Provided for non-commercial research and education use. Not for reproduction, distribution or commercial use.



**This article was published in an Elsevier journal. The attached copy is furnished to the author for non-commercial research and education use, including for instruction at the author's institution, sharing with colleagues and providing to institution administration.**

**Other uses, including reproduction and distribution, or selling or licensing copies, or posting to personal, institutional or third party websites are prohibited.**

**In most cases authors are permitted to post their version of the article (e.g. in Word or Tex form) to their personal website or institutional repository. Authors requiring further information regarding Elsevier's archiving and manuscript policies are encouraged to visit:**

**<http://www.elsevier.com/copyright>**



Available online at www.sciencedirect.com





Materials Science and Engineering C 27 (2007) 1466–1469

www.elsevier.com/locate/msec

# Study of structural microstructural and magnetic properties of very thin  $Co<sub>50</sub>Pt<sub>50</sub>$  films deposited by PLD

E. Agostinelli<sup>a</sup>, S. Laureti<sup>a</sup>, G. Varvaro<sup>a,\*</sup>, A. Generosi<sup>b</sup>, B. Paci<sup>b</sup>, V. Rossi-Albertini<sup>b</sup>, G. Scavia<sup>a</sup>, A.M. Testa<sup>a</sup>

<sup>a</sup> ISM-CNR, P.O.Box10, 00016 Monterotondo Stazione (Roma), Italy b ISM-CNR, Via Fosso del Cavaliere, 00100 Roma, Italy

Received 7 May 2006; received in revised form 12 July 2006; accepted 12 July 2006 Available online 7 September 2006

#### Abstract

Magnetic Co<sub>50</sub>Pt<sub>50</sub> films few nanometers thick have been deposited by Pulsed Laser Deposition on MgO (100) single crystal at a deposition temperature of 600  $^{\circ}$ C and then annealed "in situ" for different time. A very thin Pt underlayer (<2 nm) with an incomplete coverage is used as a possible template to favour the growth of isolated grains of the magnetic layer. The correlation between the structural, microstructural and magnetic properties has been studied as a function of the annealing time. In particular, measurements gave evidence of the presence of two competing effects of the annealing treatment: formation of hard fct-CoPt phase and thermal diffusion process between Pt and CoPt layer resulting in a reduction of the crystallographic order. © 2006 Elsevier B.V. All rights reserved.

Keywords:  $Co_{50}Pt_{50}$ ; PLD; Magnetic recording; Annealing effect; thin films

#### 1. Introduction

Chemically ordered  $L1_0$  Co<sub>50</sub>Pt<sub>50</sub> thin films, due to their high magneto-crystalline anisotropy [1–3], have received a great interest in the last years for their potential application in high density magnetic recording [4,5]. The increasing of data storage density imposes the need of a material consisting of magnetically isolated grains with a size of few nanometers  $($  < 10 nm)  $[6]$ . In such dimension range high magnetic anisotropy is needed to minimize the magnetization vector fluctuations that tend to destabilize the magnetization of recorded bits ("superparamagnetic regime").

The phase diagram of the CoPt alloy [7] shows, in the region close to the equiatomic composition, a phase transition  $(T \approx 750 \degree C)$  from a chemically disordered face-centered-cubic (fcc) structure at higher temperatures to an ordered face-centered-tetragonal (fct) structure  $(L1<sub>0</sub>$  phase) at lower temperatures constituted by alternated Co and Pt planes aligned along the [001] direction. Generally speaking, the as-deposited alloy is characterized by the disordered structure independently from the deposition temperature, and an annealing treatment is necessary to assist the formation of the long range chemical ordering [1].

This paper is dedicated to the study of few nanometers thick  $(\sim 5 \text{ nm}) \text{Co}_{50} \text{Pt}_{50}$  films deposited by Pulsed Laser Deposition (PLD) on MgO (100) single crystal. A very thin Pt underlayer  $(<$  2 nm) showing an incomplete coverage is used as a possible template for an isolated grain growth of the magnetic layer. We studied how the duration of the post deposition annealing treatment modifies the structural and magnetic properties of such system.

The peculiarity of PLD compared to other deposition techniques is the possibility of growing films from a plasma formed by high energy species  $(E_k)$  between 10–100 eV), resulting in an enhanced surface mobility of the adatoms that settle on the substrate [8]. The surface diffusion during the

<sup>⁎</sup> Corresponding author. Tel.: +39 06 90672651; fax: +39 06 90672470. E-mail address: gaspare.varvaro@ism.cnr.it (G. Varvaro).

<sup>0928-4931/\$ -</sup> see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.msec.2006.07.015

E. Agostinelli et al. / Materials Science and Engineering C 27 (2007) 1466–1469 1467



Fig. 1. Diffraction patterns of CoPt/Pt/MgO thin films annealed at 600 °C, as a function of the scattering parameter and of the annealing time. The vertical arrows indicates the positions of the Bragg reflections: (a) CoPt fct [110],  $q= 0.2405$  nm (b) Pt [111]  $q= 0.2655$ , (c) CoPt fcc [111]  $q= 0.2875$  nm, (d) MgO [200]  $q=0.2981$  nm, (e) Pt [200]  $q=0.3200$  nm, (f) CoPt fcc [200]  $q=0.3301$ .

growth, generally leads to very smooth film surface and high epitaxial degree, as required for hard disk drive applications.

#### 2. Experimental

Samples were prepared by PLD using a Lambda Physik excimer laser KrF ( $\lambda$ =248 nm) at an energy fluence of 3 J/cm<sup>2</sup> and a pulse repetition rate of 10 Hz. Depositions were performed on polished MgO (100) single-crystal substrates in a frontal geometry with a distance of 5.5 cm between target and substrate.

Films were deposited in a HV-chamber with a liquid nitrogen trap ( $P_{\text{back}} = 7 \times 10^{-6}$  Pa;  $P_{\text{growth}} = 2 \times 10^{-5}$  Pa) at  $T_{\text{dep}} = 600$  °C and for a deposition duration of 2 and 5 min for the underlayer and the magnetic layer, respectively. A carousel with Pt and a composite CoPt targets (formed by circular sectors of Co and Pt pure elements) was used to sequentially deposit the two different layers. Therefore, samples were annealed in situ at 600 °C for a time ranging from 45 up to 150 min.

Structural characterization was performed by Energy Dispersive X-ray Diffraction analysis (EDXD) by a non-commercial instrument, making use of the white radiation coming from a water-cooled X-ray W anode tube (Philips, model PW2214/20) supplied at 55 kV and 20 mA. Magnetic properties were measured at room temperature using Vibrating Sample Magnetometry (VSM) and SQUID in a field of about 2 T applied both parallel and perpendicular to the film surface. The microstructure was analyzed by commercial AFM and STM-UHV systems.

## 3. Results and discussion

Diffraction patterns of a set of CoPt/Pt/MgO samples submitted to annealing at 600 °C for different times (0' $\lt t_{\text{ann}}$  < 150′) were collected at the same scattering angle  $(2\vartheta = \vartheta_{\text{incident}} +$  $\vartheta_{reflected} = 7^{\circ}$ ) in reflection mode, but at different tilt angle  $\alpha$ , where  $\alpha = (\vartheta_{\text{incident}} - \vartheta_{\text{reflected}})/2$  [9], and are shown in Fig. 1. In all the samples contributions from MgO  $(MgO < 200)$  at  $q= 0.2984(5)$  nm<sup>-1</sup>), Pt (Pt<111>(b) at  $q= 0.2655(5)$  nm<sup>-1</sup>) and fcc-CoPt phase (fcc-CoPt<111>(c) at  $q= 0.2875(5)$  nm<sup>-1</sup>) were detected. For the annealing time up to  $120'$  <200 $\triangleright$  reflections of both Pt (e) and fcc-CoPt (f) phase are also present (at  $q=0.3200$ (5) nm<sup>-1</sup> and  $q=0.3301(5)$  nm<sup>-1</sup> respectively). Due to the different  $\alpha$  values, the relative intensities of the peaks reported in Fig. 1 are not directly comparable. However, in this way, even the very weak signals coming from the thin film, otherwise hidden by the substrate reflections, can be detected. Indeed, although the comparison is only qualitative, it can now be



Fig. 2. (a) Coercive field at room temperature as a function of the annealing time. (b) Room temperature VSM experimental curves in longitudinal (–●–) and perpendicular  $(-\triangle)$  geometry of the sample annealed for 120'.

1468 E. Agostinelli et al. / Materials Science and Engineering C 27 (2007) 1466–1469



Fig. 3. (a) STM image and (b) deep profile of the sample annealed for 120′.

noticed that the intensity ratio between  $\langle 111 \rangle$ (b) and  $\langle 200 \rangle$ (e) Pt peaks intensity increases with the annealing time, being  $11$  the thermodynamically most stable orientation. As a consequence the fcc-CoPt phase assumes either the  $\langle 111 \rangle(f)$  or the  $\langle 200 \rangle$ (f) orientations according to the underlayer crystallographic structure. For an annealing time of  $120'$  the  $L1_0$ −CoPt ordered phase was also detected as can be seen by the appearance of the <110>(a) peak (at  $q=0.2405$  (5) nm<sup>-1</sup>), which is not allowed in the cubic diffraction pattern [10]. For longer annealing duration the (a) reflection is still present but with a very low intensity and also the (b) Pt peak is barely visible. This behaviour is probably due to the thermal diffusion between Pt and CoPt layers that causes a reduction of the crystallographic order.

The variation of the coercive field  $H_c$  in the film plane with the annealing time in reported in Fig. 2a. In particular  $H_c$  increases with increasing the fct-CoPt fraction, reaching the highest value of  $2.27 \times 10^5$  A/m (2860 Oe) for  $t_{\text{ann}} = 120'$  when the magnetically hard fct phase fraction is at the maximum concentration. However, the fcc-CoPt phase is always present and, being magnetically soft, tends to reduce the coercive field. Magnetic measurements applying the field perpendicularly to the film plane, were performed in order to determine the film anisotropy of the best sample. The magnetization curve showed hysteresis also along this direction with a coercive field of  $1.99 \times 10^5$  A/m (2500 Oe) (Fig. 2b). This behaviour can be explained considering that the fct-CoPt alloy has the magnetocrystalline anisotropy axis along the [001] direction and in our case, being the fct domains [110] oriented, the *c*-axis lies on the film plane. However, if the [110] orientation presents a large angular distribution, the [001] axis can form an angle with the film surface whose perpendicular component is not zero thus contributing, at a first approximation, to the perpendicular anisotropy. The AFM image of the Pt underlayer illustrates that the film was constituted by wellseparated islands [11]. The STM analysis of the CoPt surface showed that the magnetic layer was not formed by isolated grains. Actually, although the fully coverage of the surface was not reached for this thickness, all the films were electrically continuous. Moreover, as shown in Fig. 3, the surface was constituted by very smooth islands (roughness  $< 0.2$  nm) only partially separated by deep holes whose height is comparable to the film thickness. The size of the islands, however, is quite large due to both the high deposition temperature and the thermal treatment, and further increased with the annealing time.

### 4. Conclusion

In summary, we have prepared by PLD CoPt alloy films with a thickness of few nanometers deposited onto a discontinuous Pt underlayer and studied the correlation between structural, microstructural and magnetic properties as a function of the annealing time. Both X-ray diffraction and magnetic measurements gave evidence of two competing effects of the annealing treatment: partial thermodynamic transition to the magnetically hard fct-CoPt phase from the fcc-CoPt phase and thermal diffusion process between Pt and CoPt layers that causes a reduction of the crystallographic order.

The magnetic properties can be explained by the crystallographic structure. In particular, the coercive field increases with increasing the fct-CoPt fraction and reaches its maximum value for  $t_{\text{ann}}$ = 120′. Probably a large angular distribution of [110] axis is the main source of the isotropic behaviours of the best sample.

Microstructural analysis of the CoPt surface showed that the magnetic layer is formed by very smooth islands partially separated by deep holes. The lateral size of the islands however is quite large and increases with the annealing time.

## Acknowledgments

The authors wish to thank Mr. Daniele Petrelli and Mr. Pasquale Filaci for their technical assistance in film deposition and magnetic measurements.

## References

[1] V. Parasote, M.C. Cadeville, G. Garreau, E. Beaurepaire, JMMM 198–199 (1999) 375.

E. Agostinelli et al. / Materials Science and Engineering C 27 (2007) 1466–1469 1469

- [2] R.A. Ristau, K. Barmak, L.H. Lewis, K.R. Coffey, J.K. Howard, J. Appl. Phys. 86 (1999) 4527.
- [3] H. Shima, K. Oikawab, A. Fujita, K. Fukamichi, K. Ishida, S. Nakamura, T. Nojima, JMMM 290–291 (2005) 566.
- [4] K.R. Coffey, M.A. Parker, J.K. Howard, IEEE Trans. Magn. 31 (1995) 2737.
- [5] K. Barmak, J. Kim, L.H. Lewis, K.R. Coffey, M.F. Toney, A.J. Kellock, J.-U. Thiele, J. Appl. Phys. 98 (2005) 1.
- [6] D. Weller, A. Moser, L. Folks, M.E. Best, W. lee, M.F. Toney, M. Schwickert, J. Thiele, M.F. Doerner, IEEE Trans. Magn. 36 (2000) 10.
- [7] J.M. Sanchez, J.L. Moran-Lopez, C. Leroux, M.C. Cadeville, J. Phys., Condens. Matter 1 (1989) 491.
- [8] P.R. Willmott, J.R. Huber, Rev. Mod. Phys. 72 (2000) 315.
- [9] B. Paci, A. Generosi, V. Rossi Albertini, E. Agostinelli, G. Varvaro, D. Fiorani, Chem. Mater. 16 (2004) 292.
- [10] A. Cebollada, R.F.C. Farrow, M.F. Toney, in: H.S. Nalwa (Ed.), Magnetic Nanostructures, Chapter 3, American Scientific Publisher, 2000, p. 93.
- [11] G. Scavia et al. in press.