The role of microstructural evolution during spark plasma sintering on the soft magnetic and electronic properties of a CoFe–Al₂O₃ soft magnetic composite

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ABSTRACT

For transformers and inductors to meet the world's growing demand for electrical power, more efficient soft magnetic materials with high saturation magnetic polarization and high electrical resistivity are needed. This work aimed at the development of a soft magnetic composite synthesized via spark plasma sintering with both high saturation magnetic polarization and high electrical resistivity for efficient soft magnetic cores. CoFe powder particles coated with an insulating layer of Al₂O₃ were used as feedstock material to improve the electrical resistivity while retaining high saturation magnetic polarization. By maintaining a continuous non-magnetic Al₂O₃ phase throughout the material, both a high saturation magnetic polarization, above 1.5 T, and high electrical resistivity, above 100 μΩ·m, were achieved. Through microstructural characterization of samples consolidated at various temperatures, the role of microstructural evolution on the magnetic and electronic properties of the composite was elucidated. Upon consolidation at relatively high temperature, the CoFe was found to plastically deform and flow into the Al₂O₃ phase at the particle boundaries and this phenomenon was attributed to low resistivity in the composite. In contrast, at lower consolidation temperatures, perforation of the Al₂O₃ phase was not observed and a high electrical resistivity was achieved, while maintaining a high magnetic polarization, ideal for more efficient soft magnetic materials for transformers and inductors.

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Introduction

More efficient power conversion devices, particularly transformers are needed for power grids to meet the global demands for increased energy consumption. Transformers and other power conversion devices such as motors and inductors rely on magnetic cores which must be made of soft magnetic materials [1–3]. Soft magnetic materials are not necessarily mechanically soft, but are magnetically soft, meaning the induced magnetic polarity in the material can be easily switched by an applied field and the material has relatively low magnetic coercivity ($H_c < 1000$ A/m) [3]. Some of the applied magnetic field is required to overcome the material's coercivity before a polarity can be induced in the material, causing magnetic power losses. To minimize magnetic power losses, low coercivity in soft magnetic materials must be achieved. Eddy current losses are another major source of power losses. Eddy current losses come from eddy currents produced in the core material by the switching magnetic polarity of the magnetic core. Eddy current losses can be approximated using Eq. (1) from [4],

$$P_e \propto \frac{d^2 B^2 f^2}{\rho}, \quad (1)$$

Eddy current losses (P_e) are inversely proportional to the electrical resistivity (ρ) of the core material. B is the magnetic induction in the material, f is the frequency of operation of the power conversion device, and d is the electrical domain size, or effective scale of the eddy currents [4, 5]. To minimize eddy current losses, high electrical resistivity and small eddy current domain size are necessary in a core material. Soft magnetic materials used as magnetic cores must also possess high saturation magnetic polarization ($J_s > 1.0$ T) to allow conversion devices to operate with greater power densities, converting more power with smaller core volumes. An ideal soft magnetic core should have low coercivity, high electrical resistivity, and high saturation magnetic polarization. The challenge in many state of the art soft magnetic materials, such as Si steels, has been achieving ideal magnetic properties and high electrical resistivity due to the metallic nature of most soft magnetic materials [6]. Si steels have a moderately high J_s around 1.8–2.0 T, but low electrical resistivity, approximately 0.5–0.8 $\mu\Omega\cdot\text{m}$

[6]. To reduce eddy current losses, Si steel cores are often produced as laminated sheets, effectively minimizing eddy current domain size to the thickness of individual Si steel sheets. Recently, composite materials with a soft magnetic metallic phase and an electrically insulating phase have been explored to produce more efficient soft magnetic core materials with high J_s and high electrical resistivity.

Development of soft magnetic composites (SMCs) as a design approach to more efficient soft magnetic cores began in the 1990s and gained notoriety for their potential to significantly minimize magnetic energy losses [3]. SMCs can potentially achieve high J_s , low coercivity, and high electrical resistivity, necessary to maximize power density and minimize magnetic energy losses, when a magnetically ordered phase is properly intermixed with a non-magnetic, electrically insulating phase [3, 7–10]. As noted in Eq. (1), by increasing the bulk electrical resistivity and by reducing d , electrical domain size of eddy currents of a soft magnetic material, the eddy current losses of the material are reduced [5]. SMCs can achieve high bulk electrical resistivity and reduced electrical domain sizes by fully intersecting a magnetic phase with a continuous phase which is non-magnetic and electrically insulating. Ultimately, the achievable magnetic properties of a SMC are limited by the volume fraction, morphology, and distribution, of the non-magnetic, insulating phase. The insulating phase in the SMC acts as a distributed air gap, and if not properly controlled, a high volume fraction of the non-magnetic phase detrimentally increases coercivity and reduces J_s by physically separating and inhibiting magnetic interaction between the magnetic phase [3]. Compared to discontinuously distributed non-magnetic fibers or particles, a continuous non-magnetic phase intersecting dispersed particles of a magnetic phase has been shown to be more effective at tailoring the magnetic and electronic properties of SMCs [7, 8]. A continuous phase prevents long range charge carrier transport and more effectively increases the measured resistivity of the SMC. Additionally, the thickness of the continuous phase between magnetic particles can be adjusted to tailor the magnetic interaction of the particles and thus the magnetic behavior of the SMC. The volume fraction, morphology, and distribution of the magnetic phase in an SMC can be adjusted to maintain high J_s and low coercivity, and achieve high electrical resistivity [7–10]. Development of SMCs

and their microstructures can potentially achieve more efficient properties for soft magnetic cores.

One of the most broadly effective and practical approaches to SMC development has been the consolidation of coated ferromagnetic powder particles [7, 8]. Ferromagnetic powders maximize the achievable J_s in SMCs and can be coated with an insulating layer to reliably restrict charge carrier movement in the bulk, consolidated SMC. Once consolidated, the coatings on the powder particles form a continuous phase, intersecting the ferromagnetic particles. By selecting powder particle sizes (typically on the range of 5–200 μm) and controlling the powder coating thickness, the bulk volumetric phase fraction of the insulating phase can be adjusted [7–10]. In related studies, Fe powders have been coated with organic polymers such as epoxies and resins, allowing the coated powders to be densified with relatively low consolidation pressures and temperatures. Organic polymer SMCs yielded moderate resistivity, on the order of 100 $\mu\Omega\cdot\text{m}$, but typically are not thermally stable above 300 $^{\circ}\text{C}$ and have low J_s , less than 1.0 T [11, 12]. In other approaches, consolidated SMCs made with coatings of Al_2O_3 , SiO_2 , or even CaF_2 on ferromagnetic powder particles reached high resistivities on the order of 100–1000 $\mu\Omega\cdot\text{m}$, and moderate J_s , approximately 1.0–1.5 T [13–22]. The high resistivity of Al_2O_3 containing SMCs is attributed to the high electrical resistivity of Al_2O_3 , on the order of 10^{17} $\mu\Omega\cdot\text{m}$. Additionally, the Al_2O_3 phase has a higher thermal stability than organic polymers and other insulating phases such as phosphates or fluorides which may degrade at comparably lower temperatures [19–24]. However, the processes used to coat powders with Al_2O_3 are relatively of small scale and have not enabled large production volumes of SMCs. Furthermore, the ceramic coating on metal powders requires higher temperatures and thus longer sintering and consolidation times, allowing equilibrium structures to form which may hinder magnetic and electronic properties [7, 8]. Large-scale and thick powder coating deposition techniques and consolidation processes are necessary to develop more efficient SMCs for power conversion device cores. In recent years, commercial coating deposition techniques have been developed to effectively coat large batches of metal powders with thick ceramic coatings. Industrially developed processes can produce several kilograms of powders with uniform, micrometers thick Al_2O_3 coatings reacted and

deposited onto the surfaces which can be consolidated into SMCs with controlled microstructures and properties. Non-equilibrium spark plasma sintering (SPS) consolidation processes have been developed over the years and can rapidly consolidate both metal and ceramic powders. Similar to conventional sintering, SPS is a field activated, diffusional process, which causes coalescence of powders across interfaces to form a dense, bulk body. Compared to conventional sintering, the rapid heating rates and short sinter times associated with SPS can be used to fully densify materials with minimal grain growth. In the case of SMCs, SPS can be used to rapidly consolidate metal powders coated with a ceramic while avoiding the formation of new phases or mixing of the constituent elements which may degrade the final properties of the SMC [25, 26]. In addition to more robust powder coating and consolidation techniques, ferromagnetic powders with improved soft magnetic properties can be used as the feedstock powder to achieve higher J_s .

In view of the above challenges, the objective of the present study was to investigate the feasibility of using a powder consolidation approach to synthesize a soft magnetic composite material and to investigate the resultant magnetic and electronic characteristics. To accomplish this objective, instead of Fe, the equiatomic CoFe alloy which has significantly higher J_s was used as the ferromagnetic powder to maintain high J_s and low H_c in SMCs [7, 8, 27]. Moreover, a large-scale deposition process was used to coat a large volume of CoFe powder particles with Al_2O_3 which were then subsequently densified using SPS consolidation. To produce a large volume of powders with a thick coating, on the order of several micrometers, the CoFe powders were coated by a vendor using a commercial, proprietary process which chemically reacts Al_2O_3 onto the surfaces of a fluidized bed of CoFe powders. After this coating process, a fully dense SMC with a CoFe– Al_2O_3 core-shell structure with particles of the CoFe phase fully separated by a continuous Al_2O_3 phase was then targeted by consolidating the coated CoFe powders using SPS to achieve high J_s , above 2.0 T, and high electrical resistivity, above 1.0 $\mu\Omega\cdot\text{m}$.

Materials and methods

Gas atomized, pre-alloyed equiatomic CoFe powder particles, with a particle size below 150 μm , were purchased from Oryx Advanced Materials (Fremont, CA, USA) and were used as the feedstock powder to be coated with Al_2O_3 . Approximately 1 kg of CoFe powder particles in the range of 20–150 μm were coated with 2 vol. % Al_2O_3 (2–5 μm thick) by Advanced Powder Solutions, Inc (Cypress, TX, 77429 USA) with a proprietary chemical vapor deposition process. Using a Fuji SPS-825S DR. SINTER (Fuji Electronic Industrial Co., Ltd., Kawasaki, Japan) SPS with a maximum pulsed DC output of 12 V and 8000 A, the as-coated powders were consolidated at 700 °C and 1000 °C under a vacuum of less than 5 Pa to form 5 mm OD $\times$ 2 mm HT samples. During SPS consolidation, the uniaxial load was manually held at the lower limit of the SPS, to stay below a compressive stress of 150 MPa. The samples were then polished for magnetic properties measurements and cross-sectioned for microstructural characterization.

The Archimedes method was used to measure density of as-consolidated SMC disks. To assess the relative density the measured density was compared to the theoretical density of a BCC CoFe structure with 2 vol. % Al_2O_3 . The phase composition of the as-received powders and consolidated SMCs was assessed with a Rigaku SmartLab X-ray diffractometer (XRD) equipped with a Cu K_α ($\lambda = 0.1542$ nm) radiation source. As-received coated powders were mounted in KonductoMet™ and mechanically polished to 0.04 μm to evaluate the microstructural features of the cross sections of powders. The consolidated SMCs were cross-sectioned using wire electronic discharge machining (EDM) and were mechanically polished to 0.04 μm to evaluate the microstructural features in the cross section of the SMCs, perpendicular to the SPS loading direction. The microstructures of cross-sectioned as-coated powders and consolidated SMCs were characterized using a FEI Quanta 3D field emission gun scanning electron microscope (SEM) equipped with an Everhart Thornley secondary electron (SE) detector, a pole piece mounted backscatter electron (BSE) detector and an Oxford energy-dispersive X-ray spectrometer (EDS). ImageJ software was used to measure grain sizes and coating thicknesses. All micrographs of the consolidated disks are normal to the uniaxial SPS loading direction.

The magnetic and electronic properties of the consolidated disks were measured at Sandia National Laboratories (Albuquerque, NM, USA). To evaluate the J_s and coercivity of the consolidated disks, the magnetic properties were measured using a Quantum Design MPMS-7 superconducting quantum interference device (SQUID) magnetometer. Magnetization curves were recorded from -7 T to $+7$ T at 293 K. To more accurately measure the effects of the Al_2O_3 coating and avoid surface defects from the consolidation and polishing process, the electrical resistivity through the height of the consolidated disks was measured. To measure resistivity through the height of the consolidated disks, copper strips were fitted to the circular faces of the disks and clips attached to the copper strips were used as leads. The leads were driven by a Keithley 2425 SMU set to 1 μV resolution and 10 NPLC averaging cycles to reduce noise. The current was swept from 100 mA to 1 A. All disks were isolated from the sample chuck using non-conductive, double-sided tape during measurements.

Results

The SE micrograph and corresponding BSE micrograph and EDS elemental maps of the cross sections of as-coated powders are shown in Fig. 1a, b, c, d, and e, respectively.

From the cross sections of the as-coated powder BSE micrograph in Fig. 1a, spherical powder particles with particle size on the order of 10 μm are observed. Grain features can be observed in the BSE micrographs in Fig. 1a and b, revealing that the particles have average grain size of 12 μm . In the BSE micrographs in Fig. 1a and b, the Al_2O_3 coating is observed as a dark gray phase surrounding powder particles. The Al_2O_3 coating is also observable in the EDS elemental map in Fig. 1c, where it is clear that Al_2O_3 is fully coating the CoFe powder particles. The Al_2O_3 coating is approximately 2–5 μm thick. Lastly, it should be noted the Al-rich, needle-shaped particles observed in the EDS elemental map in Fig. 1c are artifacts of the KonductoMet™ polymer the powders are mounted in for cross-sectional polishing. In Fig. 2 (a), the XRD pattern of the as-coated powders is plotted and compared with the SMCs made from the powders, consolidated at 700 °C and 1000 °C. A closer view of the Al_2O_3 peak is shown and indicated in Fig. 2b.

Figure 1 **a** BSE micrograph depicting an as-coated Al_2O_3 -coated CoFe powder particle and its grains and **b** lower-magnification BSE micrograph and **c–e** corresponding EDS elemental maps showing the Al_2O_3 coating fully surrounding the CoFe powder particles.

Figure 2 **a** XRD patterns comparing the as-received gas atomized CoFe powders, the as-coated Al_2O_3 -coated CoFe, and the coated powders consolidated at 700 °C and 1000 °C and **b** a closer view of the Al_2O_3 XRD peaks.

From the XRD pattern of the as-coated feedstock powders in Fig. 2a and b, additional Bragg's law satisfying peaks, corresponding to Al_2O_3 appear at 2θ angles of 40.289° and 44.097° in addition to the major BCC peaks for CoFe. The XRD patterns of the CoFe– Al_2O_3 composite consolidated at 700 °C and 1000 °C are also shown in Fig. 2a and b for comparison with the as-coated powder before SPS consolidation. After SPS consolidation at 1000 °C, the major peaks for a BCC CoFe structure are observed. Unlike the as-coated powder XRD pattern, an additional peak is not observable in Fig. 2b or indexed by the Rigaku SmartLab XRD software. Similar to the as-coated powder XRD pattern, the observed peaks are relatively narrow. Like the 1000 °C consolidated composite and the as-coated powder XRD patterns, the peaks of the 700 °C consolidated composite XRD pattern are relatively narrow and the major peaks for the BCC CoFe phase are observed. In addition to the

major BCC peaks, in Fig. 2b, a peak corresponding to Al_2O_3 is observed at a 2θ angle of 42.764° . However, the additional peak in the 700 °C consolidated composite XRD pattern appears at a different 2θ angle than the additional peaks in the as-coated powder XRD pattern, likely due to a phase transformation of the Al_2O_3 [28].

The BSE micrograph and corresponding EDS elemental maps of the cross section of the 1000 °C consolidated CoFe– Al_2O_3 composite are included in Figs. 3a, b, c, and d, respectively.

After SPS consolidation at 1000 °C, the relative density of the SMC reached nearly 95%. In the BSE micrograph of the 1000 °C consolidated SMC in Fig. 3a, consolidated powder particles, bounded by the Al_2O_3 coating, are observed. From the BSE micrograph and corresponding EDS elemental maps in Figs. 3a, b, c, and d the Al_2O_3 coating is distinctly observable from the CoFe powder by BSE contrast

Figure 3 **a** BSE micrograph of 1000 °C consolidated CoFe–Al₂O₃ composite and **b–d** corresponding EDS elemental maps depicting the Al₂O₃ coating fully surrounding the CoFe powder particles.

and EDS element detection. In the BSE micrograph, the Al₂O₃ phase appears nearly black and the CoFe phase appears light to medium gray with contrast within powder particles due to grain orientation. The coating is approximately 1–3 μm in thickness. Compared to the as-coated powder particles shown in Fig. 1a and b, the 1000 °C consolidated powders appear less spherical and more elongated, and the average particle size has increased to approximately 25 μm. Within the 1000 °C powder particles, the grains appear to be larger than grains in the as-coated powders and have a grain size of approximately 10 μm, the same order of magnitude as in the powder particles themselves. Figure 3a also clearly shows that the powder particles consolidated at 1000 °C are slightly deformed and packed tightly with the Al₂O₃ phase uniformly coating the boundaries between particles.

A SE micrograph and corresponding BSE micrograph and EDS elemental maps and point scans of the 1000 °C consolidated composite at higher magnification is shown for further analysis, in Fig. 4a, b, c, d, e, and f, respectively.

The polished Al₂O₃ phase has a morphologically different surface topology and appears brighter than the polished CoFe powder particle cores in the SE micrograph. Some porosity is observable between powder particles in the Al₂O₃ phase, suggesting the Al₂O₃ is not fully densified. While some small, nanometer-scale pores are found within CoFe powder particles, most likely from the atomization process, the CoFe phase appears fully densified. Upon closer inspection of the boundaries between consolidated powder particles at high magnification, some CoFe is observed between powder particles, in the Al₂O₃ coating regions. This CoFe perforation into the coating is circled in red in the BSE micrograph in Fig. 4b and can be observed upon close inspection of the EDS element maps in Fig. 4c, d, and e.

The BSE micrograph and corresponding EDS elemental maps of the 700 °C consolidated composite are shown in Fig. 5a–d, respectively.

In the BSE micrograph, in Fig. 5a, the Al₂O₃ phase, coating powder particles, appears nearly black and the CoFe particles appear light to medium gray with some contrast within powder particles due to grain orientation. The continuous Al₂O₃ coating of non-

Figure 4 Higher-magnification **a** SE micrograph and corresponding **b** BSE micrograph and EDS elemental maps of **c** Al, **d** Co, and **e** Fe. Some regions where CoFe appears perforating the Al_2O_3 coating regions, are circled in red.

Figure 5 **a** BSE micrograph and **b–d** corresponding EDS elemental maps of the 700 °C consolidated CoFe– Al_2O_3 composite, showing the distinct, continuous Al_2O_3 coating surrounding CoFe powder particles.

uniform thickness is observed between CoFe powder particles in the BSE micrograph and corresponding EDS element maps. The relative density of the 700 °C

consolidated composite was only approximately 82%, less dense than the 1000 °C consolidated composite and appeared more porous in the Al_2O_3 phase in the

electron micrographs as well. This is apparent in the regions between powder particles which are both black in the BSE micrograph and appear to have low Al content in the corresponding EDS map. Compared to the 1000 °C consolidated composite, the powders are more loosely packed in the 700 °C consolidated composite. In the EDS element maps in Fig. 5b–d, no CoFe is detected perforating the Al₂O₃ phase regions between powder particles. A higher-magnification SE micrograph and corresponding BSE micrograph is shown in Fig. 6a and b, respectively.

From the SE micrograph in Fig. 6a, isolated pores are observed (black arrows) within powder particle interiors. Between particles, and at triple junctions especially, voids and gaps in the Al₂O₃ coating are observed in the SE micrograph. Analysis of seventeen SE micrographs of the cross sections of the 700 °C SPS consolidated samples, at magnifications of 250x, 500x, and 1000x, reveals the volume fraction of the porosity is estimated to be approximately 18.89%. However, it should be noted that the threshold and analysis of the micrograph does not perfectly distinguish between porosity and the Al₂O₃ phase which can both appear dark in the micrographs. Additionally, some of the porosity in the micrograph may actually be due to pull-out of the Al₂O₃ phase during mechanical polishing and may not reflect the true density of the SMC. From both Figs. 5a and 6a, powder particles bounded by the Al₂O₃ coating are observed on the order of 10 µm. Within the powder particles, grain features are observed to be ~ 10 µm, as in the as-coated powders. Lastly, in the SE micrograph in Fig. 6a, some powder particles appear bright, but showed no compositional difference in the corresponding BSE micrographs or EDS element maps.

The measured magnetic and electronic properties and relative densities of the consolidated CoFe–Al₂O₃ composites are reported in Table 1.

Properties of the as-received coated and uncoated CoFe powders and SMCs from the previous literature are also shown in Table 1 for reference. The 1000 °C consolidated composite reached a measured J_s of 1.90 T, slightly higher than the J_s reported for the 700 °C consolidated composite. Both consolidated composites in this study had a noticeably greater J_s than previous SMCs, but not compared to the CoFe alloy, which was expected. The 700 °C sample achieved an electrical resistivity several orders of magnitude higher than the 1000 °C consolidated CoFe–Al₂O₃ composite from this work and the equiatomic CoFe alloy [27]. The DC hysteresis curves of the consolidated SMCs are shown in Fig. 7a and b.

From Fig. 7a and b, the coercivity of the 700 °C CoFe–Al₂O₃ composite was approximately an order of magnitude less than the composite consolidated at 1000 °C. As reported in Table 1, the J_s of both SMCs is less than 2.0 T. It has been shown that far greater resistivities were previously achieved in SMCs, at the cost of lowered J_s [7–9, 14–16]. Ultimately, the J_s of the composites in this study was slightly lowered compared to previous reports of the equiatomic CoFe alloy [27]. However, the J_s achieved remained higher than that of SMCs produced in previous studies because CoFe was used as the ferromagnetic phase as opposed to Fe or Fe–Si [7, 8]. While a change in J_s , proportional to the change in relative density was expected, a smaller shift in J_s was observed, suggesting the CoFe phase was fully densified at both temperatures, yielding little change. In this study, compared to the CoFe alloy, a dramatically increased resistivity was achieved by consolidating Al₂O₃-

Figure 6 Higher-magnification **a** SE micrograph and **b** corresponding BSE micrograph of the 700 °C consolidated composite, showing non-uniform porosity and voids in the Al₂O₃ coating surrounding CoFe powder particles. Some of the porosity in the powder particles is denoted by black arrows.

Table 1 Measured properties and relative densities of the as-received coated and uncoated CoFe powder as well as the consolidated CoFe–Al₂O₃ composites from this study, along with previously reported

Sample	Saturation polarization (J_s) [T]	Magnetic saturation (M_{sat}) [$A \cdot m^2/kg$]	Coercivity (H_c) [A/m]	Through resistivity (ρ) [$\mu\Omega \cdot m$]	Relative density (% of theoretical)
As-received CoFe	n.a	219 ± 1.6	1050 ± 6.1	n.a	n.a
As-coated CoFe–Al ₂ O ₃	n.a	218 ± 1.6	555 ± 6.1	n.a	n.a
1000 °C SPS CoFe–Al ₂ O ₃	1.90 ± 0.01	194 ± 1.6	382 ± 6.1	0.20 ± 7.19	94.69 ± 0.24
700 °C SPS CoFe–Al ₂ O ₃	1.88 ± 0.01	227 ± 1.6	98.9 ± 6.1	135.49 ± 7.19	82.54 ± 0.24
SMC from the previous literature [3, 6–12, 15–20]	0.3 – 1.8	n.a	150 – 400	100 – 30,000	n.a

properties of SMCs for comparison. Values of magnetic saturation are reported for the loose powders since J_s is a volumetric, density dependent measurement

Figure 7 Magnetic hysteresis curves of the CoFe–Al₂O₃ SMC after consolidation at **a** 1000 °C and **b** 700 °C. The insets in each plot show a narrower range of the B-H loop to observe the coercivity of each SMC.

coated CoFe powders. The electrical resistivity achieved in this SMC is within typical limits of previous SMC studies.

Discussion

The 1000 °C consolidated composite achieved a high relative density, nearly 95%, and is attributed to the high SPS temperature used which allowed more sintering and consolidation of the Al₂O₃ phase. There may be some discrepancy in the calculated relative density and the observed porosity in the SE micrographs because the phase fraction of the Al₂O₃ coating was targeted to be 2 vol.% Al₂O₃ in the powders but may differ in the composites due to the distribution of powder particle sizes and thus actual volume fractions of the Al₂O₃ phase. At 1000 °C, the CoFe phase appears to be fully densified, suggesting differences in density are due to partial densification of Al₂O₃ during SPS. In the SE micrograph in Fig. 4a,

the porosity observed within powder particles is on the nanometer scale and is common in gas atomized powders even near fully dense [25, 26]. At 1000 °C, 0.5 T_{melt} of Al₂O₃ and 0.66 T_{melt} of CoFe was reached allowing full sintering of the CoFe metal, but only partial sintering of the Al₂O₃ phase [25]. Additionally, any thermal expansion of the graphite dies during SPS consolidation may have caused increased uniaxial pressure above 150 MPa but the lower limit of the SPS prevented any reduction in the compressive load. The elongation and tight packing of powder particles as well as the interpenetration of the CoFe phase into the Al₂O₃ coating are attributed to the high temperature and pressure achieved during SPS. Furthermore, at a temperature above 0.5 T_{melt} in metals, grain growth is expected and was observed in the CoFe phase of the 1000 °C consolidated composite [29]. While CoFe and Al₂O₃ do not appear to have formed any new phase, as shown by the XRD pattern of the consolidated composite in Fig. 2 and Co and Fe have little to no solubility in Al₂O₃ so no

new phases or reactions are expected to take place during consolidation of this SMC [30–32]. However, in the BSE micrograph in Fig. 4b and the results of the EDS point scans shown in Fig. 4f, the CoFe phase is observed perforating the Al_2O_3 boundary phase. It is most likely that the CoFe phase has undergone plastic deformation and flow causing interpenetration into and through the regions of the Al_2O_3 coating. In the previous literature describing the processing of functionally graded materials prepared through consolidation of coated and multiphase powders, viscoplastic deformation of the softer phase of a multiphase composite has been observed and analytically modeled, especially at contact points between the softer and harder phases [33, 34]. Furthermore, at temperatures as low as $0.4T_{\text{melt}}$ metals can begin to easily experience plastic deformation and creep, and at 1000°C , the CoFe phase is at nearly $0.67T_{\text{melt}}$ which would enable the plastic flow causing the interpenetration observed in Fig. 4b [1]. Ultimately, from the previous literature and microstructural observations, it is clear the CoFe phase has perforated the Al_2O_3 phase when consolidated at 1000°C , suggesting the SMC has been over-consolidated, yet the Al_2O_3 phase appears only partially densified and sintered.

In the XRD pattern of the 700°C consolidated composite in Fig. 2a and b, the major BCC CoFe peaks and an additional peak at a 2θ angle of 42.764° was observed. From the previous literature, the additional peak most likely corresponds to the hexagonal α phase of Al_2O_3 [28]. However, a different set of additional peaks appear in the XRD pattern of the as-coated powders, at 2θ angles of 40.2859° and 44.097° . The extra peaks in the as-coated powder most likely correspond to the metastable cubic γ phase of Al_2O_3 [28]. The Al_2O_3 coating most likely undergoes a phase transformation from the metastable γ phase to the stable hexagonal α phase, which has been shown to occur at elevated temperatures and pressures in several previous works [28, 35, 36]. Additionally, the proprietary chemical vapor deposition powder coating process is a non-equilibrium process which potentially resulted in the deposition of a metastable Al_2O_3 phase on the CoFe powder particles. From the XRD patterns in Fig. 2a and b, after consolidation at 700°C , no evidence of formation of a new phase with Co or Fe was observed.

Compared to the composite consolidated at 1000°C , after consolidating the coated powders at 700°C , little to no grain growth was observed in the electron micrographs in Figs. 5a and 6a and b. At only 700°C , the Al_2O_3 coating only reached $0.35T_{\text{melt}}$ which most likely prevented full sintering and consolidation of the coated Al_2O_3 phase [25, 26]. The large gaps and voids observed between particles in the continuous Al_2O_3 coating are further indications of only incomplete densification. While some gaps and voids are from particles which have fallen out of the sample during polishing, the fact that some particles are loosely bonded at all further suggests the Al_2O_3 phase was not fully consolidated at 700°C . Furthermore, the loose packing of the CoFe powder particles and thicker, more substantial Al_2O_3 phase at triple junctions between particles also suggests the Al_2O_3 phase was not fully densified. Additionally, the particles which appear brighter in the SE micrograph in Fig. 6a most likely emit high SE signal due to charging effects of trapped electrons in the SEM [1]. These particles are most likely loosely bound and potentially able to fall out, preventing the flow of electrons into the rest of the microscopy sample. It is also possible the powder particles are well electrically insulated by the Al_2O_3 coating and isolated from the rest of the sample, also preventing the flow of electrons from the SEM beam through the sample. In either case, the presence of the bright particles further suggests incomplete densification of the Al_2O_3 phase at only 700°C . Ultimately, at only 700°C , a continuous Al_2O_3 phase intersecting CoFe particles was achieved and no new phases in the CoFe– Al_2O_3 composite were detected. However, the Al_2O_3 phase was not fully densified. Despite the incomplete densification of the CoFe– Al_2O_3 composite at 700°C , exceptional magnetic and electronic properties were observed.

In Table 1 and Fig. 7a and b, the magnetic properties of the 1000°C and 700°C consolidated composite showed significant decreases in J_s compared to the CoFe alloy; this is attributed to microstructural features of the material and the decreased volume fraction of the magnetic CoFe phase [27]. An exceptionally high J_s can be achieved by the CoFe powder particles and is attributed to the interatomic spacing of the B2 ordered BCC CoFe structure and its electron concentration [27, 37]. The ordering of the CoFe alloy is a diffusionless transformation which occurs below 700°C ; therefore, both consolidated composites

would contain B2 ordered CoFe powder particles [27, 38, 39]. However, the loose packing of powder particles, in other words, the low density of the SMC, can cause detrimental decreases to the J_s . The physical space and gaps between magnetic particles can prevent full magnetic alignment of domain walls in separated magnetic particles [3, 5, 21, 22, 40]. By significantly decreasing the volume fraction of the magnetic CoFe phase, and by physically separating the CoFe particles, the Al_2O_3 phase reduces the bulk J_s of the composite. The effect of the physical spacing between particles is more noticeable in the 700 °C consolidated composite as the Al_2O_3 phase appears far less dense than in the 1000 °C consolidated composite. However, both consolidated composites in this study achieved higher J_s than in previous SMCs and is attributed to the CoFe powder particles and relatively high densification achieved using SPS. Because nearly full densification was achieved in the 1000 °C composite, 1.90 T may be the maximum threshold J_s for this composite without reducing the volume fraction of Al_2O_3 . From this J_s of 1.90 T, using a law of mixtures calculation, it is estimated there is approximately 20% Al_2O_3 by volume in this SMC. The discrepancy from the targeted volume fraction of Al_2O_3 coated on CoFe powders is likely due to the broad particle size range consolidated in this study. Further improvement to the magnetic properties of the composite may be achieved by consolidating coated powders above a specific size range, effectively reducing the volume fraction of Al_2O_3 in the consolidated composite. Additionally, an increase in J_s proportional to the increase in density was expected, but a relatively low change in J_s is observed in the 1000 °C compared to the 700 °C consolidated SMC. This is likely because at both temperatures, the main J_s controlling phase, the CoFe, is fully densified at both 700 °C and 1000 °C. The main difference in density is due to the Al_2O_3 phase, so less dramatic changes to J_s are observed. Additionally, the percolated CoFe phase in the Al_2O_3 boundary phase can act as smaller particles of a ferromagnetic phase and may cause disruptions to the magnetization and degradation of the achievable J_s of the CoFe phase. In general, decreases to J_s in SMCs compared to the CoFe alloy are expected and can be controlled with densification and Al_2O_3 volume fractions. The J_s reported of the SMCs in this study are exceptionally high compared to previous studies of SMCs and can be further improved upon in the future.

Similarly, as shown in Fig. 7a and b, the additional phase in a SMC can detrimentally cause increases in coercivity and therefore increased hysteresis losses. While the coercivity of the consolidated composites was not specifically targeted, the coercivity of the consolidated composites in this work was relatively low and in typical range of most SMCs. In general, larger grain sizes in soft magnetic materials result in decreased coercivities, as grain boundaries and anisotropy in the crystalline lattice can disrupt the alignment of magnetic domain walls [3, 7, 8]. Despite this, the 1000 °C consolidated composite displayed a relatively higher coercivity than the composite consolidated at 700 °C, which experienced less grain growth. It is possible that the percolated CoFe pathways in the Al_2O_3 phase cause disruptions to the magnetization reversal of the larger powder particles of the composite. Nonetheless, the coercivity is typical of consolidated SMCs. The relative permeability, which was not directly targeted or studied in the 1000 °C and 700 °C consolidated samples was 4.34 and 3.84, respectively. Compared to CoFe, the relative permeability of the consolidated composites in this work is relatively low and less than ideal, although process and Al_2O_3 volume fraction adjustments could improve the relative permeability substantially. Ultimately, the coercivity and permeability of the composites produced in this study fall in the range of typical magnetic coercivities and permeabilities of previously studied SMCs.

The increased electrical resistivity observed in the CoFe– Al_2O_3 SMC can be attributed to the Al_2O_3 coating between CoFe powder particles, which has electrical resistivity on the order of $10^{17} \mu\Omega\cdot\text{m}$. In the 1000 °C consolidated composite, compared to the CoFe alloy, only a minor increase to the electrical resistivity, within statistical variance of CoFe alloys is observed. As depicted in Figs. 3b and 4b, and discussed previously, during SPS consolidation at 1000 °C, the CoFe phase is observed to have penetrated into the Al_2O_3 phase between powder particles. This interpenetration of the CoFe phase could introduce electrically conductive pathways between particles, allowing charge carrier conduction across the Al_2O_3 boundaries between powder particles. Additionally, the larger grain sizes observed in the 1000 °C sample further decrease scattering events of charge carriers, decreasing electrical resistivity, although grain size alone would not account for a several order of magnitude difference [41].

Compared to previously reported SMCs, the electrical resistivity of the CoFe–Al₂O₃ composite consolidated at 1000 °C is relatively low. Ultimately, large increases in electrical resistivity were not observed in the 1000 °C consolidated composite likely because of the flow of the CoFe phase into Al₂O₃ boundaries between particles. To further increase the resistivity, percolation of the CoFe phase into the insulating phase must be prevented.

By contrast, in the 700 °C consolidated composite, the resistivity increased by nearly 4 orders of magnitude compared to the CoFe alloy. The dramatic increase to electrical resistivity is attributed to the retention of a continuous Al₂O₃ coating during SPS consolidation with no plastic flow of the CoFe phase into the Al₂O₃ boundary, and the relatively low density of the composite. Compared to previously reported SMCs, the electrical resistivity reported in the composite consolidated at 700 °C is relatively low, but still within the typical range. As discussed previously and shown in Figs. 5a and b and 6a and b, at 700 °C, the composite was only partially densified. During SPS consolidation at 700 °C, the CoFe particles remained fully bounded by the Al₂O₃ coating, effectively creating a continuous, yet porous phase of Al₂O₃ through the CoFe particles. The irregularities in the morphology of the Al₂O₃ phase at triple junctions and between particles prevents charge carrier conduction, increasing the bulk electrical resistivity measured through the composite. This increased resistivity can result in the reduction of eddy current losses in soft magnetics by minimizing eddy currents in the bulk SMC. Additionally, eddy current losses are further reduced by limiting the electrical domain size d to the size of coated powder particles as discussed with Eq. (1) previously. If eddy currents are produced in the bulk composite, the eddy current sizes will be confined to individual CoFe powder particles and significant eddy current losses can be prevented. Ultimately, dramatic increases to resistivity in the 700 °C consolidated composite, compared to the composite consolidated at 1000 °C and CoFe alloy are attributed to the SMC having a relatively low density and a fully continuous Al₂O₃ phase separating CoFe powder particles.

Conclusions

A CoFe–Al₂O₃ composite was produced by consolidating Al₂O₃-coated CoFe powder particles via SPS, targeting a SMC with high J_s and high electrical resistivity. The powder particles were coated with a large-scale, proprietary coating deposition process. After SPS consolidation of the coated powders at 1000 °C, decreases to the magnetic properties, proportional to the volume fraction of the Al₂O₃ phase and minimal changes to the electronic properties were observed compared to the CoFe alloy. The minimal changes to the electronic properties were attributed to the interpenetration of the CoFe phase into the Al₂O₃ coatings between powder particles during SPS. At 1000 °C, plastic flow of the CoFe phase is activated and percolated through the Al₂O₃ phase. The composite consolidated at 700 °C displayed an exceptional combination of high J_s , 1.88 T, and high electrical resistivity, 135 $\mu\Omega\cdot\text{m}$, and the properties were attributed to the SMC having relatively low density in the Al₂O₃ and a continuous Al₂O₃ coating surrounding CoFe powder particles. Further densification of the CoFe–Al₂O₃ composite after consolidation at 700 °C, without uncontrolled percolation of the CoFe phase into the Al₂O₃ phase, may further improve magnetic properties of the composite. In the future, the consolidation of larger powder particle size ranges can reduce the volume fraction of Al₂O₃ and further target improved soft magnetic properties. However, larger powder particle sizes and therefore lowered Al₂O₃ volume fractions may result in reduced electrical resistivity of the final SMC. In this study, the focus was magnetic and electronic properties of these consolidated composites; future studies exploring mechanical behavior of the composite are recommended to further develop the CoFe–Al₂O₃ composite and others like it for widespread soft magnetic applications. The efficiency of soft magnetic materials may be increased by controlling secondary phase fractions and morphologies and constituent element diffusion in SMCs.

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Author's contribution

Calvin Belcher participated in conceptualization, validation, formal analysis, investigation, resources, writing the original draft, and visualization. Baolong Zheng was involved in conceptualization, methodology, validation, investigation, resources, and writing, reviewing, and editing. Benjamin E. MacDonald took part in methodology, validation, resources, and writing, reviewing, and editing. Eric D. Langlois and Benjamin Lehman contributed to methodology, validation, formal analysis, investigation. Charles Pearce and Robert Delaney were involved in methodology, validation, and formal analysis. Diran Apelian took part in writing, reviewing, and editing, and supervision. Enrique J. Lavernia contributed to resources, writing, reviewing, editing, supervision, and funding acquisition. Todd C. Monson participated in conceptualization, resources, writing, reviewing, and editing, supervision, and funding acquisition.

Conflict of interest The authors declare they have no known competing interests or personal

relationships that could have appeared to influence the work reported in this paper.

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