HIGH RESOLUTION ELECTRON MICROSCOPY STUDIES OF THE ATOMIC STRUCTURE OF INTERFACES IN MAGNETIC SANDWICHES

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Résumé: Nous présentons une étude réalisée sur des couches ultra-minces de cobalt protégées de l'oxydation par deux couches d'or. Les interfaces Au/Co de ces sandwichs magnétiques sont étudiées à l'échelle atomique par microscopie électronique à haute résolution. Une étude systématique des micrographies par diffraction optique sur banc laser, fournit des renseignements intéressants sur le raccordement entre les plans de l'or et du cobalt. Ce type de résultat sera corrélé par la suite aux propriétés magnétiques de la couche bidimensionnelle.

Abstract: We report some studies performed on ultra-thin cobalt layers protected from oxidation by two gold films. The interfaces Au/Co of these magnetic sandwiches are studied at an atomic scale by high resolution electron microscopy. A systematic study of the micrographs by optical diffraction on a laser bench gives interesting information on the adjustment between gold and cobalt planes. This kind of result will be linked, later on, to the magnetic properties of the bidimensional layer.

INTRODUCTION

The magnetic properties of very thin films, at present, are the subject of extensive investigations, because of their fundamental interest and the need of thin films with perpendicular remanent magnetization for high density magnetic recording [1,2,3]. For applications, a large magnetic anisotropy constant is wanted; this is the case for cobalt with hexagonal close packed (h.c.p.) structure: the symmetry of an hcp crystal is lower than for a fcc crystal and hence a hcp structure leads to an axial magnetic anisotropy. In very thin h.c.p. Co films, with (00.1) orientation, this axial anisotropy is reinforced by the interface anisotropy but is in competition with the shape anisotropy which tends to align the magnetization in the plane of the film. Experimental magnetism studies performed on Au/Co/Au (111) sandwiches, have shown that the remanent magnetization is perpendicular to the film for cobalt thicknesses below 1.1nm. In this thickness range, the large interface effects overcome the shape anisotropy [4,5]. Thus, it is important to know as well as possible the surrounding of the cobalt atoms which are close to the interface and the possible interfacial defects. For these reasons, we have undertaken the study of our Au/Co/Au samples by high resolution electron microscopy.

SAMPLE CHARACTERISTICS

The metal films of gold and cobalt are successively deposited

by slow evaporation in ultra-high vacuum on a polished glass substrate with very small surface roughness ($\sim 0.5\text{nm}$). Electrical resistivity, X-ray and electron diffraction experiments have already been performed on those sandwiches and are described in /6/ and /7/ where we have proved that the cobalt layer had an hexagonal structure. In the present paper we are concerned with high resolution electron microscopy: front views have been performed on sandwiches for which cobalt thickness is 3nm , the total thickness being 20nm .

Fig.1: Electron diffraction pattern on a Au/Co/Au Sandwich.

Fig.1 shows an electron diffraction pattern of this sample, the direction of observation being parallel to $.[111]$. Au corresponding to $.[00.1]$.Co. Gold and cobalt reflection are respectively labelled. G and C, dd corresponding to double diffraction between gold and cobalt; the bright spots correspond to (202) planes and the dim ones to $1/3(422)$ in cubic notation (layer spots).

HREM STUDIES AND OPTICAL DIFFRACTION

The high resolution interference images have been realized with a JEOL 200 CX microscope (accelerating voltage 200 KV, wave

Fig.2a: H.R. Electron micrography (symmetrical position $// .[110]$)

Fig.2b: Corresponding optical diffractogram.

Fig.3a H.R. Electron micrography (symmetrical position $// .[111]$).

Fig.3b: Corresponding optical diffractogram.

length 2.51 μm). Figures 2, 3 and 4 are bright field images obtained with the objective aperture n°1 on fig.1, including the (202) and 1/3 (422) reflections. The different zones of the micrographs are systematically analyzed on an optical bench by a 1mV He-Ne Laser beam. The optical diffractogram obtained corresponds to a spatial resolution of 7 nm approximatively on the crystal.

Fig. 2a and 2b show an HREM micrograph in the vicinity of a hole, and the corresponding optical diffractogram. Angle and spatial frequencies ratios, show without ambiguity that it is a cfc diagram for a direction parallel to $[110]$ (symmetrical position), for which the visualized planes are (111) and (002). Fig 3a and 3b correspond to another zone near the same hole corresponding to an orientation of

Fig 4: H.R Electron Micrography (General view of the sample)

the cfc crystal parallel to $[111]$ (symmetrical position). The planes which are visualized correspond to the $1/3(422)$. Both these observations are consistent with the existence of different gold crystallites strongly tilted one versus the other. The absence of cobalt in that part of the sandwich could be linked to the proximity of the hole. These two micrographs will be used for calibration in the following study. Fig.4 shows a HREM micrograph of the sandwich corresponding to a large zone further from a hole. The optical diffractogram realized on different parts of fig.4 are shown on fig. 5a to 5f. They enable us to follow the very rapid changes in orientation when we move from one part of the sample to the other. The planes most usually imaged are gold (111) .

Fig.5: Optical diffractogram corresponding to zone a,b,c,d,e,f, on fig.4

Zone a (bottom right on fig.4) corresponds to a direction of observation in the gold crystal, roughly parallel to $[110]$ the difference of intensity for the spots (111) as well as for (200) suggests a misorientation of a few degrees with respect to the latter direction. Moving horizontally to zone b then c we observe simultaneously (111) planes rotated by 3° with respect to those of zone a (direction $[110]$) as well as the apparition of $1/3(422)$ planes. This suggests the existence of different crystallites slightly rotated or strongly tilted with respect to those of zone a. But we must keep in mind that both upper and lower gold layer contribute to the diffraction and that it is impossible to distinguish between them from a diffraction pattern.

Moving vertically to zone d the two main orientations of the gold crystal are observed since we image simultaneously planes (111) and $1/3(422)$. Furthermore a new spatial frequency labelled 'EE' appears, corresponding to a reticular distance intermediate between gold and cobalt.

At last, on zone f and e the presence of cobalt is ascertained by the identification of a plane (10.0) of the hexagonal structure for an orientation parallel to $[00.1]$. This result is confirmed by the existence of large moiré fringes corresponding to double diffraction between gold and cobalt.

INTERPRETATION AND CONCLUSION

H.R.E.M. imaging of Au/Co/Au magnetic sandwiches followed by systematic optical diffraction can yield valuable informations on short range misorientation and tilt of the crystallites composing the layer. The average direction is parallel to $[111]_{Au}$ but locally we can find directions such as $[110]_{Au}$, the angle with the previous one being 35° . Furthermore this variation is very rapid and can arise on distances of the order of 7nm as shown by the corresponding evolution of the optical diffraction. We have also noticed that the cobalt layer was not uniform: There are little spots in the sandwich without cobalt. Our first conclusion is that the magnetic sandwiches are composed of very small regions strongly tilted one with respect to the other the general aspect being that of a buckled layer. The consequences on the magnetic properties of the specimen will be checked later on by NMR measurements.

Our second conclusion concerns the observation of reticular distances intermediate between those of Au and Co, in zones where the existence of moiré fringes suggests that cobalt is present. This result could be interpreted as thin layers of cobalt whose parameter is adjusted to that of the surrounding gold.

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