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CORE-SHELL IRON OXIDE — CARBON NANOPARTICLES MODIFIED WITH Ag. SYNTHESIS, MORPHOLOGY, MAGNETIC PROPERTIES

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Core-shell nanoparticles (CS NPs), including magnetic core — carbon — noble metal are considered now as key nanomaterials for numerous applications [1]. Properties of such structures depend critically on the preparation technique, NPs size, morphology and chemical composition. Here we investigate the structures consisted of iron oxide — carbon CS NPs modified with Ag in dependence on the Ag concentration.

Samples were synthesized by pyrolysis of $Fe(NO_3)_3 \cdot 9H_2O$ dissolved in solution of oleylamine (OLA) and oleic acid. The solution was heated in several steps. After cooling, the product was washed with a mixture of hexane and ethanol, and magnetically separated. Thus magnetite particles coated by carbon shell were formed. These particles were heated in the mixture of $AgNO_3$ and OLA to 160 °C under stirring. Last reaction proceeded for 0.5 hour. The atomic molar ratios of Ag/Fe in samples were varied from 0 to 0.34.

The synthesized NPs were investigated using transmission electron microscope (TEM) JEM-2100 (JEOL Ltd.) operating at the accelerating voltage of 200 kV, equipped with an energy dispersive X-ray spectrometer (Oxford Instruments). The powder difraction data were collected at room temperature with the Bruker D8 ADVANCE powder difractometer (Cu-K_α radiation) and linear VANTEC detector. Rietveld refinement was performed by using TOPAS 4.2 [2]. Magnetization field dependences at room temperature were measured with vibrating sample magnetometer. Mössbauer spectra were obtained with the MS-1104Em spectrometer in the transmission geometry at 300 K, radioactive source Co57 (Rh). To carry out the MCD measurements, transparent composite plates containing the nanoparticles were prepared. MCD was measured in the normal geometry: the magnetic vector and the light beam were directed normal to the plate's plane. The MCD value was measured as the difference between the absorption of right and left polarized light waves ($\Delta D = D_+ - D_-$) in the spectral range 1.2–3.3 eV in a magnetic field 13 kOe at the temperatures 90 and 300 K.

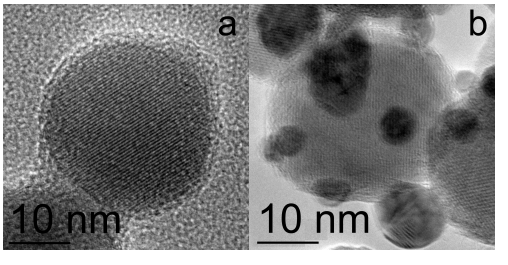

Figure 1. TEM images of samples containing no Ag (*a*), and with Ag/Fe ratio=0.25 (*b*)

Typical TEM images are shown in Fig. 1 for samples containing no Ag and with Ag/Fe ratio=0.25. Atomic planes and carbon shell are seen very well in the first case. Small Ag NPs are located around magnetic NPs. The Rietveld plot for sample with Ag/Fe=0.34 (Fig. 2). All observed peaks were indexed by two cubic phases with parameters close to Fe_3O_4 and Ag compounds. So we can say that silver

presents as separate crystalline NPs. Unfortunately, the interplanar spacing of the Fe_3O_4 crystal differs little from the crystal spacing of the other iron oxide phase — γFe_2O_3 and TEM XRD data give no ambiguous answer on the NPs crystal phase. To elucidate this point, we studied Mössbauer spectra. The analysis of these spectra showed the coexistence of two magnetic phases in the NPs core — Fe_3O_4 and γFe_2O_3.

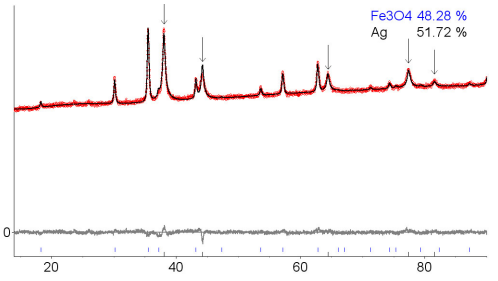

Figure 2. Difference Rietveld plot of sample with Ag/Fe=0.34. Arrows indicate Ag peaks

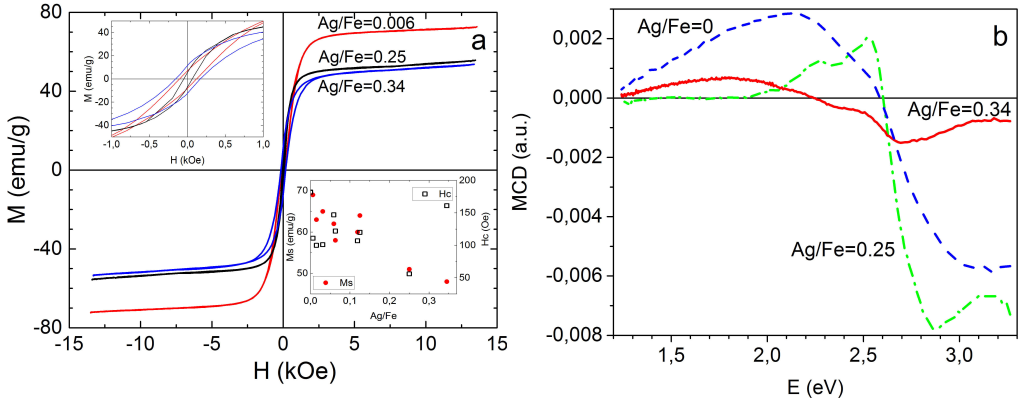

Figure 3. (*a*) — Hysteresis loops for several samples; (*b*) — MCD spectra for samples containing no Ag and with Ag/Fe ratio=0.34

Magnetization curves for all samples demonstrate hysteresis with saturation field near 3 kOe and coercive field varying from 50 to 190 Oe. These values change non-monotonous as the Ag/Fe ratio changes.

MCD spectra (Fig.3 (*b*) appeared to be very sensitive to the core magnetic phase. They reflect transition from predominantly Fe_3O_4 phase to the γFe_2O_3 phase in accordance with the Mössbauer effect data. Experiments continue to establish the relationship of the phase composition of the nanostructures with the amount of silver during their synthesis.

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