Babeş-Bolyai University Faculty of Physics

# Scientific Report for Project PN-II-RU-TE-2014-4-2360

## **October-December 2015**

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#### **1. Project Objectives**

This project intends to approach the study of the exchange spring concept in thin films experimentally as well as theoretically. An important aspect from theoretical point of view is the dimensionality effect, the variation of magnetic moments at surfaces and interfaces being correlated with magnetic anisotropy. Therefore magnetic ordering of thin films by full relativistic band structure calculations will be investigated in order to give a clear input to the experimental part of the project. Fully epitaxial SmCo<sub>5</sub>/Fe(Co)/SmCo<sub>5</sub> trilayer films are to be prepared, in which the easy magnetization axis of both hard magnetic layers is aligned along one single direction.

The dependence on the Ar pressure, temperature and thickness of the substrate will constitute an important aspect during this research project. The interlayer exchange coupling will be studied by hysteresis measurements and by DC-demagnetizing loops for different thicknesses of the soft magnetic thin film. A successful coupling together with the optimum texture provided by the epitaxial growth of the hard magnetic thin film would lead to excellent magnetic properties. We are aiming for small thicknesses for the soft magnetic material (raging from a few nm) in order to get fully coupled layers. Hysteresis curves with high coercivity and large remanence are expected.

#### 2. Results Summary for October-December 2015

In this report we present a summary of the experimental results obtained during the first stage of the project, October-December 2015. For this first stage we focused on the first objective of the project, namely the study of hard magnetic multilayers.

Ferromagnetic (FM) thin films with strong out-of-plane magnetic anisotropy have been widely studied for perpendicular magnetic recording media applications [1, 2]. Perpendicular exchange spring systems consist of exchange-coupled hard and soft magnetic layers with out-of-plane and in-plane easy magnetization axes respectively. While the hard magnetic film provides thermal stability, the soft magnetic layer reduces the reversal field. When an external magnetic field is applied, the soft layer reverses first which creates an additional effective field applied to the hard phase through exchange coupling, lowering the switching field of the whole system. The exchange-spring concept essentially opened an alternative route towards new high-performance hard magnetic materials. By associating a coercive hard magnetic phase with a large magnetization soft phase it is hoped that new high-energy product materials could be produced. It is also envisaged that exchange-coupling would favor remanence enhancement and thus low-cost isotropic materials could be obtained showing large remanence and medium coercivity. Up to now no materials have been found with properties clearly superior to those of usual hard magnetic materials or a lower cost of fabrication. Modern permanent magnetic materials, like SmCo<sub>5</sub> and Nd<sub>2</sub>Fe<sub>14</sub>B, are based on intermetallic compounds of rare-earth and 3*d* transition metals. Sm-Co intermetallics are hard magnetic materials with a high coercive field and a high, uniaxial, magnetocrystaline anisotropy, where the easy axis is aligned along the crystallographic *c*-axis. The control of the composition and the crystallographic texture are the key parameters to obtain thin films with the desired hard magnetic properties.

Understanding the effects of the Ar pressure and substrate on the hard magnetic phase is one of the main goals of the first task. Varying these two very important parameters, one can trigger the complexity of the Sm-Co phase. A Cr buffer layer forms an adequate base for growing Sm-Co by helping to maintain the in-plane anisotropy of SmCo<sub>5</sub>. Also, the Cr layer prevents the Sm atoms from interacting with the thermally oxidized Si substrate. A large magnetocrystalline anisotropy has been found in the SmCo<sub>5</sub> phase [3], an anisotropy which can overcome the magnetic instability. By tuning the pressure in the equation of deposition we can influence the hard magnetic thin film obtaining the requested magnetic coervicity and energy product that defines the hard phase. In the case of epitaxial growth, the deposited atoms grow in registry with the substrate due to atomic bonding, thereby reducing the surface energy due to the low mismatch degree. Epitaxial growth tends to occur when the lattice mismatch between the substrate and the deposited materials is small, as the elastic energy competes with the surface energy. Since most materials have distinct equilibrium lattice constants, epitaxy leads to strained thin films with lattice constants that differ from the bulk equilibrium value (at least during the initial stages of growth). In our case, the lattice mismatch between  $SmCo_x$  and Cr(110) is below the value which would lead to an uncontrolled growth of the hard magnetic thin film.

Sm-Co thin films were prepared by DC magnetron sputtering of a 1:5 Sm-Co target on a SiO<sub>2</sub>(100 nm)/Si-p(100) coated with 9 nm of Cr. The Cr layer was used as a seed layer due to the excellent lattice matching between Cr and Sm-Co, orienting the *c*-axis of Sm-Co in a direction perpendicular to the film. The Cr and Sm-Co layers were deposited in a base pressure of 7 x 10<sup>-7</sup> mbar. The deposition temperature of the Cr seed layer was 500°C, while the Sm-Co and Cr cap layers were deposited at room temperature. The deposition rates were 0.9 Å/s for Cr layers and 0.2 Å/s for Sm-Co layers. Three Cr/Sm-Co/Cr films were prepared, the Ar partial pressure for the Sm-Co deposition being  $1.37 \times 10^{-3}$  mbar,  $1.66 \times 10^{-3}$  mbar and 2.92 x 10<sup>-3</sup> mbar respectively. The Ar partial pressure value was kept constant at 5.20 x 10<sup>-3</sup> mbar during all of the Cr layer depositions. For each Sm-Co thin film that was obtained, a 2 nm cap layer was deposited from a pure solid Cr target (99.9% purity) in order to protect the film from oxidation. After deposition, the films were annealed under Ar atmosphere at a temperature of 600°C followed by guenching in water. The layer thicknesses and deposition rates were measured by reflectometry and Rutherford back-light scattering (RBS) measurements. The crystal structure of the films was investigated using a Bruker D8 Advance diffractometer with Cu  $K_{\alpha}$ radiation.

The results of the thickness measurements, Figures 1 and 2 and Table 1 respectively, show that for both the Cr and the Cr/Sm-Co/Cr films, the initial Cr seed layer is 9.20 nm thick. The Sm-Co layer is 5.42 nm thick, while the Cr cap layer thickness was determined to be around 1.84 nm.

Sample	Layer	Thickness (nm)
SiO <sub>2</sub> (100nm)/Si-p(100)/Cr film	Cr	9.20
SiO2(100nm)/Si-p(100)/Cr/Sm-Co/Cr film	Cr Sm-Co Cr	9.20 5.42 1.84

 Table 1. Cr and Sm-Co layer thickness values for the as-deposited SiO<sub>2</sub>(100nm)/Si-p(100)/Cr and SiO<sub>2</sub>(100nm)/Si-p(100)/Cr/Sm-Co/Cr film samples obtained from reflectometry measurements.



Figure 1. Measured and calculated profiles of X-ray reflectivity (a) and density profile for the asdeposited SiO<sub>2</sub>(100nm)/Si-p(100)/Cr film (b).



Figure 2. Measured and calculated profiles of X-ray reflectivity (a) and density profile for the asdeposited SiO2(100nm)/Si-p(100)/Cr/Sm-Co/Cr film (b).

The X-ray diffraction (XRD) patterns of the as-deposited and annealed films are shown in Figure 3. The Sm-Co layer in the different samples was deposited at different partial Ar pressure values, denoted by  $p_1 = 1.37 \times 10^{-3}$  mbar,  $p_2 = 1.66 \times 10^{-3}$  mbar and  $p_3 = 2.92 \times 10^{-3}$  mbar. For lower partial Ar pressures ( $p_1$  and  $p_2$ ), the as-deposited films are amorphous, no Cr or Sm-Co XRD peaks appearing in the patterns. However, for a higher partial Ar pressure ( $p_3$ ), the (210) peak of SmCo<sub>5</sub> is clearly visible, while other SmCo<sub>5</sub> peaks are absent. This indicates a texturing of the as-deposited Sm-Co layer along the (210) direction.

After annealing, the (210) peak of  $SmCo_5$  becomes visible the sample deposited at  $p_2$  due to the recrystallization of  $SmCo_5$  during annealing, the recrystallization temperature of  $SmCo_5$  being 450°C. For the sample deposited at  $p_1$  the Sm-Co layer is still amorphous. It is worthwhile to note that for the annealed film having Sm-Co deposited at  $p_3$ , the  $SmCo_5$  (210) peak intensity decreases, possibly due to the film becoming less textured because of  $SmCo_5$  growth.



**Figure 3.** XRD patterns for the as-deposited and annealed film samples. The simulated XRD patterns of Cr and SmCo<sub>5</sub> are also shown for comparison. For clarity, the XRD patterns were shifted vertically.

#### 3. Conclusions and Perspectives

The research results show that the first objective was accomplished for polycrystalline films. The effect of the partial Ar pressure on the crystal structure of the Sm-Co layer in Cr/Sm-Co/Cr films was investigated. It was found that for higher partial Ar pressures during deposition, the as-deposited film is textured along the (210) direction. After annealing, the SmCo<sub>5</sub> phase crystallizes with texture still being present in the films. For the next phase of our research, we will focus our efforts into the preparation of epitaxial hard magnetic Sm-Co and soft magnetic Fe-Co-based films, studying the effects of the substrate and deposition conditions on the structure, microstructure and magnetic properties of these multilayered films.

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