The Development of the Angle Resolved XPS Equipment at SPring-8 BL15XU

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The 3rd generation synchrotron radiation (SR) facilities have been recently built and have given us high flux and wide-energy tunable X-ray. This characteristic is useful to obtain XPS spectra excited by the high energy X-ray, which reveals the deeper layers. In this work, we used the beamline of SPring-8 BL15XU which has an revolver type undulator and a double crystal tandem monochromator and the angle-resolved XPS machine where an energy resolution is good even if X-ray energy is high up to 4500eV. For multilayers of the magnetic tunnel junction device, XPS measurement using 3000eV X-ray led to evaluate the deeper layers of a specimen and revealed the interface between Al oxide and $Fe_{50}Co_{50}$ alloy with Ta oxide or carbon protective overlayers.

1. Introduction

It is easy to recognize that high energy X-ray enables us to reveal deeper layers, since large kinetic energy of photoelectrons excited by high energy X-ray have long inelastic mean free paths (IMFPs). However, large kinetic energy of photoelectrons means that its photoionization cross-section is not so large. Therefore, XPS using high energy X-ray source, e.g. Ti X-ray tube, has not been familiar for practical use. The third generation synchrotron radiation (SR) sources, e.g. undulator, radiates the high brilliance X-ray of which energy is tunable and beam divergence is small. Therefore, an undulator can be a good source for high energy XPS, and its combination with angle-resolved XPS machine produces a powerful technique for non-destructive depth profiling [1].

2. Beamline BL15XU at SPring-8 and angle-resolved XPS

SPring-8 is one of the largest third-generation SR facilities in the world. Soft and hard X-rays are available at NIMS beamline BL15XU [2], which has high-brilliance undulators and a newly developed double crystal tandem monochromator. Our revolver undulator is the

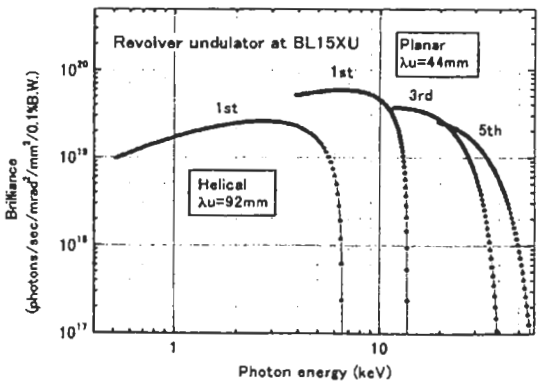

Fig.1 Brilliance of revolver undulator at SPring-8 BL15XU

combination of helical undulator and planar undulator, and the dependence of a brilliance on photon energy is shown in Fig.1, which is calculated with SPECTRA code [3]. This monochromator has two crystal pairs mounted in one vacuum chamber. One of the crystals Si(111) provides 2-20 keV photons (flux 10^{11}-10^{13} photons/sec, energy resolution $\Delta E/E=10^{-4}$), and a second one $YB_{66}(400)$ provides 1-2 keV photons (flux 10^{9}-10^{10} photons/sec, best energy resolution ΔE=0.15eV).

Our XPS system is based on the ULVAC-PHI model 10-360 analyzer with scanning capability. Double analyzer system was designed for a coincidence measurement of photoelectron and Auger electron, and/or an *in-situ* depth profile analysis under the condition where each analyzer is set at different take-off angles (TOA). These analyzers are mounted in a large UHV chamber (base pressure is $6x10^{-8}$ Pa) with 1400 mm in diameter shown in Fig.2 and put on a turntable, and a take-off angle of each analyzer can be set independently. The rotation axis of a sample manipulator is coincident with the rotation axis of double analyzer, and incident SR axis is on the horizontal plane on which analyzers move. Usual twin anode type X-ray tube (target is Mg or Ti) can be also utilized. Large kinetic energy photoelectrons, up to 4800eV, excited by hard X-ray is also available.

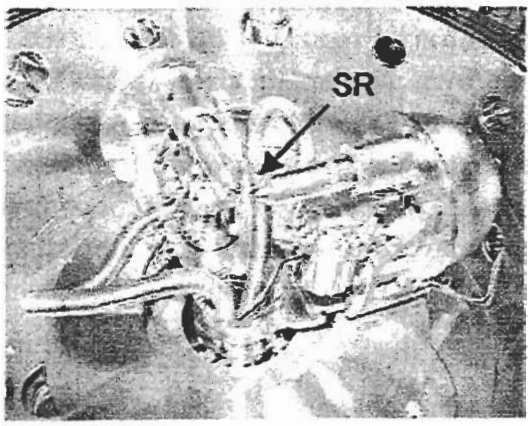

Fig.2 Photo of double analyzers in the angle resolved XPS equipment

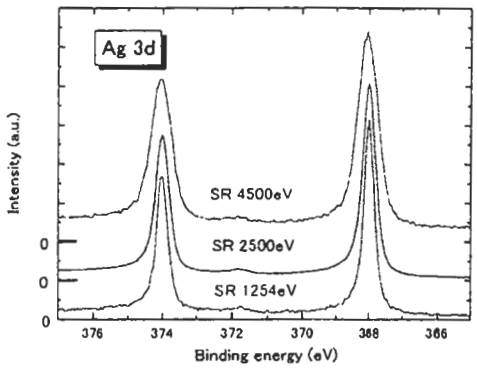

Fig.3 Ag 3d XPS spectrum excited by SR. Photo energy are set to be 1254eV, 2500eV, and 4500eV.

Figure 3 shows the Ag 3d peaks using X-ray of which energy are 4500 eV, 2500 eV and 1254 eV. 1254 eV X-ray was monochromatized by $YB_{66}(400)$ crystal. Other energy X-rays were monochromatized by Si(111) crystal. FWHM of $Ag3d_{5/2}$ peaks in Fig.3 are indicated in Table 1. It can be concluded that high energy XPS at our beamline is satisfactory to evaluate the fine spectral structure and the fine chemical shift, even if photon energy is up to 4500eV. Our best energy resolution of YB_{66} was estimated to be 0.15 eV by the rocking curve measurement.

3. High energy XPS measurements of multilayers

For many electronic devices, the analysis of the interface is very important to clarify the performance and potential of devices. Higher energy X-ray is also effective for the

Table 1 FWHM of $Ag3d_{5/2}$ peaks in Fig.3

photon energy (eV)	crystal	FWHM of $Ad3d_{5/2}$ (eV)
4500	Si	0.74
2500	Si	0.49
1254	YB66	0.39

Ta oxide (1.5nm)
Al oxide (1nm)
$Co_{50}Fe_{50}$ (3nm)
Pt Mn (20nm)
Ta (3nm)
Si oxide (200nm)
Si wafer

Fig.4 Structure of the magnetic tunnel junction device for XPS measurement

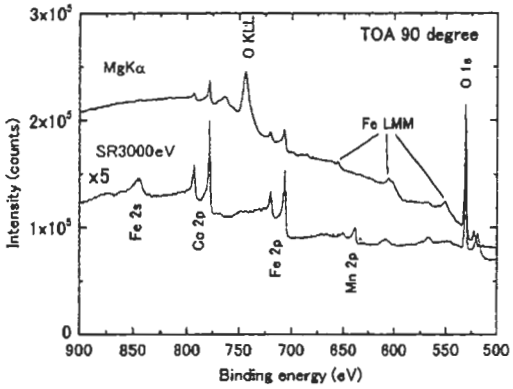

Fig.5 XPS spectra of multilayer shown in Fig.4 using Mg Ka and 3000eV X-rays at take-off angle 90 degree.

analysis of these interfaces.

In this study, we measured the interface part of the spin-polarized magnetic tunnel junction device of which structure is shown in Fig.4. In usual case, the Al oxide layer as a tunnel junction is put between ferromagnetic layers. However, in this work, there is no upper ferromagnetic layers and Al oxide is covered with Ta oxide as protective overlayer in order to analyzed the interface of the Al oxide layer and $Fe_{50}Co_{50}$ alloy layer with XPS. Al oxide layers were formed by the oxidization of Al layer in the vacuum chamber at room temperature, which was filled with oxygen for 10 minutes under a pressure of 100 Torr. This specimen without protective overlayers was synthesized with MBE machine. We have already confirmed by transmission electron microscopy that the same kind of specimens have multi-layers of uniform thickness represented in Fig.5. Ta protective overlayer was made by evaporation using usual DC sputtering and Ta natural oxide layer was formed after taking it out from a vacuum chamber.

Figure 5 shows the XPS spectra of multilayers in Fig.6 using usual the Mg X-ray tube and 3000eV photon. Take-off angle was set 90 degree in each energy case. X-ray incident angle was set 35.6 degree in Mg X-ray tube case (where polar angle was 40 degree and azimuth angle was 65 degree) and was set 45 degree in 3000 eV photon case. In comparison with Mg Ka data, 3000eV data lead us to recognize that 2p peaks of Co and Fe are more visible and Mn 2p peak of the PtMn alloy layer appear clearly.

Another advantage of SR is tuning energy in order to avoid the interference of photoemission and Auger spectra. If usual Mg Ka X-ray is used, oxygen KLL Auger peak interferes with Co 2p spectrum. If usual Al Ka X-ray is used, Co and Fe Auger peak interferes with the other 2p spectrum.

This XPS measurement indicated that the interface of Al oxide layer and $Co_{50}Fe_{50}$ layer has no oxide peaks of Fe and Co, and Al spectrum has single oxide peak. This result shows that the interface of $Co_{50}Fe_{50}$ alloy and Al oxide has an abrupt composition profile, and Ta oxide 1.5nm layer is good for protecting the underlayer from an oxidization by atmospheric oxygen during transportation of specimens to SPring-8.

When the carbon 1nm layer was used as a protective overlayer, angle-resolved SR-XPS measurement shown in Fig.6 indicates that there was Fe oxide at the interface. Here these two spectra in Fig.6 are normalized at $Fe_{3/2}$ peaks. This sample was made in the same synthetic process except for protective carbon overlayers as the sample covered with Ta oxide mentioned above was made. Carbon was coated by the RF sputtering machine which was designed for preventing specimen from damage by plasma.

By comparison with the result in the Ta oxide case, we can conclude that the carbon 1nm layer was not enough for protecting underlayers. This might result from the thickness fluctuation of carbon overlayers, since we did not check the uniformity of carbon overlayers by other techniques. In addition, SR-XPS measurement reveals that there was no Co oxide peaks, and Fe was oxidized preferentially for the carbon coated multilayer sample.

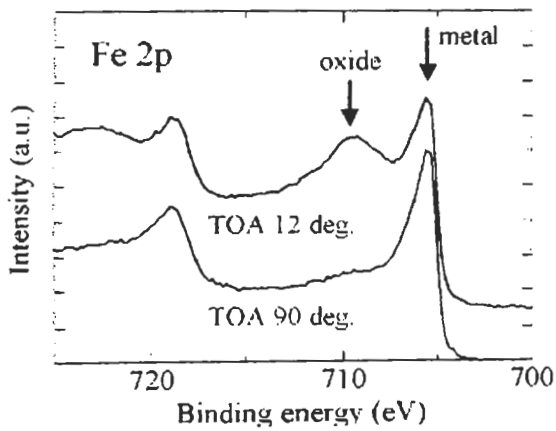

Fig.6 Normalized Fe 2p peaks of magnetic tunnel devices covered with carbon layer 1nm measured under the experimental conditions that photon energy is 2500eV and the take-off angles are set to be 12 degree and 90 degree. Example of XPS 2p chemical shift of Fe oxide is shown in ref[4].

4. Conclusions

XPS analysis using several keV high energy photons enables to reveal the deeper layers, which is too deep to analyze using conventional X-ray tube, since high energy photon increases IMFPs of photoelectrons. This simple concept was confirmed by the XPS measurements of the multilayer of Ta oxide(1.5nm) or Carbon(1nm)/ Al oxide(1nm)/$Co_{50}Fe_{50}$(3nm)/PtMn (20nm).

In this work, the conclusion mentioned above was realized by the beamline of SPring-8 BL15XU which has an revolver type undulator and a double crystal tandem monochromator and angle-resolved XPS machine.

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