Tailoring particle size via chemical composition: structural and magnetic characterisation of nanocrystalline Ni-Co alloys

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Ferromagnets embedded in a dielectric matrix display in many cases enhanced chemical and physical properties and, therefore, have potential technological applications. From this point of view, nanoparticles of ferromagnetic alloys are of particular interest since their magnetic properties can be altered by varying both chemical composition and particle size [1]. In our opinion, a promising approach to fabrication of such composites lies in the filed of intercalation chemistry.

In this work, nanoparticles of cobalt-nickel alloys encapsulated in a matrix of mixed lithiumaluminum oxides have been investigated. They were prepared by heating a precursor compound, layered double hydroxide, containing co-intercalated complexes of two metals $[LiAl_2(OH)_6]_2\{(Ni_{1-x}Co_x(EDTA)\}\cdot 4H_2O \text{ (where } EDTA - ethylenediaminetetraacetate, x = 0 ÷ 1).$ The homogeneity of the Co-Ni composition in the obtained alloys was proved by the Vegard linear dependences of the *fcc*-cell parameter as well as by supplementary examinations using EDX.

In order to characterise the microstructure and morphology thoroughly we used different methods, which provide local (HRTEM-LAED-EDX) and statistical (XRD and SAXS) information from a sample (Fig.1-2). The data obtained by these methods turned to be in good agreement with each other. It has been found that upon enrichment of Ni_{1-x}Co_x-alloy by cobalt up to x = 0.86, the particles gradually enlarge from $d_{TEM} = 6$ to 16 nm preserving their shape and size distribution. This notably differs from our previous results obtained for pure Co particles prepared in the same manner which were found to be of much larger sizes and wide size distribution [2].



Figure 1. TEM image of nanoparticles of $Ni_{0.34}Co_{0.66}$ -alloy encapsulated in a lamellar matrix of $Li_2O\cdot 2Al_2O_3$ (view along *c*-axis).



Figure 2. SAXS patterns (left) and distance distribution probability functions (right) of nanoparticles of $Ni_{1-x}Co_x$ alloys. (In parentheses: gyration radius R_g)



Figure 3. Magnetisation dependences M(T) obtained using a SQUID magnetometer in ZFC-FC regimes.



Figure 4. Ferromagnetic resonance spectra

We used FMR and SQUID magnetometry to characterize magnetic properties of the alloy nanoparticles (Fig.3-4). It has been found that $Ni_{0.14}Co_{0.86}$ ($d_{TEM} \approx 16$ nm) is ferromagnetic whereas alloys $Ni_{1-x}Co_x$ ($0 \le x \le 0.66$, $6 \lesssim d_{TEM} \lesssim 9$) are superparamagnetic at room temperature. The blocking temperatures of the latter measured in ZFC-ZC regimes occur in the range 22 K < T_b < 250 K depending on particle size. It has been also revealed that the particles form antiferromagnetic oxide shells of $Ni_{1-x}Co_xO$ that was proved by observation of bias-exchange coupling at low temperatures. The effective anisotropy constants were estimated to be $0.6-1.7\cdot10^5$ J·m⁻³ at low temperatures and $\le 2\cdot10^4$ J·m⁻³ at 20°C.

Varying the alloy chemical composition can be a promising way of tailoring the particle size and target magnetic properties. The approach studied in this work is also of interest in designing efficient bi-functional catalysts for concurrent basic and hydrogenation reactions. The comparison and peculiarities of utilisation of different instrumental methods used in this work will be also discussed.

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References:

- [1] J. Bansmann, S.H. Baker, C. Binns et al Surf. Science Reports 56 (2005) 189–275.
- [2] K.A.Tarasov, V.P.Isupov, B.B.Bokhonov *et al* J. Mater. Synth. Proc. **1** (2000) 21-27.