



Vector magnetisation measurements on thermally evaporated CoCr multilayers and solid solutions for spintronic applications

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ABSTRACT

Structure, morphology and magnetic properties of thermally evaporated Co/Cr multilayers and of the solid solution obtained by isothermal treatment of the layered samples have been studied to evaluate their potential application as high-coercivity materials for hard spin injectors in spintronic devices such as magnetic tunneling junctions. The thermal treatment was performed in a partial pressure of inert gas to allow interdiffusion of Cr and Co. Structure and composition of the materials, as well as the effects of the annealing treatment, have been investigated by field emission scanning electron microscopy and energy-dispersive X-ray analysis. The magnetic configuration of these films was investigated by magnetic force microscopy. Simultaneous measurements of two mutually perpendicular magnetisation components were performed by means of a vector vibrating sample magnetometer, and showed that the in-plane coercivity can be increased by proper thermal treatment from less than 10 Oe to about 240 Oe. However, annealing may result in a loss of compositional and morphologic homogeneity of the films.

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1. Introduction and experimental

Spin-polarised transport properties in FM/insulator/FM structures have been extensively studied in the last years for their application to data storage [1,2] owing to the large tunneling magnetoresistance (TMR) effect observed at room temperature [3].

In magnetic tunneling junctions (MTJ), the magnetic moment of the reference electrode is typically exchange-biased by an AF pinning layer to minimise the magnetostatic coupling with the soft storage layer. An efficient spin-polarised current injection is important for the development of devices based on spin manipulation, like magnetoresistive random access memories (MRAM) [4] and high density read heads [5].

In this work, two different series of thermally evaporated Co/Cr multilayers and a solid solution obtained after annealing one of the two layered samples have been investigated by a comparative study of morphology and magnetisation. This has been done to assess the multilayer properties and understand how diffusion processes induced by annealing influence their morphological and magnetic features for prospective applications of these materials as hard spin injectors. Two different stacks have been evaporated on Si(100) substrates, fixing the Co to Cr ratio (4:1) and varying

the total thickness (30 and 60 nm). They will be referred to as C1: Si/(Co₅/Cr₁)⁴/Co₆ and C2: Si/(Co₁₆/Cr₅)²/Co₁₈, where the subscript indicates the nominal thickness in nm and the superscript indicates the number of repetitions. The evaporation took place in HV (<1 × 10^{−7} Torr).

It has been reported that in Co/Cr layers, a post-deposition thermal treatment induces interdiffusion of metal atoms and allows the Co grain to segregate within the solid solution, this phenomenon being responsible for the increase of sample coercivity [6]. Annealed samples bear more resemblance to solid solutions than layered systems; however, the annealing process may result in undesired alteration of sample morphology and must be studied in detail.

As a consequence, we operated a 4 h primary annealing at 450 ± 1 °C on a C1-batch sample, preceded by a 10 min isothermal treatment at 80 ± 1 °C to promote water desorption and avoid surface oxidation phenomena. During heating, isothermal treatment and subsequent cooling a partial Ar pressure of 1 mTorr has been kept to allow heat diffusion (base chamber vacuum <5 × 10^{−7} Torr).

Morphology and grain texture of our samples were investigated by means of field effect scanning electron microscopy (FESEM) with energy-dispersive X-ray microanalysis (EDX). Magnetic images were collected at room temperature in air after out-of-plane demagnetisation by lift mode magnetic force microscopy (MFM) using commercial tips (MESP Co–Cr coated), in air at room temperature after out-of-plane demagnetisation.

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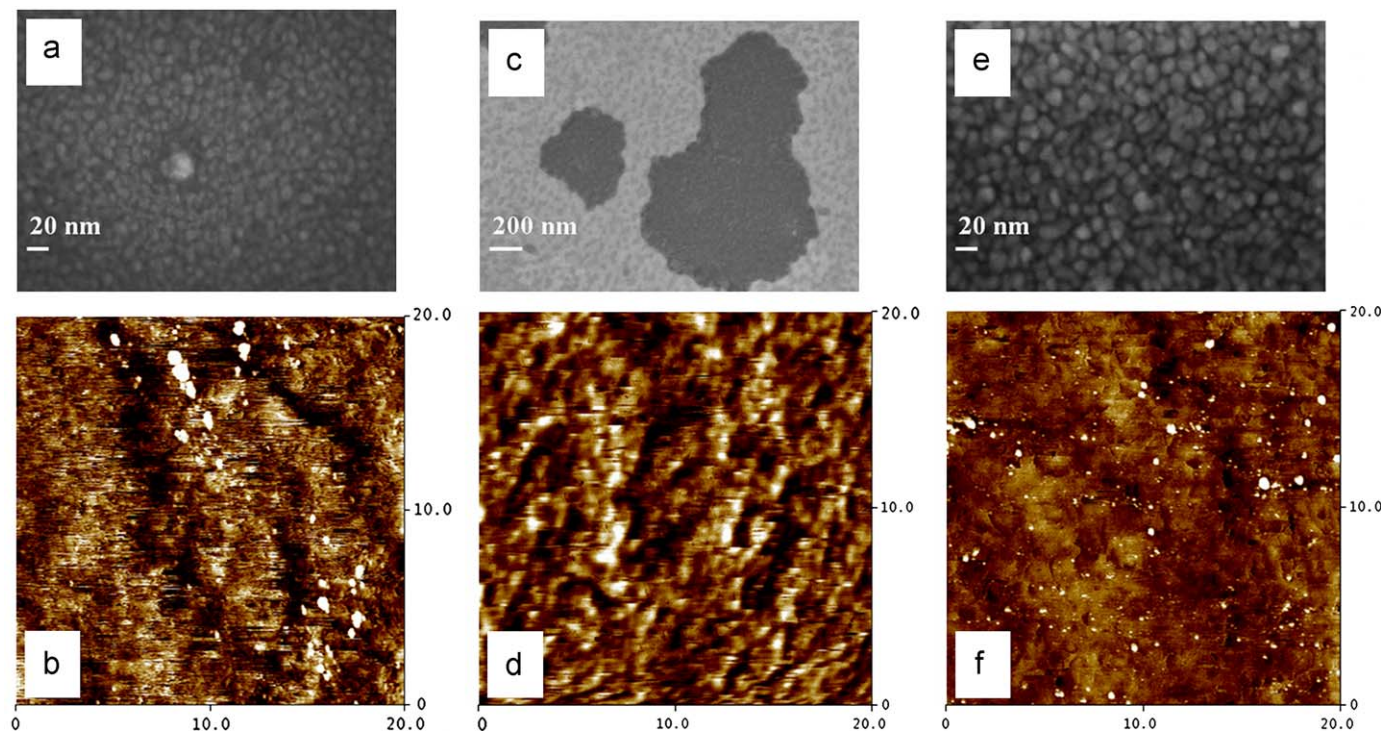


Fig. 1. (Colour online) Upper row: FESEM images of samples C1 (as-grown: (a)), C1 (annealed: (c)) and C2 (as-grown: (e)) with adapted reference bar; lower row: MFM images of the same samples after out-of-plane demagnetisation procedure. MFM images cover an area of $20 \times 20 \mu\text{m}^2$.

Room-temperature magnetisation measurements were done by means of a vector vibrating sample magnetometer (VSM) designed to simultaneously detect two mutually orthogonal magnetisation components: M_x , parallel to the applied magnetic field H_a , and M_y , orthogonal to it. Different configurations of H_a with respect to the sample plane and edges could be analysed. Details on the measurement technique are found in Ref. [7]. This advanced technique has not yet been applied to Co/Cr multilayers, although specific magnetic effects in thin films having similar compositions were studied under canted magnetic fields [8].

2. Results and discussion

FESEM images of samples C1 (as-prepared), C1 (annealed) and C2 are shown in Figs. 1a, c, e, respectively. In all cases, grains are observed to be in the nanometer range; isothermal annealing results in the formation of large defective regions (dark areas in Fig. 1c). A detailed matrix analysis of FESEM images has been implemented to characterise possible segregation effects occurring at grain boundaries and to explain the strong secondary electron contrast found in sample C1 after annealing (Fig. 1c). A recently developed technique of numerical correlation suitable to study EDX micromaps and FESEM images and described in detail elsewhere [9] shows that Co is preferentially localised in regions corresponding to a grey contrast level (gl) of $100\text{--}160^1$ (Fig. 2, brighter areas in the upper right panel; red areas in the false-colour image, left panel), whereas it is preferentially absent from regions corresponding to a gl of $40\text{--}60$ (blue regions). On the contrary, with the exception of a bright peak localised at a gl of 110 (Fig. 2, white spot in the lower right panel) the elemental signal associated to Cr is quite well distributed throughout the

sample. This demonstrates that annealing brings about Co segregation, so that in the end some regions become significantly depleted of this element.

These conclusions are supported by further analysis of FESEM images of the annealed sample. As an example, elemental spectra detected by integration along a straight-line path in the same region are shown in Fig. 3 (right panel). The integration line is indicated in white in the left panel. The blue spectrum referring to the Co K- α transition has rather low amplitude in correspondence of the darker areas of the FESEM image, indicating that these are characterized by a much lower Co content with respect to the surrounding brighter areas, where by contrast the elemental Cr signal (red spectrum) is slightly higher. For comparison, the O spectrum (in green) is reported also. It is shown that O has a more homogeneous distribution compatible with the uniform native oxide of the Si substrate.

The FESEM image of a tilted cross section of sample C2, placed perpendicular to the electron beam, is shown in Fig. 4. A part of the multilayer has been marked to show the actual stack structure: from bottom to top, the film is composed by layers of Si (black), SiO_2 native oxide (yellow), Co (blue) and Cr (green) alternating in agreement with the nominal scheme $(\text{Co}_{16}/\text{Cr}_5)_2\text{Co}_{18}$. According to the secondary electron image analysis, the actual multilayer thickness is $(\text{Co}_{14}/\text{Cr}_6)_2\text{Co}_{19}$, with a native oxide thickness of 23 nm. The section surface shows ductile flow of metal portions within each layer, as often observed in evaporated thin metallic films [9].

Magnetic force microscopy images taken on the same three samples after out-of-plane demagnetisation are reported in Fig. 1b, d, f, respectively. Samples C1 (as-prepared) and C2 are characterised by faint stripe-like (Fig. 1b) or bubble-like (Fig. 1f) magnetic domain patterns, indicating the presence of a slight out-of-plane anisotropy that locally cants the magnetisation vector off the film plane. After annealing, sample C1 develops a more pronounced out-of-plane anisotropy that accounts for the smaller and more contrasted structure of the MFM image (Fig. 1d).

¹ The numerical procedure described in Ref. [9] operates on 8 bit images (256 grey levels).

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