Interface and Annealing Effects in FeCo-Si Magnetic Multilayers

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The performance of multilayer systems used e. g. for the reflection of x-rays or neutrons critically depends on the interface structure. Especially if magnetic layers are involved, as in our case of the production of polarising neutron supermirrors, diffusion processes at the interface which reduce the magnetisation severely disturb the function of the system.
We investigated FeCo-Si magnetic multilayers which were prepared in a triode sputtering system and deposited on float glass substrates with an rms roughness of about 6 Å. While the thickness d_m of the FeCo -layers is varied from 5 to 6100 Å the Si-layers thickness was fixed at 60 Å. On top a layer of 200 Å Si was deposited to prevent oxidation during the annealing experiments.
The thicknesses of the layers were determined by RBS, x-ray and neutron reflection with errors below 5 %. The mean roughness of the interfaces was determined to be 12 ± 2 Å. Here x-ray and neutron experiments gave the same value, thus no additional magnetic roughness was seen.
Fe_XSi_{1-X} alloys show ferromagnetism for Fe concentrations above 60 at.%. We assume the existence of mixed layers at the interfaces being composed of Si and Fe and Co atoms which became nonmagnetic due to mixing with Si. Neglecting any gradients due to the diffusion process, the initial FeCo layer of thickness d_m can be divided into an effective dead layer of thickness d_D and the residual ferromagnetic layer. d_D describes only the amount of the FeCo atoms in the mixed layer, measured in Å. Within d_D the saturation magnetisation $M_S = 0$, in the residual layer M_S is equal to that of bulk crystalline FeCo. Thus the total magnetisation is given by:
$M_s(T, d_m) = M_0(T)*(1- d_D(T)/d_m)$,
where $M_0(T)$ is the specific saturation magnetisation of the crystalline FeCo bulk and $M_S(T, d_m)$ that of the magnetic layer with the thickness d_m at temperature T. d_D is assumed to have the same thickness for all samples with the same annealing history.
The magnetic measurements were performed using a SQUID from Quantum Devices Inc. Values for the experimentally determined saturation magnetisation M_S are given in the poster for the samples as produced and after annealing for 48 hours at 250, 350 and 450°C. The measurements were performed at room temperature and for the untreated samples additionally at 5 K. With decreasing d_m also M_S decreases. Fitting the values with the formula given above and $M_0(T) = 1800$ kA/m leads for the untreated samples to thicknesses for the dead layers d_D of 10 Å at room temperature and 5 Å at 5 K. Upon annealing the layers grow up to a saturation value of 40 Å, due to the finite amount of Si. Annealing for 48 hours leads to nonmagnetic thicknesses d_D of 24, 37 and 40 Å for annealing temperatures of 250, 350 and 450°C respectively.
Spin polarised neutron reflection (PNR) was also applied to determine d_D. This resulted in values of 9 ± 2 Å for the untreated samples. For three thick samples d_D was determined after annealing for 2 h and 16 h at 250°C to be 15 ± 3 Å and 18 ± 3 Å. The corresponding values from the SQUID measurements were 18 ± 1Å and 21 ± 1 Å. It seems that PNR gives in this case values which are systematically lower by 10 - 15% though they still coincide within the error limits.

Assuming simple diffusion for the formation of the interface layer:

$$d_D = 2\sqrt{Dt} \qquad \text{and} \qquad D = D_0\, e^{-\frac{E}{kT}}$$

with the diffusion constant D_0, activation energy E, temperature T and annealing time t, one finds E = 0.95 eV and $D_0 = 7.10^{-14}$ m^2/s. Sharma gives two linear expressions relating E and D_0. They are different for diffusions in either a crystalline or an amorphous material. The values given above can be fitted reasonably well by the relation for the diffusion in an amorphous material. Since Si grows amorphously during sputtering while the FeCo alloy layers consist of micro-crystals it can be concluded that the Fe and Co atoms diffuse into the Si. TEM pictures show a clear decrease in the thickness of the Si layers during annealing.
Using electron diffraction in the TEM at a sample annealed for two hours at 450°C with thick interface layers this material could be identified as consisting mainly of micro-crystals of the silicide $Fe_{0.11}Co_{0.89}Si$ which are oriented in different directions.