



Quantitative study of the effect of current density on the morphological and magnetic domain structures of electrodeposited nanocrystalline cobalt films

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ABSTRACT

We have investigated quantitatively the morphological and magnetic domain structures of nanocrystalline cobalt films electrodeposited at different current densities in the range from 40 to 50 mA/cm². The films had the thickness of about 40 μm and were prepared at room temperature from a cobalt sulfate solution with pH value of 4.5. The morphological structure of the films was revealed by atomic force microscopy (AFM), whereas the magnetic domains were imaged with the colloid-scanning electron microscopy (colloid-SEM) method. The morphological structure consisted of densely packed grains with irregular shapes. No geometric alignment and no preferred elongation of the grains were observed on the film surface. The magnetic domain structure was composed of maze and surface domains with magnetization perpendicular to the film surface. As the deposition current density was increased from 40 to 50 mA/cm², the average grain size decreased from 81 to 71 nm and the average domain size increased from 320 to 410 nm. The films crystallized mainly in the hexagonal close-packed (HCP) phase with the hexagonal axis perpendicular to the film surface. The crystallographic texture is found to decrease with decreasing grain size.

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1. Introduction

Nanocrystalline materials, including those of magnetic materials, with average grain size typically smaller than 100 nm, are currently the subject of extensive interest from both fundamental and practical points of view. This is because of their specific, improved mechanical, physical, magnetic and chemical properties in comparison with conventional microcrystalline counterparts.

Among a number of techniques developed for producing nanocrystalline materials, electrodeposition has been recognized as a technologically feasible and economically superior technique, because of its versatility, easy application and low cost [1,2]. The properties of electrodeposited materials can be tailored by controlling electrolyte composition and many deposition parameters, such as applied potential, current density, deposition temperature, deposition time, solution pH, continuous or pulsed current electrodeposition, and external magnetic field.

In this paper, the subject of interest are nanocrystalline cobalt films. Depending on the preparation method and conditions used, nanocrystalline cobalt films exhibit a wide range of morphological and magnetic properties. They are used in a number of applications and have a wide range of potential applications. For example, nanocrystalline cobalt

films are used in spintronic devices such as giant magnetoresistance (GMR) multilayers, spin valves, magnetic tunnel junctions (MTJs) and magnetic random access memories (MRAMs) [3,4], and in microelectromechanical systems (MEMS) [5,6]. Electrodeposited nanocrystalline cobalt and cobalt alloys films are currently developed as environmentally friendly substitutes for hard chrome coatings [7,8]. Very recently nanocrystalline cobalt films have been investigated as Cu capping layers to prevent oxidation of the Cu interconnects in semiconductor devices [9]. Nanocrystalline cobalt films find also applications in emerging novel voltage-controlled and current-controlled spintronic devices such as spin-transfer-torque (STT) MRAMs, and logic devices and circuits (Spin-Logic) [10–12].

The morphological structure of electrodeposited cobalt films was widely studied in the past, but observations of their magnetic domain structure were carried out only in few works (see Refs. [13–15]). In particular, the effect of current density on the morphological structure of these films was investigated qualitatively in Refs. [16–19] and quantitatively only in Ref. [1], and no research was made of the effect of current density on their magnetic domain structure. The present work reports a quantitative study of the effect of current density on the morphological and magnetic domain structures of cobalt films. The morphological structure was observed using atomic force microscopy (AFM), and the magnetic domain structure was revealed by the colloid-scanning electron microscopy (colloid-SEM) technique.

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2. Experimental

The studied cobalt films were prepared by electrodeposition at room temperature using a conventional three-electrode system. The system consisted of a polycrystalline gold plate as the working electrode with the surface area of 0.1 cm^2 , an auxiliary platinum mesh electrode of much larger surface area and a saturated calomel electrode (SCE) as the reference. The used system is described in more detail in Ref. [20]. The working electrode was successively prepared mechanically (with the use of sandpapers, down to 2000 grade), chemically (using a chromic acid cleaning mixture), electrochemically (polarization in a stock solution of $0.1 \text{ M H}_2\text{SO}_4$, within the potential range of -1.5 V to $+2 \text{ V}$, with a sweep rate of 50 mV/s), and cleaned ultrasonically to remove contamination on the surface. The used electrolyte had the following composition: 0.2 M cobalt sulfate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$), 0.6 M sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$), 0.025 M EDTA ($\text{C}_{10}\text{H}_{14}\text{O}_8\text{N}_2\text{Na}_2 \cdot 2\text{H}_2\text{O}$) and 0.1 M sulfuric acid (H_2SO_4). The solution pH was 4.5. The films were electrodeposited at current densities of 40, 45 and 50 mA/cm^2 .

The morphological structure of the cobalt films was made visible by AFM using an Omicron ultrahigh vacuum (UHV) system working at the base pressure lower than $5 \times 10^{-8} \text{ Pa}$. AFM images were taken in the contact mode using Omicron silicon cantilevers [14,15].

The magnetic structure of the cobalt films was imaged by the colloid-SEM method [21]. In this method, the studied specimen surface is decorated by fine magnetic particles and the resulting domain pattern is observed by SEM. The spatial deposition of magnetic particles on the specimen surface depends directly upon the distribution of the stray magnetic field near the surface. We used commercial suspension of magnetite (Fe_3O_4) particles [22], about 10 nm in diameter, dispersed in distilled water. The optimum concentration of the colloid was determined experimentally. After drying the colloid placed on the specimen surface, images were recorded in the secondary electron mode at 5 keV primary beam energy using a high-resolution FEI Nova NanoSEM 450 instrument equipped with field emission gun.

3. Results and discussion

Suitable values for cobalt film deposition potential were chosen on the basis of the cyclic voltammograms recorded for the system composed of the used electrolyte and gold electrode. The chemical composition on the surface of the films was studied by X-ray photoelectron spectroscopy (XPS). Some amounts of cobalt oxides and carbon were found to occur on the surface of the films. The presence of oxygen in the cobalt films grown at room temperature is consistent with the thermodynamics of the cobalt–oxygen system, and the occurrence of carbon is related to some physisorbed atmospheric contaminations (for details, see Ref. [19]).

It is important to note that the investigated cobalt films were electrodeposited at different current densities in such a way that they had (nearly) the same thickness, to eliminate the dependence of their morphological and magnetic structures on the film thickness. SEM was used to determine the thicknesses of the films. On the basis of SEM images recorded at various places of the cross-sectional area of the specimens, the thicknesses of the films electrodeposited at current densities of 40, 45 and 50 mA/cm^2 were determined to be $(39 \pm 2) \mu\text{m}$, $(40 \pm 3) \mu\text{m}$ and $(42 \pm 3) \mu\text{m}$, respectively.

Low-magnification SEM studies showed that there were cracks as well as nodules and cavities on the surface of the films (see Ref. [19] for details). However, the cracks were present in the surface region of the films, and they did not extend through the whole thickness of the films. This is in contrast to electrodeposited chromium films, where cracks are reported to be commonly observed in the volume of the films [23]. The occurrence of cracks in electrodeposited cobalt and cobalt-based films is described in a number of works [14,24–27]. This undesired effect is attributed to tensile stress in the film. Note also that the studied films showed good adhesion to the substrate despite

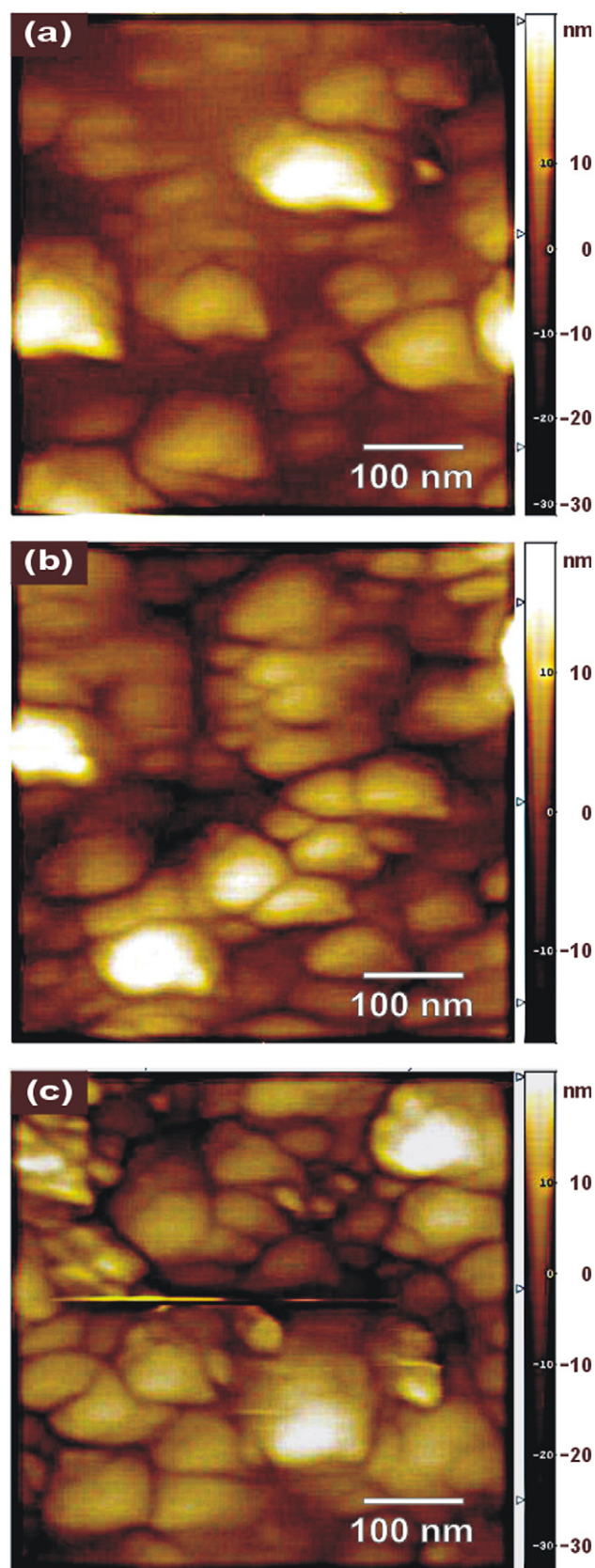


Fig. 1. AFM images of the morphological structure of cobalt films electrodeposited at current densities of 40 mA/cm^2 (a), 45 mA/cm^2 (b) and 50 mA/cm^2 (c).

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