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ScienceDirect
Journal of Magnetism and Magnetic Materials 308 (2007) 318–324
Journal of magnetism and magnetic materials
www.elsevier.com/locate/jmmm

Evolution of structural and magnetic properties of sputtered nanocrystalline Co thin films with thermal annealing

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Received 28 February 2006; received in revised form 22 May 2006
Available online 10 July 2006

Abstract

Ultrafine grain films of cobalt prepared using ion-beam sputtering have been studied using X-ray diffraction (XRD), X-ray reflectivity (XRR), atomic force microscopy (AFM) and magneto-optical Kerr effect (MOKE) measurements. As-prepared films have very smooth surface owing to the ultrafine nature of the grains. Evolution of the structure and morphology of the film with thermal annealing has been studied and the same is correlated with the magnetic properties. Above an annealing temperature of 300 °C, the film gradually transforms from HCP to FCC phase that remains stable at room temperature. A significant contribution of the surface energy, due to small grain size, results in stabilisation of the FCC phase at room temperature. It is found that other processes like stress relaxation, grain texturing and growth also exhibit an enhanced rate above 300 °C, and may be associated with an enhanced mobility of the atoms above this temperature. Films possess a uniaxial anisotropy, which exhibits a non-monotonous behaviour with thermal annealing. The observed variation in the anisotropy and coercivity with annealing can be understood in terms of variations in the internal stresses, surface roughness, and grain structure.

Keywords: Magnetic thin film; MOKE; Uniaxial magnetic anisotropy; IBS; Thermal annealing

1. Introduction

Cobalt thin films have been studied extensively in recent years because of their potential use as magnetic material in various devices. Most of the studies in literature have been done on epitaxial films deposited on substrates like Al_2O_3, GaAs, Si, Pd, Cu, W [1–7]. In case of a film deposited on Al_2O_3 $(1\,1\,\bar{2}\,0)$ substrate, the as-prepared film is a mixture of HCP and FCC phase. Thermal annealing results in growth of the FCC phase. Increasing annealing temperature above a critical value results in a reorientation transition from FCC (1 1 1) to FCC (0 0 1), accompanied by a smoothing of the surface. Recently, Kharmouche et al. have studied structural and magnetic properties of Co films evaporated on Si (1 0 0) as a function of the Co thickness [3]. The films were polycrystalline with (0 0 1) texture. It was found that surface and stress-induced uniaxial magnetic anisotropy decreases as the film thickness increases.

Nanocrystalline particles of cobalt prepared by different techniques have also been studied extensively in order to elucidate the relation of magnetic properties with the atomic structure, size and shape of the nanoparticles [8–10]. Depending on the preparation methods and temperature, the particles form either in FCC or in HCP phases. The FCC particles maintain their structure to ambient temperature without structural transformation from FCC to HCP, although in bulk it is known that FCC is stable only at temperatures above 425 °C [11]. No detailed study exists in the literature on the evolution of structural and magnetic properties of nanocrystalline Co thin films.

In the present work, evolution of structural and magnetic properties of ultrafine-grain polycrystalline cobalt films with increasing annealing temperatures has been studied with an aim to correlate the structural and morphological properties with magnetic properties.

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doi:10.1016/j.jmmm.2006.06.008

2. Experimental

Co film with nominal thickness of 45 nm was deposited by sputtering a Co target with 1 keV Ar ions incident at the angle of 45°. The film was deposited on a float glass substrate kept parallel to the target at a distance of ~16 cm. The substrate was kept at room temperature. The base pressure in the chamber was 2×10^{-7} Torr. Ex-situ X-ray reflectivity (XRR) measurements on the films were done in order to determine the deposition rate, which was found to be 4.5 nm/min. The exact thickness of the deposited film was again confirmed using XRR. Isochronal thermal annealing of the films has been done in vacuum $\sim10^{-7}$ Torr in order to avoid the surface oxidation. A turbo-molecular pump backed by a scroll pump has been used in order to achieve clean vacuum. Sample kept in an evacuated quartz tube was inserted in a pre-heated furnace. This ensures that the heating and the cooling time of the sample was about 5 min. Structure of the films as a function of thermal annealing was determined from X-ray diffraction (XRD) measurements. The magnetic properties of the samples at room temperature were characterised by ex-situ magneto-optical Kerr effect (MOKE) measurements. Measurements in the longitudinal geometry were performed with the magnetic field, H, applied along different directions in the plane of the film. Angle-dependent magnetic remanence or coercivity obtained from the MOKE hysteresis curve gives information about magnetic anisotropy in the film. The surface morphology of the film in different states (as-prepared, annealed at 200 and 500 °C) was imaged by atomic force microscopy (AFM) using nanoscope III, version 'E' from digital electronics.

3. Results and discussion

Fig. 1 gives the XRR pattern of as-prepared Co/float glass film. The reflectivity pattern was fitted using Parratt's formulism [12]. In order to get good theoretical fit of the data, it was found necessary to incorporate a thin surface layer with somewhat lower electron density. This surface layer was 1.8 nm thick with electron density ~15% less then that of the film and may be attributed to surface oxidation. The mass density of the film as calculated from the electron density obtained from the fitting of the XRR data comes out to be ~8.7 g/cm^3, which is only ~2.0% less than that of the bulk Co. The total thickness of the film was found to be 41.9 nm. The surface roughness of the film was found to be 0.6 nm, which is comparable to that of the bare substrate (~0.7 nm).

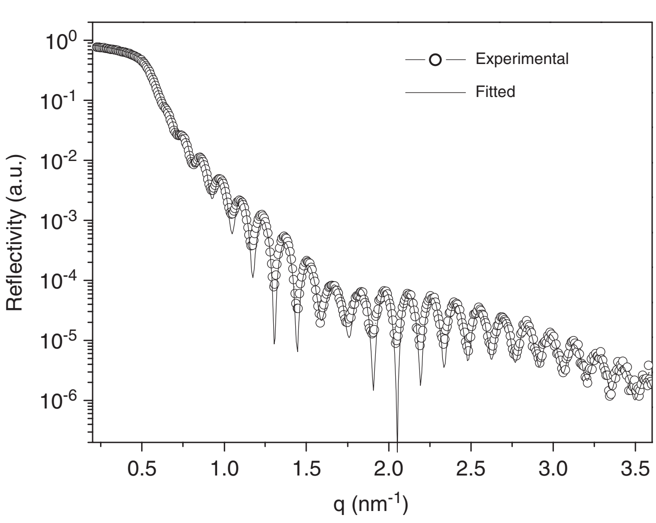

Fig. 1. Reflectivity pattern of the as-prepared Co film on the float-glass substrate. Continuous curve represents the fitting of data using Parratt's formalism.

Fig. 2 gives the XRD pattern of the as-prepared film as well as after isochronal annealing for a period of 1 h at different temperatures ranging from 200 to 500 °C. Annealing at 550 °C has been done for 5 h. The XRD pattern of the as-prepared film exhibits a broad hump at position 2θ~44.7°. This broad hump may be interpreted either in terms of an amorphous structure of the film, or arising due to overlapping of a number of crystalline peaks.

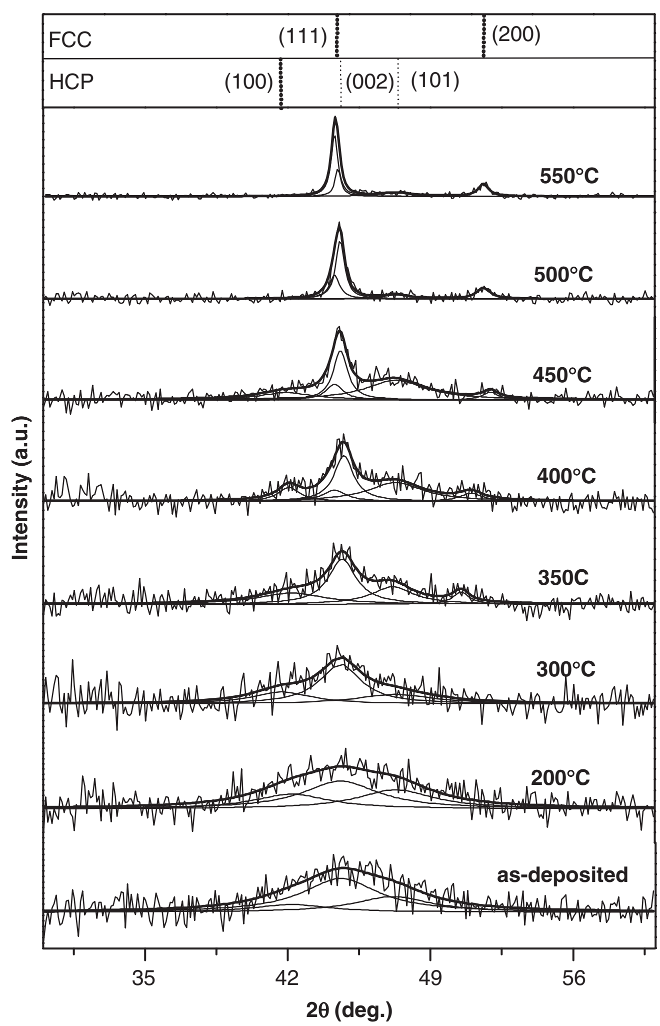

Fig. 2. X-ray diffraction pattern of the as-prepared film as well as after isochronal annealing for 1 h at different temperatures ranging from 200 to 500 °C. Annealing at 550 °C has been done for 5 h.