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**EFFECT OF FLAKE THICKNESS ON COERCIVITY OF
NANOCRYSTALLINE SmCo_5 BULK PREPARED FROM
ANISOTROPIC NANOFLAKE POWDER (POSTPRINT)**

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Effect of flake thickness on coercivity of nanocrystalline SmCo₅ bulk prepared from anisotropic nanoflake powder

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In this study, nanocrystalline SmCo₅ bulk magnets were prepared by hot-pressing of nanoflake powders fabricated via surfactant-assisted high energy ball milling. Effect of the flake thickness on magnetic coercivity of the SmCo₅ bulk was investigated. Anisotropic SmCo₅ nanoflakes with thickness between 100 and 1000 nm were prepared by varying the milling parameter of ball-to-powder weight ratio. XRD analysis revealed that as-milled flake powders possessed nanocrystalline grains with no observable oxide peaks. The coercivity of the flake powders varied between 19.9 and 21.3 kOe for 1000 nm to 100 nm thick flakes, which indicated that the flake thickness in this range had no obvious effect on the coercivity of the powders. However, the coercivity of the bulks showed a strong dependence on the flake thickness. The bulk coercivity value of 10.97 kOe corresponding to the flake thickness of 100 nm, was 80% higher compared to the bulk prepared with the flakes of 1000 nm. XRD results on compacted samples did not show any grain growth, however, Sm₂O₃ and free Co were detected in SmCo₅ bulks and their content increased with reduced flake thickness. Interestingly enough the bulk coercivity was not deteriorated with the presence of Sm oxide and Co. © 2016 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License. [<http://dx.doi.org/10.1063/1.4943015>]

I. INTRODUCTION

Nanocrystalline Sm-Co alloys possess large coercivity due to the pinning of domain walls by the nanograin boundaries.^{1,2} Intensive milling and subsequent annealing have been used to produce nanocrystalline Sm-Co magnets.³⁻⁵ However, the magnets are usually obtained in the form of randomly oriented powders with relatively low remanence due to magnetic isotropy. To achieve magnetic anisotropy in nanocrystalline magnets, research had focused on hot-deformation consolidation methods.^{6,7} In such approach however, the coercivity is destroyed due to grain growth during high temperature processing. In order to develop high performance permanent magnet both high magnetization and high coercivity are required. Recently, crystallographically textured SmCo₅ and SmCo₇ nanoflakes were prepared by surfactant-assisted high energy ball milling (HEBM).⁸⁻¹⁰ The SmCo₅ nanoflakes have attractive magnetic properties; coercivity of up to 21 kOe and maximum energy product of up to 22 MGOe.⁹ Thus, the nanoflake powders possess a potential to fabricate anisotropic nanocrystalline SmCo₅ bulk magnets with higher magnetization and higher coercivity. Anisotropic nanocrystalline SmCo₇ bulk magnets created from nanoflake powders via spark plasma sintering technique were investigated by several researchers.^{10,11} However, it is less reported on correlation between nanoflake morphology and final properties of the SmCo₅ bulk magnets. In this study, we prepared SmCo₅ nanoflakes by surfactant-assisted high energy ball milling and fabricated SmCo₅ bulks from these powders by induction hot-pressing. The effect of the flake thickness on coercivity of the powder itself and bulks was investigated.



II. EXPERIMENTS

As-cast SmCo_5 powder with typical particle size of less than $250 \mu\text{m}$ was used as the starting material for the high energy ball milling. The alloy was prepared by arc melting in argon using pure metals with a nominal composition of $\text{Sm}_{17}\text{Co}_{83}$ in order to compensate for Sm loss during processing. The ingot was then crushed and grinded down to the designated particle size. The starting powder was milled in a stainless steel vial on a SPEX 8000 mill. Milling balls with diameters of 2.4 and 4.8 mm were used and the ball-to-powder weight ratio (BPR) was varied as 0.8, 2, 4, and 10. Heptane (99.9%) and oleic acid (90%) were used as the solvent and surfactant in amounts of 55 and 30 wt. % of the starting powder respectively. After milling for 1 hour, the resultant SmCo_5 slurry was washed by acetone and then dried in vacuum. The dried powder was loaded into a die and subsequently heated to a temperature of 600°C in an RF inductive furnace under high vacuum and compacted under a pressure of 420 MPa. The obtained cylindrical shaped SmCo_5 bulk magnets were 6.3 mm in diameter and 7.5 mm in length. All powder handling was done in an argon box to prevent oxidation.

Structural and morphology characterizations of the flake powders and bulk samples were carried out by x-ray diffraction (XRD) and scanning electron microscopy (SEM). Magnetic properties of the samples were measured using a closed-circuit hysteresisgraph (model HG-700, KJS Associates Inc.) after being magnetized in a magnetic field of 100 kOe. Powder samples for magnetic characterization were prepared by mixing powders with epoxy and performing alignment in a field of 100 kOe.

III. RESULTS AND DISCUSSION

Figure 1 shows the morphology of the powders milled for 1 h under different ball-to-powder weight ratio. All conditions produced clusters of flake shaped powders with different thicknesses. It was found that the as-milled flakes had a narrow size distribution, which is more likely to be contributed by the smaller size of the milling balls. A similar result was reported in SmCo_5 nanoflake preparation by other researchers.¹² Figure 2 displays average flake thickness as a function of BPR. The flake thicknesses reported in this paper are average values estimated by measurement under SEM. The flake thicknesses reduced from 1000 to 100 nm as the BPR increased from 0.8 to 10. However, the effect of BPR on flake widths was not significant; the widths of the flakes were around 1 - 8 μm and 0.5 - 4 μm for the flake thickness of 1000 nm and 100 nm, respectively. XRD patterns of the randomly oriented flake powders are shown in Figure 3. The XRD analysis

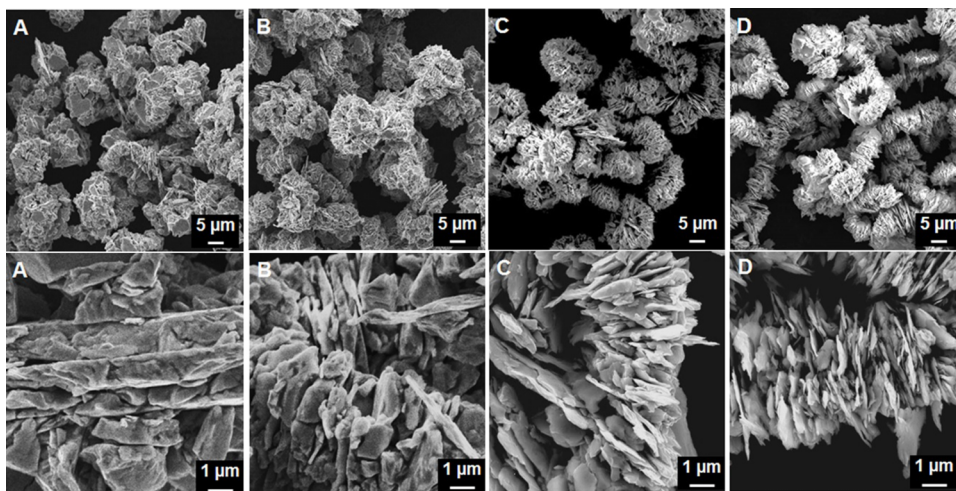


FIG. 1. SEM images of SmCo_5 flake powders milled for 1 h with ball-to-powder weight ratio of A) 0.8; B) 2; C) 4; D) 10 in low and high magnification.

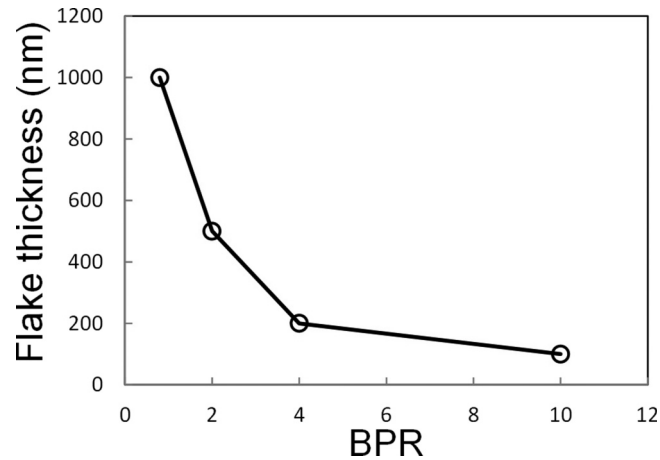


FIG. 2. Average flake thickness versus ball-to-powder weight ratio (BPR).

revealed that the milled powders exhibited only the CaCu_5 type hexagonal structure and there was no indication of oxide peaks. The broadening of the SmCo_5 peaks with increasing BPR indicated that the crystallite size became smaller. The average crystallite size of the flake powders obtained by full pattern Rietveld refinement was 17 nm and 8.1 nm for the flakes with thicknesses of 1000 nm and 100 nm, respectively. This result shows that the flakes are nanocrystalline.

Figure 4 shows demagnetization curves of the aligned flake powders measured along the parallel and perpendicular directions. The demagnetization curves have good squareness and display high coercivity. The coercivity values of flake powders are 19.91, 20.55, 21.34 and 21.14 kOe for the flake thicknesses of 1000, 500, 200 and 100 nm, respectively. Although the flake thickness reduced by 10 times, the coercivity increased only slightly. For the microngrain SmCo_5 particles, the powder coercivity was particle size dependent, i.e., increased with milling time, in some cases reaching to a maximum, and then decreasing after prolonged milling.¹³ The current SmCo_5 flakes, however, consist of nanograins with small-angle and large-angle grain boundaries, which were formed from single crystals due to localized deformation and dislocation during HEBM.¹⁴ The coercivity of such poly-nanocrystalline flake powders should be related to the special crystal structure, which could be contributed by both flake thickness and nanograin size. In our study the flakes possess higher coercivities of $\sim 20 - 21$ kOe, and there was no large coercivity change as the flake thickness varied from 1000 nm to 100 nm. The higher coercivity of the flake powders might be attributed to the nanocrystalline structure and the flake coercivity should be mainly controlled by the nanograin size.

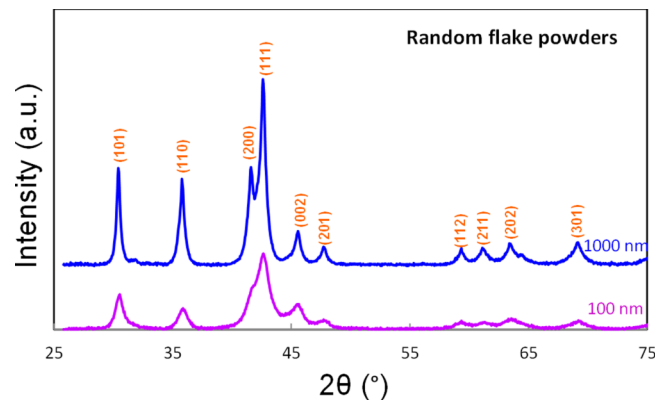


FIG. 3. XRD patterns of random flake powders with thickness of 1000 and 100 nm.

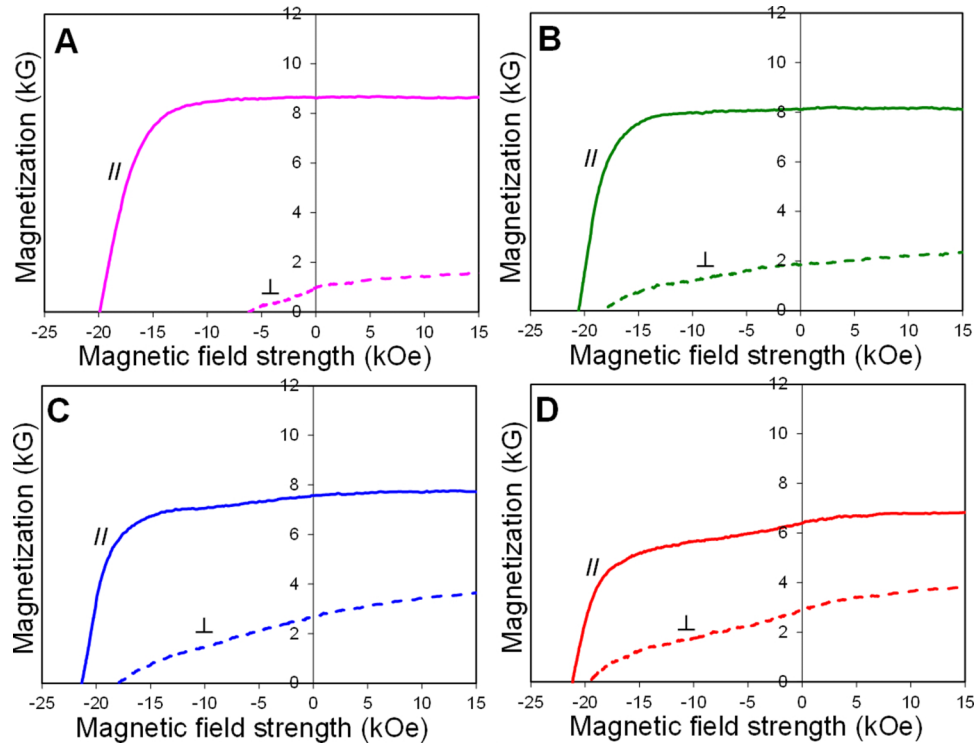


FIG. 4. Demagnetization curves of aligned flake powder samples measured along parallel (\parallel) and perpendicular (\perp) to the aligning magnetic field direction with flake thickness of A) 1000 nm; B) 500 nm; C) 200 nm; D) 100 nm.

Anisotropy of the magnetic behavior was found in all of the SmCo_5 flake powders shown in Figure 4. The degree of anisotropy DOA (which is defined $\text{DOA} = B_r(\parallel) - B_r(\perp) / B_r(\parallel)$, where $B_r(\parallel)$ and $B_r(\perp)$ is the remanence of the sample parallel and perpendicular to the magnetic aligning direction, respectively) is used to describe anisotropic behavior of the flake powders. DOA values of 0.89, 0.77, 0.65, and 0.55 correspond to the flake thickness of 1000, 500, 200 and 100 nm. The degree of anisotropy shrank with the reduced flake thickness. Same results were obtained in our previous study.¹⁵ This is probably related to two aspects. As the flake thickness is reduced, more large-angle nanograin boundaries in the flakes will form,¹⁴ which could weaken the strength of [001] out-of-plane texture of SmCo_5 flake and further influence on the anisotropy behavior of the flakes. On the other hand, as the flake size gets smaller the inter-particle friction increases, and the increased friction limits the particle rotation-orientation during alignment process.

In order to study the effect of flake thickness on the bulk coercivity, SmCo_5 bulks were prepared with the different thickness flake powders at a temperature of 600 °C and a pressure of 420 MPa. Figure 5 shows the demagnetization curves of the bulk samples prepared with the different thickness flake powders. The coercivity estimated from the magnetization curves as a function of flake thickness is shown in the inset of Figure 5. The results indicated that the bulk coercivity increased as the flake thickness was reduced. With decreasing flake thickness from 1000 nm to 100 nm, the bulk coercivity increased from 6.1 kOe to 10.97 kOe, an increase of 80%. Thus, the flake thickness does have a large influence on the bulk coercivity in comparison to the small effect of flake thickness on powder coercivity. The remanence difference for bulk samples should be related to the bulk density. The bulk densities were 6.79, 7.3, 7.4, and 8.05 g/cm³, corresponding to the flake thickness of 1000, 500, 200, and 100 nm, respectively. The density of bulk increased with decreasing flake thickness, that is, finer flakes could produce denser bulks. The bulk prepared with flakes of 100 nm thickness approached 95% of the theoretical density. The bulk density as it has an influence on remanent magnetization might have an effect on coercivity as well.¹¹ Finely distributed pores in bulks containing smaller flakes may be more effective in pinning domain walls.¹⁶

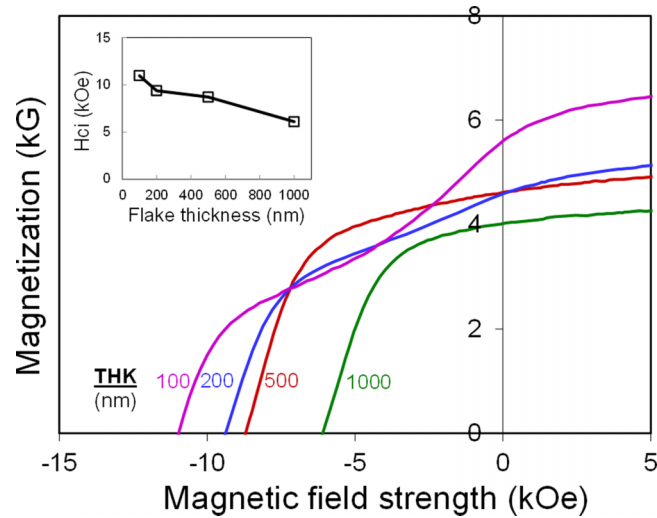


FIG. 5. Demagnetization curves of SmCo_5 bulk samples pressed at 600°C and 420 MPa with different thickness flake powders; the bulk coercivity as a function of flake thickness (inset).

It is noted that the shape of the demagnetization curve gets poorer as the flake thickness is reduced to 200 nm . For the bulk sample prepared from 100 nm flakes, a two phase magnetic characteristic with a significant shoulder in the demagnetization curve is observed (Figure 5). XRD patterns of the bulk SmCo_5 samples are shown in Figure 6. The hexagonal SmCo_5 crystal structure is maintained in all of the samples while Sm_2O_3 and free Co were detected for the bulk samples prepared with the flakes of thicknesses less than 500 nm . The peak intensity of the Sm_2O_3 and Co increased with the reduced flake thickness indicating higher volume fractions. Existence of Co beyond a minimum amount caused poor squareness and appearance of a shoulder in the demagnetization curve. The results indicated that oxidation took place during SmCo_5 bulks preparation. According to the XRD analysis of the flake powders (show in Figure 3), no oxide peaks were present, which indicates that the Sm oxidation should largely occur in the hot-pressing process. In the flake powder preparation, although the milled powders were rinsed with acetone before vacuum drying, some surfactant, OA ($\text{C}_{18}\text{H}_{34}\text{O}_2$), could still remain on the flake surface and act as a possible oxygen source. Although the powders were handled under argon protection during the whole process, the dried powders may have picked up oxygen from the experimental environment as well. Liu *et al.* reported that oxygen existed in the as-milled SmCo_5 flake powders even using a non-oxygen containing surfactant TOA ($\text{C}_{24}\text{H}_{51}\text{N}$).¹⁷

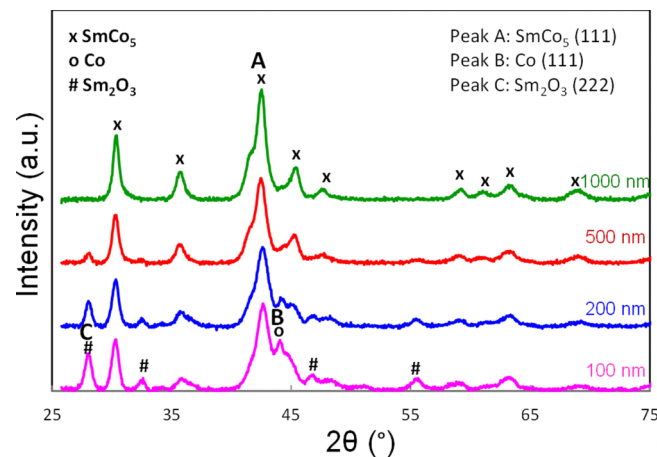


FIG. 6. XRD patterns of SmCo_5 bulk samples pressed with different thickness flake powders.

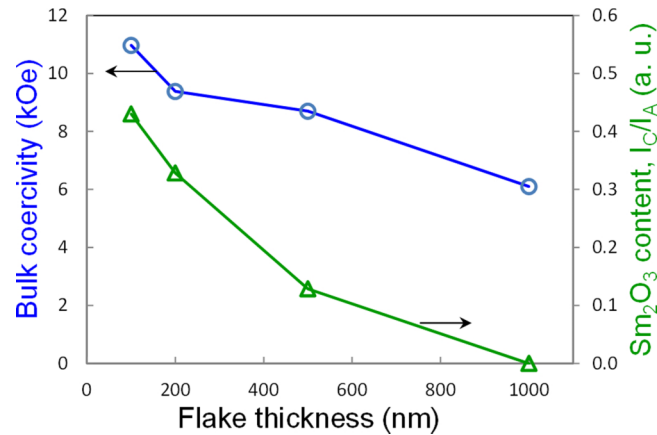


FIG. 7. Coercivity and oxide content of SmCo₅ bulk samples versus flake thickness.

As mentioned above, the relative content of each phase in the SmCo₅ bulks varied with the flake thickness. The content of oxide and Co increased with decreasing flake thickness. Figure 7 shows the flake thickness dependence of the bulk coercivity and the Sm₂O₃ content (expressed in intensity ratio of the Sm₂O₃ (222) to SmCo₅ (111) peaks shown in Figure 6) in the bulk samples. Both bulk coercivity and oxide content increased with decreasing flake thickness. For the finer flake powders, higher specific surface area to volume ratio led to the formation of more oxide after hot-pressing. It is interesting to note that increased oxide content in the bulk did not have an adverse effect on coercivity. Figure 8 shows fracture surface microstructure of the SmCo₅ bulk sample prepared with 100 nm thick flakes. Flake-shaped SmCo₅ phases are clearly visible as elongated grains, and fine particles and strips of most likely Sm₂O₃ and Co on the scale of a few tens of nanometers are distributed along the grain boundaries. Efforts to determine the composition of these nano-phases by EDS analysis were not successful probably because the phases were too small. From the XRD results of the SmCo₅ bulk samples, only Sm₂O₃ and Co phases were present along with the major SmCo₅ phase. Presence of the free Co can only be explained by selective oxidation of Sm on the surface of the flakes, and the oxide and Co are supposed to be located in the boundaries of the flakes. The tiny oxide interphase around the flakes might act as domain wall pinning sites,¹⁶ which is possible to diminish the exchange coupling between the flakes. The suitable amount of oxide existing in the SmCo₅ bulks is useful for developing the coercivity. Thus, the increase in content of oxide with flake thickness reduction did not lead to the decrease in bulk coercivity.

In this study, the bulk samples of SmCo₅ prepared from flake powders with 100 - 1000 nm in thickness had coercivity values between 6 and 11 kOe. As we know, bulk coercivity is sensitive to

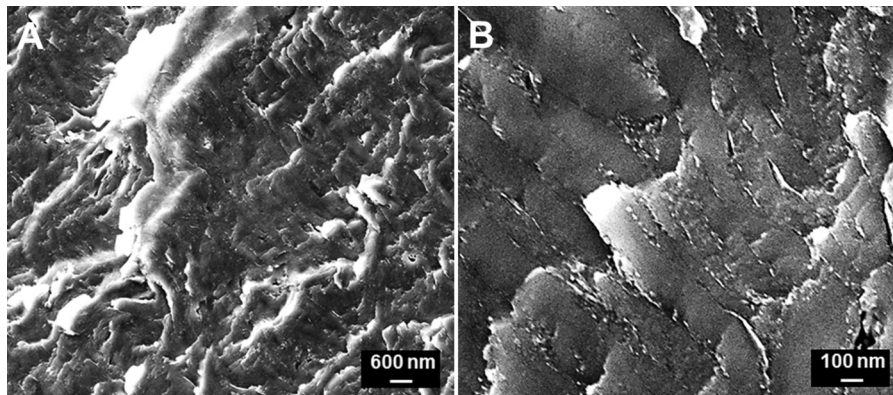


FIG. 8. SEM fracture surface images of SmCo₅ bulk sample prepared from 100 nm thick flakes in low (A) and high (B) magnification.

microstructure such as phases, grain size, grain boundary, defects, and so on. For all bulk samples, the main phase was hexagonal SmCo_5 and the presence of some oxide was not harmful to the bulk coercivity. The grain sizes estimated by Rietveld analysis were 9.9, 9.4, 10.4 and 11.9 nm for the bulk samples prepared with the flakes of 100, 200, 500, and 1000 nm in thickness, respectively. This demonstrated that the bulks still possessed their nanograin character after consolidation. However, the nanocrystalline bulk magnets did not achieve high coercivity values of the corresponding nanoflake powders. In fact, the bulk coercivity was approximately 2-3 times lower than that of the powders. Traditionally such strong coercivity reduction could be attributed to notable grain growth during sintering, however, our analysis does not support this assumption. Instead we believe another process is responsible that takes place at high temperature such as structural stress and defect relaxation.¹⁸ Ball milled powders are rich with various structural defects serving as magnetic domain wall pinning sites, thus enabling higher coercivity. As the heat treatment takes place these defects reduce in concentration allowing easier domain wall movement and therefore lower H_{ci} values. Powders with smaller flake thickness have larger surface area and therefore lead to higher residual concentration of flake boundary defects and Sm oxide in the bulks after pressing. We assume that this behavior is responsible for the observed strong dependence of the bulk coercivity on the initial flake thickness. To better understand the coercivity mechanism of SmCo_5 bulk magnets prepared with nanoflake powders, further investigations such as initial magnetization behavior and TEM analysis are underway.

IV. CONCLUSIONS

In summary, anisotropic SmCo_5 nanoflake powders prepared by HEBM process approached coercivity values as high as 21 kOe. Our results showed that coercivity of the nanoflake powders changed very little with the flake thicknesses ranging from 1000 nm to 100 nm. The coercivity of the SmCo_5 bulks prepared from the flake powders had a strong relationship with the flake thickness. The bulk coercivity significantly increased as the flake thickness decreased. The coercivity value of the bulk prepared with the flakes of 100 nm ($H_{ci} \sim 11$ kOe) was 80% higher compared to the bulk prepared with the flakes of 1000 nm ($H_{ci} \sim 6$ kOe). The bulk coercivity changed a little with the presence of Sm oxide, indicating that a suitable amount of tiny oxide distributed in the fine flake boundaries could serve as domain wall pinning sites. However, the presence of free Co beyond a minimum amount led to poor demagnetization curves of the bulks. To prepare bulk magnets with higher magnetic properties, finer flake powders are required and a non-oxygen surfactant is suggested to be utilized.

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