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PERSPECTIVE PIECE

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Functionally graded magnetic materials: a perspective to advance charged particle optics through compositional engineering

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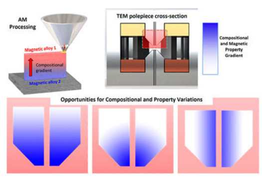
ABSTRACT
Additive manufacturing has ushered in a new paradigm of bottom-up materials-by-design of spatially non-uniform materials. Functionally graded materials have locally tailored compositions to provide optimized global properties and performance. In this letter, we propose an opportunity for the application of graded magnetic materials as lens elements for charged particle optics. A Hiperco50/Hymu80 (FeCo-2 V/Fe-80Ni-5Mo) graded magnetic alloy was successfully additively manufactured via Laser Directed Energy Deposition with spatially varying magnetic properties. The compositional gradient is then applied using computational simulations to demonstrate how a tailored material can enhance the magnetic performance of a critical, image-forming component of a transmission electron microscope.



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Spatial non-uniformities are often interpreted as flaws in a material, wherein conventional wisdom and bulk processing desires homogeneous, monolithic materials with repeatable microstructures, compositions, properties, and performance. However, many common structures have graded properties to increase their functionality and improve macroscopic performance, sacrificing less-important microscopic properties locally to improve global performance. Spatial segregation within a microstructure, either compositionally, or via grain size engineering can be formed using a variety of processes; however, these processes can also have undesired consequences such as gradient stresses, recrystallization, increased susceptibility to irradiation damage, or chemical degradation. Topology optimization is often used to introduce novel designs, such as lattice structures to reduce weight, distributing form and function [1], and compositional optimization can also be applied to introduce novel microstructures and properties [2]. Materials can be engineered on the nano-to-micro scale with compositional and microstructural gradients and heterogeneities that improve performance. Advances in the past decades with thin-film growth, self-assembly, and additive manufacturing (AM) have enabled the development of functional materials with spatially segregated properties tailored for optimal performance.

Graded magnetic materials have been utilized recently in the microelectronics industry, commonly employed with abrupt layering, for exchange-spring ferromagnetics in thin film architectures [1–3]. This method has recently been adopted into graded anisotropy spring media, moving beyond discrete layers. However, adopting this technology to larger scale, metallic components has proven challenging due to process controls, material

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anisotropies, and bridging the micro-scale to macro-scale properties with processing. Functionally graded materials (FGMs) change composition or phase to modify the properties or functional behavior. Previous synthesis of FGMs relied on thin-film growth or bonding/layering [4–7]. However, with additive manufacturing, materials are built layer-by-layer, increasing the potential for novel microstructures and forms of functional materials. AM offers the ability to print near-net-shape bulk geometries, with compositional variations as complex as the shapes themselves to tailor the material for ideal application in the desired environment. Material properties can be more easily user-controlled to improve the local and global material performance. Tailoring the local composition and microstructure affects the local properties and global performance, such as the magnetic properties of the material [8].

Magnetic functionality is important for numerous design spaces, such as space flight propulsion engines, energy conversion and transmission systems, microelectronics, and high-power density electric machines for transportation. Examples include electrical machine magnetic cores and rotors, transformers, micromotors, and transducers [1,2]. AM fabrication of a graded magnetic material would allow for unique property formulation with site-specific properties unachievable with conventional processing methods. Often applications desire switching from soft to hard magnetic materials. For example, FeCo alloys are important where high saturation and high thermal stability (high Curie temperature) is a factor, such as in aerospace systems, while FeNi alloys are important as a high-permeability soft magnetic material for shielding of electronics [2]. Bridging the gap between these two can be challenging, but offers the ability to tailor the permeability, coercivity, and saturation magnetizations within a single component geometry. Conventional bulk magnetic materials are often processed via solidification and deformation processing, melt spinning, or thermal-spraying [9,10]. However, conventional techniques are limited in their available design and composition space, which necessitates post-processing forming to fabricate complex geometry components. In contrast, additive manufacturing enables the fabrication of near-net shape components, with increased process control, allowing a wider range of control over the microstructures and compositions. AM processes, including laser powder bed fusion (LPBF), directed energy deposition (DED), and e-beam melting have been used for fabrication of soft and hard magnetic materials, utilizing bottom-up manufacturing techniques to process unique structures and compositions optimized for global material performance [9–11]. Common to the AM scheme is layer-by-layer materials printing, enabling local real-time process control on the micron scale. Selective laser melting (SLM) has been used to print Fe-Ni-V and Ni-Fe-Mo with laser engineered net shaping (LENS) [11] as well as Fe-Co-V via SLM. LENS has been used to print Fe-Si-B-Cu-Nb soft alloys with significant influence of the printing parameters. Complex microstructures and geometries have been explored via soft-magnetic functional composite [9] and cold-spraying [10], but until recently not with AM directly. LENS of Fe-Ni, Ni-Fe-V, and Ni-Fe-Mo alloys has been performed by Mikler [11], with the scan speed greatly influencing the build magnetic properties. Additionally, Fe-Co (Hiperco-like) alloys have been printed by LENS previously [12], enabling production of near net-shape forms without post-process working, with the magnetic properties falling within the range of conventionally-processed Hiperco.

However, joining two distinct magnetic materials to one another has remained a challenge, due to differences in compositions and properties between dissimilar soft magnetic materials. However, recently some work has advanced the field of graded magnetic materials, with fabrication via spark plasma sintering (SPS) [13], while A.M. Chaudhary et al. [14] printed CoFe to NiFe with AM and showed how increasing Fe content increases the saturation magnetization. Heer [15] used LENS to print a graded 316 SS to 430 SS material, spanning the non-magnetic to magnetic space. Finally, graded Fe-Si-B-Nb-Cu [16] with changing Si:B ratios have been printed. Soft and hard magnetic materials together in the same process can open new doors for designing tailored topologies of magnetic devices and fields without the costs of tooling and post-processing [17]. Graded magnetic materials hold potential for spatial variation in mechanical and magnetic properties (i.e. grading from a structural to magnetic material or grading from one magnetic material to another) in applications for magnetic machines, but the concepts and advances in manufacturing can be applied to a new application space: magnetic lenses for charged particle optics (CPOs).

In this letter, we explore graded magnetic materials for CPOs to gain enhanced control over the beam used for imaging nanoscale features. Like optical lenses, magnetic lenses are used for bending, focusing, and magnifying charged particle beams. Naturally, there are many similarities in the principles behind which both lens types accomplish these tasks. One potential application of graded magnetic materials is for CPOs. In the transmission electron microscope (TEM), the image-forming optic is the objective lens, and its construction and composition greatly affect the performance of the TEM. The lens materials are commonly made up of solenoidal coils of wire surrounded by monolithic, uniform ferromagnetic materials such as soft Fe [18]. This has been the mode of operation for decades and has resulted in good TEM performance. However, if instead of monolithic materials, the magnetic field of the CPOs can be tuned using diverse materials configurations, the performance of the TEM could be strategically tuned. Along this vein, using materials with graded properties, such as graded magnetic materials instead of a monolithic ferromagnetic material may be able to alter the performance of the CPOs and the subsequent microscope optics. In this work, we discuss the fabrication of a graded magnetic material via AM, present preliminary properties of printed materials, show with computational modelling how graded magnetic properties can be used in the TEM objective lens geometry, and propose the ability to design future functional magnetic materials with geometries and compositions for charged particle optics applications.

As a proof of concept, this paper investigates the application of graded magnetic materials for charged particle optics. Graded magnetic materials are additively manufactured via Laser Engineered Net Shaping (LENS) demonstrating how variations in the microstructure can play important roles in tailoring the magnetic properties. Then, we explore the ability to tune the electron optics in a TEM using graded magnetic materials with computational work presenting conceptual designs for how the magnetic flux magnitude changes with corresponding changes in the material properties. Finally, we choose TEM objective optics to illustrate graded AM of magnetic materials for two reasons: (1) the geometry of pole pieces is critical to their performance, and (2) the flux in in pole pieces is very high compared to most magnetic applications, around the saturation flux (2.4 T) of the best magnetic materials. Functionally graded magnetic materials may improve the performance characteristics of CPOs lenses via spatial control of saturation, which would help in thermal management and lens efficiency.

The limited space in the pole piece gap of a TEM necessitates material magnetic properties optimized within the spatial constraints. To address the feasibility of fabricating graded magnetic materials, a Hiperco-Hymu graded material was 3D printed with LENS, as shown schematically in Figure 1(a). Hyperco50 (Fe-49Co-2 V) is a Fe-Co-V soft magnetic alloy with a high-saturation magnetization and Hymu80 (Fe-80Ni-5Mo) is a Fe-Ni-Mo alloy with a high permeability. These were chosen for their high-saturation properties and ability to shield stray magnetic fields. A 12.7 mm diameter x 76 mm tall gradient rod was deposited on a mild steel build plate using and RPMI 222 blown powder directed energy deposition system. $\sim$ 144 layers of the Hymu80 material were first deposited after which the composition was linearly transitioned to Hiperco50 over $\sim$ 12 layers (3 mm-thick gradient region) in $\sim$ 7 volume percent increments followed by deposition of $\sim$144 layers of Hiperco50. A laser power of 650 watts was used with a scan speed of 16.9 mm/s and a layer thickness of 0.25 mm. Development of the printing process (including printing parameters) is detailed individually for both alloys in previous publications [19–21]. Figure 1(b–c) shows the microstructure of the as-printed graded alloy, with the change in composition corresponding to a change in grain size, as indicated in the EBSD data.

Figure 1 shows an exemplar functionally-graded Hiperco50-Hymu80 specimen, which can be successfully printed via DED without macroscopic defects. The authors believe this is among the first direct examples for printing one magnetic material on another. Previous work has controlled order within a Fe-Co alloy to a few AM build layers, but this approach has not yet been explored for graded Hiperco-Hymu structures [22]. AM has been used to process traditionally brittle Fe-Si and Fe-Co alloys without macroscopic defects, showing how AM is enabling the development of high-performance soft magnetic alloys [23]. Graded Fe-Co-316 SS has been shown to be successfully printed with stable microstructures in the gradation region [24]. However, as Figure 1 shows, graded microstructures from Hiperco to Hymu were successfully printed with minimal porosity for the first time to the best knowledge of the authors. Full analysis of the printed microstructures will be published in future work.

Magnetic materials frequently have nonlinear magnetic flux response for a linear magnetic field. With AM, we can tailor the nonlinear response by modifying the local composition and material microstructure. The magnetic properties, including permeability, B-H curves, and magnetic saturation must be determined for the global part and locally to decipher the compositional effects on properties. Figure 2 shows one concept in which the ability to tailor compositions and therefore properties is explored for optimizing material performance in a TEM objective lens geometry. Example B-H curves of a multi-material configuration consisting of mixed Co-Fe and Ni-Fe are shown in Figure 2(a) and demonstrates the possibility for tailoring properties. In the TEM objective lens geometry, shown in Figure 2(b), although the physical geometry is highly constrained, the ability to grade the magnetic properties significantly expands the design space. Figure 2(c–e) shows just a few examples of how compositional variations can be designed within the pole piece geometrical constraints, leading to corresponding variations in the global magnetic performance. These gradations in magnetic properties represent potential design opportunities that could

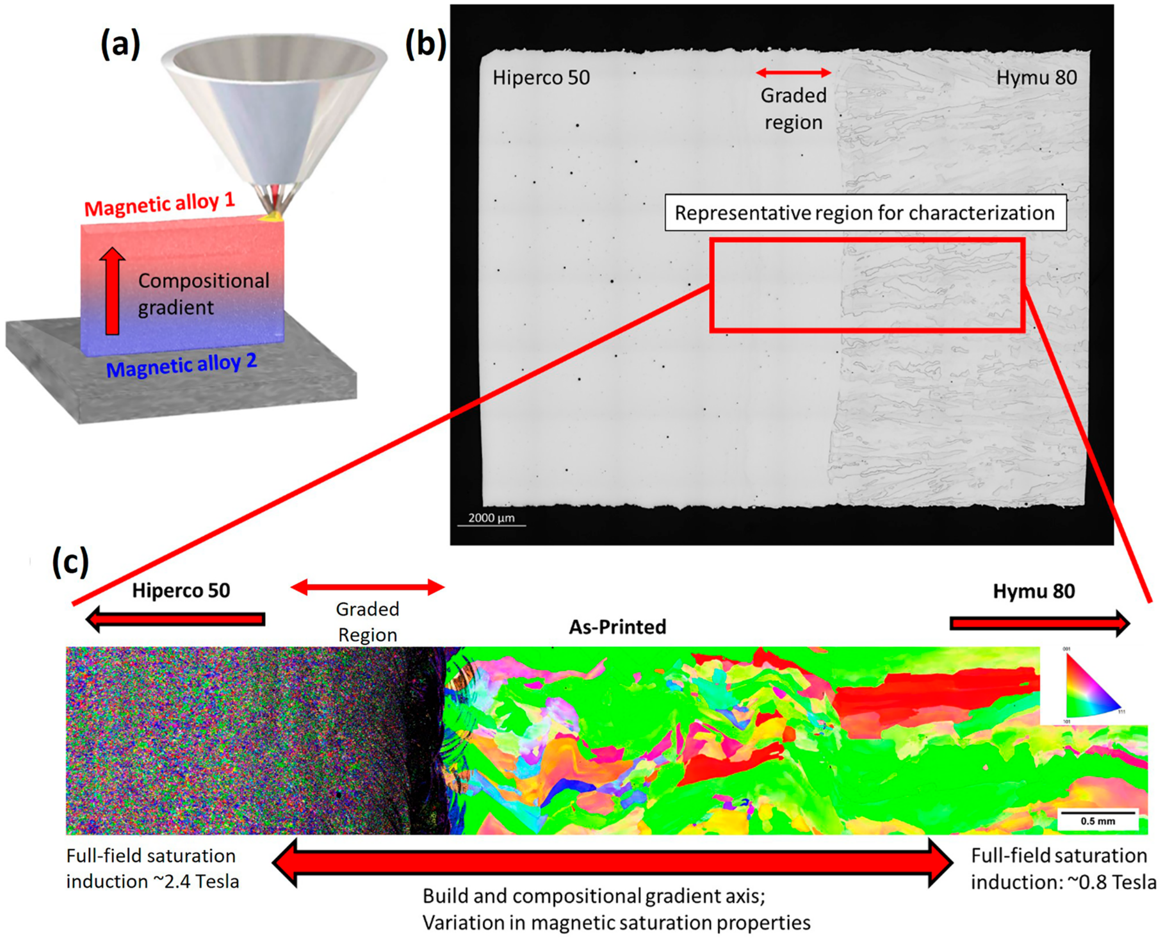

Figure 1. (a) Schematic illustrating the general approach to compositional grading through laser beam directed energy deposition. (b) Representative image of specimen with a gradation between Hiperco 50 (high-saturation induction) and Hymu 80 (low-saturation induction) soft magnetic alloys. (c) Representative EBSD IPF-Z map across gradation of specimen illustrating significant changes in microstructure and full-field saturation induction magnetic properties.

be created with compositional variation, such as graded cobalt content as shown in Figure 2(c), or graded grain structure variations tailored via AM process parameters. Figure 2(f–h) displays example experimental magnetic property data for the printed Hiperco-Hymu alloy shown in Figure 1, demonstrating the ability to tailor the magnetic properties on both a global and local scale through compositionally graded microstructures.

Figure 2 shows that the magnetic properties of materials can be tailored through AM, with designs tailored for a TEM objective lens. Figure 2(f–h) show experimental properties of the successfully printed graded magnetic alloys from Figure 1, showing variation in the magnetic properties depending on the composition of the alloy, and ability to tailor B-H properties for desired functionality. The 24 and 50 mm graded Hymu/Hiperco rings were wound 60 and 64 times respectively with 30 AWG Jonard Tools (Elmsford, NY) insulated wire. The rings were then connected to an Iwatsu (Tokyo, Japan) SY-8218 B-H Analyzer for alternating current (AC) hysteresis characterization. Hysteresis loops were collected at sinusoidal frequencies of 0.01–1 kHz over a magnetic field range of −700–700 A/m. The B-H Analyzer recorded relative permeability (μ_r), max magnetic flux density (B_m), and core loss (P_c) at these frequency ranges, which were used to generate data plots for comparison of the rings.

To verify if the graded magnetic materials successfully printed would be applicable in charged particle optics applications, such as in TEMs, a computational CPOs methodology is adopted to approximate gradation in material magnetic properties in a TEM objective lens geometry. Magnets are used to collimate electron beams [25] therefore, use of graded magnetic materials may enhance the performance and collimation abilities and allow for decreased physical size. Magnetic lenses in a TEM use the magnetic fields generated by coils of wire to shape the electron beam trajectory. In a conventional TEM, a soft iron yolk controls the stray magnetic fields, while the Co-Fe alloy Permendur is commonly used as the pole piece for its high magnetic saturation. Pure Fe,

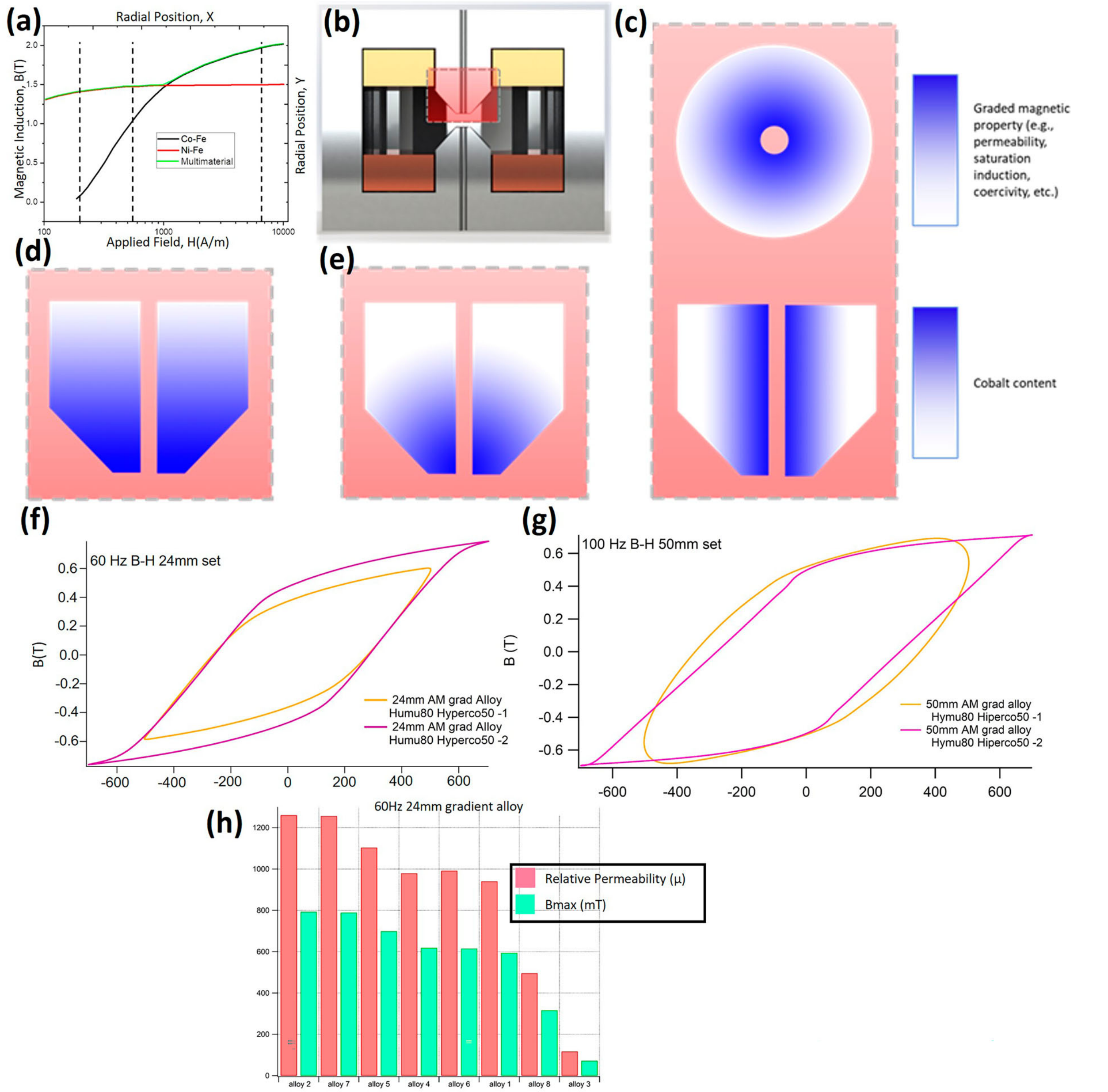

Figure 2. (a) Comparison of magnetization curves for monolithic and multi-material pole pieces, wherein multi-material constructions enable retention of higher field-dependent magnetic induction compared to monolithic versions, (b) notional example of pole piece construction, (c)–(e) represent example pole piece gradations that lead to spatially-dependent magnetic properties and composition. Experimental data of (f–g) B-H curves for two graded Hiperco-Hymu alloys measured at 60 Hz (f) and 100 Hz (g) showing the ability to tailor the magnetic properties with additive manufacturing processes, and (h) magnetic permeability (red) and maximum magnetic field (green) for 8 different composition alloys.

while cheaper, does reduce the performance of the lens due to its reduced maximum flux density at the same field strength.

Modelling of CPOs with the accuracy required to precisely control particle trajectories is a technically involved field of study with complexity emerging from the large span of length scales inherent to large field distributions and small particle position and trajectory distributions, as well as the complexity of the equations for the Lorentz force on a charged particle. Furthermore, CPOs with fields that require expansions fifth order and higher – such as hexapole and higher-order multipole lenses – require special care when modelling the fields to ensure the higher-order components of the field expansion are correctly output by the modelling software, which will be based on finite elements (FE) in most cases. Nevertheless, modelling CPOs with a first-order (paraxial) ray approximation is usually the first step in designing an

CP accelerator or microscope since image plane coordinates can be achieved with enough accuracy to get a rough idea of the placement of lenses. To the best of our knowledge, FE packages which simulate Maxwell's equations do not come with built-in methods to grade the magnetic properties of materials. We show here a general method to achieve graded magnetic properties using the common FE package COMSOL (Stockholm, Sweden) without creating additional plugins for extant FE packages. We use the specific example of graded saturation flux density, but the general approach may be extendable to other magnetic properties such as hysteresis.

To probe the effects of magnetic properties changes on charged particle trajectories, COMSOL Multiphysics was employed. COMSOL's magnetic fields (MF) physics module handles magnetically homogeneous materials – materials whose flux versus field does not vary within its volume – but there is currently no built-in method for handling magnetically heterogeneous materials to the best of our knowledge. Thus, modelling such materials, including graded magnetic materials, requires a workaround.

The flux magnitude $||B||$ is computed for every point in the material domain using the system of equations 1–3. Equation 1 relates the field H to the solenoid current J_e. Equations 2a and 2b provide the field-flux relationship for homogeneous and heterogeneous (graded) materials, respectively. For soft-iron and other homogeneous magnetic materials, the function f_h is the typical $B - H$ constitutive relationship, which has a rapid rise to saturation at low applied external fields, as shown in Figure 3.b.1. The main idea for incorporating heterogeneous materials is that the function f_g not only varies with the field magnitude at each point, but with the spatial coordinates of the point (x, y, z), hence the relationship $f_g = f_g(H, x, y, z)$.

$$\nabla \times H = J_e \tag{1}$$

$$B_{\substack{homo-\\geneous}} = \frac{f_h(H)H}{H} \tag{2a}$$

$$B_{graded} = \frac{f_g(H, x, y, x)H}{H} \tag{2b}$$

$$\nabla \cdot B = 0 \tag{3}$$

To show how this is implemented in a common FE package, we produced the flux $||B||$ and flux-field ratio $B/||H||$ distributions using the FE package COMSOL (Stockholm, Sweden) which are shown in Figure 3 (3.a.2, a.3, b.2, and b.3). The field $||H||$ produced by the solenoids is used along with the constitutive relationship of Figure 3.b.1 to determine the flux magnitude $||B||$. This methodology was produced using the following procedure:

(1) calculate H and $||H||$ from the solenoids; denote the latter as H_1,
(2) calculate $H_2 = \frac{H_1}{r}$ with $r = \sqrt{x^2 + y^2}$ where the origin is the center between the pole piece faces and the axis of symmetry is the $z-$coordinate, and,
(3) determine $||B||$ from the linear curve shown in Figure 3.a.1 where the x-axis comes from H_2.

Figure 3 illustrates how the magnetic flux magnitude, B, responds to the magnetic field magnitude, H, for both the homogeneous (subfigure 3.b) and graded (subfigure 3.a) approaches. Figure 3.c illustrates the theoretical principle. Curves 1–3 represent typical magnetic material $B - H$ curves with varying saturation flux density magnitudes. These are represented by f_h in Equation 2a. The curve labelled 'Saturation-Intersection Line' (SIL) is the $B - H$ relationship used in the graded-material simulations input by the user when the FE package asks for the constitutive relationship. Due to the sharp rise and sudden plateau of typical magnetic-material $B - H$ curves, the SIL intersects their saturation flux density.

In contrast, Figure 3.a illustrates the behavior of the modelled graded pole piece. Unlike the homogeneous material case of Figure 3.b.1, – where the curve can directly be represented by f_h – the heterogeneous curve of Figure 3.c, cannot be directly represented by f_g. This is because f_g is a function of the spatial coordinates in addition to the field. This is illustrated in Figure 3.c by the fact that the B/H ratio is no longer constant throughout the volume.

For a graded magnetic material, assuming that each point in the volume of the lens has similarly-shaped $B - H$ curve but with different saturation flux densities (different plateaus), going from step 2 to step 3 in the above methodology is like moving diagonally along the different plateaus of the curves as a function of the spatial coordinate. The spatial function chosen for illustration here is a radial decay away from the axis of symmetry, hence the inverse-radius relationship in step 2, but any spatial function is possible. This is why the flux in the lens can take on distributions that cannot be achieved in passive homogeneous lenses, as can be seen by comparing Figure 3a.2 and b.2. Furthermore, because of the approximately logarithmic nature of the constitutive relationship in soft-iron materials, the ratio $B/||H||$ is approximately constant throughout the volume of homogeneous lens, as is seen in Figure 3.b.3, while the effect of grading can be clearly seen in Figure 3.a.3.

Figure 3.b illustrates how the flux from a homogeneous magnetic lens typically behaves when that lens is a double pole piece of mirrored frustum geometry, common in the objective lens of a TEM. For a homogeneous soft-iron material, instead of a linear relationship,

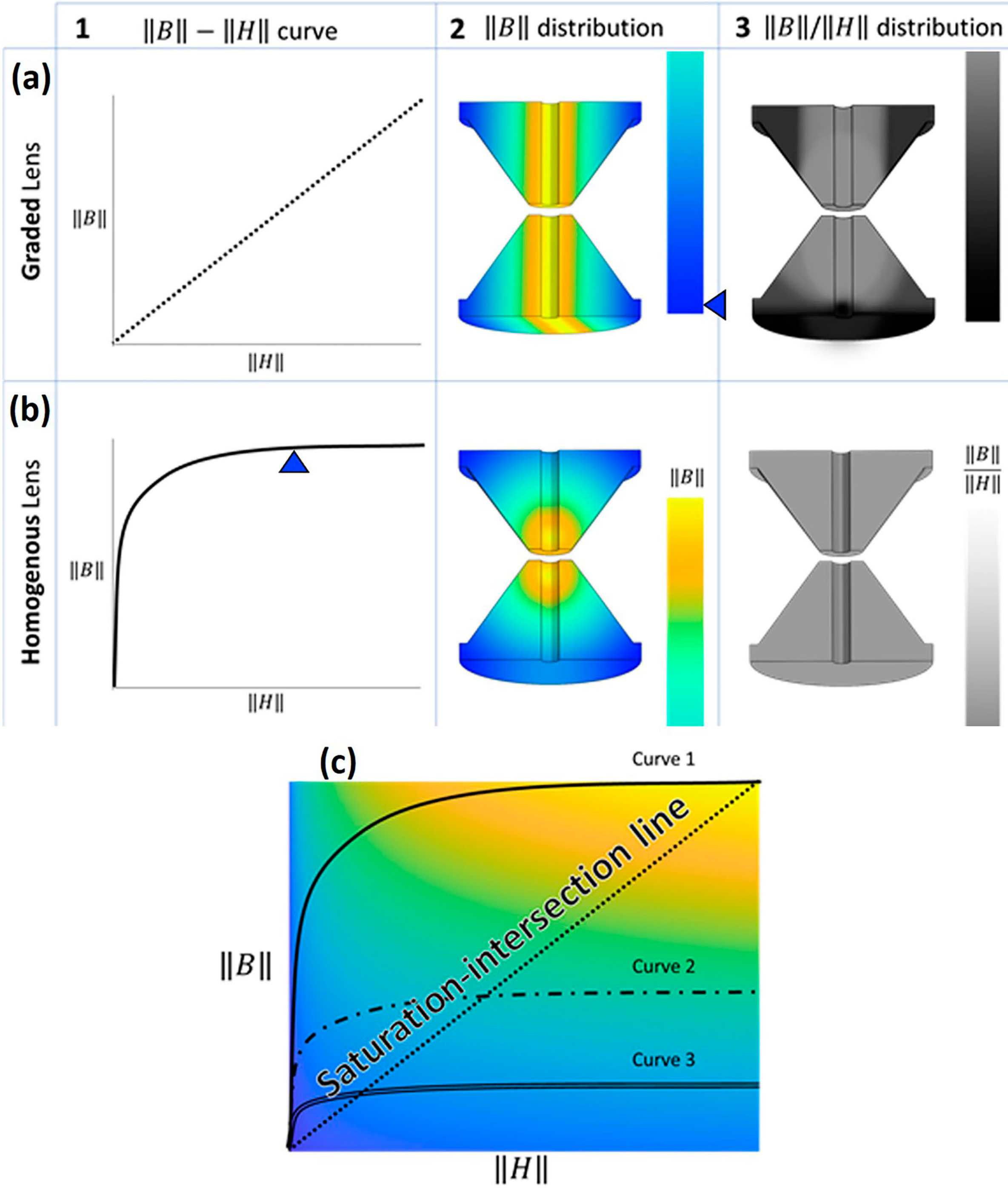

Figure 3. Modelling of magnetic lenses with graded magnetic properties, in this case the saturation flux density is graded throughout the lens volume. (a) An example radial gradation of the magnetic flux in a heterogeneous (graded) lens. (b) A conventional homogeneous lens. (c) Illustration of the path through different B-H curves that occurs when the lens is graded spatially.

it would be the common relationship shown in Figure 3.b.1. The two solenoids in this case are close to the lenses and mirrored as the lenses are: about the plane between the faces of the pole pieces. Satisfying Equations 1, 2a, and 3 results in high flux near the faces of the pole pieces as shown in the flux heat map of Figure 3.b.2. The ratio B/H is effectively constant throughout the volume, as shown in Figure 3.b.3. This is because the entire volume is saturated and the slope of the $B-H$ curve at saturation is constant.

Once the soft magnetic material is saturated, the magnetic induction will only increase linearly in direct proportion with the magnitude of the applied flux, causing the excess magnetic flux to leak out of the core, creating spatial variation in the magnetic field and increasing the variation across the optical axis. Therefore, an ideal material will saturate at a higher value, or have a composition that limits the spread of the stray magnetic flux when saturation is achieved. Such a property of a pole piece could be achieved through having a graded composition pole piece, enabled through a functionally graded magnetic material.

In the common case, Figure 3.b.2, the magnetic flux is strongest right at the pole piece gap and the *B/H* is constant everywhere. Meanwhile a graded lens in Figure 3.a.2, graded in the style shown, has a non-constant *B/H* radially, causing a strong magnetic flux throughout the height of the lens, altering the trajectory of the electron though the entire pole piece. B/H can now be a controlled variable, classically assumed to be constant with uncontrolled manufacturing variations, throughout the volume of the pole piece. This advancement permits charged particle physicists to tailor the charge particle trajectory and the resulting CPOs.

In this work, functionally graded soft–hard magnetic materials are printed. Compositional optimization can be performed to optimize a material global performance by tailoring the local composition and properties. In application to the objective lens of a TEM, conventional stacked iron cores include tooling and machining costs, associated waste material costs, and may have variations between the as-fabricated monolithic plates. However, with functionally graded magnetic materials, there is the opportunity for design of optimized geometries with limited need for post-processing machining, therefore limited material waste, and process control over the microstructure and desired properties.

Additionally, soft magnetic materials commonly used in the pole piece include Co-rich materials, which have been identified as a conflict mineral. By functionally grading the magnetic material, the amount of rare earth metals and conflict minerals can be diminished. Cobalt is a critical mineral for many industries, let alone the magnetics industry, and significant amounts are extracted in the Democratic Republic of the Congo, a concern for supply chain and human rights issues [26]. The sustainability of magnet materials has also been questioned regarding rare-earth metals in permanent magnets [27]. The drive to replace rare-earth and conflict metals in magnets has spurred interest in alternative compositions. This letter hopes to show that compositionally grading materials can decrease dependence on and use of any one element, demonstrating the ability to design novel, more sustainable materials to achieve the same ends; in this example, offering the same or improved magnetic capabilities so as to optimize local properties for enhanced global performance [28]. In addition to providing the opportunity to alter the electron trajectories in a TEM, 3D printed magnetic lenses may find applications in other industries that need complex geometries, such as prisms. 3D printing magnetic lenses may be able to control and alter saturation in locally specific regions of the lens, something not easily possible with conventional magnetic materials.

As with any AM process, challenges in repeatability and porosity will decrease permeability, saturation, and overall magnetic performance. The work presented here represents a preliminary microstructure, as evident by the slight porosity in the as-built materials in Figure 1, and explored a limited scope of compositional gradation, both in elemental composition and physical dimensions. Future work will explore larger physical gradients to extend the variation in magnetic properties and reduce the abruptness of the microstructural transition. Also, this printed part in this study was not an optimized geometry for CPOs insertion opportunities, as it was a 12.7 mm diameter rod and not a larger, more complex shape necessary for CPOs applications. Future work will explore AM processing of larger, application-relevant geometries for CPOs and beyond. However, magnetic-based topology optimization can inform unique designs that optimize electromagnetic properties and performance on the global scale. Additive manufacturing has only increased the opportunities for locally tailored material design for global property optimization. In this letter, a graded Hiperco-Hymu structure is printed via DED with spatially tailored magnetic properties, and graded designs are proposed for applied charged particle optics in the objective lens of a TEM. However, the design, perspectives, and innovations described here can be applied to numerous other applications, such as energy conversion and electric motors industries.

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Disclosure statement

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