Extended Abstract of PSA-19

P-16

Chemical state analysis for buried Al_2O_3 layer of Y_2O_3/Al_2O_3 multi-layer film using laboratory HAXPES

Shinsuke Nishida[1,*], Shinya Yasunaga[1], Shinya Otomo[1], Hirokazu Sasaki[1]
[1]*Furukawa Electric Co., Ltd., 2-4-3 Okano, Nishi-ku, Yokohama, Kanagawa 220-0073, Japan*
*shinsuke.nishida@furukawaelectric.com

(Received: May 30, 2019; Accepted: July 29, 2019)

We examined the practicability and chemical state analysis of laboratory HAXPES using monochromatic Cr Kα excitation. The acquisition time of nine min and the FWHM of 1.03 eV was obtained by Au 4*f* measurements. We also clarified the relationship between the oxygen gas flow rate and the chemical state of the Al_2O_3 layer around the Y_2O_3/Al_2O_3 interface for the buried Al_2O_3 layer of Y_2O_3/Al_2O_3 multi-layer film. Our findings demonstrate that laboratory HAXPES with Cr Kα is capable of nondestructive chemical state analysis with practical throughput.

1. Introduction

Hard x-ray photoelectron spectroscopy (HAXPES) offers larger information depth compared to conventional x-ray photoelectron spectroscopy (XPS) using monochromatic Al Kα (hv = 1486.6 eV), and can perform bulk sensitive measurement [1]. HAXPES has been realized in synchrotron radiation facilities, as the photoionization cross section becomes extremely small [2], and it is not easy to secure machine time. This makes it difficult to use it for quick feedback on the manufacturing processes and quality control. Recently, laboratory HAXPES using monochromatic Cr Kα (hv = 5414.9 eV) has been put into practical use, and it has now become easier to measure HAXPES spectra in the laboratory [3–5].

The purpose of our study is to develop analytical techniques with practical throughput using laboratory HAXPES. In this paper, we focused on Y_2O_3/Al_2O_3 multi-layer film, which is often used in electronic and optical devices. Chemical state analysis of the Al_2O_3 layer around the Y_2O_3/Al_2O_3 interface in the Y_2O_3/Al_2O_3 multi-layer film sample was performed.

2. Experimental

We used a Y_2O_3 (15 nm)/Al_2O_3 (180 nm) multi-layer film sample deposited on a metal alloy substrate by magnetron sputtering in this study. Samples A, B, and C with difference oxidation states of the Al_2O_3 layer were prepared by changing the oxygen gas flow rate at the time of sample preparation. The oxygen gas flow rate was set to decrease in the order of sample A, B, and C.

All conventional XPS and HAXPES measurements were performed with the laboratory HAXPES system (PHI Quantes, ULVAC-PHI, Inc.) installed at the Yokohama Works of Furukawa Electric Co., Ltd.. It is equipped with a dual scanning x-ray source composed of a conventional soft x-ray source (Al Kα) and a hard x-ray source (Cr Kα). The spot size of both x-ray beams was 100 μmϕ (25 W at Al Kα, 50 W at Cr Kα). All narrow scan spectra were taken at the pass energy of 55.0 eV, at the acceptance angle of ±20° and the take-off angle (TOA) (from average surface of the sample) of 45°–90°. Charge neutralization was not used in any of the measurements.

The Au plate was used to evaluate the basic performance of the laboratory HAXPES system.

3. Results and discussion

3.1. Practicability of laboratory HAXPES

The acquisition time and the energy resolution were evaluated using the Au 4*f* photoelectron peak with the TOA of 45°. **Figure 1**(a) shows the Au 4*f* spectrum obtained from the Au plate by Al Kα excitation. The acquisition time of the Au 4*f* spectrum was two min (0.6 s per point, 200 total points). The full width at half maximum (FWHM) of the Au $4f_{7/2}$ peak was 0.89 eV.

The acquisition time of the Au 4*f* spectrum obtained

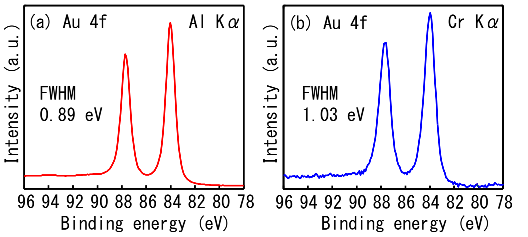

Fig. 1 Au 4*f* spectra obtained from Au plate by (a) Al Kα excitation and (b) Cr Kα excitation.

with Cr Kα excitation was nine min (2.7 s per point, 200 total points), as shown in **Fig. 1**(b). The FWHM of the Au $4f_{7/2}$ peak was 1.03 eV. The energy resolution of Cr Kα excitation was comparable to that of Al Kα excitation, and the acquisition time of the Cr Kα-excited peak required for a sufficient S/N ratio did not exceed ten times that of the Al Kα-excited peak.

These results demonstrate that laboratory HAXPES measurement using the Cr Kα sources can be performed with practical throughput.

3.2. Chemical state analysis of Al_2O_3 layer around Y_2O_3/Al_2O_3 interface

Chemical state analysis of the Al_2O_3 layer in three Y_2O_3/Al_2O_3 multi-layer film samples was performed in order to provide feedback to the manufacturing processes. **Figure 2**(a) shows the Al 2*p* spectra obtained at the TOA of 90° with Al *K*α excitation. Chemical state analysis of the Al_2O_3 layer by Al Kα excitation was difficult due to the thick Y_2O_3 layer. In contrast, from Cr *K*α excitation, a high-intensity Al 1*s* peak was detected in three samples at the TOA of 90°, as seen in Fig. 2 (b). Al 1*s* spectra exhibited a peak position very close to that of the Al_2O_3 phase, and had shoulders on the lower binding energy side corresponding to the sub-oxide (AlO_x) phase. In addition, weak peaks in the metallic Al phase were also detected in samples B and C. The above three peaks were assigned in accordance with reference peak positions determined by measuring the surfaces of bare Al_2O_3 and pure Al reference single film samples.

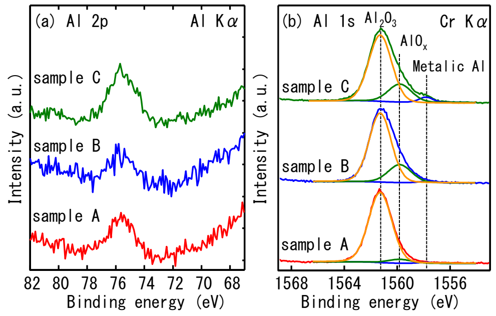

Fig. 2 (a) Al Kα-excited Al 2*p* spectra and (b) Cr Kα-excited Al 1*s* spectra obtained from sample A, B and C.

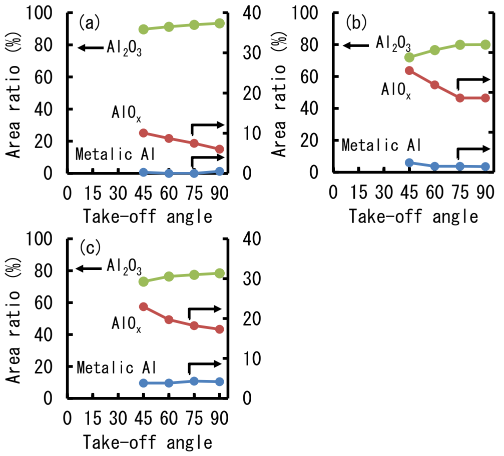

Fig. 3 Area ratios of Al_2O_3, AlO_x and metallic Al peaks of Al_2O_3 layer in Y_2O_3/Al_2O_3 multi-layer film as a function of TOA: (a) sample A, (b) sample B, and (c) sample C.

Angle-resolved measurements were performed to evaluate the Al-bonding components at the Y_2O_3/Al_2O_3 interface. The area ratios of Al_2O_3, AlO_x and metallic Al peaks in the Al 1*s* spectra for samples A, B, and C are plotted in **Fig. 3** as a function of the TOA. For all samples, the AlO_x component increased as the TOA was decreased, indicating that the AlO_x component exists around the Y_2O_3/Al_2O_3 interface.

4. Conclusion

The relationship between the oxygen gas flow rate and the chemical state of the Al_2O_3 layer around the Y_2O_3/Al_2O_3 interface for the buried Al_2O_3 layer of Y_2O_3/Al_2O_3 multi-layer film was clarified by laboratory HAXPES. We have demonstrated that laboratory HAXPES with Cr Kα is capable of nondestructive chemical state analysis with practical throughput.

5. References

[1] K. Kobayashi, *et.al., Appl. Phys. Lett.* **83**, 1005 (2003).

[2] J. J. Yeh and I. Lindau, *At. Data Nucl. Data Tables* **32**, 1 (1985).

[3] M. Kobata, *et.al.*, *Anal. Sci.* **26**, 227 (2010)

[4] T.Hishida, *et.al.*, *Jpn. J. Appl. Phys.* **54**, 083201 (2015).

[5] H.Song, *et.al.*, *Appl. Surf. Sci.* **433**, 1148 (2018).