

INFLUENCE OF SYNTHESIS CONDITIONS ON THE PROPERTIES OF $\text{Fe}_x\text{Co}_y\text{O}_z$ - NANOPARTICLES IN SiO_2 SOL-GEL FILM

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Abstract:

Synthesis conditions influence on structure of $\text{Fe}_x\text{Co}_y\text{O}_z$ - nanoparticle in SiO_2 - sol-gel coating is discussed. The samples are synthesized by hybrid sol - gel method including following stages: TEOS hydrolysis, introduction into sol of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, deposition of film-forming sol by spin-coating on silica substrate, heat treatment at the temperature from 200 up to 900 °C. The Synthesis conditions influence on a specific structural property of sol gel films is investigated by AFM.

Keywords: nanoparticle, AFM, $\text{Fe}_x\text{Co}_y\text{O}_z$, phase change, primitive and face-centered cubic lattice.

1. Introduction

Unique physical characteristics of nanoparticles arising due to surface or quantum-size effects, are object of intensive researches now [1]. The special place in this investigation is taken by the magnetic characteristics of nanoparticles. Most difference (sometimes very essential) between compact magnetic materials and the same are detected and the theoretical base which is capable to explain the majority of observed effects.

A number of general methods of nanoparticles obtaining [2] is developed now; the majority of them can be used for obtaining of magnetic nanoparticles. But the synthesis of magnetic nanoparticles has the essential singularities; they can be formulated as the requirements to know-how of synthesizing of magnetic nanoparticles. It is necessary to synthesize the nanoparticles of the given size and shapes, at all events, the dispersion on the sizes should be small (5-10 %) and can be controlled. Differently, changing the sizes, shapes, structure of nanoparticles, it is possible in definite limits to control the magnetic characteristics of materials on the base of magnetic nanoparticles. All this allows to use the materials including nanoparticles in perspective systems of a record and storage of information, for creation of new permanent magnets, in systems of magnetic cooling, as magnetic sensors etc.

Using the sol - gel technology it is possible to synthesize the sol - gel matrix with different structure possessing much higher thermostability and chemical stability than polymer matrices.

The main purpose of the research is the investigation of magnetic properties sol-gel films including ferromagnetic $\text{Fe}_x\text{Co}_y\text{O}_z$ nanoparticles. Control of the nanoparticles size in sol-gel coatings by doping at various sol-gel process stages by different ratio of such elements as iron and cobalt will allow to use these films in electronics,

as data carriers with high density, and also as sensors of electromagnetic radiation.

2. Experimental

The formation of sol-gel film takes place as a result of hydrolysis of film-form solution chemical agents and their interplay to surface layer of a substrate. For example, film of a silicic acid derived as a result of a full hydrolysis of solutions applicable alkyne compounds, are consolidated at the expense of covalent linkages Si-O-Si. The formation of such band was conducted by a way of hydrolysis and polycondensation of tetraethoxysilane (TEOS) and alkyl-displaced alkoxy silane and ferrum carbonate and cobalt nitrate in organic solution.

The main stages of sol-gel process are showed in Fig. 1.

Then the film-form solutions were deposited on a substrate by spin-coating or dip-coating.

Silicon wafers and quartz glass were applied as the substrates. Just after the deposition the samples were heat treated stepwise in a muffle during 20 minutes at the temperature from 200 to 900 °C.

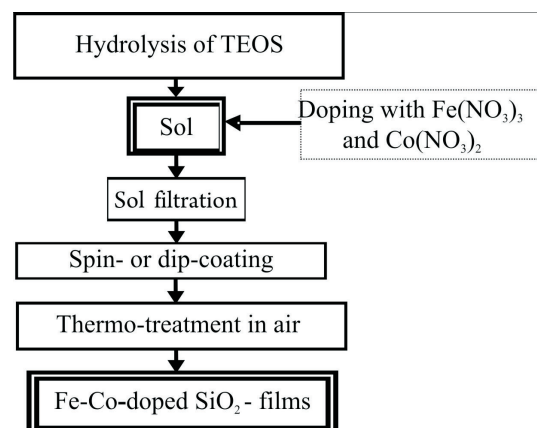


Fig. 1. The main stages of sol-gel process.

$\text{Fe}_x\text{Co}_y\text{O}_z$ sol-gel films were transparent and homogeneous with light green colour before heat treatment and from golden up to red after heat treatment at 500 °C within 15 minutes.

The analysis of phase changes in the synthesized matrixes has been carried out using the XRD method. The examination of doped film surfaces has been performed by AFM (SOLVER P 47 - PRO «NT-MDT») which allows for studying structural formations in the nanometer scale.

The magnetic characteristics of the formed silicate sol-gel films doped with $\text{Fe}_x\text{Co}_y\text{O}_z$ nanoparticles have been studied by ballistic method of construction of hysteresis

loop of ferromagnetic materials at different strength of external magnetic field.

3. Results

In Figure 2 the surfaces images of $\text{Fe}_x\text{Co}_y\text{O}_z$ SiO_2 sol-gel films on the silicon plane (100) are shown. Films are deposited by dip-coating and annealed on air at 800 °C. As can be seen, sol - gel coating with different ratio between ferrum and cobalt compounds, have the brightly expressed features of structure. The sample in Figure 2(a) has not the expressed features of structure. At the ratio of ferrum and cobalt compounds equal 1:1 on the AFM image are clearly visible nanoparticles with the mean size 50-60 nm and cubical shape. With a large degree of accuracy all particles have the identical size. The size of nanoparticles increase with the increasing of cobalt content. The main role on the forming of cubical nanoparticle shape is played the crystalline pattern with the symmetry of the silicon plane (100)). By XRD data, the type of structure of nanoparticles is face-centered (the temperature heat treating is 800 °C). The unit cell of $\text{Fe}_x\text{Co}_y\text{O}_z$ nanoparticle, erased in the SiO_2 -film doped with ferrum and cobalt compounds (1:1), is shown in Figure 3. The image of the unit cell is obtained through the program Powder Cell 2.0 [3].

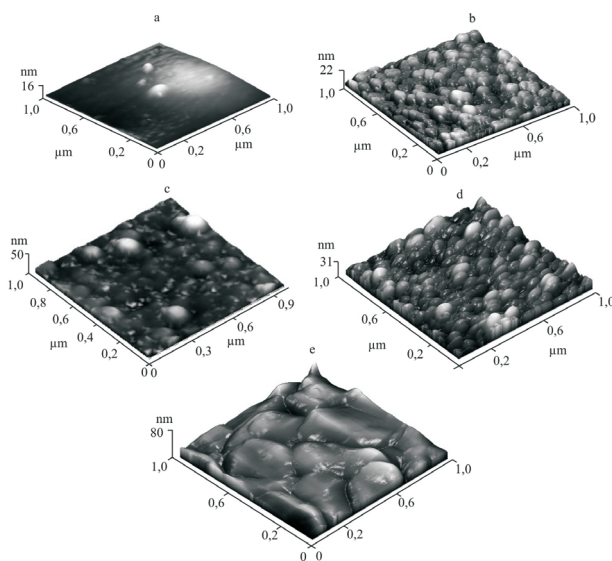


Fig. 2. AFM images of $\text{Fe}_x\text{Co}_y\text{O}_z$ - SiO_2 sol-gel films on the silicon plane after heat treatment on air at the temperature 800 °C with the ratio between ferrum and cobalt compounds: a-1:0,25; b-1:1; c-1:0,5; d - 1:2; e-0:1.

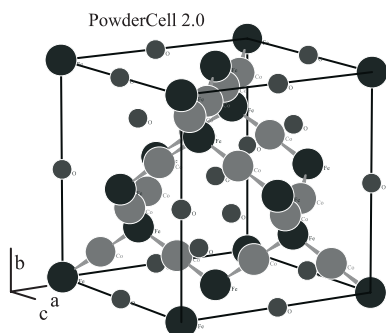


Fig. 3. The unit cell of $\text{Fe}_x\text{Co}_y\text{O}_z$ nanoparticle, erased in the SiO_2 -film doped with ferrum and cobalt compounds (1:1).

The hysteresis loop for $\text{Fe}_x\text{Co}_y\text{O}_z$ -doped film-forming sol is characterized by the magnetization $B_s = 12$ Gs, the coercive force $H_c = 68$ a/m, and the residual magnetic induction B_r about 2 Gs. The hysteresis loop of xerogel differs from that of film-forming sol by the magnetization value $B_s = 14$ Gs.

4. Conclusion

SiO_2 -films with $\text{Fe}_x\text{Co}_y\text{O}_z$ nanoparticles are synthesized by sol-gel on silicon substrate under heat treatment temperature 800 °C. At the ratio of ferrum and cobalt compounds 1:1 the nanoparticles with the mean size 50-60 nm and cubical shape are arise. The size of nanoparticles increase up to 500 nm with the increasing of cobalt content. The main role on the forming of cubical nanoparticle shape is played the crystalline pattern with the symmetry of the silicon plane.

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