

Effect of Sm:Co ratio in SmCo nanocomposites on the structural, morphological and magnetic properties

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This work reports the synthesis of SmCo based magnetic nanocomposites (MNCs) with varying Sm:Co ratio, prepared using chemical route technique followed by post synthesis heat treatment at 900 °C. The prepared samples were characterized using high resolution field emission scanning electron microscopy (HRFESEM), Energy Dispersive X-ray spectroscopy (EDS), X-ray Diffraction (XRD) and dc-magnetization to analyze the effect of Sm:Co ratio on the morphological, structural and magnetic properties of the MNCs. The HRFESEM images revealed the irregular shape morphology in a size range of 62 – 92 nm. The EDS maps and spectra confirmed the existence of Sm, Co and Oxygen in the samples. The XRD analysis revealed the presence of metallic cobalt, Sm₂O₃ and Sm₂Co₅ with varying phase fractions. The highest magnetization of 125.8 emu/g has been obtained for Sm:Co::1:1 corresponding to a magnetic anisotropy value of 1.44 x 10⁶ erg/cm³. Sample with highest Co (>60%) showed highest saturation magnetization which was reduced with decreasing Co content. Thus, the magnetization behavior is found to be dependent on the nature of phases and their phase fractions.

Keywords: Magnetic Nanocomposites, Sm:Co Ratio, HR-FESEM, dc-Magnetization

INTRODUCTION

Magnetic nanocomposites (MNCs) are emerging as promising artificially engineered materials showing applications in advanced information storage and processing systems [1, 2]. Strong permanent magnetism of these materials is most suitable for applications including ultra-high-density data storage and processing, triboelectrification, gas detection sensors etc [3]. The highly sought after magnetic anisotropy crucial for many advanced technological applications is one of the primary intrinsic properties of these MNCs. Magnetic anisotropy helps to create and stabilize the magnetic domains which are essential for the retrieval and storage of magnetic data. Magnetic anisotropy also regulates the energy losses in numerous other devices like transformers and inductors [4]. MNCs integrating both soft and hard magnetic phases, coupled via exchange interactions, construct an integral part of a wide range of advanced technologies spanning from

electronics to the energy industries [2]. The coexistence of high magnetic anisotropy and large saturation magnetization makes these materials attractive for spintronic devices. The strategic combination of soft and hard magnetic phases is typically aimed at maximizing the intrinsic magnetic attributes of the MNCs, where, soft magnetic phases like FeCo, FeNi contribute to high magnetization, while hard phases such as Sm₂Co₅ provide coercivity and magnetic anisotropy [5]. Amongst many, SmCo-based MNCs are particularly interesting due to their pronounced magnetic anisotropy, which originates from the crystal field effects of Sm³⁺ ions in the hexagonal lattice [6].

The SmCo phase imparts high coercivity and magnetocrystalline anisotropy, while metallic Co enhances saturation magnetization and facilitates rapid magnetization reversal. Additionally, the hard phase stabilizes the soft phase through exchange coupling, preventing demagnetization [7]. Moreover, rare-earth elements, with their unique 4f electron configurations, play a crucial role in creating strong

magnetocrystalline anisotropy in compounds like SmCo₅, Sm₂Co₁₇, and Nd₂Fe₁₄B, making these materials essential for high-performance permanent magnets. The development of hard/soft nanocomposite magnets offers a promising path to reduce rare-earth content while still achieving high magnetic strength. The combination of hard magnetic phases with soft magnetic phases optimizes the performance of resulting MNCs. Recent findings show that varying the hard magnetic phases in the composites leads to the notable changes in their structure and magnetic behavior due to hard-phase engineering effect [8].

Besides, MNCs exhibit superior magnetic properties at nanoscale compared to their bulk counterparts, primarily due to the size as well as shape dependent ferromagnetic behavior. Considering the size reduction at nanoscale, chemical synthesis methods promote the growth of MNCs consisting of multiple phases resulting in single-phase-like exchange-coupled behavior, rather than a simple mixture of distinct soft and hard phases. High-density nanostructures are essential for enhancing the performance of bulk nanocomposite magnets. Prior studies showed that interfacial mixing of Co and Fe improves exchange coupling and energy product in Sm-Co/Fe systems. A graded interface further boosts saturation magnetization and energy product [9]. Moreover, chemical synthesis techniques are effective to produce dimensionally and morphologically controlled nanostructures. In case of multi-component compounds, solution methods give rise to the efficient coalition between the exiting components enhancing the overall properties of the material. Therefore, in this work, we have employed a solution-based approach to synthesize SmCo MNCs. Additional modification is carried by performing the reactions in the presence of a magnetic field in order to channelize the dimensional dependence magnetic properties. The samples were prepared by varying Sm:Co ratio to optimize the phases present in the materials with a post-synthesis annealing at 900 °C in the presence of hydrogen. The samples were investigated for structural, morphological, and magnetic properties through XRD, HR-FESEM, and DC magnetization measurement techniques.

EXPERIMENTAL

All the chemicals used in this study, including Samarium chloride hexahydrate: SmCl₃ · 6H₂O (364.81 g/mol); cobalt chloride hexahydrate: CoCl₂ · 6H₂O (237.93 g/mol), NaOH (40 g/mol), hydrazine hydrate: N₂H₄·H₂O (64%) were purchased from Sigma-Aldrich with high purity > 98% and used without further treatment.

The SmCo based MNCs were synthesized via a chemical route with varying stoichiometric ratio of Sm³⁺ and Co²⁺ from 1:1 to 1:5. The precursor salts were dissolved in 50 mL of deionized water, to prepare a 0.4 M solution, homogeneously using magnetic stirring at 90 °C, followed by the addition of 4 g of NaOH. Then, 4 mL of N₂H₄·H₂O was added dropwise. Hydrazine not only increases the alkalinity of the solution but also reduces the metal salts to their metallic form. A vigorous reaction was followed, leading to the formation of a black precipitate separated magnetically. After washing many times through DI water and ethanol, it was dried

followed by high temperature annealing at 900 °C in the presence of hydrogen at a flow rate of 120 ml/min.

The synthesized samples were characterized using X-ray diffraction (XRD), High resolution field emission scanning electron microscopy (HR-FESEM), High resolution transmission electron microscopy (HR-TEM), energy dispersive X-ray spectroscopy (EDS) and dc-magnetization. The XRD patterns were obtained at PANalytical X'pert pro X-ray diffractometer (λ Cu-K α = 0.154 nm) in the range $20^\circ \leq 2\theta \leq 80^\circ$ (step size = 0.016715). HR-FESEM micrographs and EDS spectra were scanned using JSM-7900 F microscope manufactured by Jeol. The HR-TEM images were obtained using JEM 2100 F(FE-TEM) / 200 kV. The magnetization measurements were carried out at the VSM module of the cryogen free versa lab developed by quantum design.

RESULTS AND DISCUSSION

The HRFESEM images of the SmCo nanocomposites prepared at 90 °C and annealed at 900 °C post-synthesis, are displayed in Fig. 1 (a-e). The effect of annealing is clearly visible on the morphology of the MNCs. However, in the present case when the MNCs were subjected to the post-synthesis annealing at high temperatures, the morphology is modified. The MNCs display little elongation with a narrow range of distribution. The size distribution histograms are displayed in the insets of respective Fig. 1 (a-e). The average size of the MNCs is found to be increased from ~ 62 nm to ~ 92 nm with increasing in Sm:Co ratio from 1:1 to 1:5. The varying size is shown in Fig. 1 (f) indicating that the change in the Sm:Co ratio influences the particle growth.

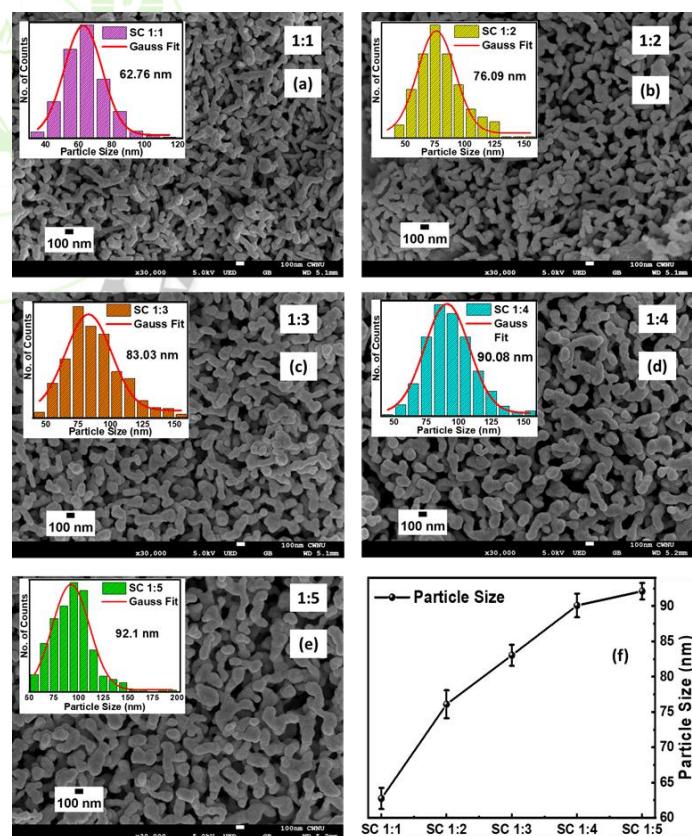


Figure 1. (a-e) HE-FESEM images of SmCo nanocomposites with ratio 1:1, 1:2, 1:3, 1:4 and 1:5, respectively, respective insets show

the particle size distribution histograms; (f) variation of average particles sizes with the Sm:Co ratio.

Fig. 2 (a-c) demonstrates the EDS maps of the samples corresponding to Sm:Co ratio of 1:1, 1:3 and 1:5, respectively. The elemental distribution appears uniform as the ratio remain low, however, the cobalt content becomes more visible as the ratio increases. This additionally suggests the enhanced contribution of cobalt in forming metallic cobalt and SmCo₅. **Fig. 2 (a'-c')** indicates the EDS spectra of the samples confirming the presence of Sm, Co and Oxygen as the major elements. The inset of **Fig. 2 (a'-c')** shows the atomic% of the elements showing the enhancement in the content of Sm with increasing ratio.

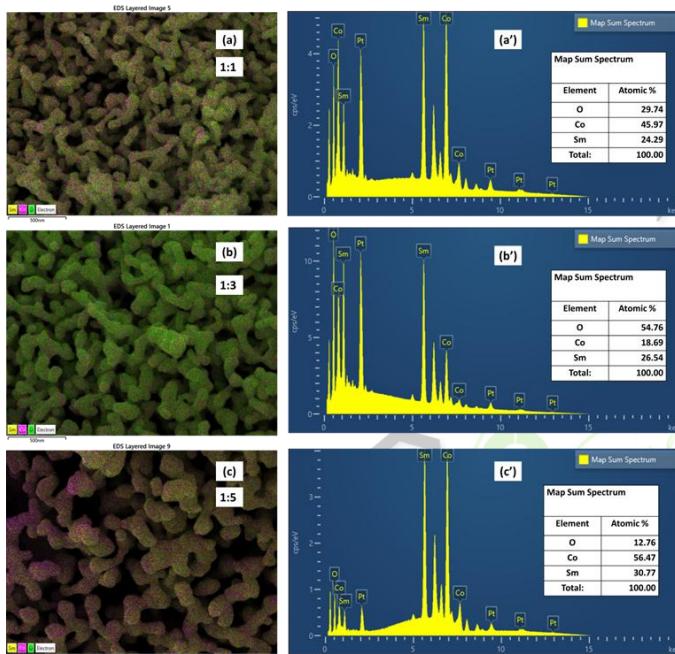


Figure 2. (a-c) represents the elemental distribution of Sm (yellow), Co (pink) and O (green); (a'-c') EDS spectra showing presence of Sm, Co and O as major elements, respective insets show the atomic% of the elements.

The nature of the phases is identified by analyzing the XRD patterns shown in **Fig. 3 (a)** by performing Rietveld refinement (not shown here). It is observed that the MNCs consists of three phases: Sm₂O₃ (cubic: I a -3), Co (hcp: P63/mmc) and SmCo₅ (hexagonal: P6/mmm). The phase fraction of the phases varying with Sm:Co ratio is displayed in **Fig. 3 (b)**. It is observed that at low Sm:Co ratio, metallic cobalt is more as compared to other two phases, however, as the Sm:Co content increases, the fraction of metallic cobalt starts to decrease while that of Sm₂O₃ and SmCo₅ starts to increase.

Further, the magnetic response of the MNCs is recorded through magnetization (M) vs magnetic field (H) hysteresis curve. The MH hysteresis curves corresponding to Sm:Co::1:1, measured in a temperature range of 300 K \leq T \leq 50 K, are displayed in **Fig. 3 (c)**. The saturation magnetization of the samples is found to be 125.8 emu/g and is not affected by the change in measurement temperature from 300 K to 50 K, however, the coercivity has been found to be increased from 60 Oe at 300 K to 129 Oe at 50 K. The change in coercivity points towards the increase in magnetic

anisotropy of the samples. The law of approach to saturation magnetization is used to calculate the magnetic anisotropy.

$$M = M_s \left(1 - \frac{a}{H} - \frac{b}{H^2} \right) + \chi H \quad (1)$$

$$0.0762 \frac{K_1^2}{M_s^2}$$

Where, $b =$

$$K_s = \frac{\mu_0}{4} M_s^2 \quad (2)$$

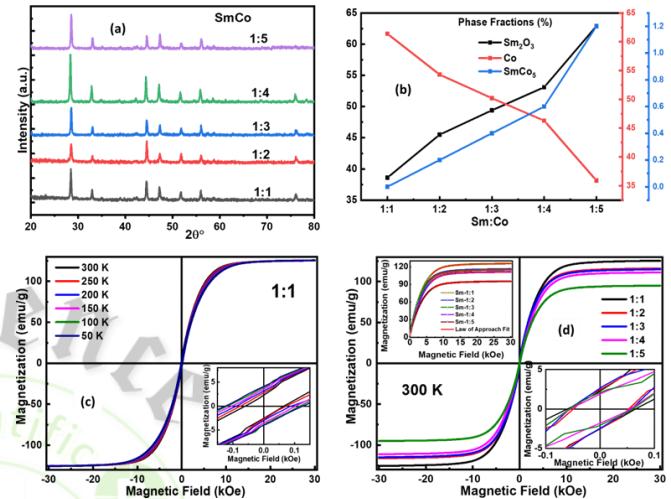


Figure 3 (a) XRD patterns of SmCo nanocomposites; (b) Variation trend of phase fractions of phases SmCo₅, Sm₂O₃ and Co present in the nanocomposites; (c) Magnetization vs magnetic field hysteresis curves at various temperatures, inset shows the magnetization behavior in narrow range of magnetic field; (d) Magnetization vs magnetic field hysteresis curves at 300 K, insets show the near zero field response and Law of Approach fit of magnetization in high field range.

The highest magnetic anisotropy is found to be 1.44×10^6 erg/cm³ for Sm:Co::1:1 which decreased as the Sm:Co ratio increased. The magnetic anisotropy is to be proportional to the saturation magnetization according to equation (1). The investigation of SmCo₅/Sm₂Co₇ by Xu et al., showed that the enhancement in c-axis texture improves the magnetocrystalline anisotropy of the system [10]. Teng et al., have also reported the existence of magnetic anisotropy in SmCo based system due to phase transformations among many phases [11]. The soft/hard magnetic SmCo based nanocomposites exhibit phase dependent variation in the magnetic anisotropy. The exchange coupling between various SmCo phases is an undeniable aspect that influences the magnetic attributes of the material [12–14]. Interestingly, the saturation magnetization is not found to vary significantly from 300 K to 50 K for SmCo based MNCs [15, 16]. On the other hand, the saturation magnetization changes substantially with the Sm:Co ratio showing highest value of 125.8 emu/g for 1:1 and reducing further with increase in the Sm:Co ratio to 94.5 emu/g for 1:5 as shown in **Fig. 3 (b)**. These observations may be possibly due to the different magnetic attributes of the phases present in the MNCs. At 1:1, metallic cobalt has the highest fraction (> 60%) and therefore, dominates the overall magnetization behavior making the nanocomposite soft magnetic in nature. The fraction of SmCo₅ increases with increase in Sm:Co ratio, albeit remains less than 2%. Due to this reason, hard magnetic nature does not have

significant effect on the overall magnetization. On the other hand, since, Sm_2O_3 is a non-magnetic phase, therefore, does not contribute to the magnetization. Thus, all the phases altogether give the soft magnetic characteristic of the MNCs indicating dominating effect of metallic cobalt.

CONCLUSION

In summary, the synthesis of SmCo based magnetic nanocomposites (MNCs) with varying Sm:Co ratio have been carried using chemical route technique. The prepared samples were characterized using high resolution field emission scanning electron microscopy (HRFESEM), Energy Dispersive X-ray spectroscopy (EDS), X-ray Diffraction (XRD) and dc-magnetization to analyze the effect of Sm:Co ratio on the morphological, structural and magnetic properties of the MNCs. The HRFESEM images revealed the irregular shape morphology within 100 nm size. The EDS maps and spectra confirmed the existence of Sm, Co and Oxygen in the samples. The amount of the elements has been found to vary with the Sm:Co ratio. The XRD analysis revealed the presence of three primary phases including metallic cobalt, SmCo_5 and Sm_2O_3 with varying phase fractions. The magnetization behavior of these phases has been found to influence the overall magnetic response of the samples. The samples were found to have a dominance of soft magnetic cobalt at lower Sm:Co ratio with a saturation magnetization of ~ 125.8 emu/g. SmCo based nanomagnets show excellent thermal stability at high temperatures. The SmCo based nanocomposites prepared in this work can be further optimized to increase to coercivity, remnant magnetization and other magnetic properties to enhance the scope of their utilization in state of art devices.

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