

I Electric Drive Technologies

I.1 Electric Drive Technologies Research

I.1.1 Bottom-Up Soft Magnetic Composites (Sandia National Laboratories)

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Project Introduction

In order to meet 2025 goals for enhanced peak power (100 kW), specific power (50 kW/L), and reduced cost (3.3 \$/kW) in a motor that can operate at $\geq 20,000$ rpm, improved soft magnetic materials must be developed. Better performing soft magnetic materials will also enable electric motors without rare earth elements. In fact, replacement of permanent magnets with soft magnetic materials was highlighted in the Electrical and Electronics Technical Team (EETT) Roadmap [1] as a R&D pathway for meeting 2025 targets. Eddy current losses in conventional soft magnetic materials, such as silicon steel, begin to significantly impact motor efficiency as rotational speed is increased. Soft magnetic composites (SMCs), which combine magnetic particles with an insulating matrix to boost electrical resistivity (ρ) and decrease eddy current losses, even at higher operating frequencies (or rotational speeds), are an attractive solution. Today, SMCs are being fabricated with values of ρ ranging between 10^{-3} to 10^{-1} $\mu\text{ohm}\cdot\text{m}$ [2], which is significantly higher than 3% silicon steel ($\sim 0.5 \mu\text{ohm}\cdot\text{m}$) [3]. The isotropic nature of SMCs is ideally suited for motors with 3D flux paths, such as axial flux motors. Additionally, the manufacturing cost of SMCs is low and they are highly amenable to advanced manufacturing and net-shaping into complex geometries, which further reduces manufacturing costs. There is still significant room for advancement in SMCs, and therefore additional improvements in electrical machine performance. For example, despite the inclusion of a non-magnetic insulating material, the electrical resistivities of SMCs are still far below that of soft ferrites ($10 - 10^8 \mu\text{ohm}\cdot\text{m}$).

We are developing SMCs from the bottom up, with a final objective of creating composites with high magnetic material loading (and therefore high magnetization) while increasing the value of ρ several orders of magnitude over current state-of-the-art SMCs. To accomplish our goals, we are starting with particles of γ' -Fe₄N, which have a saturation magnetic polarization (J_s) of 1.89 T, or slightly greater than Si steel [4] and a ρ of $\sim 2 \mu\text{ohm}\cdot\text{m}$ [5]. In our bottom-up approach we begin by coating the magnetic particles with a diamine, which chemically reacts directly with epoxide terminated monomers to form a cross-linked epoxy composite. This “matrix-free” approach to composite formation will not suffer from the same nanoparticle aggregation and phase separation effects commonly observed in most nanocomposites [6]. Furthermore, it should ensure better separation between magnetic particles and significantly reduce or eliminate inter-particle eddy currents. A precedent already exists for the use of epoxies in electrical machine construction [7, 8]. Additionally, it is possible to design epoxy systems with glass transition temperatures (T_g) well in excess of the target maximum motor operating temperature of 150 °C [9] as was documented in last year’s annual progress report. Furthermore, composites

have been successfully demonstrated in high-speed motors [10] and even flywheels rotating at speeds up to 60,000 rpm [11].

Objectives

The project objective is to develop high-magnetization, low-loss iron-nitride-based soft magnetic composites for electrical machines. These new SMCs will enable low eddy current losses and therefore highly efficient motor operation at rotational speeds up to 20,000 rpm. Additionally, iron nitride and epoxy composites will be capable of operating at temperatures of 150 °C or greater over a lifetime of 300,000 miles or 15 years.

Approach

A high-level overview of our approach is:

1. Convert commercially available mixed-phase iron nitride powder to nearly phase-pure γ -Fe₄N
2. Coat iron nitride particles with diamine molecules (part A of epoxy chemistry)
3. Combine surface functionalized particles with epoxide terminated monomers (part B of epoxy chemistry)
4. Fabricate SMC parts by adding mixture from #3 into a hot-pressing die
5. Evaluate and test the fabricated SMC part
6. Optimize SMC magnetic volume loading, magnetic properties, and physical properties

Results

Development of a lab scale hot pressing setup

In order to progress beyond the 65 vol.% Fe₄N achieved in our iron nitride/epoxy composites during FY 2020, we needed to move beyond curing our SMC samples in a mold and begin using a hot press for part fabrication. For this reason we built a lab scale hot pressing setup. An image of the hot pressing setup is displayed in Figure I.1.1.1. A zoomed in image of the die, thermocouple, and heating band inside of the press can be seen in Figure I.1.1.2. The specific components of our hot pressing setup are as follows: A 20 ton E-Z press (P/N 0012-6306) from International Crystal Laboratories (ICL); a CSI32R-C24 Benchtop Temperature Controller from Omega; an R-type (Pt 13% Rh/Pt, P13R-020-12) thermocouple, also from Omega; heating bands of various sizes from TempCo which are rated for 120V AC. The heating bands are constructed of mica insulated steel on the inner surface and stainless steel on the outer surface. The die for producing cylindrical test samples was a 3/8" ID stainless steel die from Carver. When fabricating toroids, a toroidal die from Electrodes, Inc. machined out of I-82 graphite was used. The ID of the pressed toroids was 6 mm and their OD was 9 mm.



Figure I.1.1.1. Lab scale hot pressing setup used for SMC part fabrication.

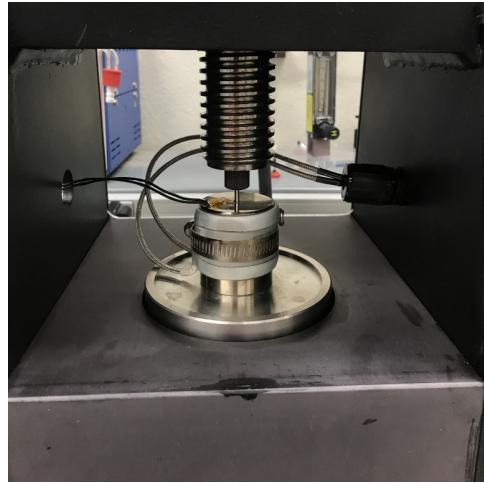


Figure I.1.1.2. Close up image of a die for hot pressing SMC samples. A heating band surrounds the die and the temperature is monitored with a thermocouple.

Prior to sample fabrication, control over the temperature inside the die was evaluated. As can be seen in Figure I.1.1.3, the temperature inside the die routinely stabilizes within 10 °C of the temperature set point.

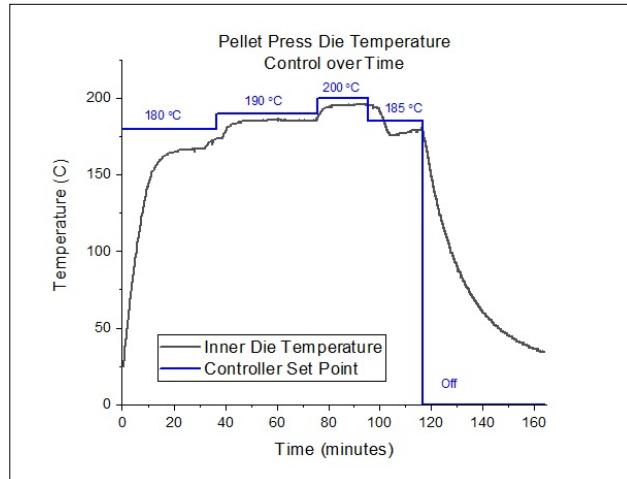


Figure I.1.1.3. Temperature inside the hot pressing die in relation to the temperature set point.

Iron nitride/epoxy composite fabrication via hot pressing

Iron nitride based SMCs were constructed using our new hot pressing setup. The processing parameters were optimized such that Fe₄N loadings \geq 70 vol.% could be achieved. Prior to adding the uncured iron nitride/epoxy mixture, the die was coated with boron nitride (BN) spray to serve as a release agent. A SMC with 71.4 vol.% Fe₄N was achieved using a pressure of 500 MPa and a temperature of 180 °C. The sample was pressed for 18 hours (overnight) and allowed to cool for 2 hours before removing from the die. The sample can be seen in Figure I.1.1.4.



Figure I.1.1.4. Iron nitride based SMC containing 71.4 vol.% Fe_4N . In the images located in the center and right hand side, the sample has been cut using a diamond saw prior to insertion in a magnetometer.

The sample's magnetic properties were characterized using a vibrating sample magnetometer (VSM) from Quantum Design. The magnetic hysteresis curve is plotted in Figure I.1.1.5. The sample achieved a saturation magnetization (M_s) of $144 \text{ Am}^2/\text{kg}$. As a comparison, bulk iron has a M_s of $217 \text{ Am}^2/\text{kg}$. When converted to saturation magnetic polarization (J_s) the SMC has a J_s of 0.96 T . This is nearly double that of soft ferrites ($J_s \sim 0.5 \text{ T}$), and half the value of Si steel ($J_s = 1.87 \text{ T}$). This puts iron nitride/epoxy SMCs in good standing amongst other insulating soft magnetic materials. Further increases in both M_s and J_s can be expected as the volume loading of iron nitride is increased further through additional process and material improvements.

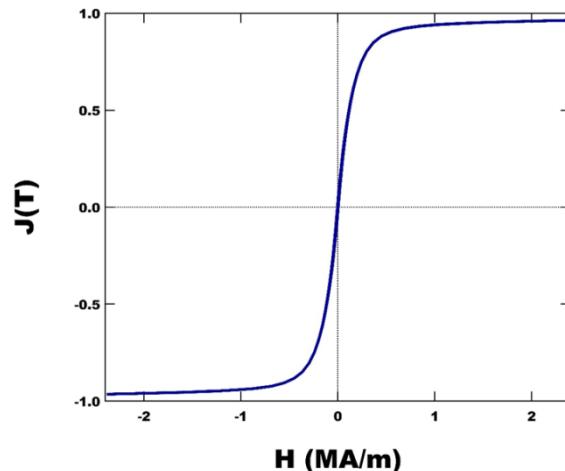


Figure I.1.1.5. Magnetic hysteresis curve (plotted as J vs. H) for an iron nitride based SMC containing 71.4 vol.% Fe_4N .

Thermal Characterization of iron nitride/epoxy SMCs

To ensure our magnetic composites are well designed for electric motor operation, it is also important to characterize, and perhaps even tune, the thermal conductivity of the samples. Additionally, understanding the thermal conductivity of our composite samples will be important for the consortium members attempting to integrate our bottom-up SMCs into their motor designs. Sandia has partnered with EDTC consortium member NREL to complete thermal conductivity measurements of our epoxy-based composites. NREL's thermal characterization setup requires $2'' \times 2''$ square samples no more than 2 mm thick. There is also a requirement that the square faces be flat and co-planar for high quality data to be collected. A photograph of NREL's thermal characterization apparatus is displayed in Figure I.1.1.6

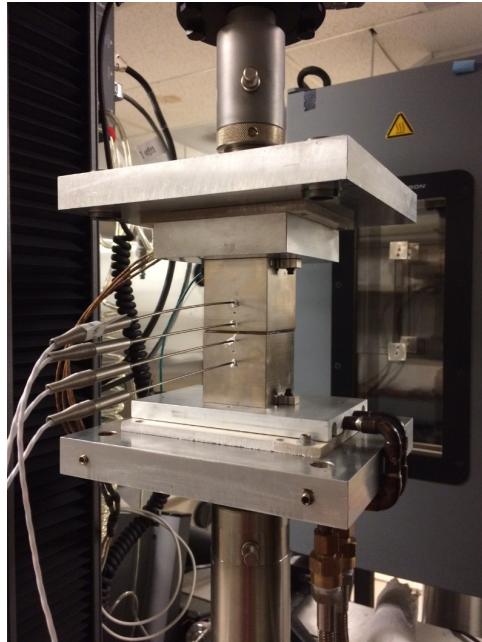


Figure I.1.1.6. Apparatus at NREL for bulk and thermal resistance measurement.

Both neat epoxy and Fe₄N/epoxy samples with 60 vol.% iron nitride loading were prepared. Uncured samples were added to a 3D printed mold and cured at temperature. Cured samples were polished to ensure the square faces were both smooth and co-planar with one another. Figure I.1.1.7 displays a 2" x 2" 60 vol.% Fe₄N in epoxy sample shipped to NREL for thermal characterization.

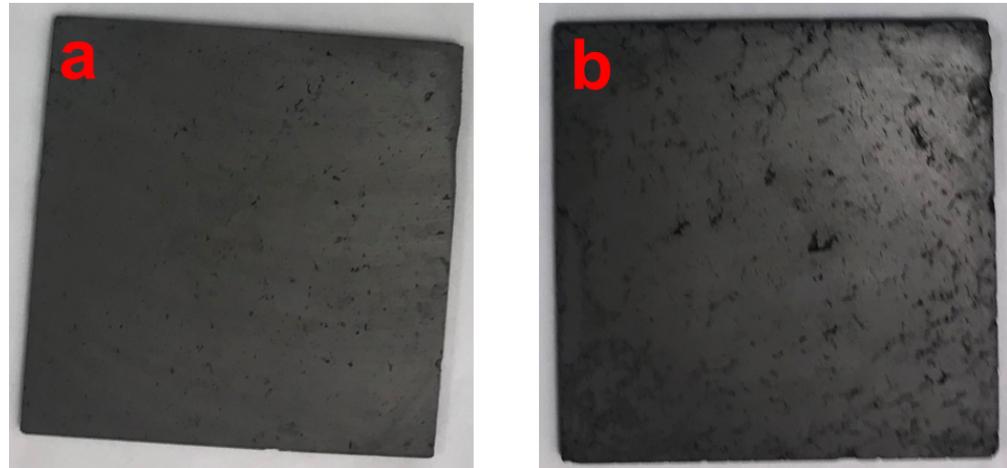


Figure I.1.1.7. Front and back of a 2" x 2" 60 vol.% Fe₄N in epoxy sample shipped to NREL for thermal characterization.

Thermal conductivity data collected at NREL for neat epoxy samples at three different temperatures (45 °C, 100 °C, and 150 °C) are shown in Figure I.1.1.8. Figure I.1.1.9 contains the data for the 60 vol.% Fe₄N/epoxy composites. Neat epoxy samples averaged a thermal conductivity of 0.24 W/m·K and iron nitride filled epoxy samples averaged 1.8 W/m·K.

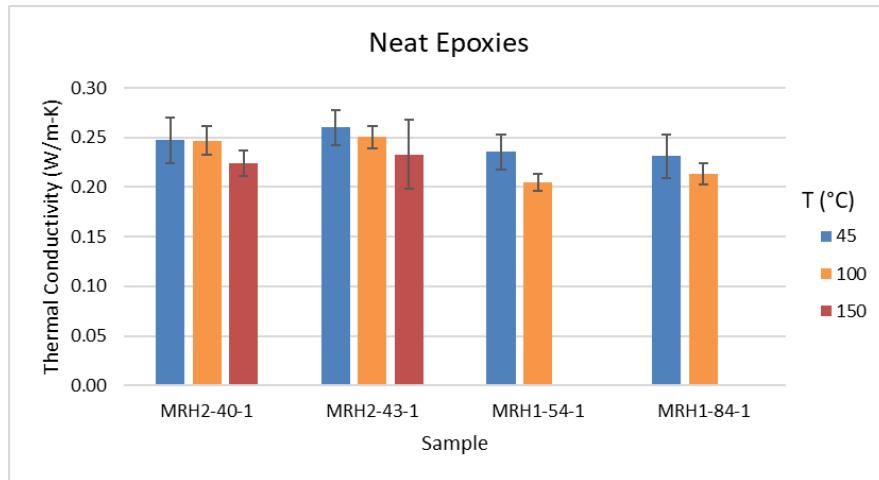
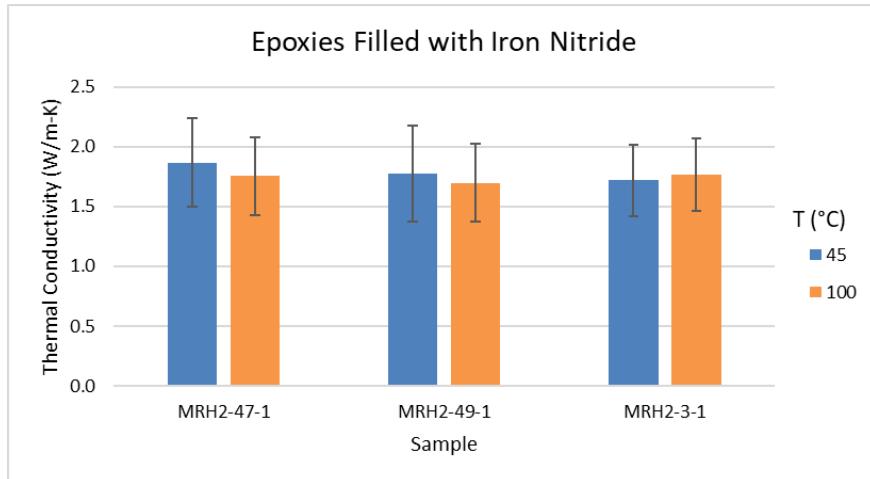


Figure I.1.1.8. Thermal conductivity results for neat epoxy samples collected at three different temperatures.

Figure I.1.1.9. Thermal conductivity results collected at both 45 °C and 100 °C for epoxy composites containing 60 vol.% Fe_4N .

Conclusions

During FY21, significant progress was made in the fabrication and characterization of iron nitride ($\gamma\text{-Fe}_4\text{N}$) based magnetic composites for electric motors. The reader should keep in mind that these materials also show substantial promise as inductor cores for electric drive power electronics. A lab based hot pressing setup was constructed and used to produce Fe_4N based SMCs with an iron nitride vol.% loading $> 70\%$. The J_s of these samples was nearly 1 T, which is double that of the leading state-of-the-art insulating soft magnetic material (ferrite). Additionally, further increases in J_s for Fe_4N based SMCs are imminent. Samples were fabricated for thermal conductivity measurements by consortium member NREL and the thermal conductivity for 60 vol.% Fe_4N in epoxy composites was higher than expected (1.8 W/m·K). This higher thermal conductivity could prove helpful in the cooling of electric motors constructed with Fe_4N based SMCs. Future work will focus continuing to increase magnetic material volume loading and enhance magnetic performance in both electric motor and motor drive applications. Additionally, during FY 2021 we will continue to collaborate with EDTC consortium NREL and investigate the mechanical strength of Fe_4N /epoxy composites.

Key Publications

1. T.C. Monson, B. Zheng, R. Delaney, C. Pearce, Y. Zhou, S. Atcity, E. Lavernia, Synthesis and Behavior of Bulk Iron Nitride Soft Magnets via High Pressure Spark Plasma Sintering. *Journal of Materials Research*, (2021). DOI: [10.1557/s43578-021-00379-z](https://doi.org/10.1557/s43578-021-00379-z).
2. G. Ouyang, B. Jensen, W. Tang, J. Schlagel, C. Pan, B. Cui, K. Dennis, D. Jiles, T.C. Monson, I. Anderson, M.J. Kramer, and J. Cui, Near Net Shape Fabrication of Anisotropic Fe-6.5%Si Soft Magnetic Materials. *Acta Materialia* **201**, 209-216 (2020). DOI: [10.1016/j.actamat.2020.09.084](https://doi.org/10.1016/j.actamat.2020.09.084).

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Acknowledgements

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