Electron Yield XAFS Study of Evaporated Co/Pd Multilayers with Various Thickness Ratios of Co to Pd Sublayers: Simulations of the Co K-edge XAFS and Fourier Transforms

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M. CHOI, J. H. JOO,[†] S. K. KIM,^{††} J. S. KANG,^{††} Y.-P. LEE^{††} S.-C. SHIN,^{†††} and S. M. HEALD^{††††}*Department of Physics, Catholic University Medical College, Seoul, 137-701, Korea*[†]*Materials Design Laboratory, Korea Institute of Science and Technology, Seoul, 136-791, Korea*^{††}*Basic Science Branch, Research Institute of Industrial Science and Technology, Pohang, 790-600, Korea*^{†††}*Department of Physics, Korea Advanced Institute of Science and Technology, Taejeon, 305-701, Korea*^{††††}*Brookhaven National Laboratory, Upton, NY 11973, USA*

Electron-yield XAFS measurements were made on e-beam evaporated Co/Pd multilayers with various sublayer thicknesses and different thickness ratios of Co to Pd sublayers. The Co K-edge and the Pd K-edge XAFS data were obtained for the Co/Pd multilayers with the sublayer thicknesses of 3Å/4Å, 15Å/4Å, 3Å/15Å, 2.1Å/13.5Å, and 2.2Å/4.5Å. The Fourier transforms of the Co K XAFS for most samples show a splitting of major peak, and the magnitude ratio of these split peaks varies systematically with the thickness ratio of the Pd sublayer to the Co sublayer, whereas the Fourier transforms of the Pd K XAFS for the same samples do not show a splitting of peaks. As a preliminary analysis, the Co K XAFS and the split peaks in the Fourier transform for the Co/Pd(3Å/4Å) case were simulated by using the FEFF calculations, and the Co K XAFS and the major peak in the Fourier transform for the Co/Pd(15Å/4Å) case were also simulated consistently.

KEYWORDS: Electron-yield XAFS, multilayers, FEFF

§1. Introduction

Co/Pd multilayers with a perpendicular magnetic anisotropy are considered to be promising as magnetooptical recording device materials. The magnetic and magnetooptical properties of Co/Pd multilayers have recently been studied extensively,¹⁻³⁾ and the properties such as the magnetic anisotropy, the coercivity, and the Kerr rotation which are useful for magnetooptical recording have been shown to depend strongly on the sublayer thickness and on the interdiffusion at the interfaces. The tunability of these properties is valuable in practical applications, but the microstructural aspect of this sublayer thickness dependence has not been studied fully. Especially, the perpendicular anisotropy is deemed to originate from the interface anisotropy. However, the atomic structure and the interdiffusion in the interface region have not been characterized so far. The electron-yield XAFS measurements were made on e-beam evaporated Co/Pd multilayers with various sublayer thicknesses to investigate the structural variation in the atomic environment for various thickness ratios of Co to Pd sublayers. Also the atomic interdiffusion at the interfaces will be studied in the future with the glancing angle method.^{4,5)}

§2. Experimental

Co/Pd multilayers with a few atomic layers of Co were deposited onto float-glass substrates with a base pressure of $0.5 \sim 1.5 \times 10^{-7}$ mbar by alternating the electron-beam and thermal evaporation of Co and Pd in an ultra high vacuum(UHV) physical vapor deposition(PVD) system(Leybold L 560 UV). The pressure during the de-

position was $0.5 \sim 2.0 \times 10^{-6}$ mbar. A quartz crystal oscillator interfaced with XTC(crystal thickness controller) was used to monitor the layer thickness, and the evaporation rate of $0.1 \sim 0.3$ Å/sec was used. Co/Pd multilayers with various sublayer thicknesses such as 3Å/4Å, 3Å/15Å, and 15Å/4Å were prepared in this system and had the total thickness of about 100 or 200 Å. Two samples of 3Å/4Å were prepared with and without a predeposition of a 65 Å thick Pd layer. Also a 230 Å-thick Co film and a 120 Å thick Pd film were prepared. Two other samples were deposited onto quartz substrates by e-beam evaporation of Co and Pd in a different vacuum system maintained at about 2×10^{-6} Torr during the deposition, and had the total thickness of 1300 Å with the Co/Pd sublayer thicknesses of 2.1Å/13.5Å and 2.2Å/4.5Å. Using a He-flow electron detector, electron-yield XAFS measurements were made on these samples and a 7μ thick Co foil at the beamline X-11A of the National Synchrotron Light Source. The electron beam energy was 2.523 GeV and the current was about 200 mA. A N₂ gas flow was used for an I₀ chamber. Si (111) monochromator crystals in a two-crystal mode were used and the resolution was about 1 eV at the Co K-edge energy. The energy were calibrated by using a Cu K-edge near-edge feature and harmonics were rejected by detuning. Figure 1 shows a typical raw data. The samples were characterized with X-ray diffraction(XRD) and some magnetic measurements will be also made on the same samples.

§3. Results and Discussion

Figure 1 shows a typical raw data of electron-yield XAFS spectrum and a fluorescence corrected spectrum. The data collected in electron detection mode were cor-

* Present address: Department of Material Science and Engineering, Kunsan National University, Kunsan, 573-360, Korea.

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Fig. 1. Comparison of the XAFS spectra before and after the fluorescence correction.

rected by the fluorescence correction program (FLCORR) which divides out the energy dependence of the I_0 chamber and thus enables the comparison of the data with transmission data. The Co K XAFS data for Co/Pd samples with various thickness ratios of Co/Pd were Fourier transformed, and the major peaks for the 3Å/15Å and 3Å/4Å samples showed a splitting. The Fourier transform peak for the 15Å/4Å sample was similar to that for pure Co. These Fourier transforms are shown in comparison in Fig. 2. The quality of data for the 3Å/4Å sample with a predeposition of Pd was better, and the data for that sample was used in the analysis. The Fourier transform of the Pd K-edge XAFS for the same sample did not show a splitting, and the XRD also showed a single sharp peak. The previous XRD studies did not show a sharp peak for a Pd thin film, showed only a weak bump for a Co-Pd alloy thin film, and showed no peak for a Co thin film.⁶ In comparison, the major XRD peaks for Co/Pd multilayers were sharp and corresponded to the d spacings of the (111) plane, and the (111) d spacing for Co/Pd multilayers with different thickness ratios of Co/Pd are known to be roughly equal to the weighted average of the (111) d-spacing values of Co and Pd. Since the multilayer samples are only about 100 Å or 200 Å thick, the small satellite peak for $n=-1$ near the main peak is hard to detect without a thin film attachment, and only the 15Å/4Å sample showed a good satellite peak. The XRD results will be presented in another paper after the satellite regions are more carefully scanned with a very high power X-ray diffractometer. Though an XRD peak around 40° cannot give an accurate d-spacing value, the d spacing for a multilayer sample roughly corresponded to the weighted average of Co and Pd d values. Whereas the split peaks in the Fourier transform of the Co K XAFS for the 3Å/4Å showed a separation of 0.46 Å. A similar splitting of the Fourier transform peak was also observed in the case of Co/Al multilayers,⁷ and it was of interest whether the split peaks come from the separation of two subshells or the different backscattering amplitudes of different atomic shells at the same radial distance can result a splitting

Fig. 2. Fourier transforms of the Co K-edge XAFS for Co/Pd multilayers with different thickness ratios of Co to Pd sublayers. (The y-scale on the right side is for the dotted line.)

of the Fourier transform peak due to the interference effect. Thus the Co K XAFS and the Fourier transform for the 3Å/4Å sample were simulated with the FEFF calculations. In the beginning of the simulation process, first the Co K-edge XAFS data collected in transmission mode for 7μ foil of pure Co was simulated by using the FEFF parameter values to check the accuracy of the FEFF values. The XAFS of a backscattering Co shell around a Co atom was synthesized using FEFF calculations with $R_1=2.5071$ Å, and the Co K XAFS for 7μ thick pure Co foil was simulated very well with the parameter values of $N_1=12$, $S_0^2=0.72$, $R_1=2.49$ Å, and $\sigma_1^2=0.0062$ Å². The fluorescence corrected XAFS for the 230 Å thick Co film was almost identical to the XAFS for the 7μ thick foil except for a smaller amplitude, and a good fit with FEFF values was obtained for the 230 Å-thick Co film data with $N_1=12$, $S_0^2=0.73$, $R_1=2.49$ Å, and $\sigma_1^2=0.0073$ Å². Then the XAFS of a Pd shell around a Co central atom was synthesized using FEFF calculations with $R_1=2.641$ Å of a CoPd alloy, and the XAFS for the 3Å/4Å was fitted with a Co standard shell and a Pd shell thus synthesized by varying the parameters until the experimental XAFS could be simulated well with these two shells. A good fit was obtained with $N_1=8.71$, $R_1=2.60$ Å, $\sigma_1^2=0.0244$ Å², $\Delta E_0^1=-2.3$ eV, $N_2=3.29$, $S_{0,2}^2(\text{Pd-shell})=0.85$, $R_2=2.60$ Å, $\sigma_2^2=0.0073$ Å², and $\Delta E_0^2=-3.5$ eV. When the Fourier transform

Fig. 3. Comparison of the experimental and simulated k^3 -weighted XAFS and Fourier transforms.

of the XAFS for the $3\text{\AA}/4\text{\AA}$ sample was simulated using these parameters, the simulated Fourier transform showed split peaks with a wider separation even though the two shells were seemingly at the same radial distance. Thus this simulation result showed that the positions of the split peaks cannot be taken for the actually separated positions of two subshells. By using the same ΔE_0 values, the XAFS for the $15\text{\AA}/4\text{\AA}$ sample was also simulated and a good fit was obtained with $N_1=9.70$, $R_1=2.53\text{ \AA}$, $\sigma_1^2=0.0141\text{ \AA}^2$, $S_{0,2}^2(\text{Pd-shell})=0.85$, $N_2=2.30$, $R_2=2.56\text{ \AA}$, and $\sigma_2^2=0.0076\text{ \AA}^2$. The Fourier transform for the $15\text{\AA}/4\text{\AA}$ case was also simulated with these parameters. The results of these simulations for the $3\text{\AA}/4\text{\AA}$ sample are shown in Fig. 3. These Co/Pd multilayers have a preferred orientation, and the (111) planes are parallel to the sample plane. A Co atom in this configuration has 6 in-plane atoms, 3 top layer atoms, and 3 bottom layer atoms as nearest neighbors. In case of a good commensurability and little interdiffusion, a Co atom in the $3\text{\AA}/4\text{\AA}$ can have about 8.71 Co atoms and 3.29 Pd atoms as nearest neighbors. However the electron detection was made with the X-ray polarized in the sample plane, and thus the effective signal of 6 in-plane atoms corresponds to that of 9. Also the effective number of top or bottom 3 atoms is only 1.5. When this polarization effect is taken

into account, a Co atom in the $3\text{\AA}/4\text{\AA}$ sample would seem to have 9.58 Co atoms and 2.42 Pd atoms as nearest-neighbors. But with $N_1=9.58$ or with a smaller number of N_1 around 8, the unique beating feature around $6.3\text{ \AA}^{-1}$ in the inverse Fourier transform of k^3 -weighted XAFS for the $3\text{\AA}/4\text{\AA}$ case could not be simulated well. Therefore it can be concluded that a Co atom has less Co atoms and more Pd atoms as nearest neighbors than predicted for a case of no interdiffusion at the interfaces. There must be some interdiffusion and intermixing of Co and Pd atoms across the interfaces. Due to a large lattice mismatch, interface atoms would have to adjust to an average lattice spacing but the lattice relaxation may be gradual and for the $15\text{\AA}/4\text{\AA}$ sample the Co atoms in the central region of the $15\text{ \AA}$ thick sublayer may still have a smaller than average lattice spacing. It thus seems that the simulation results for the $15\text{\AA}/4\text{\AA}$ case are reasonable and the much smaller (larger) number of Co (Pd) nearest neighbor atoms than expected for a case of no interdiffusion also indicates a considerable amount of interdiffusion at the interfaces. The Co atoms which are smaller and lighter may experience more severe lattice relaxations and may have a larger structural disorder due to various near-neighbor configurations. However for the $3\text{\AA}/4\text{\AA}$ case, the σ^2 value for the Co shell is too large, and a possibility of two different Co-Co distances for in-plane and out-of-plane atomic pairs will be investigated in the future by polarization dependent XAFS measurements using an electron detector operating at low temperature.

§4. Conclusion

Electron yield XAFS spectra were measured for evaporated Co/Pd multilayers with various sublayer thicknesses and different thickness ratios of Co to Pd sublayers. For the $3\text{\AA}/4\text{\AA}$ and $15\text{\AA}/4\text{\AA}$ samples, the k^3 -weighted XAFS and Fourier transforms were simulated, and from the simulation results, it can be concluded that there must be some interdiffusion and intermixing at the interfaces. The simulation parameters for the $15\text{\AA}/4\text{\AA}$ sample seem to be reasonable. However the fit for the $3\text{\AA}/4\text{\AA}$ sample cannot be considered to be final, since the σ^2 value for the Co shell is too large. Whether there are two different Co-Co distances due to the intermixing or the anisotropy needs to be investigated further.

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References

- 1) P. F. Garcia: J. Appl. Phys. 63 (1988) 5066.
- 2) S.-C. Shin and A. C. Palumbo: J. Appl. Phys. 67 (1990) 317.
- 3) W. R. Bennet, C. D. England, D. C. Person, and C. M. Falco: J. Appl. Phys. 69 (1991) 4384.
- 4) S. M. Heald, H. Chen, and J. M. Tranquada: Phys. Rev. B38 (1988) 1016.
- 5) H. Chen and S. M. Heald: J. Appl. Phys. 66 (1989) 1793.
- 6) S. K. Kim: Thesis, POSTECH, 1991.
- 7) J. H. Joo, M. Choi, J. G. Lee, and S. M. Heald, *Proceedings of the 3rd Synchrotron Radiation User's Workshop*, POSTECH (1991) 295.

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