Single Domain SmCo₅@Co Exchange-coupled Magnets Prepared from Core/shell Sm[Co(CN)₆]·4H₂O@GO Particles: A Novel Chemical Approach

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SmCo₅ based magnets with smaller size and larger maximum energy product have been long desired in various fields such as renewable energy technology, electronic industry and aerospace science. However, conventional relatively rough synthetic strategies will lead to either diminished magnetic properties or irregular morphology, which hindered their wide applications. In this article, we present a facile chemical approach to prepare 200 nm single domain SmCo₅@Co core/shell magnets with coercivity of 20.7 kOe and saturation magnetization of 82 emu/g. We found that the incorporation of GO sheets is responsible for the generation of the unique structure. The single domain SmCo₅ core contributes to the large coercivity of the magnets and the exchange-coupled Co shell enhances the magnetization. This method can be further utilized in the synthesis other Sm-Co based exchange-coupled magnets.

SmCo₅ permanent magnets, with extraordinary magnetocrystalline anisotropy energy about 10⁸ erg/cc as well as high curie temperature over 700 °C, have been laying an irreplaceable role in aerospace, motors, electronics and automotive industries. Especially in recent years, the booming development of modern electronics and industries put forward stringent requirement for SmCo₅ magnets that they should simultaneously hold smaller size and larger maximum energy product $$(BH)_{\text{max}}$$ ¹⁻⁷. Fortunately, a size-dependent behavior of ferromagnetism makes it possible. As is well known, the coercivity of a single crystalline permanent magnet increases with the reduction of grain size from multi-domain range to critical single domain size, and then decreases for smaller grain⁶⁻⁹. Therefore, single domain SmCo₅ magnets are believed to have a good chance possessing superior ferromagnetic performance than commercial bulk SmCo₅ magnets¹⁰⁻¹⁵. Zhang and co-workers synthesized 7 nm single domain Sm-Co NPs with controlled size and composition via reductive annealing of CaO-coated Sm-Co oxide NPs¹⁴. However, those NPs were so minute that they not only held diminished ferromagnetic properties (coercivity = 7.2 kOe) but also endured easily oxidation. On the other hand, Hadjipanayis and co-workers prepared single domain Sm-Co₅ particles with average size from 176 nm to 376 nm by mechanochemical method¹⁶. Though the particles have wide size distribution, the coercivity of the magnets can reached up to 23.7 kOe with increase of the average size to 300 nm, which is comparable to commercial SmCo₅ magnets.

Another route to produce minimized magnets with enlarged $$(BH)_{\text{max}}$$ lies in the exchange coupling of magnetically hard and soft phases in magnets. It is accepted that rare earth based exchange-coupled magnets might be next generation of permanent magnets which hold larger maximum energy product with less use of rare-earth elements¹⁷⁻²⁵. Liu and co-workers synthesized nanostructured Sm-Co/Fe bulk magnets by warm compaction method and found that the $$(BH)_{\text{max}}$$ of products increased from 10 to 17 MG-Oe with the contents of Fe increase from 0 to 25%¹⁸. In addition, they fabricated sintered Sm-Co/Fe magnets with $$(BH)_{\text{max}}$$ of 18 MG-Oe²⁷⁻²⁸, and further manifested that the formation of intermediate layer by interphase diffusion of Co and Fe is the origin of...
enhanced energy product\textsuperscript{21,24,28–32}. Similarly, Skomski and co-workers also implied the importance of interphase layers to the single-phase like behavior of the SmCo\textsubscript{5} based magnetic composite\textsuperscript{33}.

Despite the significant progress in the two strategies mentioned above, it is still an arduous task to prepare desired SmCo\textsubscript{5} magnets due to the obstacles in controlled synthesis of highly ferromagnetic single domain SmCo\textsubscript{5} magnets as well as hardship in introducing both effectively exchange-coupled soft phases and uniform interphase layers.

Herein, we report a bottom-up method to chemically synthesize exchange-coupled single domain SmCo\textsubscript{5}@Co core/shell magnets with an amorphous interphase layer by reductive annealing of Sm[Co(CN)\textsubscript{6}]\textsubscript{4}·H\textsubscript{2}O@graphene oxide particles. The as-synthesized SmCo\textsubscript{5}@Co magnets have an average size of 200 nm, and exhibit enhanced coercivity of 20.7 kOe and saturation magnetization of 82 emu/g. In our strategy, graphene oxide (GO) sheets played a vitally important role in the fabrication of the mesoscopic high performance magnets. During the preparation of precursor, the wrapping of GO sheets restrained the growth of Sm[Co(CN)\textsubscript{6}]\textsubscript{4}·H\textsubscript{2}O crystals so that the size of resulted Sm[Co(CN)\textsubscript{6}]\textsubscript{4}·H\textsubscript{2}O@GO particles would meet the requirement for production of 200 nm SmCo\textsubscript{5}@Co magnets. Afterwards, during the high temperature reduction reaction, GO sheets helped to sustain the SmCo\textsubscript{5} particles as single crystals, which is a fundamental requirement for the formation of single domain magnets. Besides, by controlling the interphase diffusion during the annealing process, GO sheets also facilitated the generation of Co shell and interphase layer which increased saturation magnetization and promoted exchange coupling effect in the magnets, respectively.

Results

Morphology and structure of single domain SmCo\textsubscript{5}@Co magnets. SmCo\textsubscript{5}@Co magnets were prepared by a reductive annealing process of Sm[Co(CN)\textsubscript{6}]4·H2O@GO as precursor. X-ray diffraction (XRD) analysis was used to characterize the chemical structure of the product (Figure 2a). The XRD pattern matched well with standard SmCo\textsubscript{5}, either in the position or in the intensity of the peaks (JCPDS No. 27-1122), indicating the formation of SmCo\textsubscript{5} phase. A small peak at 44° corresponded to the standard pattern of cubic Co (JCPDS No. 15-0806) indicates the co-existence of cubic Co along with a dominant SmCo\textsubscript{5} phase. Transmission electron microscopy (TEM) was then utilized to look into the morphology of the products. According to the TEM images, instead of generating bulk magnets or agglomerated particles with random size and morphology, the products were consisted of 200 nm isolated particles (Figure 2b, c). High resolution transmission electron microscopy (HRTEM) images further manifested the detailed configuration of the as-synthesized particles. Figure 2d is the edge structure of a typical particle as shown in Figure 2c. From the HRTEM image, it can be seen that the exterior part of the particle had several nanocrystalline domains, and the lattice spacing values of 0.180 nm and 0.201 nm are close to the inter-plane distance of 0.177 nm and 0.205 nm in the [111] planes and [200] planes in cubic Co (JCPDS No. 15-0806), respectively. This observation implies the external part of the particle is composed of polycrystalline Co phase. Then, closed to the exterior Co shell, a coherent amorphous layer with thickness of 2 nm was observed as marked by the dot lines in Figure 2d. Based on previous study, such intermediate layer was formed by interphase diffusion between soft and hard phase during high temperature process, and would enhance the exchange-coupled effect as well as sustain the integrity of hard phase by blocking excessive diffusion\textsuperscript{21,24,28–32}. In the interior part, as shown in Figure 2d, the lattice fringes were all along the same direction, which demonstrates the single crystalline nature of the inside part. The lattice spacing value of 0.210 nm fitted well to the interplane distance of 0.212 nm in [111] planes in hexagonal SmCo\textsubscript{5}. HRTEM image of an arbitrary interior part also exhibited concordant parallel fringes with a lattice spacing value of 0.210 nm, which suggests the core of the particle as single crystalline SmCo\textsubscript{5} (Figure 2e). In addition, selected area electron diffraction (SAED) pattern of the particle with hexagonal symmetric diffraction spot further proves the generation of single crystalline SmCo\textsubscript{5}. Considering the single crystalline nature as well as a diameter smaller than critical size of single domain of SmCo\textsubscript{5} (~0.7 μm), the interior part is determined as single domain SmCo\textsubscript{5}. Magnetic force microscopy (MFM) images also demonstrate the single domain structure in SmCo\textsubscript{5}@Co particles (Figure S3a, b). According to the image, magnetic moments were all along the same direction and no domain wall structure could be found. Consequently, the as-synthesized magnets (200 nm) possess a multi-grain Co shell and single domain SmCo\textsubscript{5} core with an amorphous intermediate layer. Note that the interaction with the lattice of Sm-Co matrix might be responsible for the formation of metastable cubic Co\textsuperscript{26–39}.

To further investigate the structure of products described above, element analysis by a scanning transmission electron microscopy (STEM) scan along with energy dispersive X-ray spectroscopy (EDS) line scan was performed (Figure 3). From the STEM image (Figure 3a), the particles had average size of 200 nm, which are in good agreement with the TEM observation. The EDS line scan was applied on the edge of the particle as indicated by the red dot line in the inset of Figure 3a. Note that the inset is an amplified image of the blue box in Figure 3a and the scale bar represents 5 nm. The profile of the line

![Figure 1](https://www.nature.com/scientificreports/)

**Figure 1.** (a) TEM image of Sm[Co(CN)\textsubscript{6}]\textsubscript{4}·H\textsubscript{2}O@GO particles. (b) TEM image of the interface between Sm[Co(CN)\textsubscript{6}]\textsubscript{4}·H\textsubscript{2}O and GO layers as indicated by the red square in (a).
A scan was displayed in Figure S4 and four points (denoted by b, c, d and e in Figure 3a) were picked to present the distribution of chemical composition in detail. The corresponding EDS patterns of the four points were shown in Figure 3b–e, respectively. In the EDS patterns, only Co was detected in external points (Figure 3b–c) while both Sm and Co were observed in the relatively internal point (Figure 3e), which confirm the formation of SmCo$_5$@Co core/shell magnets. In addition, EDS scan of an area of the sample suggests that the Co content in the SmCo$_5$@Co magnets is 4%. Thus, the overall schematic illustration for the fabrication of SmCo$_5$@Co magnets was displayed in Figure 4.

Magnetic properties of single domain SmCo$_5$@Co magnets. Physical property measurement system (PPMS) was used to characterize the room temperature magnetic properties of SmCo$_5$@Co particles (Figure 5). The magnets exhibited a saturated magnetization of 82 emu/g with a remnant magnetization of 62 emu/g (the inset of Figure 4), which defines $M_r/M_s$ ratio of the as-synthesized magnets.
as 0.75, verifying the effective exchange-coupling between hard and soft phases. Moreover, the magnets have a room temperature coercivity of 20.7 kOe, which is so far one of the largest room temperature coercivity obtained from chemical synthesized SmCo5 based magnets with such size to the best of our knowledge. The enhanced coercivity is originated from the 200 nm single domain SmCo5 core, and the large saturation magnetization is attributed to the exchange-coupled effect between SmCo5 and Co. In addition to that, according to the B-H hysteresis loop, the magnets held a superior (BH)max of 10 MG Oe, which is comparable to bulk SmCo5 magnets prepared from sintered or warm compaction methods.

Discussion

The incorporation of GO sheets has great impact on the generation of SmCo5@Co magnets. To demonstrate the importance of GO sheet, Sm[Co(CN)6]4H2O crystals without GO wrapping were directly utilized as the precursor and underwent the same procedure as the synthesis of SmCo5@Co particles. The XRD pattern of the product fitted well with the standard SmCo5 pattern (JCPDS No. 27-1122) (Figure 6a), suggesting the formation of SmCo5 phase. Additionally, an unknown peak appeared at 43° which might be conducted by defects or lattice mismatch from Sm evaporation. TEM image of the product implies that the as-prepared particles were severely aggregated and had irregular shapes as well as random sizes ranging from 10 nm to 600 nm (Figure S5a). HRTEM images indicate the microstructures of the particles. As shown in Figure 6b, there was neither a core/shell structure nor an amorphous layer existed in the exterior part. Instead, assembly of SmCo5 nanocrystals was found as marked by the dot circles in Figure 6. The lattice spacing value of 0.241 nm and 0.221 nm corresponded to [110] and [200] planes of SmCo5, respectively (ICPDS No. 27-1122). HRTEM image of the interior part showed a similar structure as the outer part. As can be seen in Figure 6c, the inner part was also consisted by nanosized grains and the lattice spacing value of 0.210 nm and 0.247 nm fitted well with inter-plane spacing of [111] and [110] planes of SmCo5, respectively (ICPDS No. 27-1122). SAED of an isolated as-synthesized particle shown in Figure S5b further supports the result from HRTEM images (Figure 6d). A ring pattern in the SAED indicates the constitution of random-orientated nanocrystalline SmCo5 grains within the structure.

Thus, during the high temperature annealing process, GO sheets have at least three indispensable contributions to the generation of highly ferromagnetic single domain SmCo5@Co magnets. Firstly, GO sheets will restrain the growth of particles and confine the size of magnets to 200 nm. In a meantime, they also impede inter-particle fusion, which facilitates the production of isolated particles. Secondly, wrapping of GO sheets will help to sustain magnetic hard phase as single crystalline during collapse of -(CN)- bridge and formation of SmCo5 under high temperature treatment. Finally, GO sheet seems to obstruct interphase diffusion of Co so that Co shell along with an amorphous intermediate layer are deposited and formed on the surface of SmCo5 hard phase.

The Sm[Co(CN)6]4H2O@GO can be also employed to synthesize a series of SmCo5 based magnets. By increasing Co/Sm ratio from 3.5 to 3.7 in the starting material, as can be seen in Figure S7b, the intensity for the reflection of Co phase was increased while a small peak for the [303] plane of Sm2Co17 appeared, which is the peak with maximum intensity in the standard pattern of Sm2Co17 (JCPDS No. 19-0359). Moreover, after increasing Co/Sm ratio to 4 in the starting material, the reflection peaks of Sm2Co17 phase arose in the XRD pattern of the product as indicated by the solid triangles in Figure.
were observed in the generated magnets, respectively. We took SmCo5@40% Sm2Co17 as an example to manifest detailed structures of magnets with larger content of Sm2Co17. Figure S10a is the scanning transmission electron microscopy (STEM) image of the magnet, while the EDS profiles in Figure S10b and S10c correspond to the scan line 1 and 2 in Figure S10a. Based on the profiles, similar with SmCo5@9% Sm2Co17 magnets, the exterior part of the produced magnet had larger Co/Sm ratio while interior part held a smaller Co/Sm ratio, which suggests the presence of SmCo5@Sm2Co17 core/shell structure. HRTEM analysis was performed on the edge of the magnet (Figure S10c). According to the HRTEM image, in the exterior part, the magnet had an apparently thicker shell than that of SmCo5@9% Sm2Co17 magnets, and the lattice spacing values of 0.189 nm and 0.209 nm are close to the inter-plane distance of 0.187 nm and 0.210 nm in the [223] planes and [220] planes in Sm2Co17 (JCPDS No. 19-0359), respectively. In the interior part, the lattice fringe pattern exhibited single crystalline nature, and the lattice spacing value of 0.212 nm fitted well to the inter-plane distance of 0.212 nm in [111] planes in SmCo5 (JCPDS No. 27-1122). Thus, with increase of Sm2Co17 content in the product, the as-synthesized SmCo5@40% Sm2Co17 magnets have thicker Sm2Co17 shell while the interior part still hold single crystalline SmCo5 core.

Demagnetization curves of as-prepared magnets with different composition were shown in Figure 7. The curves show that all the magnets exhibited single-phase demagnetization behavior. Moreover, with the increase of Sm2Co17 amount in the magnets, the coercivity of magnets decreases from 20.7 kOe to 7.1 kOe while the saturation magnetization increased from 82 to 114 emu/g. Table 1 summarized magnetic properties of as-synthesized SmCo5 magnets with various compositions.

Interestingly, Sm2Co17@Co magnets can be also prepared by tuning the Co/Sm molar ratio to 7 in the starting materials. According to the XRD results, the peaks of resulted magnets matched well with the standard patterns of Sm2Co17 (JCPDS No.19-0359) and Co (JCPDS No. 15-0806) (Figure s11a), indicating the co-existence of Sm2Co17 and Co. HRTEM image of the edge of a random particle was shown in Figure S11b. Similar with what was founded in SmCo5@Co, the HRTEM image also manifests that the outer layer was composed of multi-domain outer shell and inner part is formed by single crystalline core. In addition, the lattice spacing value of 0.205 nm in the inner part matched well with inter-plane distance of 0.205 nm in the [200] planes of Sm2Co17. Therefore, Sm2Co17@Co magnets were also synthesized from Sm[Co(CN)6]·4H2O@GO precursor. Moreover, based on PPMS characterization, the as-synthesized Sm2Co17@Co magnets exhibited...
favorable room temperature coercivity of 5.7 kOe and saturation magnetization of 124 emu/g (Figure S11c).

In summary, we developed a facile route to synthesize core/shell structured single domain SmCo5@Co magnets with size of 200 nm via a reductive annealing of Sm[Co(CN)6]·4H2O@GO precursor. The as-synthesized SmCo5@Co particles exhibited enhanced (BH)_max with large coercivity and saturation magnetization. We found that the GO sheets played a vitally important role in the generation of single domain SmCo5 phase as well as the core-shell structure. In addition, this strategy can be extended to the synthesis of the SmCo5 based nanoparticles and nanocomposites. The magnetic behavior and other intermetallic magnets.

| Table 1 | Summary for the magnetic properties of as-synthesized SmCo5 magnets with various compositions |
|---------------------------------------------|
| SmCo5 with | 9% | 17% | 40% | 51% |
| Hc (kOe) | 20.7 | 19.1 | 16.1 | 8.5 | 7.1 |
| Ms (emu/g) | 82 | 85 | 90 | 102 | 114 |
| Mr (emu/g) | 62.46 | 55.83 | 61.06 | 53.51 | 55.37 |
| (BH)_max (MG·Oe) | 10 | 6.5 | 8 | 4.5 | 4.4 |

Methods

In a typical procedure of synthesizing SmCo5@Co magnets, Sm[Co(CN)6]·4H2O GO particles were used as precursor, and cobalt acetylacetonate (Co(acac)) fine powder was added to tune the Co/Sm molar ratio. Moreover, Ca and KCl were applied as reducing agent and high temperature solvent, respectively.

Synthesis of Sm[Co(CN)6]·4H2O crystals. 0.7 g (2 mmol) K2[Co(CN)6] (95%) and 0.89 g (2 mmol) Sn(NO3)3·6H2O (99.95%) were each dissolved in 5 ml DI water. 0.4 ml 6.5% (volume ratio) HNO3 was added to K2[Co(CN)6] solution. The H2O was added to K2[Co(CN)6] solution. Then, the mixture was kept without disturbing for 7 days until the generation of yellow crystals. Finally, the crystals were washed with DI water for 3 times and dried.

Synthesis of SmCo5@Co and Sm2Co17@Co magnets. GO was prepared from graphite powder by a modified Hummers method. 80 mg of the GO was exfoliated in 80 ml DI water by sonication for 1 h to generate GO sheets. The GO sheet was collected and dispersed again in 5 ml DI water. Then, 0.89 g (2 mmol) Sn(NO3)3·6H2O (99.95%) was added into the GO solution and the mixture were shaken for 1 h. 0.7 g (2 mmol) K2[Co(CN)6] (95%) was dissolved in 5 ml DI water, and 0.4 ml 6.5% (volume ratio) HNO3 was added to K2[Co(CN)6] solution. Then, the mixture was kept without disturbing for 7 days until the generation of brown SmCo5@Co nanoparticles. Finally, the resultant was washed with DI water for 3 times and dried.

Characterization. X-ray crystallography measurement of Sm[Co(CN)6]·4H2O were made on a Rigaku RAXIS-IV image plate area detector with graphite monochromated Mo Kα radiation. Transmission electron microscopy (TEM) was taken on an FEI Tecnai T20 microscope. High-resolution TEM (HRTEM) and EDS line scans were carried out on an FEI Tecnai F30 microscope. X-ray diffraction (XRD) patterns were obtained using a Rigaku DMAX-2400 X-ray diffractometer equipped with Cu Kα radiation. The accelerating voltage and current were 40 kV and 100 mA, respectively. The magnetic properties were measured on a Quantum Design PPMS 9 Tesla system. TEM images were taken on a VEECO Nanoscope III, microscopy.

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Author contributions
Y.H. and S.G. proposed the conceptual idea, and participated in the analysis of results, discussing and writing the manuscript, and provided financial support through grant application. Y.C. performed the synthesis and XRD experiments of samples. L.J. provided the synthetic method and X-ray Crystallography results. C.G. and S.W. characterized and analyzed the magnetic properties of samples. D.S. provided the MFM and AFM images of samples.

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