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Observation of Patterns by Magnetic Force Microcopy in Fe-alloys with Shape Memory Effect

M. I.N. da Silva, J. C. González and M. S. Andrade[1]
North Carolina State University, Analytical Instrumentation Facility,
Raleigh, NC, 27695-7531, U.S.A.
[1]Laboratório de Nanoscopia, Setor de Tecnologia Metalúrgica – CETEC, Av. José Cândido da Silveira, 2000, Belo Horizonte-MG, 31170-000, Brazil.

ABSTRACT

In this study, we investigated the magnetic domains of a FeMnSiNiCr stainless steel sample using Magnetic Force Microscope (MFM). We compared the magnetic patterns obtained by scanning the sample with three coated probes with different magnetic properties: Medium magnetic moment (MM), low magnetic moment (LM), and low coercivity (LC). The probe-surface separation was varied between 25 to 300 nm in order to quantify the magnetic microstructure of the sample. A simple model for the probe-sample interaction was used to interpret the contrast change as a function of the probe-surface separation. The experiment showed that the average maximum frequency decreases with the probe-surface separation and the intensity of the frequency is the strongest for the MM probe. X ray diffraction experiments were used to identify the different phases present in the sample. The X-ray diffraction experiments together with the MFM showed that α-phase islands surrounded by a γ-phase matrix are responsible for the magnetic properties of the sample.

INTRODUCTION

Fe-based alloys with Shape Memory Effect (SME) [1] have great technological importance. The SME effect correspond to a martensitic transformation in the material and may be defined as the property of recovering its original shape during a thermal cycle after a material has been apparently deformed in a permanent way. In order to maximize the SME, it is important to eliminate or reduce at minimum other phase present in the material that not contribute to the SME. In our case the ferromagnetic phase α play an important role reducing the SME efficiency, being important the study and characterization of the magnetic microstructure of the sample. Magnetic force microscopy (MFM) has proven to be a useful tool for imaging the magnetic microstructures in a variety of magnetic materials [2]. This technique has characteristics such as high spatial resolution and minimum sample preparation. MFM is based on the interaction between a magnetic sample and a magnetic coated Atomic Force Microscopy (AFM) probe or tip. The AFM tip can be coated with a variety of materials with different magnetic properties. The contrast observed in this kind of microscopy depends strongly of the magnetic interaction between the sample and the AFM tip as well as the modifications that the magnetic AFM tip can induce on the magnetic microstructure of the sample. The choice of magnetic coating for the AFM tip can reveal fine microstructure in the magnetic domains of the sample or simply modify the microstructure itself leading to an erroneous characterization of the

sample. In this work, the magnetic domains of a FeMnSiNiCr stainless steel sample, with strong SME, were studied using the MFM technique. We compared the magnetic patterns obtained by scanning the sample with three coated probes with different magnetic properties: Medium magnetic moment (MM), low magnetic moment (LM), and low coercivity (LC). The tip-surface separation was varied from 25 to 300 nm in order to quantify the magnetic microstructure of the sample. A simple model for the magnetic tip-sample interaction was used to interpret the contrast change as a function of the tip-surface separation, for all the three AFM tips. X ray diffraction experiments were used to identify the different phases present in the sample.

EXPERIMENTAL DETAILS

A Digital Instruments Dimension 3000 Scanning Probe Microscope (SPM) was used in Tapping/Lift™ mode. In this mode the tip oscillates close to its mechanical resonance frequency. These measurements are based on a two-pass technique for each scanline. In the first pass, the topographical data from the surface of the sample is acquired. The tip is then raised up and a second pass is done keeping a constant separation between the tip and the surface of the sample (lift height). During the second pass, the shift in the resonant frequency induced by the interaction of the dipolar field of the sample with the magnetic moment of the tip is acquired at each point of the scanline. The lift mode virtually eliminates the cross link with the topography of the sample during the MFM measurements.

The AFM tips used in this work were Silicon cantilevers with pyramidal tips from the Digital Instruments MESP series. The first tip used in this work was a standard MESP tip (MM) coated with 50 nm of CoCr alloy. This tip is considered to have a medium coercivity with a value of approximately 400 Oe, as well as a medium magnetic moment of approximately 10^{-13} emu. The second tip used was a MESP-LM tip (LM) coated with 15 nm of CoCr alloy. This tip has a coercivity of approximately 400 Oe, and a magnetic moment three times lower than the MM tip, both of which are considered low. The third tip was a MESP-LC tip (LC) coated with a NiFe alloy. With a coercivity value less than 1 Oe and a magnetic moment similar to the MM tip, the coercivity and the magnetic moment values of this tip are considered low.

The FeMnSiNiCr stainless steel used in this work has the following composition (weight %): 66Fe-14Mn-6Si-9Cr-5Ni. An ingot was prepared by induction melting of purity materials under an argon atmosphere. A square sample of 1×1 cm^2 of area and 2 mm thick was cut from the ingot, embedded in epoxy, and polished with a diamond past of 1 μm grain size in order to obtain a flat surface. Following this process the sample was rinsed with DI water, dried with Ar, and mounted in the SPM to be imaged.

The X-ray experiment was carried out in a Rigaku Geigerflex 2037 diffractometer. Monochromatic CuKα radiation was used. The data was collected in the 2θ mode.

RESULTS AND DISCUSSION

Figure 1 shows an X-ray diffractogram of the sample at room temperature. X ray results showed the presence of three phases in the sample: a paramagnetic γ–phase, a ferromagnetic α-phase, and a tetragonal crystal structure σ-phase. Based on these results, we expected the MFM measurements to reveal small magnetic areas surrounded by a large paramagnetic matrix. Due to the fact that the γ-phase and σ-phase are both of paramagnetic nature, it was not possible to

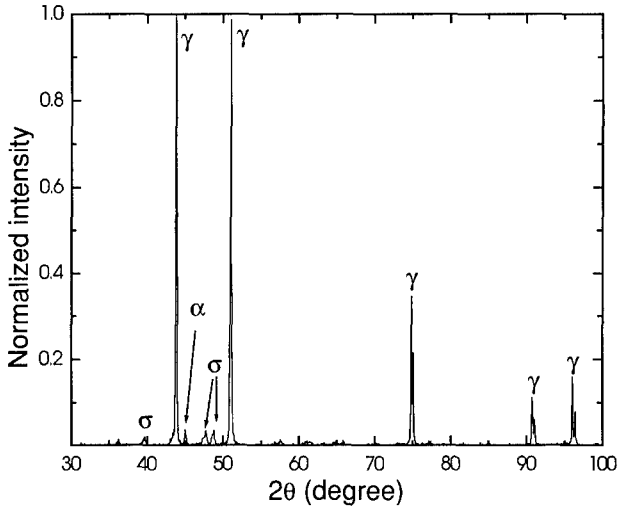

Figure 1. X-ray diffractogram obtained at room temperature.

differentiate the two phases in the MFM measurements. Only the magnetic areas of the α-phase can clearly be distinguish from the rest of the matrix.

Figure 2 presents the MFM measurements obtained with the three different tips. A topographic image of the surface of the sample is shown in Figure 2(a) with z scale of 15 nm. The sample is relatively flat and only small scratches from polishing are visible. Figures 2(b) through (d) are MFM images obtained with the MM, LC, and LM tips, respectively, from the same region of the sample as shown in 2(a). Figures 2(b) through (d) present an interesting magnetic microstructure not observed in the Figure 2(a). Z scale is 12 Hz for figure 2(b) and 2 Hz for figure 2(c) and 2(d). In these images, two distinct patterns are easily identified: a lighter region, which is paramagnetic in nature as well as a larger ferromagnetic region with characteristic dark and light stripes.

Images 2(c) and (d) have an apparent larger lateral resolution than 2(b). This "loss" of resolution in Figure 2(b) is caused by the remagnetization effect [3] that the high coercivity and medium moment tip induces into the low coercivity sample. A reverse in contrast of the images shown in 2(b) and (d) is observed in Figure 2(c). This change in contrast is caused by the realignment of the magnetic moment of tip [4]. In this case, the dipolar field from the sample is high enough to rotate the moment of the low coercivity tip, which leads to the misinterpretation of the direction of the dipolar field of the sample. In the case of Figure 2(d), the high coercivity and low moment tip reads the true magnetic microstructure of the sample without inducing a remagnetization of the domains in the sample with the resulting lost of resolution, as observed in the case of the MM tip. In addition, there is no realignment of its own moment that can cause a misinterpretation of the direction of the dipolar field of the sample, as in the case of the LC tip. As can be seen from the results, the choice of coating material, and as a consequence the magnetic properties, of the tip are extremely important to evaluate the real magnetic

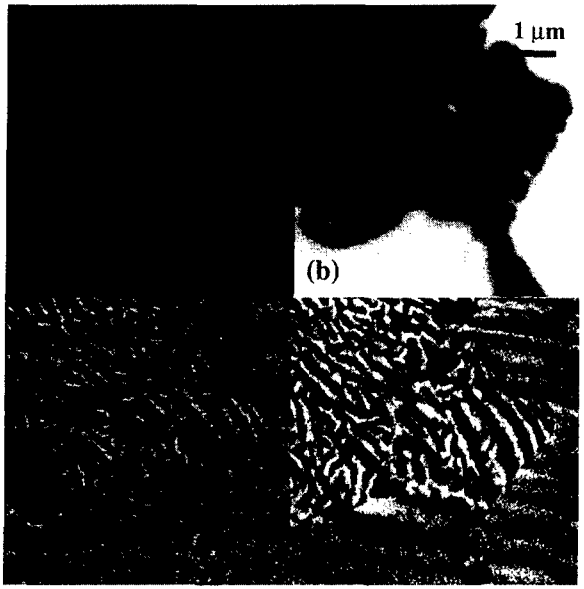

Figure 2. Images of the sample. Topographic image (a), MFM image using MM tip (b), MFM image using LC tip (c), and MFM image using LM tip (d).

microstructure of the sample. We think the best option is to choose a tip with a magnetic moment low enough to produce a magnetic field lower than the coercivity field of the sample, but with coercivity field larger than the dipolar field produce by the sample. However, the magnetic moment of the tip has to be relatively large in order to maximize the interaction with the sample.Figure 3 shows MFM images of the sample, taken with the MM tip at different lift heights. Figure 3(a) is the topographic image of the surface of the sample. Figures 3(b) through (h), are MFM images of the same region with a tip-surface separation of: 25, 50, 100, 150, 200, 250, and 300 nm, respectively. The z scale is 30 nm for image (a) and 80 Hz for images (b) through (h). In these images the magnetic α-phase appears as an island in the center of the images surrounded by the paramagnetic phase. As the tip-surface distance increases, the frequency shift of the cantilever decreases indicating a weakness of the magnetic signal from the sample. The frequency shifts as a function of the tip-surface separation for the MM tip and LC tip are shown in Figures 4(a) and 4(b).

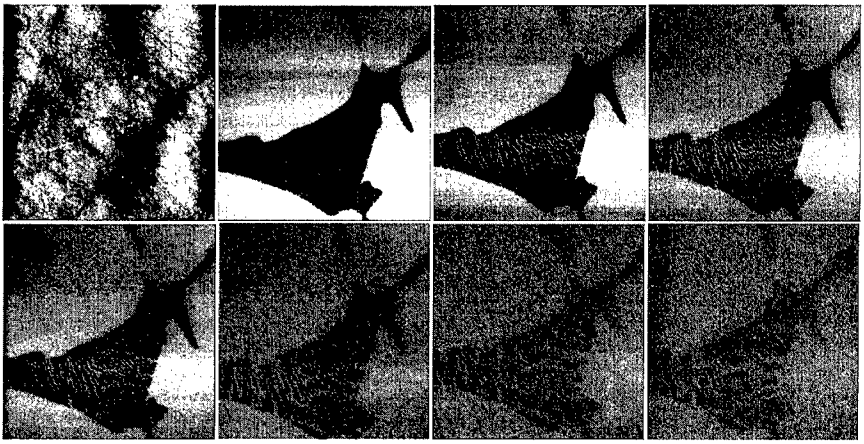

Figure 3. Topographic image (a) of a 50 μm x 50 μm field of the sample, (b) through (h) are MFM images with a tip-surface separation of 25, 50, 100, 150, 200, 250, and 300 nm, respectively.

In order to understand the decrease in the frequency shift with the lift height, a very simple model [5] for the tip-sample interaction was developed. This model considers the interaction between a magnetic probe of magnetic dipole m along the z direction with the stray magnetic field H_z from the sample. In order to calculate the stray field H_z from the sample a very simple approximation is made: considering the magnetic domains can be represented by disks of radius b and magnetization M along the z direction at a distance h from the surface of the sample. The magnetic force gradient Δf_0 in the direction z can be expressed as following equation:

$$\Delta f_0 = \frac{\omega_0}{2k} m \frac{\partial^2 H_z}{\partial z^2} = \frac{3\pi b^2 \omega_0 M m}{k} \frac{z+h}{\left[(z+h)^2 + b^2\right]^{5/2}} \qquad (1)$$

where z is the tip-surface separation, k is the spring constant, and ω_0 is the natural frequency of the cantilever.

The best least squares solid lines in Figure 4 shows fit of expression (1) to the experimental data and the values of the parameters are summarized in Table I.

The fairly good fit to the experimental data shown in Figure 4 corroborates the validity of the model used. Note that the size and depth of the domains found by the MM tip are almost two (1.78) times larger than the respective values found by the LC tip, confirming the remagnetization effect induced by the MM tip in the structure domain of the low coercivity sample. However, the two tips sense the magnetic domains at approximately the same depth.

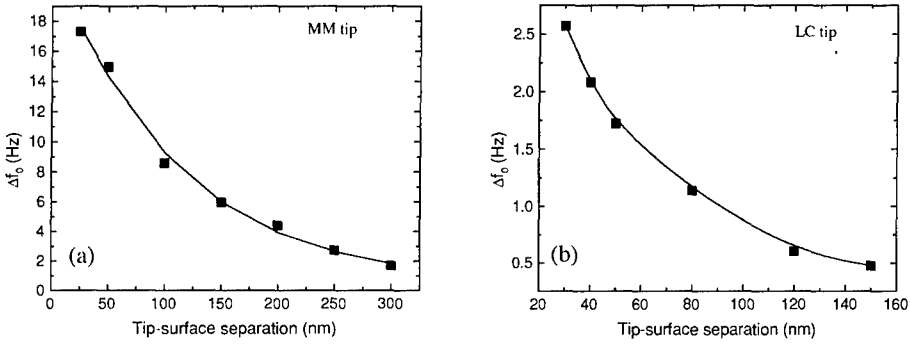

Figure 4. Frequency shift versus tip-surface separation for MM tip (a) and LC tip (b). The squares are experimental data and the lines are the fit using expression (1).

Table I. Parameters for the two tips, HM and LC, obtained by fitting experimental points with expression (1).

Tip	ω_0 (kHz)	b (nm)	h (nm)	Mm' (nm^2)
MM	**55.35**	**187**	**156**	**6.39**
LC	**77.98**	**105**	**130**	**5.00**

CONCLUSION

The MFM technique has been shown to be a powerful tool to identify ferrite phases in Fe-alloys with shape memory effect. Different magnetic patterns were observed when MM, LC, and LM tips were employed. A broadening of the magnetic domains due to the remagnetization effect induced by the MM tip in the sample was observed. A contrast inversion was observed in the MFM images produced by the LC tip. This contrast inversion was related to the rotation of the magnetic moment of the low coercivity tip induced by the relatively strong dipole field of the sample. A high coercivity and low magnetic moment tip proved to be ideal for the study of magnetic microstructure of this kind without inducing misinterpretation of size and orientation of the magnetic domains in the sample. A simple model was used to understand and quantify the tip-sample interaction and the magnetic microstructure of the sample.

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