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Magnetic Force Microscopy Studies of Superparamagnetic Nanoparticles

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Abstract. We have tested ability of superparamagnetic nanoparticles embedded in matrix to be detected by the magnetic force microscopy (MFM). The sample was characterized by X-ray diffraction and by further magnetic studies such as the temperature and field dependence of magnetization ($M$) that served as the starting point for an optimal setup for the MFM imaging. To demonstrate the magnetic origin of the phase contrast, the images were taken both with the magnetic and non-magnetic probes. Because of the strong inter-particle interactions arising from the small distances among the particles, only the clusters of the magnetic nanoparticles have been imaged. The optimization of scanning parameters that depend on the sample properties, as well as the possibility to measure the contrast arising from the single particle embedded in matrix will be discussed.

Superparamagnetic metal oxide nanoparticles have been promising candidates for applications in many areas, favored by variety of routes allowing their fabrication with well-controlled size and shape \cite{1, 2, 3}. Large group of applications of the nanoparticle-based ceramic materials is closely connected with their magnetic properties that are usually studied by macroscopic methods such as measurements of magnetization, a.c. susceptibility and magnetization isotherms. Also the microscopic properties of these materials are investigated by methods sensitive to the local magnetic structure, such as neutron scattering or Mössbauer spectroscopy. However; the interpretation of the hereby obtained results is not always clear and do not provide the entire information about the material. Thus the magnetic force microscopy (MFM) could be the useful local probe, allowing direct observation and visualization of magnetic domain structure, namely the single-domain superparamagnetic nanoparticles in our case.

Despite the detection of the pure magneto-static interaction arising from magnetic nanoparticles has been demonstrated by Agarwal et al. in 2008 \cite{4}, interpretation of these experiments is still complicated by presence of other long-range interactions. Thus one of the crucial points in imaging of magnetic nanoparticles/aggregates is to have the sample in a form, which minimizes the contribution of the topography to the phase contrast. Because of this reason, we have used magnetic nanoparticles embedded in a silica matrix as testing samples for the MFM.

The magnetic sample constituted of the Fe\textsubscript{3}C particles embedded in SiO\textsubscript{2} matrix has been prepared by the convenient sol-gel method. The X-ray diffraction patterns have been taken with the Rigaku XRD...
diffractometer operating with the Mo Kα radiation, the particle size was estimated using the Sherrer formula. The magnetic measurements were taken with the MPMS7 and PPMS9 devices up to the magnetic field of 7 T in the temperature range 2 - 600 K. The zero field cooled (ZFC) and field cooled curves (FC) were measured in the field of 10 and 50 mT, respectively, the magnetization isotherms were measured at 2, 10, 300, 450 and 550 K, respectively. The AFM/MFM images were captured with the Multimode V Veeco microscope, the phase images were taken in the lift scan height of 100 nm and in the static magnetic field of approximately 0.2 T [4] at room temperature. Both the magnetic (MESP) and non-magnetic probes (RTESP) by Veeco were used to prove the magnetic origin of the phase contrast.

The XRD pattern reveals the peak positions corresponding to those of the Fe3C nanoparticles (Fig. 1) with the average particle diameter of 8.5 nm. The ZFC-FC curves (Fig. 2) measured in small magnetic field point at the superparamagnetic nature of the system, the blocking temperatures of the major and the biggest fraction of particles, TMAX and TDIFF, shift to the lower temperatures with increasing field. The discrepancy of these temperatures is a consequence of a significant particle size distribution within the sample (Fig. 1). The low temperature part of the FC curve (inset in Fig.2) points at the presence of the strong inter-particle interactions [5], suggesting the particles are close to each other. To image the magnetic moments of single nanoparticles, it is necessary to have them separated at least in distances longer than the curvature of the tip apex [6].

![Figure 1. The XRD pattern of the sample.](image1)

![Figure 2. The ZFC-FC curves taken in the field of 10 and 50 mT, respectively. The low temperature part in the inset.](image2)

The magnetization isotherms measured at 2, 10 and 300 K exhibit the hysteresis, as is expected for a SPM system in the blocked state. The magnetization isotherm taken at 300 K (Fig. 3) is the important input for the interpretation of the MFM images, highlighting that the saturated magnetization, Ms, is reached in the field of approximately 1 T. Placing the sample on the microscope holder, not all the magnetic moments of the particles are oriented in the direction of the external magnetic field and thus the magnetic contrast should be observed in the MFM phase images.

The average magnetic moment of the particle $\mu = 3.8 \times 10^3 \mu_B$, (which is sufficient for imaging of the particles with the MESP tip [3]) was calculated from the un-hysteretic magnetization isotherms taken at 450 and 550 K by fitting with the weighted sum of Langevin functions with the log-normal distribution of magnetic moments [7] (Fig. 3).
Figure 3. Left—magnetization isotherm at 300 K, the line highlights the value of magnetization in the field of the microscope holder, 0.2 T. Right – isotherm taken at 550 K (dotted) with the fit of the weighted sum of Langevin functions (line), with the distribution of the magnetic moments in the inset.

The images of the sample taken with the non-magnetic tip exhibit the phase contrast that is lower than 1°. Because this contrast in not caused by the magnetic forces, it can be interpreted just as the noise arising from the surface topography, as is demonstrated in Fig. 4. So the topography induced error of the phase contrast measurement could be set to 1°. On the contrary, the images taken with the magnetic tip exhibit the presence of both the positive and negative contrast on the phase image, up to 80° (Fig. 5), as a consequence of the attractive and repulsive forces acting on the magnetic tip. Because both the images taken with the non-magnetic and magnetic probes were taken in the same lift scan heights, operating with almost similar amplitudes, it serves as the evidence that the contrast on the flattened parts of the phase images taken with magnetic tip arises from the long-range magnetic forces. Despite the pure topography, the presence of large areas of the same phase contrast in the flattened areas of the sample, belonging to the fraction of particles with the same orientation of magnetization (yellow ellipse, Fig. 5), confirms the presumption the particles are close to each other and only the clusters of particles have been imaged.

Figure 4. Image taken with the non-magnetic tip. From the left: amplitude, phase image and the cross sections below. The phase contrast represents the measurement error caused by the topography.
Figure 5. Images taken with the magnetic tip in the LSH 100 nm. From the left: height, amplitude error and the phase images, respectively. The flattened parts of the sample (one of them is highlighted by the ellipse) depict the areas of the same contrast – clusters of magnetic nanoparticles.

It could be said in conclusion that it was possible to observe magnetic contrast on the sample of SPM nanoparticles embedded in the silica matrix. Unfortunately because of the high density of the particles in matrix, it was possible only to detect the contrast arising from the clusters of nanoparticles, not the contrast arising from single nanoparticles. To visualize the magnetic contrast of single nanoparticles embedded in matrix, it seems to be necessary to prepare the sample in which the particles are diluted enough, within the distances that are larger than is the curvature of the tip apex. It is also vital to prepare the sample with the smooth surface by adjusting the temperature and the time of drying of the SiO₂ matrix, to minimize the topography caused artefacts in the phase images.

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References