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Preparation, characterization and magneto-optical investigations of electrodeposited Co/Au films

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Abstract

Au/Co(4–8 ML)/Au single magnetic layers and Au(8 ML)/Co(4 ML)/Au(8 ML)/Co(8 ML)/Au bilayer were sequentially grown by electrodeposition on an Au(1 1 1) buffer layer electrodeposited on Si(1 1 1). The technique used in this work provides full control on the structure and the chemical composition of the different layers (no alloying) as well as on the chemistry at interfaces. scanning tunneling microscopy (STM) and atomic force microscopy (AFM) imaging and X-ray diffraction measurements show that *atomically flat continuous* Co(0 0 0 1) layers (4–8 ML) can be grown in epitaxy with the Au(1 1 1) substrate and that the 2 nm-thick spacer is also a continuous Au(1 1 1) layer. The Co ultrathin layers (4 and 8 ML) exhibit perpendicular magnetic anisotropy. The lateral magnetic homogeneity and magnetization reversal process have been investigated by scanning magneto-optical Kerr effect (MOKE) magnetometry and global Kerr microscopy. The correlation between magnetization switching behaviour in each layer of the Co-bilayer stack has been evidenced from in-depth sensitive MOKE measurements and microscopy. The strong coupling observed between the two Co layers is attributed to magnetostatic interaction at domain wall boundaries.

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1. Introduction

Electrodeposition has been used in the late 1990s to grow thin multilayers [1] exhibiting significant giant magnetoresistance (GMR). The technique proved to be highly efficient to grow 1D nanowires inside porous membranes [2,3]. More recently it was also proven that self-ordered electrochemical growth can be achieved by electrodeposition on pre-structured surfaces [4]. With respect to magnetic materials, electrochemistry stands as a powerful method for preparing epitaxial ultrathin film and nanostructures over large surfaces [5]. For example, electrodeposited Au/Co(t_{Co})/Au ultrathin films grown on mica [6] show quite similar magnetic properties than the best evaporated-grown samples [7]. In particular, they exhibit out-of-plane magnetic anisotropy for Co layer thickness (t_{Co}) up to 2 nm (i.e. 10 ML), and highly square hysteresis loops. For $t_{Co} > 2$ nm, the easy magnetization axis reorients into the film plane. These electrodeposited films would be appropriate to manufacture cheap media for magnetic recording or spintronic applications.

Only few works were devoted so far for checking the correlation between morphology and magnetic properties of electrodeposited structures. This is the aim of this article, where archetypal single magnetic layer Au/Co(t_{Co})/Au sandwiches, but also Au/Co(t_{Co1})/Au/Co(t_{Co2})/Au magnetic bilayer structures were selected. Our study focuses on

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ultrathin Co film structures with perpendicular magnetic anisotropy ($t_{Co} < 10$ ML).

The fabrication of films and multilayers is first described. Their morphological homogeneity has been checked by atomic force microscopy (AFM) and in situ scanning tunneling microscopy (STM). Their structure has been determined by X-ray diffraction. The local magnetic properties of these films were tested by using scanning magneto-optical Kerr effect (MOKE) magnetometry, but also by MOKE microscopy [7]. It has been recently demonstrated that MOKE can provide in-depth magnetic information in multilayer structures, even if the magnetic layers are composed with the same material [8,9]. This technique is very useful in the case of cobalt bilayer structures because it allows studying separately the magnetization reversal of each layer.

2. Preparation, structure and morphology of the magnetic ultrathin film structures

2.1. Sample preparation

The H–Si(1 1 1) substrate surface was prepared by controlled chemical etching in 40% NH_4F with ~50 mM $(NH_4)_2SO_3$ to remove dissolved oxygen [10]. A miscut angle of 0.2°, oriented towards $\langle 1\,1\,-2 \rangle$, allowed obtaining a staircase surface structure. After etching, an ohmic contact was realized by applying an InGa eutectic on the rear side of the sample which was mounted on a rotating electrode (1750 rpm) with only the (1 1 1) polished face exposed to the plating solution (the sides of the sample were protected with an electrolytic scotch tape, Struers). The 60 ML (14 nm)-thick Au(1 1 1) buffer layer was electrodeposited under potentiostatic mode at—2 V for 300 s from an aqueous electrolyte containing 0.1 mM $HAuCl_4$ + 0.1 M K_2SO_4 + 1 mM KCl + 1 mM K_2SO_4 (pH 4) onto an n-type H-terminated Si(1 1 1) electrode. In what follows, all potential are quoted with respect to the Mercury Sulfate Electrode (MSE).

The freshly prepared Au/Si(1 1 1) substrate was rapidly mounted in a 5 ml electrochemical cell for sequential Co and Au electrodepositions (static electrode, no solution stirring). Fig. 1 explains the procedure by presenting the variations of the electrode potential and of the current during the whole process. After 60 s at −0.8 V, the first step corresponds to Co electrodeposition (phase I) at −1.6 V for 30 s (case of a 4 ML-thick Co layer) from a 1 mM $CoSO_4$ aqueous solution in 0.1 M K_2SO_4 + 1 mM KCl (pH 4) as supporting electrolyte. At the end of the deposition, the electrode potential is ramped to −1.15 V, where Co neither grows nor dissolves. A Co counter electrode was used in phase I. In phase II ($130\,s < t < 330\,s$) the deposit is stabilized by maintaining the sample potential at −1.15 V while progressively exchanging the Co solution with the supporting electrolyte: after having pumped out the 2 mL of solution from the cell the same volume of supporting electrolyte was injected. The operation was repeated eight

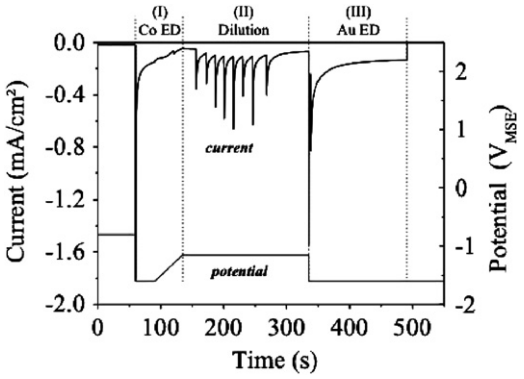

Fig. 1. Plot of applied electrode potential (bottom curve) and current (top curve) as a function of time during the fabrication of a Au/Co/Au multilayer. Phase I: Co electrodeposition (ED) at −1.6 V for (case of a 8 ML Co film). Phase II: Exchange of the Co solution while the electrode potential is set at −1.15 V, where the Co layer is stable. Phase III: Au electrodeposition for 150 s at −1.6 V after the injection of $HAuCl_4$. See text for details.

times to reduce the Co^{2+} concentration in the 10 μM range. Upon each injection of solution, the transient current corresponds to the reduction of protons (the peak arises from solution convection). Phase III corresponds to the deposition of gold capping layer: 0.5 ml of a 10 mM $HAuCl_4$ solution (final concentration 1 mM) were first added to the solution, while simultaneously shifting the electrode potential to −1.6 V. This potential appeared to be the optimum to obtain a *continuous* gold layer (depositing Au at 1.15 V leads to a discontinuous film). Gold deposition time was 150 s (phase III).

The thickness of the cobalt layers was derived from calibration curves using the stripping method [11]. For a given deposition time, the error on the cobalt thickness was less than 10%. For the magnetic bilayer, the thickness of the gold spacer layer was estimated to be 1.7 ± 0.8 nm (taken as 2 nm in the following).

2.2. Sample characterization

To determine the film structure, a home-built four-circle diffractometer with a Kα1 Cu source ($\lambda = 1.5405$ Å) and a monochromator were used. The sample configuration is represented later in Fig. 2a. The X-ray beam is horizontal and parallel to XX′. The entrance slit was $1 \times 0.025\,mm^2$ wide; the slit before the detector was $6° \times 2.3°$. Before measurements, the sample plane was carefully adjusted for setting the normal **n** along the rotation axis ZZ'. For grazing incidence measurements or ψ-scans, the incidence angle was $\theta_i = 0.4°$ or 0.6°. The corresponding Bragg conditions are obtained with the settings (h, γ) reported in Table 1 for Si, Au and Co, respectively. For a *quantitative* comparison of different deposits, the XRD spectra were normalized with respect to the intensity of the direct beam, exposure time and exposed surface area. The origin, for $\psi = 0$, corresponds to the $[2\,\bar{2}\,4]$ diffraction peak of the silicon substrate.