Beyond the qualitative description of complex magnetic nanoparticle arrays using FORC measurement

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Abstract

First-order reversal curve (FORC) measurements are broadly used for the characterization of complex magnetic nanostructures, but they can be inconclusive when quantifying the amount of different magnetic phases present in a sample. In this paper, we first establish a framework for extracting quantitative parameters from FORC measurements conducted on samples composed of a single type of magnetic nanostructure to interpret their magnetic properties. We then generalize our framework for the quantitative characterization of samples that are composed of 2–4 types of FeCo magnetic nanowires to determine the most reliable and reproducible parameters for a detailed analysis of samples. Finally, we conclude that the parameter with the best quantification potential, backfield remanence coercivity, does not require the full FORC measurement. Our approach provides an insightful path for fast, quantitative analysis of complex magnetic nanostructures, especially determination of the ratios of magnetic subcomponents present in multi-phase samples.

Introduction

Advancement of nanotechnology has extensively expedited the emergence of novel magnetic nanostructures, such as magnetic nanowires (MNWs), in various research areas, including medical treatment [1–5], environmental science [6, 7], and quantum devices [8–12]. These magnetic nanostructures have opened numerous opportunities for scientists in different disciplines such as nanomedicine, molecular biology [13–16], applied physics, and nanostructured materials [17–22]. In all of these applications, it is crucial to know the characteristics of the magnetic nanostructures, which may inhibit or enhance their use depending on the application. Unfortunately, the high yielding nanofabrication processes of magnetic nanostructures do not allow perfectly identical production, leading to variation in their magnetic characteristics and functionalities [23]. Besides that, the lack of a coherent framework for data extraction and analysis means that current techniques for the quantitative characterization of magnetic nanostructures are inefficient both at the research level and at the industrial level.

Magnetic nanostructures have been characterized by measuring their magnetization using the major/minor hysteresis loops and/or first-order reversal curves (FORC) for decades. Unfortunately, speed and accuracy are competing criteria, where hysteresis loop measurements are relatively fast yet provide significantly limited information compared to FORC measurements which are more time consuming because more data points are needed [23–25]. For example, hysteresis loops typically provide saturation magnetization and coercivity information, so they are sufficient to describe a magnetic structure that contains only a single type of uncorrelated magnetic subcomponents, such as arrays of non-interaction MNWs. However, they fail to fully describe complex magnetic nanostructures, such as arrays of interacting MNWs. On the other hand, FORC offers a comprehensive insight into the qualitative and partially quantitative interpretation of any complex magnetic nanostructure, but the measurements typically involve 100x more data points. Theoretical models,
such as the mean field model, have been used to interpret the information in FORC diagrams [26–30] but these models usually only consider a perfect arrangement of building blocks with homogenous properties due to computational limitations. These perfect structures are not well matched with experimental structures. Therefore, to describe the complex magnetic nanostructure and analyze their functionality, it is helpful to step beyond the conventional data analysis and representation of FORC data to precisely describe magnetic nanostructures.

Mayergoyz [31–33] proposed the current standard FORC measurement as an identification technique via the classical Preisach model [34], which describes magnetic hysteresis loops as a superposition of numerous independent relays, called hysterons. Hysterons represent the switching of single MNWs with rectangular hysteresis loops, such as those of isolated MNWs acting like Stoner-Wohlfarth particles. Experimentally, FORC measurements begin by applying a large magnetic field, as follows:

\[
\rho = \frac{1}{2} \frac{\partial^2 M(H, H_r)}{\partial H \partial H_r}
\]

In FORC analysis, \( \rho \) is plotted as a heat-map with the axes representing the coercive field (x-axis, \( H_c = \frac{1}{2}(H-H_r) \)) and the interaction field (y-axis, \( H_u = \frac{1}{2}(H + H_r) \)).

Traditionally, the quantitative analysis of the FORC data is done using the projection of FORC distribution on the \( H_r \)-axis and \( H_u \)-axis, called coercivity distribution (\( P_{Hc} \)) and interaction distribution (\( P_{Hu} \)), respectively, see figure 1(a). Here, quantitative analysis means quantifying the coercivity and the interaction fields, not quantifying the amount of the magnetic components [26, 35]. Intuitively, the definition of the coercivity and interaction fields is not appropriate for quantifying the amount of each magnetic subcomponent present in a sample. For example, if a magnetic nanostructure contains both interacting and non-interacting magnetic subcomponents, the interaction field distribution of the magnetic nanostructure only represents the interaction field distribution of the interaction subcomponents regardless of the amount of the non-interacting subcomponents exist.

To overcome these shortcomings, one solution is to take only one derivative from the FORC data to calculate the switching field distributions [36–39]. In this approach, the switching field distributions are functions of both \( H \) and \( H_r \) fields leading to 2D heat-maps that inherently share the complex data interpretation and analysis of the FORC method [40–42]. To further suppress these limitations, we propose to decompose the irreversible and reversible switching fields (ISF and RSF, respectively) and only investigate the ISF distributions at the \( H = H_c \) and \( H = 0 \), figure 1(a). Note the ISF at \( H = H_r(P_{Hr}) \) can be calculated by projecting the FORC heat-maps in \( H_r \) axis [23, 43, 44] as it is the residual magnetic moment at the reversal field. An equivalent parameter to this is the backfield remanence coercivity (BRC), see figure 1(b), which is the ISF at \( H = 0 \). The BRC can be determined by taking a derivative of the magnetization at the zero applied field, which is known to be backfield remanence magnetization (BRM), see figure 1(b). It is essential to emphasize that the only difference between the BRC and
of the MNWs in various combinations. To determine the most reliable and reproducible parameter for quantitative analysis of the volume ratios and types of those parameters for the individual types and the combinations of our MNWs. The results are discussed to engineer the aforementioned parameters, helping to illustrate the nature of those parameters. We then extracted MNWs arrays because their sizes and the interwire distances can be varied to provide the opportunity to approach on FORC diagrams of various types of MNWs arrays and their various combinations. We chose MNWs measured along the easy axis, parallel to the MNWs axis. Samples with the smallest diameter were shown to consist of single magnetic subcomponents and the PHc and PHu. First, since magnetic switching between two stable magnetic equilibrium states is always irreversible, these parameters contain all magnetic subcomponent responses regardless of their concentration in the sample. Second, they can be determined by measuring ~5 points on each FORC curve versus ~100 points per curve, which significantly accelerates the measurements without adding complexities into the measurement protocols [24]. Any moment measurement, standard or fast, such as MOKE-FORC [25, 45] and AC FORC methods [37, 46, 47], can be used to measure these 5 points. In addition, complex data processing and smoothing are not required [27, 40–42, 48].

In the following sections, we extract and examine the aforementioned quantitative parameters (PHc, PHu, PHr, BRM, and BRC) concealed in FORC diagrams using a rigorous statistical analysis. To do so, we practice our approach on FORC diagrams of various types of MNWs arrays and their various combinations. We chose MNWs arrays because their sizes and the interwire distances can be varied to provide the opportunity to engineer the aforementioned parameters, helping to illustrate the nature of those parameters. We then extracted those parameters for the individual types and the combinations of our MNWs. The results are discussed to determine the most reliable and reproducible parameter for quantitative analysis of the volume ratios and types of the MNWs in various combinations.

Experimental and statistical approaches

Iron cobalt (FeCo) MNWs were electrodeposited into track-etched polycarbonate templates with a broad range of diameters (and fill factors)—30 nm (0.5%), 50 nm (1.0%), 100 nm (2.0%), and 200 nm (12%)—at room temperature. The electrolyte consisted of 0.4 M boric acid, 1 mM malonic acid, 0.3 M ammonium chloride, 0.3 mM sodium dodecyl sulfate, 6 mM ascorbic acid, 0.2 M iron sulfate, and 0.1 M cobalt sulfate at pH 3. The concentration ratio of the iron sulfate to cobalt sulfate was chosen 2:1 in order to achieve a Fe to Co atomic ratio of Fe65Co35. This composition is known to have the highest saturation magnetization [49] so it enables measurements of even very small numbers of MNWs. Figure 2 shows the FORC distributions of the four types of MNWs measured along the easy axis, parallel to the MNWs axis. Samples with the smallest diameter (fill factor), 30 nm (0.5%), had inter-wire distances of ~450 nm with inter-wire distance to diameter ratios of 15. This large inter-wire distance presents a fairly symmetric rectangular magnetic hysteresis loop with a narrow FORC distribution, as predicted by micromagnetic simulations when there are negligible magnetic interactions [50]. By contrast, the inter-wire distance to diameter ratio decreases, the MNW stray fields interact, leading to sheared hysteresis loops with vertically broadened FORC distributions, see figure 2(d). For example, the samples with the largest diameter (fill factor) of 200 nm (12%) had inter-wire distances of ~556 nm and inter-wire distance to diameter ratios of 2.78, where the resulting FORC distribution broadens vertically indicating a large interaction field between the MNWs. The inter-wire distance to diameter ratio for samples with diameters of 50 nm and 100 nm is 9 and 7.3, respectively.

![Figure 2. The FORC distributions for different types of the FeCo MNWs with diameter (fill factor) of (a) 30 nm (0.5%), (b) 50 nm (1%), (c) 100 nm (2%), and (d) 200 nm (12%).](image-url)
Several combinations were created with at least two different types of the MNWs, and FORC measurements were repeated (Figure 3). We first extract the PHc, PHu, PHr, BRM, and BRC parameters for the individual samples and combinations. To analyze the reliability and reproducibility of the extracted parameters for quantitative description, the parameters for the combined samples were fit to the corresponding parameters of the individual types of MNWs. The fitting quality was evaluated using the root mean square (RMS) error of the difference between the ‘experimental data’ and ‘recreated curve’, which is the weighted summation of the corresponding parameters of the individual types. The RMS error was minimized to find the optimum weights for the volume ratio of each type of MNW in the combination.

Results

Here, we plot the PHc, PHu, PHr, BRM, and BRC parameters for the individual MNWs and their combinations. We describe the magnetic properties that these parameters represent to establish a framework for optimizing their efficiency for achieving the best quantitative description of any magnetic nanostructure. The fitting quality and volume ratios will be discussed in the discussion section.

Figures 4 and 5 depict the PHc and PHu distributions calculated by taking the integral from the FORC distributions over all Hu and Hc, respectively. The location of the peaks in Figure 4 shows the average coercivity of the MNWs, where the sample with MNW diameters (fill factor) of 30 nm (0.5%) yields the maximum coercivity (∼0.685 kOe) and 200 nm (12%) MNWs has the minimum coercivity (∼0.163 kOe). These distributions have extra features (e.g. local minima in the top row of figure 4) that are difficult to interpret physically, and they complicate analysis of multiple component samples. Physically, these extra features cannot fully be due to interaction fields, but they have been previously shown to be potentially from thermal fluctuations [51]. Other magnetic measurements, such as ferromagnetic resonance, have shown that interaction fields cause broadening and/or shifts of the PHc peak [52–55], but not necessary inducing extra local minima/maxima. Similar problems arise with PHr distributions in Figure 6. PHr is the interaction field distribution which has a different complication in its distribution that will make it difficult to use in quantification of magnetic subcomponents in multiphase samples. Namely, the jagged peak due to the projections of the famous nanowire ‘T’ or ‘wishbone’ which have been explained in other works [37, 44]. Here, we simply state that these complicated distributions inhibit accurate quantification of phases, such as the MNWs used in this paper.

On the other hand, the PHu distributions are smooth functions, Figure 6. The PHu peak indicates the average coercivity of the sample, similar to PHc distributions, as it is related to the derivative of the upper branch of the hysteresis loop [36]. These results are similar to the demonstration of AC susceptibility plots in [35] where two magnetic phases could be more easily visualized that in standard FORC heat-maps. For most of the combined samples, the PHu plots show a clear mixture of single component features, according to the
Figure 4. Coercivity distribution ($P_{Hc}$), determined by taking an integral over $H_u$ from FORC distribution, for individual and different combinations of the MNWs as indicated in the legend.

Figure 5. Interaction distribution ($P_{Hu}$), determined by taking integral over $H_c$ from the FORC distributions, for individual and different combinations of the MNWs as indicated in the legend.

Figure 6. The $P_{Hr}$ distributions for individual and different combinations of the MNWs as indicated in the legend.
volume ratio of the samples in the combination. The shift of the peaks indicates the amount of each MNW relative to another [27, 29].

The $P_{1H}$ distributions maintain a sharp peak even for very small volume ratios, such as the 30 nm and 200 nm combination where the volume ratio was 1.3%. This sensitivity exists because the $P_{1H}$ not only depends on the MNWs coercivity and volume ratio but also on the irreversibility fraction (defined as irreversible magnetization to total magnetic moment). Note that the irreversibility fraction is a measure of (1) the external energy (provided by an external field, also known as Zeeman energy) that is required to switch the magnetization direction, and (2) the stability of the magnetization direction once switching occurs. For example, non-interacting MNWs with very small diameters magnetized along their easy axis experience magnetic reversal by a coherent rotation mechanism, and the irreversibility fraction is 100%. On the other hand, for interacting MNWs with large diameters, the interaction field reduces the irreversibility fraction. Simply, this means the accuracy of using $P_{1H}$ for subcomponent identification can be enhanced by controlling the irreversibility fraction of the MNWs in the combination, especially when the volume ratio is very small.

Figure 7 shows the BRM results of single MNW samples and several combinations. Since the samples have different magnetic moments, we normalized the BRM with respect to their saturation backfield remanence (remanence of the major hysteresis loop). From figure 7, it can be seen that the BRM of any combination is always valued between the BRMs of the individual MNWs in the combination. Therefore, the volume ratio can be determined by finding the BRM shift of the combination. Practically, two features characterize the BRM [57–60]: (1) the field where it is zero, which is average coercivity of the MNWs, and (2) its slope, which is correlated to the MNW interaction field. Thus, the accuracy of the BRM can be enhanced by manipulating these two parameters.

Figure 8 provides the BRC distribution of the MNWs, calculated by taking a derivative of the BRM with respect to the reversal field. As experimentally observed, the BRC of single MNWs describes a maximum that is centered on the inflection points of the BRM curves, as can be seen clearly in figures 7 and 8. Practically, the behavior of the BRC of the combinations can be demonstrated by two parameters: (1) the amplitude of each local peak (determined by the volume ratio of the MNWs), and (2) the relative location of the peaks (indicating the coercivities of the MNWs present in the sample). Therefore, similar to $P_{1H}$, one can quantitatively describe the type and volume ratio of the MNWs in the combination by just analyzing these two features. It should be emphasized that the accuracy of the BRC depends on both the amount and coercivity of component MNWs. Therefore, combinations of MNWs can be designed for optimal quantitative description by combining high coercivity and low coercivity types of MNWs. For example, combination of 30 nm diameter MNWs with 200 nm diameter MNWs was easy to quantify with high accuracy due to completely distinct peaks in their BRC distributions.

**Discussion**

Figure 9 summarizes the fitting results of each parameter. Specifically, figure 9(a) shows the results for the combinations including two types of the MNWs and figure 9(b) shows the results for a single combination with
all four MNW types. The fit volume ratio ($\chi$) was found by minimizing the RMS error, and it is compared with the known $\chi$. According to figure 9, all parameters give $\chi$ values within reasonable accuracy, and the $P_{HR}$ results seem to be slightly better than the others. $P_{HR}$ and BRC should have relatively similar results because they both depend on the subcomponents’ coercivities, irreversibility fraction, and amount of each type of the MNWs. However, as was already mentioned, there is a difference between $P_{HR}$ and BRC in that $P_{HR}$ is a measure of the residual magnetization at $H = H_r$ while the BRC is a measure of the residual magnetization at $H = 0$. Since the $P_{HR}$ is measured at the reversal point, the applied field overcomes the interaction fields. Thus, the $P_{HR}$ shows the residual magnetization purely dependent on the initial magnetization state with negligible effect from interaction fields. On the other hand, since the applied field is zero while measuring the BRC, the magnetostatic energy of the system is lower when the interaction fields reverse some MNW magnetizations. To examine the strength of these parameters for combinations with more than two types of MNWs, we repeated the measurements on the combination of all four MNWs types, see the ESI and figure 9(b). In figure 9(b), the first, second, third, and fourth known $\chi$ show the volume ratio of MNWs with the diameters of 30 nm, 50 nm, 100 nm, and 200 nm to the total volume of the MNWs, respectively. The accuracy of these parameters for the combination of all types, figure 9(b), highlights the advancement of this analysis in sensitive applications, such as barcoding.
Conclusion

In this work, we established a framework for quantitative data extraction and analysis using the FORC measurement. We showed that the $P_{1H}, P_{1H^+}, P_{BRM},$ and $BRC$ can be readily extracted for quantitative analysis of the MNWs, which is not possible with the conventional representation (heat-maps) of FORC data. Our experimental observation indicates that $P_{1H}$ has slightly a better capability for quantifying the volume ratio ($\chi$) of the MNW combinations because it employs the effects of the irreversibility while minimizing effects of interwire interaction fields. Furthermore, these parameters are able to estimate the volume ratios of the individual types of MNWs in a combination containing several types of MNWs within reasonable accuracy. This finding opens numerous opportunities in applications such as nano-barcoding.

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