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# EXAFS STUDY OF SPUTTERED C₀/AI MULTILAYERS BY ELECTRON DETECTION: STRUCTURAL DIFFERENCE DUE TO DEPOSITION CONDITION

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## ABSTRACT

EXAFS measurements and X-ray diffraction analysis have been made on magnetron sputtered Co-Al multilayers with modulation wavelengths of 1 to 2 monolayers and sputtering Ar gas pressure of 3 to 7 mTorr. X-ray diffraction results cannot clearly identify the phases present in the samples because the major diffraction peaks of different phases overlap. Preliminary analysis of the EXAFS data shows that Co atoms exist in two or more different phases, i.e.  $\alpha$ -Co, CoAl(CsCl typc), and possibly a solid solution of Co and Al with some Co atoms occupying the substitutional sites of fcc Al.

### INTRODUCTION

Multilayers or layered structures are materials of great interest, since they exhibit a variety of properties which can be useful in certain applications. Especially some Co/nonmagnetic metal multilayers are promising as magnetic recording media<sup>1</sup>. An important factor in understanding multilayers is a structural analysis. X-ray diffraction and cross-sectional TEM are widely used in structural studies of multilayers in the area of semiconductor research. But the application of X-ray diffraction to a disordered system with layers and interfaces has limitations and for very thin multilayers (< few hundred angstroms) XRD cannot give accurate information about the internal structure. XTEM is a direct observation method and HRTEM can give even atomic level information, however the specimen preparation is destructive and may damage the original structure of multilayers made of materials with different sputtering yields during the thinning by ion milling. Moreover, multilayers with disordered interfaces cannot be analyzed by those techniques. In such studies, EXAFS is valuable as a non-destructive analytic tool. The Co-Al multilayer system, which has a 13.6% lattice mismatch(d<sub>Al(111)</sub>=2.34Å, d<sub>Co(0002)</sub>=2.02Å), was chosen for the present study. We have measured the electron-yield EXAFS of Co-Al multilayers deposited at three different sputtering Ar gas pressures and the

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modulation wavelengths of 1 to 2 layers.

#### EXPERIMENTAL

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Co-Al multilayers were deposited onto float-glass substrates by a two-source dc-planar magnetron sputtering method while rotating the substrate. During the deposition from two sputter sources, the water cooled substrate was rotated at 16.6 rpm. Two cylindrical shields are used to prevent cross-contamination from the other source. The thickness was about 300Å. The sputtering Ar gas pressure and the thickness ratio of Co/Al were varied for different samples. EXAFS measurements of those multilayers were made on the beamline X11A at the National Synchrotron Light Source located at Brookhaven National Laboratory, using the electron detection technique<sup>2</sup>.

#### RESULTS AND DISCUSSION

The raw data were analyzed by the EXAFS analysis package developed by the University of Washington. In order to get the phase identification, thin film X-ray diffraction patterns for the samples were obtained using a Rigaku DMAX-III diffractometer with a thin film attachment which fixes the X-ray incidence angle. For three samples with different sputtering Ar pressure of 3, 5 and 7 mTorr, XRD patterns show only one clear peak at  $2\theta = 44.9$  degree which may be Al(200), Co(0002) or CoAl(110). As the thickness of Al layer increases, the diffraction peak gets more pronounced at all three Ar gas pressures as shown in Fig. 1. In Fig. 2, the Fourier transforms for those samples with various Al layer thicknesses deposited at 7 mTorr of Ar pressure, show peaks between 1.5 and 2.8 Å of radial distance and give some information on the phases present in the multilayers.

Material	Туре	N(1st)	R(1st)	N(2nd)	R(2nd)	N(3rd)	<b>R</b> (3rd)
Al	FCC	12	2.86	6	4.04	24	4.95
α-Co	HCP	12	2.49(6)	6	3.53	2	4.07
			2.50(6)				
CoAl	CsCl	8(Al)	2.47	6(Co)	2.86	12(Co)	4.04

\* R is in angstroms and in CoAl a Co atom is at the origin.

Table.1 Structural data calculated from Pearson's Handbook.(rcf.3)

The splitting of major peak in the region of nearest neighbor contribution for  $Co_1Al_2$  deposited at 7 mTorr clearly shows that, in the multilayer film, Co atoms exist in more than one phase. As shown in Table 1, in pure  $\alpha$ -Co, a Co atom has 6 nearest neighbors on its basal plane at the distance of 2.49Å and 6 others on top and bottom at the distance of 2.50Å. The most probable second phase Co atoms can form with Al atoms is CoAl of the CsCl type structure. In CoAl, a Co atom has 8 Al atoms as the nearest neighbors at the distance of 2.47Å and 6 Co atoms as the 2nd near neighbors at the distance of 2.86Å. Since the backscattering phase values of Co and Al atoms are quite different, an Al shell and a Co shell at such a close distance would result in a large interference effect<sup>4</sup>. Thus the peak positions may not accurately represent the actual shell positions. A quantitative fitting has not been completed yet, and for now a possibility of even a third phase cannot be ruled out. In pure Al, an Al atom has 12 nearest neighbors at the distance of 2.86Å. Thus if some Co atoms are in substitutional sites of fcc Al, a Co atom may have Al nearest neighbors at the distance of 2.86Å.

Whether a Co atom has a Co shell(CoAl) or an Al shell(solid solution) at the distance of 2.86Å can be determined by a fitting because of the large difference in the backscattering phase between Co and Al.

Therefore a careful fitting of the Fourier transforms as well as the inverse transformed EXAFS will be attempted to confirm the presense of these possible phases. The Fourier transform peaks for  $Co_1Al_1$  multilayer films shown in Fig. 3 also strongly indicated the presence of more than one phase. Thus a fitting will be also attempted to determine whether more than one of  $\alpha$ -Co, CoAl, and Al(Co) phases exist in these multilayer films.

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Fig. 1 Thin Film X-ray diffraction results.  $CuK\alpha$ , incidence angle=1.5 degree. (a)  $Co_1Al_1$ ,  $Co_1Al_{1.5}$ ,  $Co_1Al_2$  (from top to bottom) deposited at 3 mTorr. (b)  $Co_1Al_1$ ,  $Co_1Al_{1.5}$ ,  $Co_1Al_2$  (from top to bottom) deposited at 7 mTorr. In both cases, the peak at 44.9 degree grows as the Al layer thickness increases.



Fig. 2 Fourier transformed EXAFS showing that the increasing thickness of Al layer induces the peak splitting, which may be due to the presence of two or more phases.



Fig. 3 Fourier transformed EXAFS showing the effect of sputtering Ar gas pressure on the multilayer structure. As the gas pressure increases from 3 mTorr to 7 mTorr, the position of the major peak shifts a little.

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