doi:10.1017/S1431927614012331
Microsc. Microanal. 20 (Suppl 3), 2014

Arrays Produced by Electron Beam Nanolithography from Fe_3O_4 Compressed Nanoparticles Targets

Geronimo Perez[1], Elisa Maria Baggio Satovitch[2], Guillermo Solorzano[3]

[1] Materials Metrology Division/Instituto Nacional de Metrologia, Duque de Caxias, Brazil.
[2] Experimental Physics Coordination/Centro Brasileiro de Pesquisas Físicas, Rio de Janeiro, Brazil.
[3] Materials Engineering Department/Pontifícia Universidade Católica, Rio de Janeiro, Brazil.

The semi-metallic Fe_3O_4 films have attracted interest by the characteristic of combining a 100% spin polarization with a high Curie temperature [1] and have a relatively high conductivity [1]. These have been of great interest due to the properties of spin in an insulating material, therefore, are candidates for spintronic applications [2], such as magnetoresistive devices or magnetic tunneling junctions [1, 3]. These magnetic tunneling junctions containing magnetite have been successfully fabricated [3, 4]. It has also been of great interest to study the transition temperature Verwey and transport properties observed in thin films of magnetite [4].

Periodic arrays of basic shapes were produced by electron beam lithography followed by sputtering: a 1 μm squares array and arrays of circles of 1 μm, 500 nm and 250 nm in diameter formed of a magnetite film 80 nm thick.

Before the production of magnetite arrays, several magnetite thin films were produced using the sputtering RF (radio frequency source) deposition system. The thin films were deposited on silicon substrates. The formation of the magnetite after the deposition was confirmed by x-ray diffraction (XRD) and vibrating sample magnetometer (VSM). The magnetite sputtering targets were produced by compression of magnetite nanoparticles previously produced by chemical method of co-precipitation from mixing of iron salts and ammonium hydroxide.

The magnetic properties of the magnetite films were studied by VSM, at longitudinal and transverse (perpendicular to the magnetic field direction) directions. Some samples showed a magnetic saturation near 85 emu/cm^3 at longitudinal direction (easy magnetization direction).

Once selected the optimal sputtering parameters, the nanolithography with lift-off process was started. The first step is the preparation of the on silicon substrate covered of PMMA (polymethylmethacrylate) resist 250 nm thick by spin coat method. At the second step, this substrate was written by electron beam nanolithography and later, immersed into acetone solution for some seconds to remove the exposed areas making the mask. Then, the magnetite film was deposited, by RF sputtering, onto the lithographed mask. Finally the sample was immersed in acetone until all the PMMA film has been lifted-off. The film thickness, shape, size and separation between the figures which comprise standards lithographed can influence the ease with which the mask is withdrawn from PMMA.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) images provide additional topographical information. The SEM is more accurate to determine the diameter of the shapes of a pattern; on the other hand, the AFM provides good thickness information. Figure 1 (a) shows the 1 μm circle array and figure 1 (b) shows a detailed image of a 1 μm circle. Figure 2 show the AFM topography images of the shapes corresponding to different arrays.

Mechanical test showed that the deposited magnetite thin films exhibit good adhesion and abrasion resistance.

References:
[1] H. Takahashi, *et al.*, J Appl Phys, 2003,93: 8029–8031.
[2] X. L. Tang, *et al.*, Journal of Solid State Chemistry 179 (2006) 1618–1622.
[3] K. I. Aoshima and S. X. Wang, J. Appl. Phys. 93 (2003) 7954.
[4] L. Pan, *et al.*, Thin Solid Films 473 (2005) 63–67.

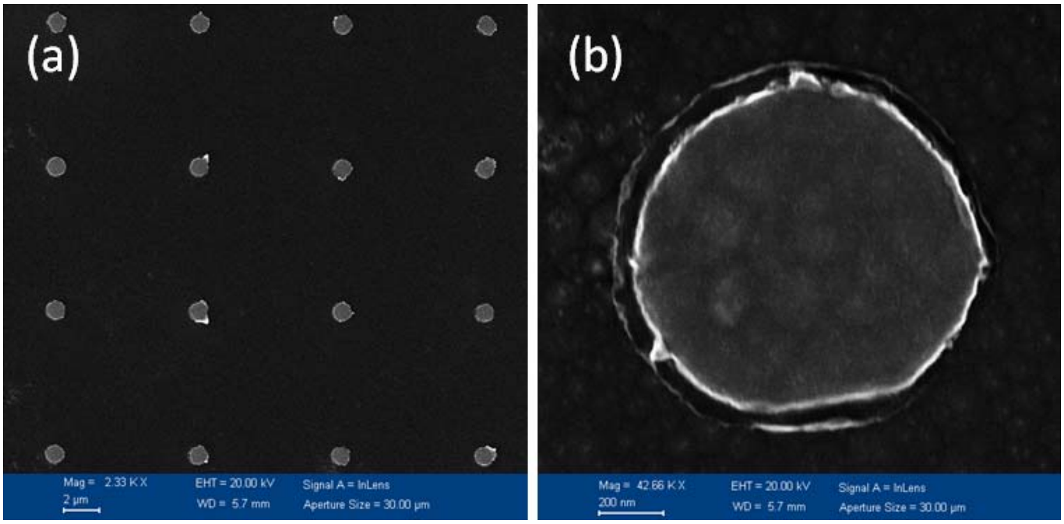

Figure 1. SEM micrographs of: (a) Magnetite array of circles of 1 µm diameter, (b) detail of a circle of the same array.

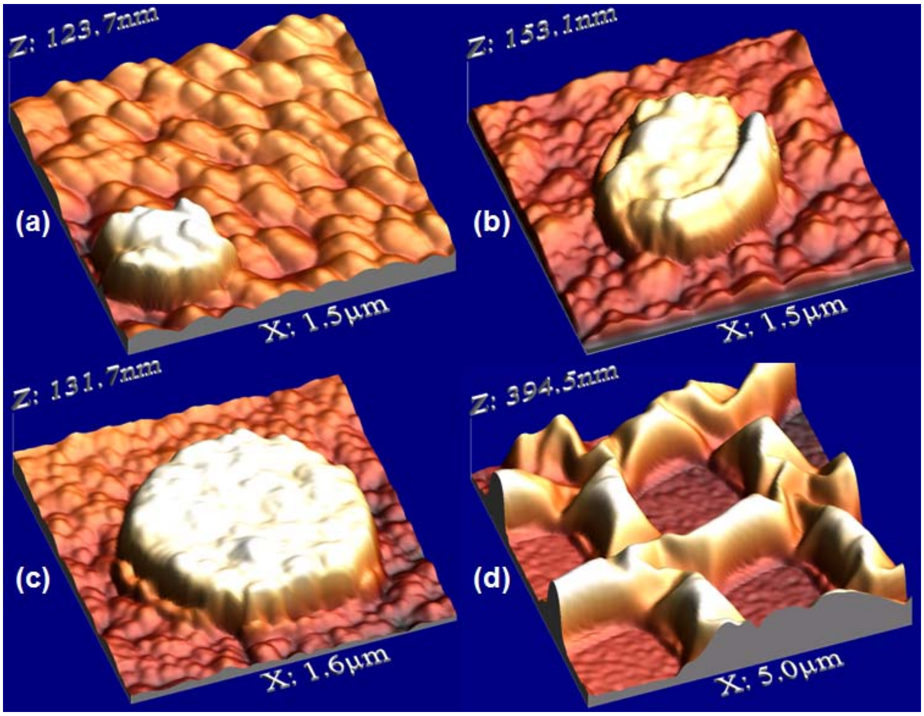

Figure 2. AFM images of the shapes corresponding to different arrays: (a) 250 nm circles, (b) 500 nm circles, (c) 1 µm circles, (d) 1 µm squares.