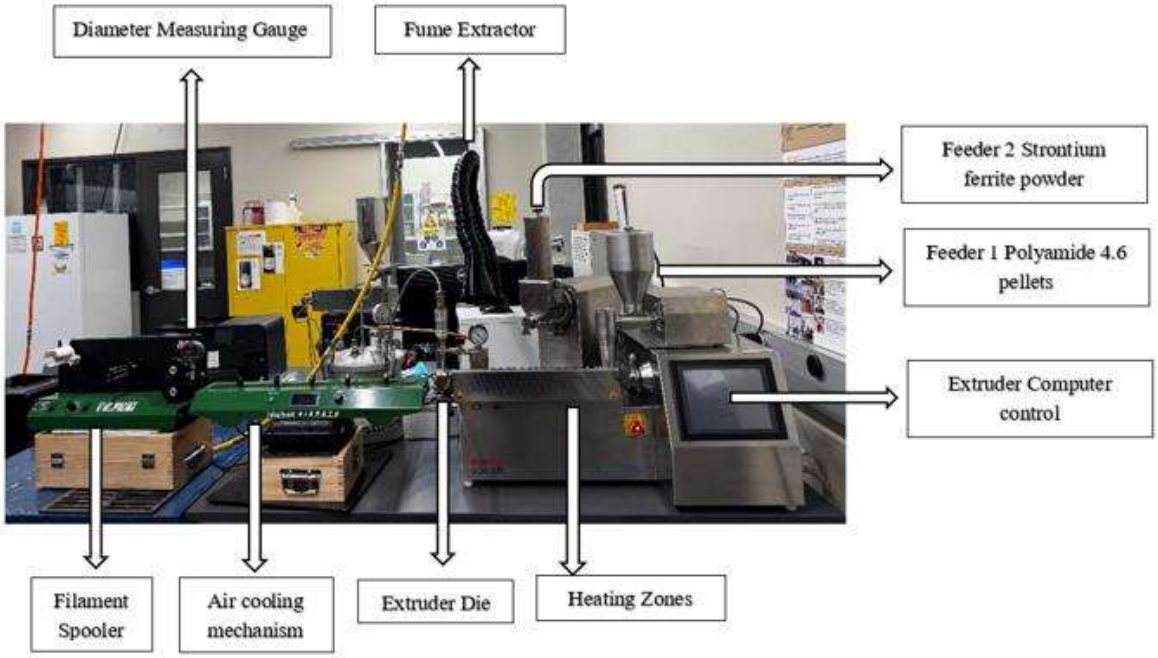


Figure 4. Twin screw extrusion technology using Thermoscientific twin-screw extruder (Process 11).

2.3 Materials Characterization

The fabricated bonded magnetic composite was subjected to materials characterization techniques such as scanning electron microscopy to evaluate the microstructures, thermal analysis – thermogravimetry and differential scanning calorimetry to evaluate thermal properties, and vibrating sample magnetometry to evaluate the magnetic properties.

The microstructure of the bonded magnetic composite was investigated using a JEOL scanning electron microscope using a high vacuum secondary electrons signals detection mode. The thermal properties of the bonded magnetic composite were evaluated using a TA instrument Simultaneous Differential and Thermogravimetric Analyzer (SDT 650). This equipment can conduct both TGA and DSC in a single experimental run. The magnetic performance of the fabricated bonded magnetic composite was investigated via a Microsense Biaxial EZ-9 vibrating sample magnetometer (VSM) as presented in **Figure 5**. This was achieved by applying a magnetic field from -2.2kOe to +2.2kOe using a sweep rate of 400Oe/s on a vibrating composite sample positioned between two large electromagnets. The primary magnetic properties evaluated from the magnetic hysteresis loop are magnetic saturation (Ms), remanence (Mr), and coercive field (Hc).

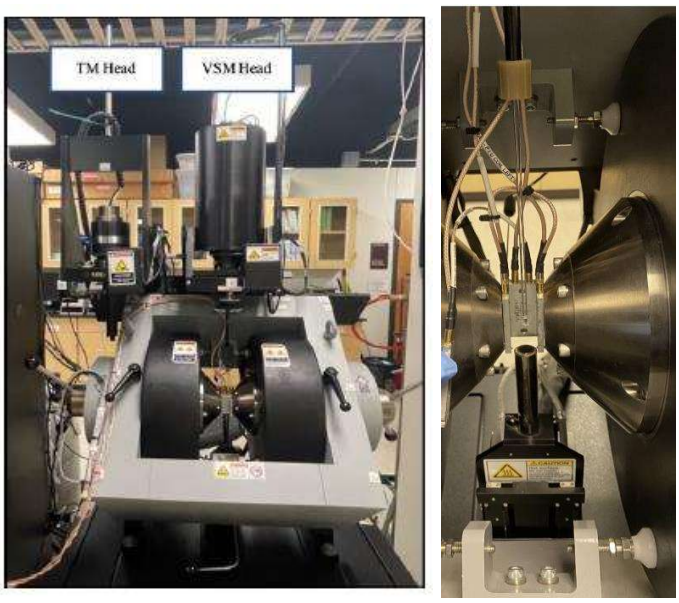
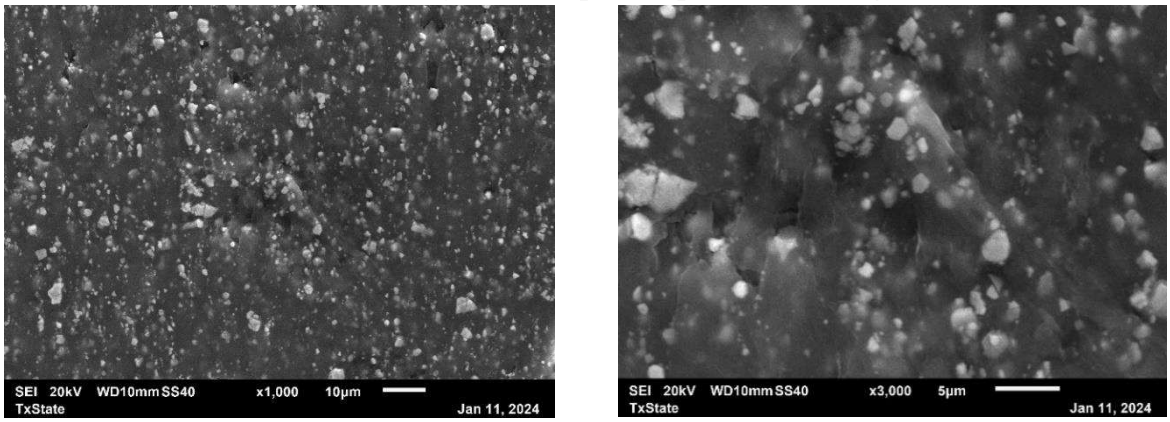


Figure 5. Vibrating Sample Magnetometer (left) VSM perpendicular rod loaded in between two large electromagnets (right).

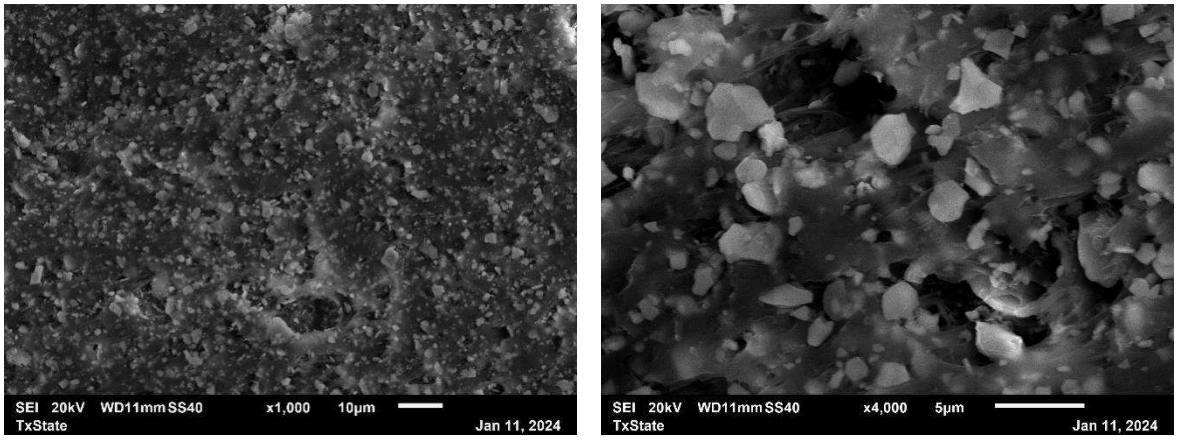
3. RESULTS AND DISCUSSION

3.1 Morphological Characteristics

The dispersion quality and morphology of the 20 and 40 wt.% strontium ferrite bonded magnets composite are presented in the SEM images in **Figure 6**. The strontium ferrite magnetic powders were observed to retain their platelet morphology evenly dispersed in the polyamide 4.6 binders. Agglomeration of the strontium magnetic fillers can promote the formation of complex magnetic domains and prevent consistent magnetization, thereby reducing the ferrimagnetism and permanent magnetic properties of the bonded magnetic composites. In addition, uniform dispersion of the magnetic fillers in the matrix of the composite prevents stress site formation and facilitates durable interfacial bonding that can promote the structural integrity of the bonded magnetic composites. There was no appreciable agglomeration of the strontium ferrite platelets in the microstructures of the bonded magnetic composites presented in **Figure 6.**



(a)



(b)

Figure 6. SEM images of (a) 20 wt.% strontium ferrites (b) 40 wt.% strontium ferrites bonded magnetic composites.

3.2 Thermal Performance

The thermal performance of the bonded magnetic composites was evaluated using the data obtained from the thermogravimetry (TGA) and differential scanning calorimetry measurements (DSC). **Figure 7** shows the thermogravimetry plots of the magnetic composites were obtained in accordance with the ASTM E2550 – standard test method for thermal stability [13].

The thermal stability of a composite is evaluated based on its decomposition temperature. As shown in **Figure 7**, there was an early onset of decomposition of the magnetic bonded composites when compared with the neat polyamide 4.6 binder. The observable reduction in the onset of decomposition temperature of the bonded magnetic composite could have stemmed from the appreciable packing fraction of 20 and 40 wt.% of strontium ferrite in the nylon matrix. The magnetic domain sites distributed within the polyamide 4.6 matrix could be a source of catalytic degradation of the aliphatic polyamide 4.6 considering that strontium ferrites metal powders only exhibit a moderate thermal conductivity which could promote heat accumulations, thereby facilitating an early decomposition of the polyamide 4.6 binder in the magnetic composites. This is similar to the observation reported by Turski Silva et al. [14] on the early onset of decomposition of polyamide 4.6 composites compared to the neat one. According to the mass loss evaluation as shown on the TGA plots, the 20wt.% magnetic composite was 21.92% of strontium ferrites and the 40 wt.% magnetic composite was 40.03% of strontium ferrite, which indicates a very little margin of error of $< \pm 3\%$ in the packing fraction of strontium ferrite in the magnetic composite. This consistency between the target and actual packing fraction suggests that the fabrication of the bonded magnetic composite via the twin screw compounding process was adequately optimized.

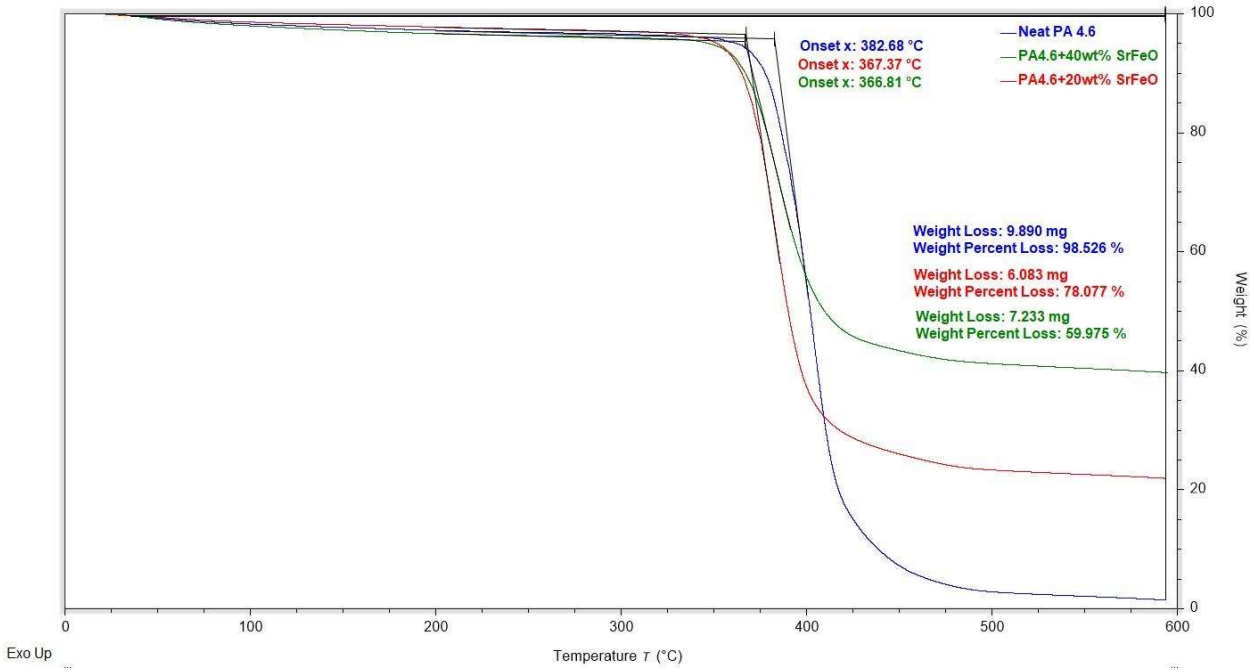


Figure 7. Thermogravimetry plot of strontium ferrite bonded magnetic composites.

The melting and crystallization transition properties were evaluated on the DSC scans as observed in Figure 10. These thermal transitions were obtained in accordance with **ASTM D3418-15-** standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry (DSC) [15].

According to the DSC scans in **Figure 8**, it could be observed that the neat polyamide 4.6 exhibits an onset of melting at 290.73 ^{o}C which was not appreciably altered by the inclusion of the strontium ferrite magnetic powders in the bonded composite. This is imperative for high performance bonded magnetic application that the fabricated bonded strontium magnetic composite is thermally resistant such that the melting point of the polyamide 4.6 binder of the magnetic composite would not depreciate but maintained or rather enhanced. Additionally, the melting transitions evaluation of the neat polyamide 4.6 binder via the DSC was helpful in determining the extrusion temperature profile baseline for efficient fabrication of the strontium ferrite bonded magnetic composites. The crystallization behavior of the magnetic composites was monitored by evaluating the percent crystallinity according to the mathematical relationship presented in the equation below. According to equation 1, ΔH_m represents the heat (enthalpy) of crystallization, φ is the packing fraction of strontium ferrite magnetic powders and ΔH_o is the heat of fusion of 100% crystalline polyamide 4.6, with a unanimous value reported in the literature as 210J/g and X_c (%) is the estimated percentage crystallization.

$$X_c\,(\%) = (\Delta Hm/(1-\varphi)\,\Delta H_o)\,X\,100 \qquad (1)$$

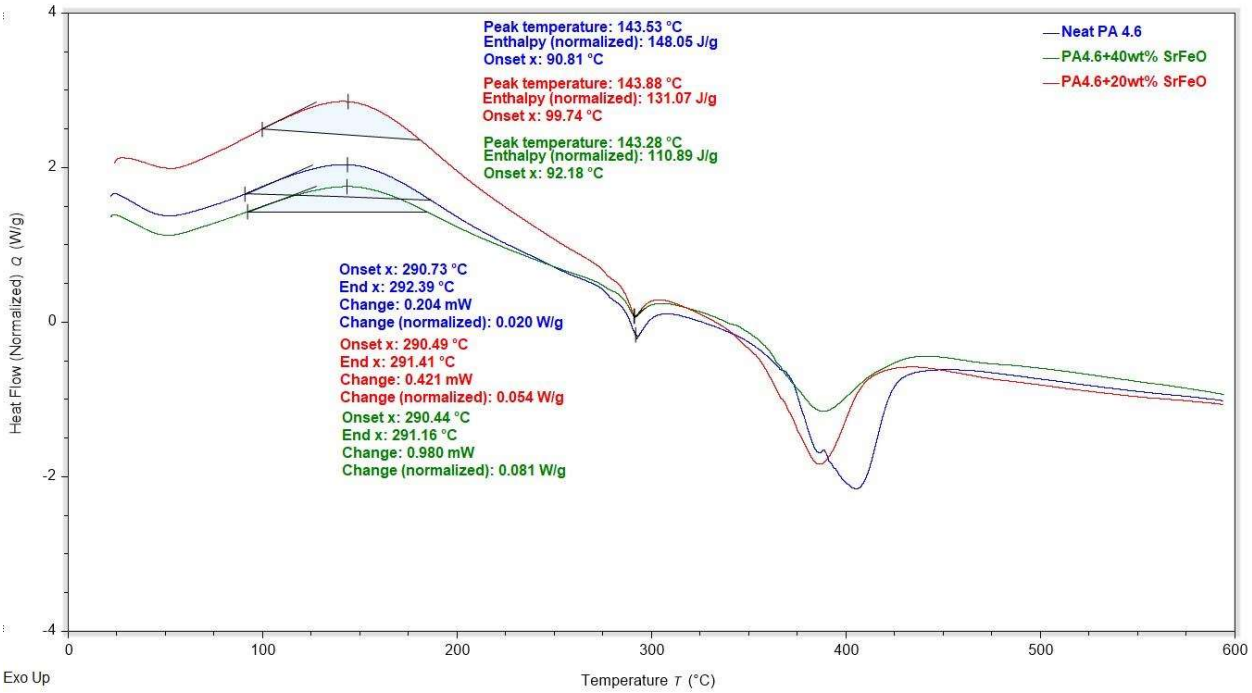


Figure 8. Differential scanning calorimetry plot of strontium ferrite bonded magnetic composites.

Using equation 1, the percentage crystallinity was evaluated as 70.5, 79.9 and 88% for neat, 20wt%, and 40wt% strontium ferrite bonded magnets respectively. The crystallinity of the bonded magnets was observed to increase by 13% and 25% for 20 and 40wt% strontium ferrite loading fraction respectively. The appreciable percentage crystallization increase observed in the bonded magnetic composite could be traced to heterogeneous nucleation phenomenon, where the metal powders act as nucleation sites in the polyamide 4.6 binder. This agrees with the work of B. Weidenfeller et al. [16], where they reported similar improvement in % crystallization of polyamide 4.6 nanocomposite. This enhancement in % crystallinity experienced in the bonded magnetic composite indicates more ordered crystalline domains, making the amorphous molecular chains of the binder stiffer. Such observation has been reported to translate to higher dimensional stability and better mechanical properties of the composite [17, 18].

3.3 Magnetic Performance

The magnetic properties of the fabricated strontium ferrite bonded magnet were characterized to evaluate their performance at room temperature. This was achieved by obtaining the magnetic hysteresis loops and analyzing the intrinsic magnetic properties and magnetic anisotropy of the bonded magnets.

(a) Magnetic anisotropy

The magnetic anisotropy of the fabricated strontium ferrite bonded magnets was evaluated from the RT hysteresis loop. In order to fully understand the orientation and anisotropy behavior of the strontium ferrites platelets in the bonded magnets, a magnetic field angle hysteresis measurement was conducted such that the magnetic hysteresis of the investigated sample was obtained in all

360^{o} rotational direction at every 15 degrees. The investigated sample has a cylindrical geometry, and the sample was positioned on a perpendicular VSM rod in an orientation that is parallel (horizontal-0 degrees) and perpendicular (vertical – 90 degrees) to the applied magnetic field (H).

The obtained hysteresis loop from the field angle hysteresis measurement at RT is presented in **Figure 9** and **Figure 10** for 20 and 40% strontium ferrite bonded magnetic composite respectively. It could be observed that when the cylindrical filament sample was positioned parallel to the applied magnetic field, there were different induced magnetization signals and magnetic hysteresis loops when rotated from 0 degrees to 360 degrees. This is an indication that the fabricated bonded magnets exhibit magnetocrystalline anisotropy, where it requires more energy to magnetize or align the magnetic dipole moment in one direction than in other directions, thereby resulting in an easy axis and hard axis [19]. The easy axis is considered the axis that's most energetically favorable, which requires the smallest applied magnetic field to attain saturation, which can be observed as the field angle with the most squared hysteresis loop in **Figure 9a** and **Figure 10a**. In **Figure 9b** and **Figure 10b**, where the cylindrical bonded magnetic composite sample was positioned perpendicularly to the applied field (H), a similar magnetic signal shown as hysteresis loops overlapping one another was observed when rotated from 0 to 360 degrees. This suggests that the perpendicular orientation has similar magnetization energy, which appears to exhibit the same magnetic signal obtained for the easy axis in the parallel orientation. The anisotropy characteristics exhibited by the investigated strontium ferrite bonded magnetic composite are important to develop strong permanent magnetic devices, where their magnetic properties can be maximized via the preferred easy directions.

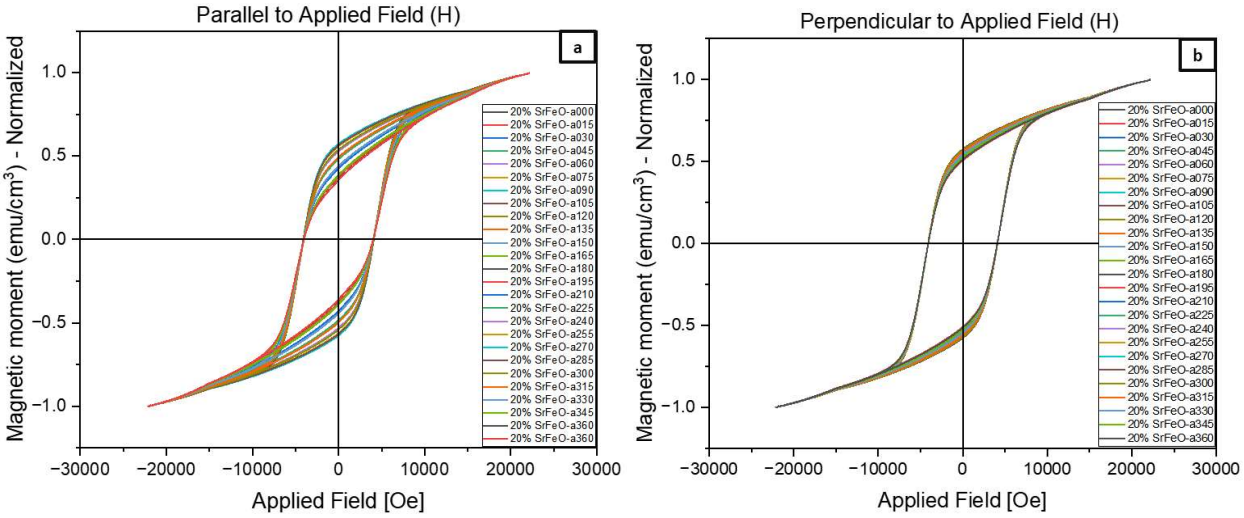


Figure 9. Magnetic hysteresis loop of 20 wt.% strontium ferrite bonded magnetic composites.

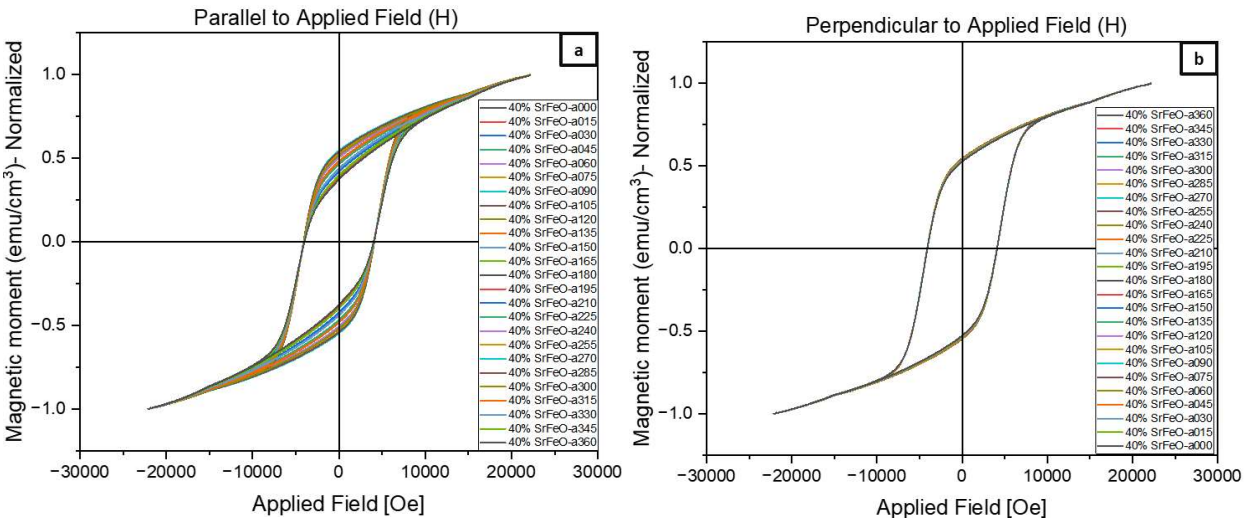


Figure 10. Magnetic hysteresis loop of 40 wt.% strontium ferrite bonded magnetic composites.

In order to fully understand the magnetic anisotropy behaviour of the fabricated bonded magnets, **Figure 11** shows the S-values (also known as hysterisis curve squareness) obtained for each magnetic hysterisis loop for every field angle measurement in both parallel and perpendicular orientation in **Figure 9** and **Figure 10**. S-values is an anisotropy parameter obtained as the ratio of remanent magnetization to saturation magnetization (Mr/Ms), which indicates the degree of alignment of the magnetic moment in a magnetic material. It is noteworthy that the direction or orientation with the highest S-value signifies where the easy axis lies in a magnetic material. As shown in **Figure 11**, the highest s-values were recorded at 90 degrees and 270 degree field angles measurement. This indicates the two prefered easy direction to magnetize the fabricated anisotropic strotium ferrite bonded magnets, and the other directions with lowest s-values (such as 0 and 180) as the hard directions. This is consistent with what was reported in the literature as the easy axis which is a line that lies parallel to two preferred directions that are 180-degree angles apart [20]. It was observed that the easy direction (e.g. 90 degrees) lies perpendicular to the extrusion direction of the strontium ferrite-bonded magnetic monofilament, while the hard direction (0 degrees) is parallel to the extrusion direction. This anisotropy behavior suggests the existence of a flow-induced anisotropy with an easy axis that is perpendicular to the extruded filament's cylindrical axis. Such anisotropy could have originated from the effect of the shear flow on the orientation of the strontium ferrite platelets near the wall of the twin screw extruder nozzle. This is in agreement with the observation reported by Ahmed et al.[21] for a freely extruded nylon 12/strontium ferrite magnetic composite filament, where the highest degree of alignment (easy direction) was reported in the radial direction of the extruded filament. The intrinsic magnetic properties obtained for both the easy and direction for both the 20 and 40 wt% have been summarized on **Table 1**. It is evident that the easy direction presents higher values of magnetic properties for both packing fractions which can be maximized or enhanced when the magnetic composites filaments are printed in a magnetic field assisted additive manufacturing (MFAAM) to produce magnetic devices.

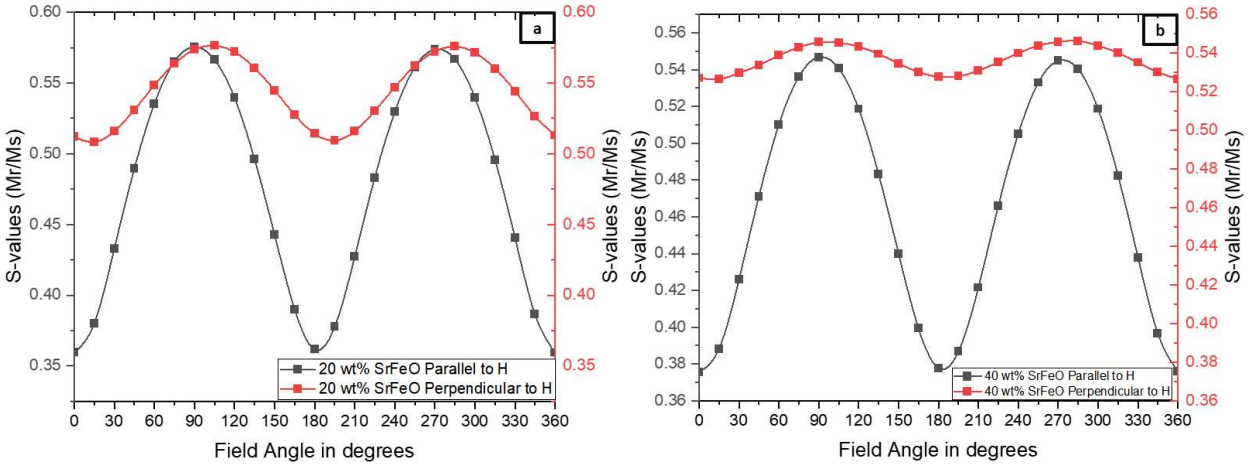


Figure 11. S-values of (a) 20 wt.% (b) 40wt.% strontium ferrite bonded magnetic composites.

Table 1. Magnetic properties (Mr, Ms, Hc & S-values) of the strotium ferrite bonded magnetic Composites.

		Mr (emu/cm^3)	Ms (emu/cm^3)	Hc (Oe)	S-values
Parallel - 20% SrFeO	Easy Axis 90°	209.43	363.91	4102.91	0.57546
	Hard Axis 0°	139.73	388.72	4006.42	0.35947
Perpendicular 20% SrFeO	Easy Axis 90°	214.71	373.84	4097.29	0.57351
	Hard Axis 0°	191.39	374.38	4083.36	0.51196
Parallel - 40% SrFeO	Easy Axis 90°	192.951	352.898	4104.03	0.54676
	Hard Axis 0°	144.618	384.956	4000.56	0.37567
Perpendicular 40%SrFeO	Easy Axis 90°	196.827	360.616	4106.12	0.54580
	Hard Axis 0°	192.059	364.794	4086.44	0.52648

4. CONCLUSIONS

High performance bonded magnetic composite was successfully fabricated via the twin screw extrusion technology, by loading a packing fraction of 20 and 40 wt.% strontium ferrite magnetic powders in a polyamide 4.6 binder to produce a 3D printable monofilament viable for fused filament fabrication 3D printing.

The microstructure of the bonded magnetic composites exhibited a considerable uniform dispersion of platelets morphology. This suggests that the platelets shape of the strontium ferrite magnetic powders, which is a contributing factor to its magnetic anisotropy, was retained in the magnetic composite. In addition to enhanced interfacial bonding between the magnetic fillers and polyamide 4.6 binder in the composite, a uniform dispersion of the strontium ferrites particles in the magnetic composites would prevents formation of complex magnetic domains caused by agglomeration, which can lead to decline in ferrimagnetic properties.

There was no observable change in the melting transitions of the bonded magnetic composites and the melting temperature of the polyamide 4.6 binder was not altered by the inclusion of strontium ferrite magnetic particles. This suggests that the bonded magnetic composites are

thermally resistant which is essential for high performance bonded magnetic applications. The crystallization behavior of the bonded magnetic composites exhibits an increment of 13 and 20% for 20wt.%and 40wt% loading fraction of strontium ferrites. This improvement in crystallinity suggests more ordered crystalline domains, making the amorphous molecular chains of the polyamide 4.6 binder stiffer which can translate to higher dimensional stability and better mechanical properties of the bonded magnetic composite.

The bonded magnetic composites exhibited a significant magnetic anisotropy with the easy axis perpendicular to the extrusion direction. Remanence (Mr), which is the most important properties for magnetic application was significantly increased by 49.8% along the easy direction in comparison to the hard axis. This suggests that the fabricated bonded magnets can be viable in producing anisotropic bonded magnetic devices where the intrinsic magnetic properties can be maximized along the easy direction.

5. ACKNOWLEDGEMENT

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