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Atomic force microscopy and magnetization investigation of a water-based magnetic fluid

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Abstract

Atomic force microscopy (AFM) and magnetization measurements were used to unfold the particle-size polydispersity profile of a magnetite-based magnetic fluid sample. The sample preparation and the experimental conditions used to obtain the AFM image are described. The differences found in both the average diameter (D_m) and particle-size dispersion (σ) values obtained from the two techniques are discussed.

Keywords: Atomic force microscopy; Magnetization curves; Magnetic fluids; Particle-size determination

Nanometer-sized particles have attracted a lot of attention, not only because of their basic properties but also due to industrial applications. In particular, the physical, chemical, and physico-chemical properties of magnetic fluids (MFs) are strongly influenced by the details of the distribution in size and shape of the dispersed colloidal nanomagnetic particles. Scanning probe microscopy has emerged as a powerful technique in the analysis of nanometer-sized objects. Besides the usual topographic analysis performed by the atomic force microscopy (AFM), a long list of modified systems based on magnetic [1] and electric [2] characteristics of the tip have been proposed in recent years. Despite of the breakthrough brought about by the AFM technique, sample preparation and artifact observation are still the limiting issues for a wider use of this technology. Therefore, comparison between the data obtained from the AFM technique and traditional techniques, such as magnetization [3], is of great help in establishing the AFM technology for the in-line application. In this work, we report on contact mode AFM imaging of nanometer-sized spherical magnetite (Fe_3O_4) originally dispersed as a stable MF sample. Except for a recent work [4] AFM has not been used to image magnetic nanoparticles in MFs, particularly due to the difficulties introduced by both the absence of a sample preparation procedure and the AFM experimental conditions. The particle-size histogram obtained from the AFM imaging is compared with the particle-size histogram obtained from magnetization measurements.

The MF used in this work was obtained by chemical precipitation of a mixture of iron (II) and iron (III) ions in alkaline aqueous medium. Peptization and stabilization of the magnetite precipitate in water was performed by surface coating the magnetite nanoparticles with a first layer of dodecanoic acid followed by a second layer of an ethoxylated polyalcohol [5]. One drop of freshly peptized MF sample was deposited on top of a 3×3 mm mica substrate. The MF drop was left to dry on top of the horizontally positioned mica substrate in ambient air, under a vertical magnetic field of about 300 G. The AFM used in this study was a Topometrix 2000 Explorer operating in the contact mode and at ambient air. Standard 200 μm V-shaped Si_3N_4 cantilevers with integrated

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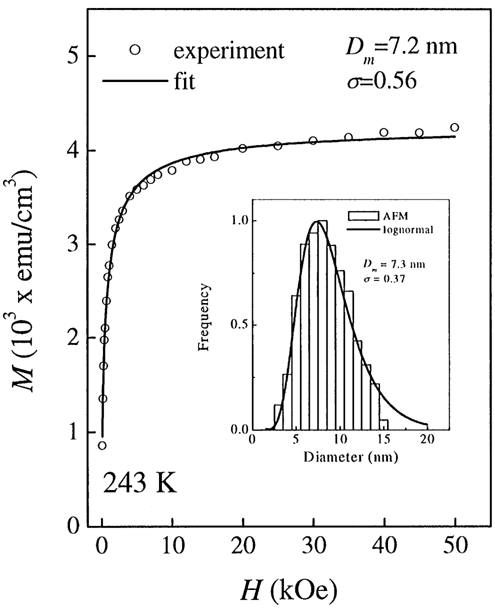

Fig. 1. The field dependence of the magnetization, at about 243 K. Open circles are the experimental data while the full line represents the best fit of the data according to the standard approach. The inset shows the particle size histogram obtained from the AFM data.

pyramidal tips and spring constant of 0.032 N/m was used. The loading force (normal force) was set around 10 nN. The AFM system was calibrated according to the procedure described elsewhere [6]. The magnetization measurements were performed in the range of 4–260 K using a commercial VSM system, previously calibrated with a nickel-standard sample.

The inset of Fig. 1 shows the particle histogram obtained from a typical AFM picture of the surface morphology, taken from an $1.52 \times 1.52\,\mu m^2$ area at a scan rate of 10 μm/s. The solid line in the inset of Fig. 1 represents the best curve fit of the particle-size histogram using the lognormal distribution function [7] with a mean particle diameter of $D_m = 7.3 \pm 0.1$ nm and a standard deviation of $\sigma = 0.37 \pm 0.01$. Open circles in Fig. 1 represent the field dependence of magnetization (M) at about 243 K while the solid line represent the best fit of the data according to the following equation $M(H; D_m, \sigma) = \int L_1(H, D)P(D)\,dD$, where $L_1(H, D) = \coth(\xi) - (1/\xi)$ is the first-order Langevin function and $P(D)$ is the lognormal distribution function. Note that $\xi = \mu H/kT$, with $\mu = M_S V = (\pi/6) M_S D^3$. The model described by $M(H; D_m, \sigma)$ is the traditional approach used to unfold the particle-size polydispersity in MFs [3]. The particle-size distribution obtained from the fitting of the magnetization data, averaged out by the lognormal distribution function, gives $D_m = 7.2 \pm 0.1$ nm and $\sigma = 0.56 \pm 0.01$. As far as the mean particle diameter is concerned the AFM and magnetization data give about the same value. However, the higher standard deviation observed from the magnetization data (0.56) in comparison with the value obtained from the AFM data (0.37) would be explained by the presence of agglomerates (dimer, trimer, etc). The influence of agglomerates upon the magnetic birefringence data obtained from MFs has been recently addressed [8].

In summary, this work provides very useful information concerning the use of the atomic force microscopy for magnetic fluid characterization. The MF sample preparation for contact mode AFM measurements as well as the experimental parameters used during the AFM measurements are described here. The average particle size obtained from the AFM data is very close to the average particle size obtained from the magnetization data. However, the significant difference observed in the standard deviation obtained from the magnetization measurements, in comparison with the AFM data, would be an indication of particle agglomeration.

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