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Investigation of thick cobalt films electrodeposited on gold substrates

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ABSTRACT

We have investigated the morphological and magnetic structures of thick cobalt films obtained by electrodeposition on polycrystalline gold substrates. The deposits became more and more inhomogeneous with increasing deposit thickness. The morphological structure of the films consisted of nanocrystalline grains roundish in shape and having sizes of the order of 100 nm. The magnetic domains were of the order of 500 nm in size and exhibited sufficiently high perpendicular anisotropy. The deposits were found to possess mainly the hexagonal close-packed crystalline structure with sufficiently strong [0001] preferred orientation. The average domain size slightly increased with decreasing average grain size.

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1. Introduction

Magnetic films attract much attention from both fundamental and technological points of view. On the fundamental side, they exhibit different magnetic properties, such as magnetic anisotropy, magnetic microstructure, coercivity and magnetoresistance, depending on their thickness, chemical composition, morphological structure, crystallographic structure and preparation conditions. From the technological point of view, magnetic films find a wide range of applications in the areas of magnetic and magneto-optic recording and magnetic sensors.

In particular, cobalt and cobalt-based films, deposited by various techniques on different substrates, have been intensively investigated in recent years. This appears to be related to the fact that depending on the film thickness as well as the preparation method and conditions used, these films exhibit a very wide range of magnetic properties, and consequently a very wide range of applications. For example, cobalt and cobalt-based films are used in spintronic devices such as giant magnetoresistance multilayers, spin valves, magnetic tunnel junctions and magnetic random access memories [1,2], in micro/nanoelectromechanical systems (MEMS/NEMS) such as actuators, sensors, motors, generators and frictionless microgears [3,4], and cobalt-based films are used as high-density magnetic recording materials [5,6].

Recently, studies have been performed of cobalt films obtained by pulsed laser deposition on sapphire [7], silicon [8], carbon [8] and glass [9] substrates, by electron beam evaporation on GaAs [10], sapphire [11] and silicon [12] substrates, by electron-beam glancing angle deposition on silicon substrates [13], by thermal

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evaporation on glass [14], silicon [15] and NaCl [14] substrates, by sputtering on glass [16] and silicon [16,17] substrates, by chemical vapor deposition on glass [18] and silicon [19] substrates, by electrodeposition on platinum [20], copper [20,21], gold [22], silicon [23] and graphite [21] substrates.

Electrodeposition is a film deposition technique which offers a number of advantages, such as precisely controlled near room temperature operation, low-energy requirements, rapid deposition rates, capability to handle complex geometries, low cost and simple scale-up with easily maintained equipment. In addition, the properties of materials can be tailored by controlling solution compositions and deposition parameters. Because of these advantages, electroplated materials are widely used for the fabrication of magnetic recording heads and MEMS/NEMS [3,4].

This Letter presents a study of thick cobalt films electrodeposited on gold substrates. The morphological structure of the films was observed using scanning electron microscopy (SEM) and atomic force microscopy (AFM), while the magnetic structure was imaged by magnetic force microscopy (MFM). The morphological structure of electrodeposited cobalt films has been extensively investigated in the past (mainly by SEM), but observations of their magnetic structure are in fact very rare (see a review article [24]). To the authors' knowledge, only recently has the magnetic structure of electrodeposited cobalt been studied in Refs. [23,25–27] by a high-resolution technique such as MFM.

2. Experimental

Thick cobalt films were prepared by electrodeposition using a three-electrode system. The system consisted of a polycrystalline gold plate as the working electrode with the surface area of



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0.1 cm², an auxiliary platinum mesh electrode of much larger surface area and a saturated calomel reference electrode. More details on the system used are given in Ref. [28]. The working electrode was successively prepared mechanically (with the use of sandpapers, down to 2000 grade), chemically (using a chromic acid cleaning mixture) and electrochemically (polarization in a stock solution of 0.1 M H₂SO₄, within the potential range of -1 to +2 V, with a sweep rate of 50 mV/s). The electrolyte solution contained 0.2 M CoSO₄·7H₂O, 0.6 M Na₃C₆H₅O₇·2H₂O and 0.1 M H₂SO₄. The solution pH was 4.5. The cobalt films were electrodeposited at room temperature and at constant current density of 20 mA/cm². The films of different thickness were obtained by varying the deposition time.

The investigation of the morphological structure of the films was carried out by SEM using a Tescan Vega 5135 MM instrument and by AFM using an Omicron instrument. The scanning electron microscope was equipped with conventional tungsten filament and images were recorded in the backscattered electron mode at 30 keV primary beam energy. AFM images were taken in the contact mode using Omicron silicon cantilevers. The MFM study of the magnetic structure of the films was made with a NT-MDT instrument operated in the dynamic mode using MikroMasch silicon cantilevers with tips magnetized along the tip axis, which was perpendicular to the specimen surface. In this case, MFM senses the vertical component of the derivative of the force between the specimen and the tip [29,30]. The image signal was detected as the phase or amplitude shift of an oscillating cantilever. The tips used were coated with a FeCoNi film of about 60 nm in thickness. The coercivity of the tips was approximately 8 kA/m.

The MFM measurements were carried out by the two-pass method. For each raster line, the first pass was made very close to the specimen surface and yielded knowledge of the surface topography (AFM). The second pass then followed the recorded topography but at an increased scan height. Of course, the tip-specimen distance must be large enough to eliminate (or minimize) the short-range forces that provided the topographic contrast. Then in the second pass the tip is affected only (mainly) by long-range magnetic forces and the corresponding MFM image of the specimen surface is obtained. All the MFM images shown in this Letter were collected with a tip-specimen distance of 100 nm.

3. Results and discussion

SEM was used to determine the thicknesses of the studied cobalt films. On the basis of SEM images recorded at various places of the cross-sectional area of the specimens, the thicknesses of the cobalt deposits under investigation were determined to be $(14 \pm 1) \mu m$, $(28 \pm 2) \mu m$ and $(55 \pm 5) \mu m$. The first two films were relatively uniform in thickness, in contrast to the third film. None of the films detached from the substrate despite the fact that they were thick (up to 55 μm in thickness), thus the polycrystalline gold substrates may be considered suitable.

Figure 1a–c show low magnification SEM images of the surface of the cobalt deposits 14, 28 and 55 μ m in thickness, respectively. At the presented scale, an inhomogeneous character of the films surface is clearly revealed, especially in the case of the thickest film shown in Figure 1c. Elevations and cavities in the form of stripes (of the order of 100 μ m in width), as well as smaller scale cavities and some cracks, are present on the surface of the films. The deposits become more and more inhomogeneous when the deposit thickness is increased, as proved also by the corresponding Fourier transform patterns presented in the insets of Figure 1. It was verified that the topographic stripes run in the direction parallel to the direction of the current flow during the electrodeposition process, but the exact reason for their occurrence is not known. The cracks were



Figure 1. Low magnification SEM images of the surface of cobalt films 14 μ m (a), 28 μ m (b) and 55 μ m (c) thick. The double headed arrow in each image is parallel to the direction of the current flow during the electrodeposition process. The insets present the two-dimensional Fourier transform of the images.

present in the surface region of the deposits, and they did not extend through the whole thickness of the deposits, as evidenced in SEM cross-sectional and surface images of the deposits. This is in contrast to electrodeposited chromium films of similar thickness, where cracks are reported to be commonly observed in the volume of the films [31]. The presence of cracks in electrodeposited cobalt and cobaltbased films is reported in many papers (e.g. Refs. [20,28,31]). The most likely reason for this undesired effect is the presence of tensile stress in the film [3,28,32]. In general, there are several possible sources for generation of tensile stress. These include (i) interfacial stress between the film and the substrate, (ii) crystallographic texture and grain size, (iii) coalescence and stress evolution during the film growth, and (iv) hydrogen adsorption/ desorption. The latter mechanism, related to the incorporation of hydrogen into the deposit (adsorption) during electrodeposition and subsequent diffusion out of the deposit (desorption) after the interruption of electrodeposition, is found to be the most likely dominant stress mechanism [32].

The tensile stress in the investigated cobalt deposits is found not to be very large because the cracks occurred only in the surface region of the deposits and the deposits exhibited good adhesion to the substrate. There are few ways to reduce the stress (and consequently to reduce or eliminate the cracks) in electrodeposited film. These ways include, for example, the use of pulse electrodeposition [32], adding a stress reducer (such as saccharine, thiourea, benzene sulfamide, benzene sulfonic acid, etc.) to the electrolytic solution [3,32], increasing the temperature of the electrolytic solution [32] or the application of magnetic field during the electrodeposition process [28].

AFM was used to study the morphological structure of the films with high resolution. Figure 2a–c show original AFM images of the surface of the cobalt films 14, 28 and 55 μ m thick, respectively; the insets present the two-dimensional Fourier transform of the images. The morphological structure is composed of grains. The grains are packed very densely, they are roundish in shape and possess sizes at the nanometer range. Note, however, that in the case of the 28 μ m thick film the grains form clusters. In this sense, the morphological structure of the film 28 μ m thick is different from that of the films 14 and 55 μ m thick.

AFM images taken from sufficiently small film surface area, smaller than about $2 \times 2 \mu m$, usually showed some directionality, i.e. the grains exhibited preferred orientations (which in general varied when moving from one film surface area to another). This is the case for the images of Figure 2a and c, as proved by the corresponding Fourier transform patterns in the form of an ellipse (presented in the insets). Nevertheless, images recorded from sufficiently large film surface area, larger than about $5 \times 5 \mu m$, exhibited in fact no directionality, i.e. the grains ran in random directions in the image plane. In general, no preferred elongation of the grains was observed.

The original AFM images usually did not possess uniform background intensity, i.e. some regions were displayed as considerably darker or brighter (cf. Figure 2). As a result, the visibility of grains in such regions was unsatisfactory. To obtain better quality images, appropriate for quantitative analysis of grain sizes, the original images were subjected to the procedure of image differentiation. After differentiating of the images from Figure 2, the corresponding images presented in Figure 3 were obtained. On the basis of such images as those in Figure 3, the grain size distributions and average grain sizes in the plane of the investigated films were derived using the standard linear intercept method [33-35]. In this method, a large number of test straight lines running in random directions is used; the method is illustrated in the image of Figure 3a, where five test lines are drawn. The object size (the grain size in the considered case) *S* is determined from the formula [33]:

$$S = \left(2/\pi\right) \left(\sum_{i} l_{i}\right) \middle/ \left(\sum_{i} n_{i}\right)$$
(1)

where l_i is the length of the *i*th test line and n_i is the number of intersections of the *i*th test line with object boundaries.



Figure 2. AFM images of the surface of cobalt films $14 \mu m (a)$, $28 \mu m (b)$ and $55 \mu m (c)$ thick. The double headed arrow in each image is parallel to the direction of the current flow during the electrodeposition process. The insets present the two-dimensional Fourier transform of the images.

We used the standard linear intercept method with 1000 intercepts counted for each AFM image. The grain sizes were in a wide range; they varied from about 30 to 230 nm, from about 40 to 250 nm and from about 40 to 220 nm for the films of 14, 28 and 55 μ m in thickness, respectively. The average grain sizes were



Figure 3. Images (a–c) were obtained by applying a differentiation procedure to the AFM images from Figure 2a–c, respectively. On the image (a), five test straight lines are additionally superimposed to illustrate the standard linear intercept method used for determining the grain sizes.

 (123 ± 9) nm, (130 ± 14) nm and (111 ± 6) nm for the films of 14, 28 and 55 μ m in thickness, respectively. Moreover, the root mean square (rms) values of the surface roughness, obtained directly by AFM measurements using images taken at different places on the specimen surface, were (20 ± 3) nm, (30 ± 3) nm and (80 ± 20) nm

for the films of 14, 28 and 55 μ m in thickness, respectively. These results show that the 55 μ m thick film had the roughest surface (as expected from SEM study, cf. Figure 1) and the smallest grains.

Thanks to the application of SEM and AFM, useful complementary information on the morphological structure of the specimen could be obtained. Although both techniques probe the surface of the specimen, SEM is certainly better suited for investigating large areas of the surface, whereas AFM allows for the study of small areas with high sensitivity and high spatial resolution. Moreover, in contrast to SEM, AFM provided additional information in the direction perpendicular to the specimen surface, i.e. information on the surface roughness. Note also that in the case of a specimen composed of very small grains, SEM fails due to its insufficient spatial resolution, as reported for example in Ref. [36].

Figure 4a-c present MFM images of the cobalt films 14, 28 and 55 µm in thickness, respectively. The magnetic structure consists of maze stripe domains. In some regions the magnetic domains were more maze in character (like those in Figure 4b), and in other regions they were elongated, more like stripes than maze in character (like those in Figure 4a and c). In the case of the elongated domains, they were typically from about 8 to $14 \,\mu\text{m}$ in length. The reason for the undulation of the domains (the maze structure) is the reduction in the magnetostatic energy at the cost of a larger total domain wall area. In general, the domains ran in random directions in the film plane, i.e. no directionality in the orientation of the domains in the film plane was observed. The domains are displayed as bright and dark areas in the MFM images of Figure 4, which proves unambiguously that they have opposite magnetization components perpendicular to the film surface. This means also that the magnetostatic interaction between the domains and the tip was substantially non-perturbing. And more precisely, during scanning process the tip was not remagnetized by the stray fields of these domains each time when passing from one domain to a neighboring domain (i.e. at the domain walls), providing information on the direction of their magnetization component perpendicular to the film surface [37].

The observed domain structure is characteristic of materials with sufficiently high perpendicular magnetic anisotropy [7,11,35,38– 41]. Due to the lack of geometric alignment of the grains (the anisotropy of the demagnetizing field), the magnetic anisotropy of the studied films is obviously of crystalline origin (the magnetocrystalline anisotropy). In general, cobalt films can crystallize in the hexagonal close-packed (HCP) or face-centered cubic (FCC) phase. These two phases have very different magnetic properties. The HCP phase possesses only one magnetic easy axis [0001] (i.e. along the hexagonal axis), while the FCC phase has four easy axes [111] (i.e. along the cube diagonals). Moreover, the FCC phase of cobalt has magnetocrystalline anisotropy much lower (about 20-30 times) than the HCP phase [42,43]. What is most important here, the former phase cannot be the source of perpendicular magnetic anisotropy. As a consequence, the investigated cobalt films have mainly the HCP crystalline structure. As proved by the MFM study, the films possess perpendicular magnetic anisotropy high enough to overcome the shape anisotropy of the film (the anisotropy of the demagnetizing field) which tends to align the magnetization parallel to the film surface. Because of the polycrystalline character of the films (Figure 2), this in turn means that the films exhibit sufficiently strong crystallographic alignment (texture) of the cobalt grains with the hexagonal axis (being the only easy axis of magnetization) perpendicular to the film surface, i.e. the films exhibit sufficiently strong [0001] preferred orientation. To determine quantitatively the degree of crystallographic texture, investigations by the X-ray Schulz pole figures [7] or the electron backscattered diffraction (EBSD) method [34] are required.

For obtaining quantitative data about the sizes of magnetic domains in the studied films, we used the standard linear intercept



Figure 4. MFM images of cobalt films $14 \mu m (a)$, $28 \mu m (b)$ and $55 \mu m (c)$ thick. The double headed arrow in each image is parallel to the direction of the current flow during the electrodeposition process.

method with 1000 intercepts counted for each MFM image. The average domain sizes were determined to be (560 ± 20) nm, (530 ± 20) nm and (610 ± 30) nm for the films 14, 28 and 55 μ m in thickness, respectively. Taking into account the average grain sizes of the studied films, one can notice a correlation between the domain size and the grain size. Namely, the domain size increases with decreasing grain size. On the other hand, it is widely

known that the magnetic domain size becomes larger as the magnetic anisotropy of the specimen decreases [44,45]. In the case of the studied films, the magnetic anisotropy is apparently higher when the crystallographic alignment of the grains is better. As a consequence, the crystallographic texture is found to increase with increasing grain size. The occurrence of such dependence for electrodeposited thick cobalt films was indicated in Ref. [24]. It is also worth noting that reported recently in Ref. [46] electrodeposited cobalt films with very high [0001] preferred orientation possessed similar morphology (with grains roundish in shape and having sizes in the range of about 50–100 nm) to the films studied by us.

In the case of the films investigated in this work, the average domain size was few times larger than the average grain size, which means that each magnetic domain usually consisted of many crystalline grains. As a general rule, when the average grain size is about 40 nm or less, a strong exchange coupling among the grains occurs [47,48]. This is not the case for the studied films, in which the interactions between the grains are expected to be predominantly magnetostatic in nature. It is worth noting that the presence of a strong exchange interaction in cobalt films composed of grains smaller than about 30 nm in size leads to wide magnetic domains of a few micrometers in size [49].

In contrast to previous studies, we performed a detailed investigation of the grain and magnetic domain structures of the thick cobalt films electrodeposited on polycrystalline gold substrates, by high-resolution techniques of AFM and MFM, respectively. The average grain and domain sizes for the films were determined using the standard linear intercept method. The magnetic domain structure determines the hysteresis loop of the material, and the domain configuration is affected by the grain structure. The obtained results show the correlation between the domain size and the grain size. A detailed knowledge and understanding of the grain structure, magnetic domain structure and the correlation between these structures are not only of basic research interest, but also of practical significance. From the practical point of view, they are important for tailoring of the materials with the required properties. The knowledge of the magnetic domain behavior in relation to the grain structure of the specimens is also significant for theoretical modeling of magnetic properties.

4. Conclusions

A study has been made of the morphological and magnetic structures of thick cobalt films obtained by electrodeposition on polycrystalline gold substrates. The films were 14, 28 and 55 μ m in thickness, as revealed by SEM. They were electrodeposited from a cobalt sulfate bath with pH value of 4.5 at room temperature and at constant current density of 20 mA/cm². The morphological structure of the deposits was imaged by SEM and AFM, whereas their magnetic structure was made visible with MFM.

Despite their large thickness, the films did not detach from the substrate, thus in this regard the used polycrystalline gold substrate can be recognized as suitable. Nevertheless, at the micrometer scale the morphology of the deposits was inhomogeneous in nature. Elevations and cavities in the form of stripes (of the order of 100 μ m in width), as well as smaller scale cavities and some cracks, were present on the surface of the films. The deposits became more and more inhomogeneous as the deposit thickness was increased. But it is necessary to note that the deposits were found not to be highly stressed because they showed good adhesion to the substrate and the cracks occurred only in the surface region of the deposits.

The smaller-scale morphological structure of the deposits was composed of nanocrystalline grains which were roundish in shape, possessed sizes of the order of 100 nm, and generally exhibited no preferred elongation and no geometric alignment. The recorded images of the grain structure were subjected to digital processing and analysis for obtaining quantitative information on grain sizes. The average grain sizes for the investigated films were determined using the standard linear intercept method. Thanks to applying the SEM and AFM techniques, useful complementary information on the morphological structure of the films could be obtained.

The magnetic structure of the films was composed of domains. The domains formed a maze pattern and possessed sizes of the order of 500 nm. The domain structure was characteristic of materials with sufficiently high perpendicular magnetic anisotropy. As a consequence, the deposits were found to crystallize mainly in the HCP structure with sufficiently strong [0001] preferred orientation. The average domain sizes for the studied films were determined using the standard linear intercept method. The obtained results show that the average domain size slightly increased with decreasing average grain size.

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