High-temperature FORC study of single- and multi-phase permanent magnets

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First-order-reversal-curves (FORCs) are a nondestructive tool for characterizing the magnetic properties of materials comprised of fine (micron- or nanoscale) magnetic particles. FORC measurements and analysis have long been the standard protocol used by geophysicists and earth and planetary scientists investigating the magnetic properties of rocks, soils, and sediments. A FORC can distinguish between single-domain, multi-domain, and pseudo single-domain behavior, and it can distinguish between different magnetic mineral species.[1] More recently, FORC has been applied to a wider array of magnetic material systems, because it yields information regarding magnetic interactions and coercivity distributions that cannot be obtained from measurements of a material's major hysteresis loop alone. In this article, we discuss the FORC measurement and analysis technique and present high-temperature FORC results for multi-phase permanent magnets.

Magnetization measurements and first-order-reversal-curves

The most common measurement that is performed to characterize a material's magnetic properties is measurement of the major hysteresis loop M(H) using either a vibrating sample magnetometer or superconducting quantum interference device magnetometer. The parameters that are most commonly extracted from the M(H) loop are the saturation magnetization M_s, the remanence M_r, and the coercivity H_c. In a hysteresis loop measurement, the measured coercivity H_c is the weighted coercivity of the entire ensemble of magnetic particles that constitute a magnetic material, thus if the material contains more than one magnetic phase, it is hard to discern between these phases.

FORCs[2] can give information that is not possible to obtain from the hysteresis loop alone. This includes the distribution of switching and interaction fields, and identification of multiple phases in composite or hybrid materials containing more than one phase.[3,4] A FORC is measured by saturating a sample in a field H_{sat}, decreasing the field to a reversal field H_a, then measuring moment versus field H_b as the field is swept back to H_{sat}. This process is repeated for many values of H_a, yielding a series of FORCs. The measured magnetization at each step as a function of H_a and H_b gives $M(H_a, H_b)$, which is then plotted as a function of H_a and H_b in field space. The FORC distribution $\rho(H_a, H_b)$ is the mixed second derivative, $\rho(H_a, H_b) = -(1/2)\partial^2 M(H_a, H_b)/\partial H_a \partial H_b$.

The FORC diagram is a 2D or 3D contour plot of $\rho(H_a, H_b)$. It is common to change the coordinates from (H_a, H_b) to $H_c = (H_b - H_a)/2$ and $H_u = (H_b + H_a)/2$, where H_u represents the distribution of interaction or reversal fields, and H_c represents the distribution of switching or coercive fields.

High-temperature FORC results for multi-phase permanent magnets

To demonstrate the utility of the FORC measurement and analysis protocol for magnetic property measurements, the characterization of high-temperature magnetic properties of materials is presented. Measurements were conducted for a synthetically produced three-phase magnet by mixing together, in approximate equal mass proportions, three different single-phase magnets: Sr-ferrite powder (Hoosier Magnetics), BaSr ceramic (Magnet Sales & Manufacturing – Integrated Magnetics), and sintered NdFeB (Magnequench). All magnetic measurements were performed using a Lake Shore Cryotronics MicroMag vibrating sample magnetometer with a high-temperature furnace, which allows for variable temperature measurements from room temperature to 800°C. All measured magnetization data are presented in terms of the magnetic moment (emu) as a function of field (Oe) and temperature (°C). There are a number of open source FORC analysis software packages such as FORCinel[5] and VARIFORC,[6] although in this paper, custom analysis software was used to calculate the FORC distributions and plot the FORC diagrams.

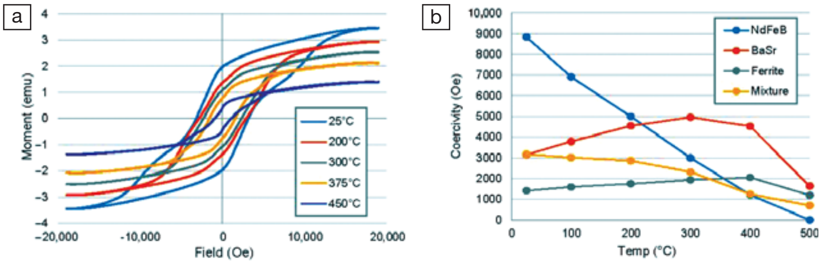

Figure 1. (a) M(H) at T = 25°C, 200°C, 300°C, 375°C, and 450°C for a three-phase mixture of Sr-ferrite powder, BaSr ceramic, and sintered NdFeB, and (b) H_c versus T plots for the individual samples and the three-phase mixture.

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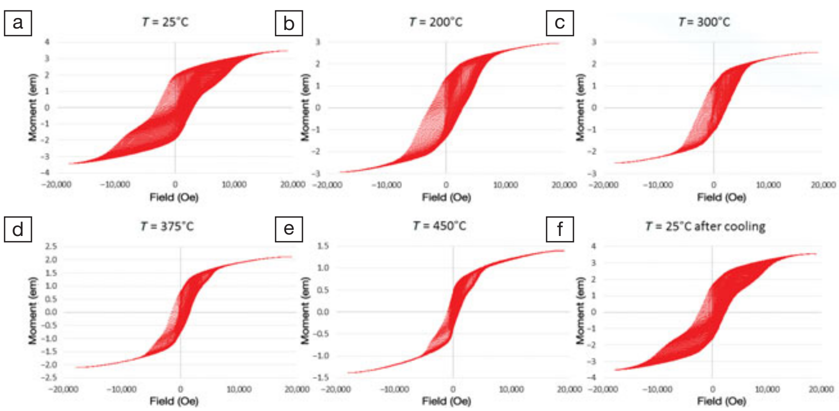

Figure 2. Measured first-order-reversal-curves at T = (a) 25°C, (b) 200°C, (c) 300°C, (d) 375°C, (e) 450°C, and (f) 25°C after cooling from 450°C for a three-phase mixture of Sr-ferrite powder, BaSr ceramic, and sintered NdFeB.

Figure 1 shows the hysteresis M(H) loops at temperatures of T = 25°C, 200°C, 300°C, 375°C, and 450°C (a) and the temperature dependence of the coercivity (b) for the individual samples and three-phase mixture. The M(H) loops at 25°C and 450°C show evidence of a two-step and thus two-phase behavior, while the loops at intermediate temperatures essentially exhibit single-phase behavior. There is no suggestion of three-phase behavior in any of the measured M(H) loops.

Figure 2 shows the measured FORCs, and **Figure 3** shows the 2D FORC diagrams at each temperature. At 25°C, there are three peaks in the FORC distribution corresponding to each phase. At 200°C, the BaSr and NdFeB peaks are not separable because their coercivities are very similar; although at temperatures above 200°C, each phase is again distinguishable, with the NdFeB peak shifting toward lower switching fields coincident with the decrease in its coercivity with increasing temperature. The Sr-ferrite and BaSr peaks initially shift toward higher switching fields and then move toward lower switching fields with increasing temperature. This coincides with the temperature dependence of their coercivity, as determined from their M(H) loop measurements.

From FORC measurements of the single-phase samples, it is known that the ridge feature (see Figure 3) is related to the NdFeB. The ridge continuously shifts toward lower switching fields and less negative interaction fields with increasing temperature, and is believed to be due to temperature-induced magnetostatic interactions. Also there is a fourth peak of unknown origin present in the distribution at 300°C located between the Sr-ferrite and NdFeB peaks. At 450°C, there is no feature associated with the NdFeB because its coercivity is small, and the ridge has entirely disappeared. Finally, the last FORC diagram shows results at 25°C after cooling from 450°C and depicts the reemergence of the NdFeB peak and ridge. In comparing the FORC diagrams at 25°C before warming and after cooling, there are obvious differences owing to irreversible changes in the material resulting from thermal cycling.

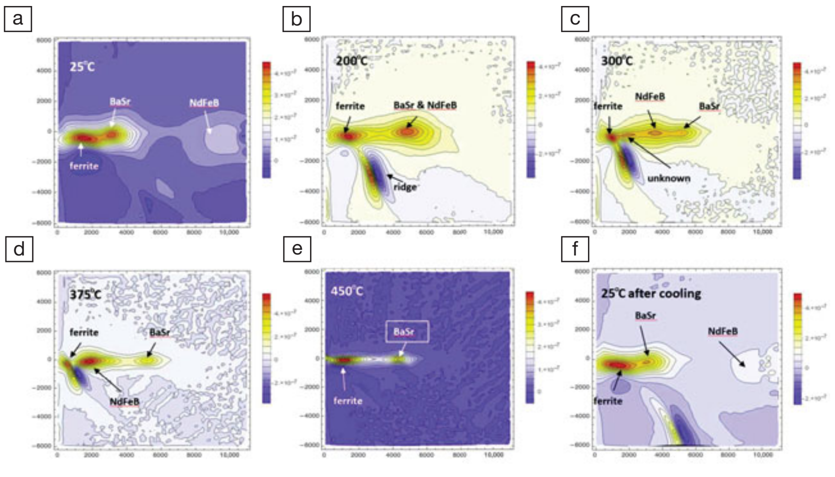

Figure 3. First-order-reversal-curve diagrams at T = (a) 25°C, (b) 200°C, (c) 300°C, (d) 375°C, (e) 450°C, and (f) 25°C after cooling from 450°C for a three-phase mixture of Sr-ferrite powder, BaSr ceramic, and sintered NdFeB.

Conclusions

FORC analysis has been shown to be very useful for characterizing interactions and coercivity distributions in an array of magnetic materials, including magnetic nanowire arrays, permanent magnets, magnetic recording media, exchange-biased magnetic multilayer thin films, and natural magnets. We have shown the evolution at high temperatures of the distribution of switching and interaction fields as determined from FORC analysis for a three-phase mixture of three single-phase magnets. The results demonstrate the utility of FORC analysis for differentiating phases in multi-phase magnetic materials. *The results shown in the figures are based on unpublished data.*

References

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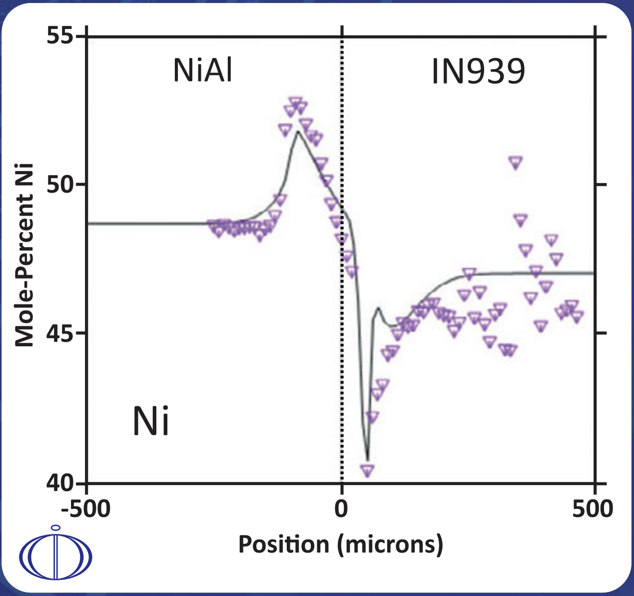

Diffusion in ordered phases