SAND2022-16891R

I Electric Drive Technologies

I.1 Electric Drive Technologies Research

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

Todd Monson, Principal Investigator
Sandia National Laboratories
P.O. Box 5800, MS 1415
Albuquerque, NM 87185
E-mail: tmonson@sandia.gov

Susan Rogers, DOE Technology Development Manager
U.S. Department of Energy
E-mail: susan.rogers@ee.doe.gov

Start Date: October 1, 2018 End Date: September 30, 2024
Project Funding (FY22): $125,000 DOE share: $125,000 Non-DOE share: $0

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 ≥ 20,000 rpm, improved soft magnetic materials must be developed. Better performing soft magnetic materials will also enable rare earth free electric motors. 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 increases. 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} μohm·m [2], which is significantly higher than 3% silicon steel (~0.05 μohm·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 ($10 - 10^8$ μohm·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'-Fe_4N, which have a saturation magnetic polarization (J_s) of 1.89 T, or slightly greater than Si steel [4] and a ρ of ~ 2 μohm·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 2020'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'-Fe_4N
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 fabricated SMC parts
6. Optimize SMC magnetic volume loading, magnetic properties, and physical properties

Results

Mechanical Characterization of iron nitride/epoxy SMCs

To ensure our magnetic composites are well designed for electric motor operation, it is important to characterize, and in some cases tune, their mechanical strength. Additionally, understanding the mechanical properties of our composites will be important for the consortium members attempting to integrate our bottom-up SMCs into their motor designs. Sandia once again partnered with EDTC consortium member NREL to complete mechanical measurements of our epoxy-based composites.

A significant amount of this fiscal year's effort was devoted to fabrication of dog bone structures for mechanical evaluation at NREL. ASTM standard D638 was a guide for choosing the size and shape of our dog bone test structures. Three test structures each of the following compositions were fabricated: neat epoxy, 30 vol.% Fe_4N, 40 vol.% Fe_4N and 65 vol.% Fe_4N. The first step in the fabrication process was to create a CAD drawing of an anti-mold for the dog bone structure and then 3D print that anti-mold. Next, the anti-mold is used to fabricate a silicone mold for the dog bone structure. An image of a silicone mold used for dog bone fabrication is displayed in Figure I.1.1.1. Once the silicone mold was completed, an appropriate amount of iron nitride powder was SPEX milled with our diamine, 4-aminophenyl sulfone (4-AS). After the surface of the iron nitride particles were coated with the 4-AS, they could be mixed with the second part of our epoxy, a triepoxide molecule: N,N-diglycidyl-4-glycidyloxyaniline (NND). This uncured mixture is then transferred into the silicone mold where it undergoes an initial cure at 180 °C for 4 hrs. After this initial curing step the test piece is transferred to a jig to prevent bowing during the final high temperature cure (see Figure I.1.1.2). The final high temperature cure occurs at 255 °C for 12 hrs. in tube furnace under flowing Ar with 3% H_2. X-ray diffraction (XRD) confirmed no oxidation of the Fe_4N occurred during this final high temperature curing step. After cooling, the dog bone structures are polished to remove most remaining surface defects and to minimize or eliminate camber. An example of a final polished dog bone structure prior to mechanical measurement can be seen in Figure I.1.1.3.

Figure I.1.1.1. Silicone mold for the fabrication of dog bone test structures.

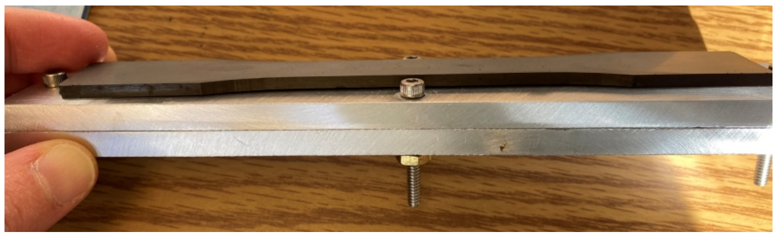

Figure I.1.1.2. Jig to prevent bowing of dog bone structures during final cure with a completed test structure resting on top.

Figure I.1.1.3. Dog bone structure ready for measurement at NREL.

The dog bone structures next underwent tensile testing at NREL. See Figure I.1.1.4 for a picture of a Fe_4N/epoxy composite undergoing tensile testing. The values obtained from tensile testing (elongation at yield, tensile strength, and tensile modulus) for our neat 4-AS/NND are comparable to other epoxies. The stiffness (tensile modulus) of the composite samples increases proportionally with the volume loading of iron nitride powder. Additionally, as iron nitride vol.% increased from 0 to 65 vol.% the tensile strength of the composite test structures decreased by approximately 18 MPa. For a full summary of the tensile testing results please see Table I.1.1.1.

Figure I.1.1.4. Picture of a Fe_4N/epoxy composite undergoing tensile testing at NREL.

Table I.1.1.1 Summary of Fe_4N/epoxy composite tensile testing results.

Fe_4N vol.% loading	Elongation at yield (%)	Tensile strength (MPa)	Tensile modulus (GPa)
0	9.7	51.7	3.8
30	6.2	35.7	10.3
40	5.4	32.7	15.4
65	3.5	33.0	33.0

Iron nitride/epoxy composite fabrication via hot pressing

Once again this fiscal year, additional iron nitride based SMCs were constructed using our in house hot pressing setup. The processing parameters were optimized such that Fe_4N loadings ≥ 75 vol.% could be achieved. A SMC with 75.3 vol.% Fe_4N 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.1.1.5.

Figure I.1.1.5. 75.3 vol.% Fe_4N in epoxy SMC. The shorter cylinder was cut using a diamond saw for VSM analysis.

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.1.1.6. This sample achieved a saturation magnetic polarization (J_s) of 1.19 T. This is more than double that of soft ferrites ($J_s \sim 0.5$ T), and nearly two-thirds the value of Si steel ($J_s = 1.87$ T). This puts iron nitride/epoxy SMCs in good standing amongst other state-of-the-art insulating soft magnetic materials. Further increases in J_s can be expected as the volume loading of iron nitride is increased further through additional process and material improvements.

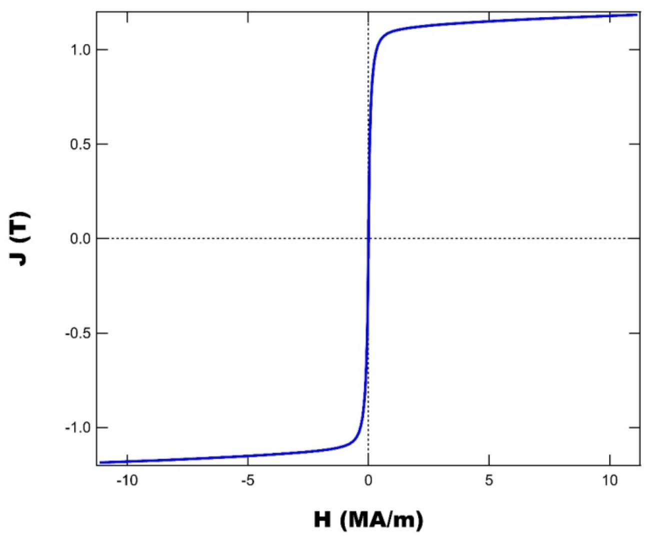

Figure I.1.1.6. Magnetic hysteresis curve (plotted as J vs. H) for an iron nitride based SMC containing 75.3 vol.% Fe_4N.

Fabrication of Larger SMCs

As our capability to produce Fe_4N/epoxy samples improves we are progressing to the fabrication of larger samples. This is a key step towards producing inductor cores for motor drives and soft magnetic parts for electric motors. We have acquired die that enable us to hot press toroids with an outer diameter (O.D.) of 35 mm and squares of 2 cm x 2 cm. During the upcoming fiscal year we will transition to fabricating specialized stator soft magnetic components. A photograph of newly acquired die for fabricating larger toroids and cubes is displayed in Figure I.1.1.7

Figure I.1.1.7. New die for pressing larger toroids and cubes.

Conclusions

During FY22, important progress was made in the fabrication and characterization of iron nitride (γ'-Fe_4N) based soft magnetic composites for electric motors. Please keep in mind that these materials also show substantial promise as inductor cores for motor drives. A lab based hot pressing setup was used to produce Fe_4N based SMCs with an iron nitride vol.% loading > 75 %. The J_s of these samples was nearly 1.2 T, which is more than double that of a leading state-of-the-art insulating soft magnetic material (ferrite). Additionally, further increases in J_s for Fe_4N based SMCs are still possible. Samples were fabricated for mechanical testing by consortium member NREL. The mechanical strength of our neat custom epoxy formulation is equivalent to other commercially available epoxies. Although the tensile strength of the composite test structures decreased by approximately 18 MPa as iron nitride vol.% increased from 0 to 65 vol.% this will have no impact on the use of iron nitride based SMCs in stator designs. In order to adopt Fe_4N based SMCs in rotor construction it may be necessary to utilize carbon fiber sleeves for added mechanical strength. However, there is considerable precedence already for the use of carbon fiber sleeves in electric motor construction [12]. Future work will focus on continuing to increase magnetic material volume loading and enhancing magnetic performance in both electric motor and motor drive applications. We will also increase the size of our fabricated SMC parts and begin constructing soft magnetic stator parts for prototype motor designs in collaboration with EDTC university and lab partners. Finally, during FY 2023 we will continue to collaborate with EDTC consortium member NREL and complete additional measurements on the thermal conductivity of Fe_4N/epoxy composites at elevated temperatures.

Key Publications

1. A.B. Kustas, D.F. Susan, T.C. Monson, "Emerging Opportunities in Manufacturing Bulk Soft-Magnetic Alloys for Energy Applications: A Review," *Journal of Materials*, **74**, 1306-1328 (2022). DOI: 10.1007/s11837-021-05019-9.

2. T.C. Monson, B. Zheng, R. Delaney, C. Pearce, Y. Zhou, S. Atcitty, E. Lavernia, "Synthesis and Behavior of Bulk Iron Nitride Soft Magnets via High Pressure Spark Plasma Sintering," *Journal of Materials Research*, **37**, 380-389 (2022). DOI: 10.1557/s43578-021-00379-z.

References

[1] US Drive, "Electrical and Electronics Technical Team Roadmap," *Partnership Plan, Roadmaps, and Other Documents* 2017.

[2] H. Shokrollahi and K. Janghorban, "Soft magnetic composite materials (SMCs)," *Journal of Materials Processing Technology,* vol. 189, no. 1-3, pp. 1-12, 2007, doi: 10.1016/j.jmatprotec.2007.02.034.

[3] J. S. Corporation. *Super Core^{TM} Electrical steel sheets for high-frequency application*. (2017). JFE Steel Corporation. [Online]. Available: http://www.jfe-steel.co.jp/en/products/electrical/catalog/f1e-002.pdf

[4] J. M. D. Coey, *Magnetism and Magnetic Materials*. New York: Cambridge University Press, 2010.

[5] T. C. Monson *et al.*, "Soft Magnetic Multilayered FeSiCrB–Fe_4N Metallic Glass Composites Fabricated by Spark Plasma Sintering," *IEEE Magnetics Letters,* vol. 10, pp. 1-5, 2019, doi: 10.1109/LMAG.2019.2906832.

[6] M. Qu *et al.*, "Magneto-photo-acoustic imaging," *Biomed. Opt. Express,* vol. 2, no. 2, pp. 385-396, 2011/02/01 2011, doi: 10.1364/BOE.2.000385.

[7] M. Magazine. https://magneticsmag.com/new-structural-adhesive-from-delo-for-magnet-bonding-has-high-temperature-stability/ (accessed.

[8] Crosslinktech. http://www.crosslinktech.com/products-by-application/featured-electric-motor-products.html (accessed.

[9] M. Bond. https://www.masterbond.com/techtips/how-optimizing-glass-transition-temperature-tg (accessed.

[10] A. Schoppa and P. Delarbre, "Soft Magnetic Powder Composites and Potential Applications in Modern Electric Machines and Devices," *IEEE Transactions on Magnetics,* vol. 50, no. 4, pp. 1-4, 2014, doi: 10.1109/TMAG.2013.2290135.

[11] P. Mason, K. Atallah, and D. Howe, *Hard and soft magnetic composites in high speed flywheels*. 1999.

[12] V. C. d. Nascimento and S. D. Sudhoff, "Non-Axisymmetric Structural Analysis of High Speed Rotor Orthotropic Retention Sleeve," in *2019 IEEE International Electric Machines & Drives Conference (IEMDC)*, 12-15 May 2019 2019, pp. 797-804, doi: 10.1109/IEMDC.2019.8785324.

Acknowledgements

This work is supported by the DOE Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Office. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. The views expressed in the article do not necessarily represent the views of the U.S. Department of Energy or the United States Government. We wish to thank Melinda Hoyt, Charles Pearce, Sophia Shaar, and Sydney Fultz-Waters for their fabrication of magnetic composites; Robert Delaney and Charles Pearce for their assistance with magnetic characterization; and Mark Rodriguez for his help with X-ray diffraction data collection and analysis. We would also like to acknowledge key collaborators within the electric drive systems consortium: Kevin Bennion (NREL), Emily Cousineau (NREL), Iver Anderson (Ames Lab), Jun Cui (Ames Lab), and Matt Kramer (Ames Lab).