Effect of thermal fluctuations on the recoil loops of $SmCo_5$/Fe nanocomposite system

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The correlation between the recoil loop openness and the activation volume has been studied for the $SmCo_5/\alpha$-Fe nanocomposite powder material prepared by mechanical alloying. The α-Fe phase content in the nanocomposite powders was adjusted by varying the amount of α-Fe addition in raw materials and the postannealing temperature after mechanical alloying. It was found that the recoil loop openness increases with increasing α-Fe phase content. More interestingly, there is a linear relation between the openness of recoil loops and the reciprocal activation volume, indicating that the recoil loops are related to thermal fluctuation. The large open area of the recoil loops for nanocomposite magnets is attributed to the fact that low anisotropy in the α-Fe phase leads to unstable magnetization under thermal fluctuation. © *2009 American Institute of Physics*.
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I. INTRODUCTION

Exchange-coupled hard/soft nanocomposite magnets have attracted tremendous attention due to their high potential energy product $(BH)_{max}$ above 100 MG Oe. The key to reach the high energy products in the nanocomposite magnets is effective exchange coupling between the hard and soft phases. One of the most effective ways to characterize the exchange-coupling strength in the nanostructured permanent magnets is to measure recoil loops of the nanocomposites.[1–9] The open recoil loops are usually observed in hard/soft exchange-coupled nanocomposite magnets while not in single-phase magnets. Recently, the investigation of recoil loops has attracted attention again since the element-specific recoil loop measurements on Sm–Co/Fe exchange spring thin films showed that the open recoil loops present not only in the soft layer but also in the hard Sm–Co phase layer.[8] Our recent work showed that thermal fluctuations influence the openness of recoil loops in the FePt/Fe_3Pt nanocomposite magnets.[9] In this work, we focus on the influence of soft-phase content and annealing temperature on the recoil loop openness of the $SmCo_5$/Fe nanocomposites prepared by mechanical alloying. The correlation between the recoil loop openness and the activation volume has also been studied.

II. EXPERIMENTAL DETAILS

The powder mixtures of $SmCo_5+x$ wt % α-Fe ($x=0-30$) were mechanically milled for 4 h in a high-energy ball-milling machine (SPEX8000M), where the commercial $SmCo_5$ and α-Fe fine powders with particle size around 40 μm were purchased from *Alfa Aesar*. The ball to powder ratio is around 10–15:1 in weight. The as-milled powders were annealed at temperatures ranging from 500 to 800 °C for 30 min under vacuum better than 1×10^{-4} Pa. The morphology and crystalline structure were characterized by scanning electron microscopy (SEM), transmission electron microscopy, and x-ray diffraction (XRD) using Cu $K\alpha$ radiation. The composition of the compacted samples was checked by energy dispersive x-ray analysis in SEM. Magnetic properties were measured with superconducting quantum interference device magnetometer with a maximum applied field of 70 kOe. To measure the magnetic viscosity coefficient, a large field was applied and subsequently ramped to the target field, which will be held for approximately 2×10^3 s for magnetization monitoring. This procedure was then repeated with successive increase in the desired measurement fields.

III. RESULTS AND DISCUSSIONS

The open recoil loops were observed in all samples studied in this work. Figure 1 shows examples of the recoil loops

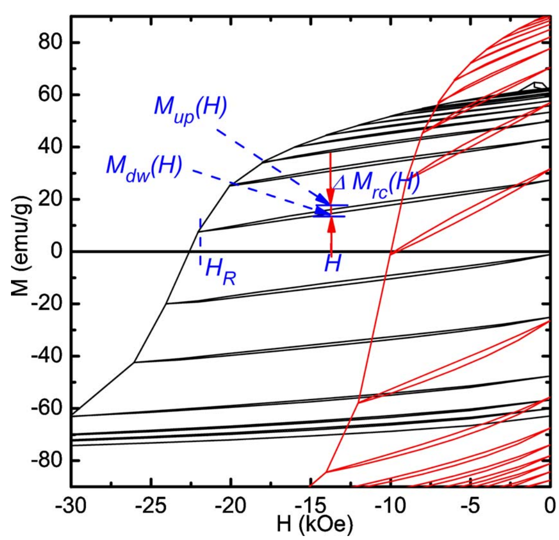

FIG. 1. (Color online) Recoil loops of $SmCo_5$ single phase (with large coercivity) and $SmCo_5/\alpha$Fe nanocomposite with 20 wt % α-Fe addition (with small coercivity) after annealing at 550 °C for 30 min.

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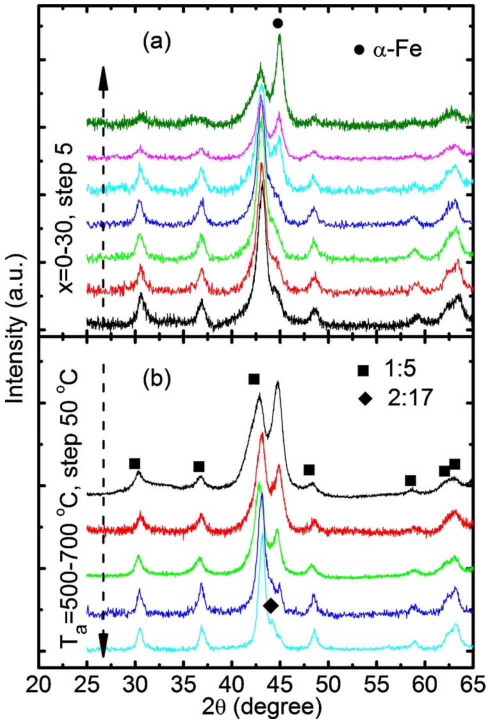

FIG. 2. (Color online) XRD patterns of $SmCo_5/\alpha$-Fe nanocomposite powders with (a) different α-Fe additions after annealing at 550 °C for 30 min and (b) different postannealing temperatures with α-Fe content of 25 wt %.

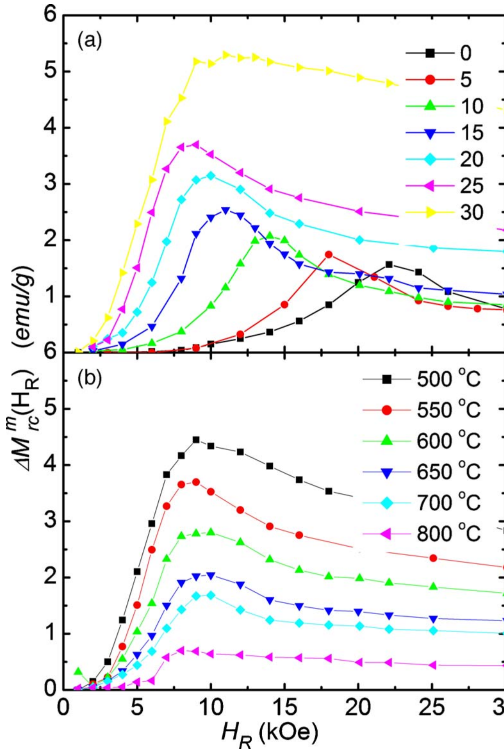

FIG. 3. (Color online) The dependence of recoil loop openness $\Delta M_{rc}^{m}(H_R)$ on the maximum applied field H_R for $SmCo_5/\alpha$-Fe powders with (a) different α-Fe additions after annealing at 550 °C for 30 min and (b) different postannealing temperatures with α-Fe content of 25 wt %.

of the $SmCo_5$ single phase and the $SmCo_5/\alpha$-Fe nanocomposite powders after annealing at 550 °C for 30 min. It was found that the recoil loops of the nanocomposite magnets are relatively open, while those of the single-phase magnets are less open. These results are very similar to those reported previously.[1–9] To quantitatively characterize the openness of recoil loops, we use $\Delta M_{rc}(H)=[M_{\mathrm{up}}(H)-M_{\mathrm{dw}}(H)]$, where $M_{\mathrm{up}}(H)$ and $M_{\mathrm{dw}}(H)$ are the magnetizations of the upper and lower branches on the recoil loops under applied field H (see Fig. 1). The maximum $\Delta M_{rc}(H)$ in each recoil loop (with a maximum applied reversal field H_R) was then defined as $\Delta M_{rc}^{m}(H_R)$.

To study the effect of soft phase on the recoil loop openness, we adjusted the soft-phase content in the $SmCo_5/\alpha$-Fe nanocomposite magnets by tuning the amount of α-Fe addition in raw powders or the postannealing temperature after ball milling. Figure 2(a) shows the XRD patterns of $SmCo_5/\alpha$-Fe nanocomposite powders with different amounts of α-Fe addition after annealing at 550 °C for 30 min. It was found that the relative intensity of α-Fe peaks increases with increasing the amount of the α-Fe addition, indicating the increase in the α-Fe phase content in the final $SmCo_5/\alpha$-Fe nanocomposite powders. The peak width analysis of the XRD patterns shows that the average grain size of both $SmCo_5$ and α-Fe phases in the powder annealed at 550 °C for 30 min is around 10 ± 2 nm determined by Scherrer formula, which is around the efficient exchange coupling length, so that the magnetic moments in the soft phase can be exchange coupled by those in the hard phase. Figure 2(b) gives the XRD patterns of the $SmCo_5/\alpha$-Fe nanocomposite powders with 25 wt % of α-Fe addition after postannealing at different temperatures. The fast drop of the α-Fe peak intensity with increasing annealing temperature indicates the decrease in α-Fe phase content in the nanocomposite, which could be attributed to the atomic diffusion at high temperatures. This was confirmed by the formation of 2:17 phase in the 700 °C annealed sample as shown in Fig. 2(b). In addition, the grain size of both SmCo and α-Fe phases increases with increasing annealing temperature. Especially for the α-Fe phase, the grain size increases from 8 ± 1 nm for 500 °C annealed sample to 28 ± 3 nm for 700 °C annealed sample.

Figure 3 shows the dependence of recoil loop openness $\Delta M_{rc}^{m}(H_R)$ for the $SmCo_5/\alpha$-Fe powders with different amounts of α-Fe addition and different postannealing temperatures. Similar to our recent results on $FePt/Fe_3Pt$ nanocomposite magnets, the peak of $\Delta M_{rc}^{m}(H_R)$ curves corresponds to an applied field close to coercive field, which means that the recoil loop openness is related to the magnetization reversal behaviors. Moreover, it was found that $\Delta M_{rc}^{m}(H_R)$ increases with increasing amount of α-Fe addition and decreasing postannealing temperature. Therefore, one can conclude that the recoil loop openness increases with increasing soft-phase content in the nanocomposite magnets.

It was reported in our recent work that the large recoil loop openness of the nanocomposite magnets is attributed to the unstable magnetic moments in the soft phase due to the thermal fluctuation.[9] The higher soft-phase content in the nanocomposite magnets, the larger openness of the recoil loops was observed due to the larger amount of unstable magnetic moments. To study the relation between the recoil loop openness and the thermal fluctuation, the activation vol-

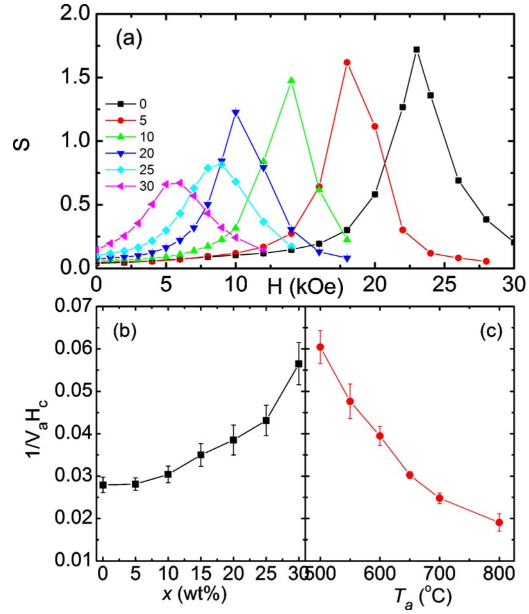

FIG. 4. (Color online) The dependence of magnetic viscosity parameter on external field for the $SmCo_5/\alpha$-Fe nanocomposite with different α-Fe addition and the dependence of $1/V_aH_c$ on (b) α-Fe phase content and (c) annealing temperature.

ume V_{ac} was determined by $V_{ac}=k_BT\chi_{irr}/M_sS$, where k_B, S, and χ_{irr} are Boltzmann's constant, magnetic viscosity parameter, and irreversible susceptibility, respectively.[6,10–12] Figure 4(a) shows the measured dependence of magnetic viscosity parameter on external field for the $SmCo_5/\alpha$-Fe nanocomposite with different α-Fe addition as an example. The V_{ac} can be calculated and the dependence of $1/V_{ac}H_c$ on the amount of α-Fe addition and postannealing temperature was then given in Figs. 4(b) and 4(c), respectively. One can see that $1/V_{ac}H_c$ increases with increasing amount of α-Fe addition while decreases with increasing the postannealing temperature. This trend is similar to the dependence of peak value of $\Delta M_{rc}^{m}(H_R)$ on the soft-phase content and the postannealing temperature (see Fig. 3). Figure 5 shows the dependence of ΔM_{rc}^{pm} on $1/V_{ac}H_c$ where we define the peak value of $\Delta M_{rc}^{m}(H_R)$ as ΔM_{rc}^{pm}. The black square and red circle represent the data for the samples with different α-Fe phase contents and different postannealing temperatures, respectively. Interestingly, there is a linear correlation between ΔM_{rc}^{pm} and $1/V_{ac}H_c$, implying that the thermal fluctuation played an important role on the recoil loop openness. Combining with our previous results[9] on $FePt/Fe_3Pt$ nanocomposite magnet, this linear correlation may be applied to all hard/soft nanocomposite systems.

IV. CONCLUSION

The soft-phase content in $SmCo_5/Fe$ nanocomposite powders was adjusted by tuning the addition amount of α-Fe powders and the interdiffusion during postannealing. It was found that the recoil loop openness increases with increasing α-Fe phase content. The large open area of the recoil loops for nanocomposite magnets compared to that of single phase magnets is attributed to the unstable magnetic moments in α-Fe phase, which can be reversed during the thermal fluctuation. A quantitative analysis confirms a linear relation between the openness of recoil loops and the reciprocal activation volume, confirming that the recoil loops are related to thermal fluctuations.

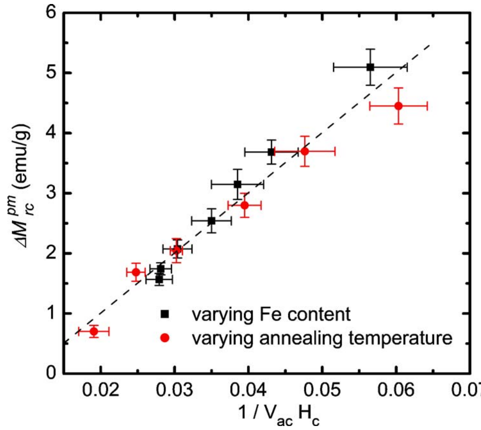

FIG. 5. (Color online) The dependence of ΔM_{rc}^{pm} on $1/V_{ac}H_c$ for samples with different α-Fe phase contents and different annealing temperatures.

ACKNOWLEDGMENTS

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