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ESRF

Experiment Report Form

ESRF	**Experiment title: X-ray diffraction and reflectivity of MBE grown Co-Ag superlattices**	**Experiment number**: 25-02-663
Beamline: BM25B	**Date of experiment: CRG-beam time** from: 10/12/2008 at 8:00 to: 14/12/2008 at 8:00	**Date of report**: 1/09/2009
Shifts:12	Local contact(s)**: Dr. Pilar FERRER**	*Received at ESRF:*

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Introduction

The aim of these measurements was to use Synchrotron X-ray diffraction and reflectivity (XRD, XRR) to investigate the microstructure of different types of Co-Ag films that exhibit interesting magnetic properties [1]. Metallic multilayer films are challenging systems in order to provide materials with new specific properties, becoming especially relevant for ultra-high density magnetic recording, spintronics or magneto-plasmonics. We focused on Co/Ag superlattices (SL) with ultra thin (2-6 Å) Co layers, since perpendicular magnetic anisotropy [1,2] and oscillations of the magneto resistance (MR) and exchange coupling [3,4] in Co/Ag multilayers have only been reported for very thin Co layers (<10 Å). Previous reports [1-8] have shown that achieving Co/Ag multilayers with flat, laterally continuous and compositionally abrupt interfaces is particularly difficult, also pointing out the singular and still controversial features of the Co/Ag system respect to other metallic multilayers. Several groups [6-8] even indicate that a granular system is actually formed when Co layers are very thin, invoking it to explain the magnetic properties observed for this thickness range. Microstructure seems thus to play a relevant role on the magnetic behaviour of Co/Ag multilayers.

Experimental

The samples of this study were grown by molecular beam epitaxy (MBE) on clean surfaces of single-crystal substrates with an intermediate Ag buffer layer. Growth was performed at the ICMM institute in Madrid and routinely characterized by "in situ" reflection high energy electron diffraction (RHEED) and Auger electron spectroscopy (AES), as well as by conventional X- ray (Cu*K*α) diffraction and reflectivity (XRD, XRR). Two types of sample series were studied at BM25B (see layer sequence in Fig. 1).

* 1st sample set: Old Co/Ag SL samples grown on Si(111) substrates. They consist of N=5 periods of the [Ag(t_m)/Co(t_n)] SL sequence, keeping fixed the Co layer content (t_n) and varying that of Ag (t_m) from about 3 Å

to 15 Å. These samples have been previously studied [1] by VSM, MOKE, FMR, AFM and TEM (vibrating sample magnetometry, magneto-optic Kerr effect, ferromagnetic resonance, atomic force microscopy and transmission electron microscopy, respectively).

* 2nd sample set: New samples, grown just few days before the ESRF measurements and which had not been characterized by AFM, VSM, MOKE, FMR,… (such measurements are now underway). Their nominal growth parameters are detailed in Table 1. In comparison to the first sample set, the new SLs have thicker periods (i.e., thicker Ag layers) and also a higher number of periods (N), in order to facilitate the detection of the SL periodicity at low angles (XRR). Superlattices with similar parameters were grown on Si(111)-7x7 substrates and also on clean MgO(001) surfaces, to compare results on two substrates and orientations.

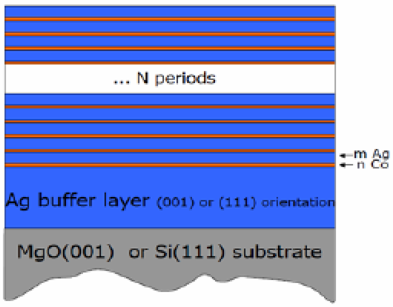

Fig.1: *Sample layer sequence*

sample	*substrate*	*BL t(Å)*	t_n(Å)	t_m(Å)	*P(Å)*	*N*
#1252	MgO(001)	Ag 200 + 200	3,5	36,7	40	15
#1253	Si(111)	Si 2500 + Ag 200 + 200	3,5	36,7	40	15
#1255	MgO(001)	Ag 200 + 200	1,8	18,4	20	30
#1256	Si(111)	Si 2500 + Ag 200 + 200	1,8	18,4	20	30
#1251	MgO(001)	Ag 400	21.3 x = 0.36	36,7 1-x = 0.64	58	15

Table 1: *Nominal growth parameters of the 2nd sample set.*

Results

Experiments at BM25-B involved XRR, XRD and reciprocal space mapping (RSM) runs using the six-circle diffractometer. Measurements performed in the θ/2θ geometry confirm that Ag planes parallel to the sample surface have a (001) orientation for films grown on MgO(001) substrates, and a (111) orientation for those on Si(111), in agreement with our RHEED and XRD data for these samples and also previous reports on Ag-Co SLs [1-3,5]. On the other hand, while for the first set of samples we did not find clear evidences of SL formation, the experiments performed on the second sample set were rather successful.

In particular, for films grown on MgO(001) surfaces, clear SL features (undoubtedly related to the Co-Ag SL periodicity) were found on the XRR spectra and also around intense Ag reflections in the experiments at BM25-B, whereas XRR and XRD runs performed on the same samples with our conventional laboratory XRD techniques in Madrid did not reveal such features. Superlattice peaks are nicely observed (up to the 4th order for sample #1252 and up to the 2nd order for sample #1255) in "L-scans" and RS maps recorded around the (111) reflection of Ag. Such results, which are among the best reported for Ag-Co multilayers, demonstrate the growth of well ordered and good quality coherent SL films, even for the case of extra-thin Co layers, and do not support interpretations in terms of granular systems for our samples.

Excellent reflectivity spectra are obtained for the Co/Ag SLs grown on MgO(001) substrates. Although the SLs grown on Si(111) still display good reflectivity features, when samples with the same nominal SL parameters are compared, a real degradation of the XRR spectra is observed for samples grown on Si (111) respect to those on MgO(001).

Experiments at BM25-B have also provided evidences of SL formation on Si(111) substrates (e.g., the XRR spectra of sample #1253 display a SL feature similar to the first one observed for #1252, both samples having the same nominal periodicity). Nevertheless, the detection of SL features was more difficult for films grown on Si(111) than on MgO(001), probably due to both intrinsic (lower XRR signal) and external (technical) reasons; note that "L- scans" cannot be performed in this case around the most intense Ag reflection: (111). Concerning XRD experiments, sets of peaks periodically distributed have been observed around the Ag(111) signal in a θ/2θ geometry for #1253 and #1256 samples; however, a detailed analysis shows that they cannot be interpreted in terms of the SL periodicity.

The XRR results of the 2nd sample set reveal that the morphology of the layers and interface definition is slightly better for the superlattices of smaller period, on both MgO(001) and Si(111) substrates.Taking into

account the evolution of the RHEED patterns during the MBE growth of these samples, such improvement is interpreted in relation to the thinner Co layers involved (Table 1).

In summary, experiments at BM25B have been successful in providing evidences of high quality SLs, specially for samples grown on MgO(001) substrates. They demonstrate that the growth protocols and parameters used for the 2nd set of samples (Table 1) are adequate to preserve a coherent epitaxial and layered arrangement of Co and Ag films within the SL, even for extremely thin Co layers. This high degree of control manufacturing model systems of Co-Ag superlattices is crucial to address a reliable investigation of the magnetic or magneto-plasmonic properties on these heterostructures. One of the next challenges is to find evidences of SL formation for Co-Ag multilayers similar to those of Table 1 but with still thinner Ag layers (t_m = 1.5 -0.3 nm), to complete the investigation in the range of highest interest and controversy from the point of view of the magnetic properties [1,3,4].

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