The Study on Al₂O₃/Nb Interface by SAX Method

Han Xing Liu, Han Lin Zhang, Hai Lan Ren, Shi Xi Ouyang & Run Zhang Yuan

Advanced Materials Research Institute, Wuhan University of Technology, Wuhan 430070, China

(Received 13 December 1994; accepted 12 March 1995)

Abstract: Small area X-ray photoelectron spectroscopy technique is employed to study the chemical bonding of metal–ceramic interfaces. Nb, Al₂O₃ and Nb/Al₂O₃ interface samples are chosen for the analyses. The results show that there is a chemical reaction at the interface. Nb donates electrons to surface O atoms with an Nb–O bond forming at the interface and Nb has an Nb⁵⁺ state at the interface.

1 INTRODUCTION

Interfaces between ceramics and transition and noble metals are of practical importance and it is of interest to understand the bonding between them. In view of the complex nature of atomic interactions between metals and ceramics, our understanding of their structures is still poor. In recent years, there has been considerable interest both theoretical and experimental. The chemical and structural aspects of metal-ceramic bonding have been characterized in many instances by electron microscopy. In particular, recent advances in HREM have allowed this technique to provide both structural and chemical information about the interfaces at an atomic level. 1-3 Several recent studies by Rühle and co-workers have shown no reaction layer forms at the interface of the Nb/Al₂O₃ system by HREM.^{4,5} The surface science approach, including X-ray and ultraviolet photoelectron spectroscopies, Auger electron spectroscopy, mass spectroscopy, low energy electron loss spectroscopy and low energy electron diffraction to metal-ceramic interfaces studies provide information about chemical, electronic and atomic structures of the interface. In the present paper, small area X-ray photoelectron spectroscopy (SAX) technique is used to determine the bonding of Nb/Al₂O₃ interfaces.

2 METHOD

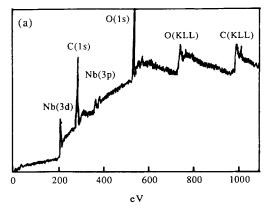
The metal-ceramic interfaces can be produced by

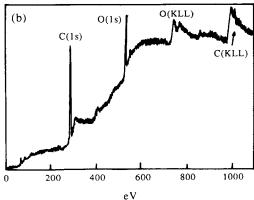
many methods, such as internal oxidation, diffusion bonding, MBE technique. In the present study the samples used for the researches are formed by the hot pressing method, the bonding temperature is 1050°C and the pressure is 3.5 MPa. the orientation of Nb and Al₂O₃ is Nb(110)/Al₂O₃ (1010), Nb[001]/Al₂O₃[0001]. In order to analyse the chemical bond at the metal-ceramic interfaces, three types of sample (Nb, Al₂O₃, Nb/Al₂O₃) are employed for the XPS study. Because of the small area of the cross-section of the interface, the information obtained by normal XPS for the interfaces would be very small compared with the information of no interface area in the XPS dot. The diameter of the dot for small area XPS is only 150–250 μ m. All experiments of SAX XPS are performed on ESCALAB MKII multi-technique electron spectrometer. Small samples of Al₂O₃, Nb, and Nb/Al₂O₃ are fixed on Ni substrate, and the spectroscopy of every sample should contain a few signals from the Ni substrate. The SAX analyses of the Nb/Al₂O₃ interface are performed on the cross-section of the interface.

3 RESULTS AND DISCUSSION

Figure 1 presents XPS spectroscopy for three samples which did not contain any of the Ni information. Figure 2 is the electron binding energy of Nb(3d) of pure (solid) Nb (Fig. 2(a)) and the interface (Fig. 2(b)). Figure 2(a) shows Nb(3d) has two states — the metallic Nb peaks and the Nb^{X+}

198 Han Xing Liu et al.





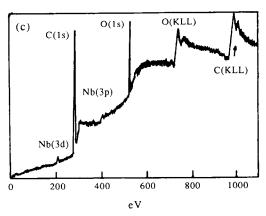


Fig. 1. The XPS spectroscopy for (a) Nb; (b) Al_2O_3 ; (c) Nb/Al_2O_3 .

peaks. The peaks can be divided into two groups using a mathematics method. One of them (203·17eV and 205·8 eV) corresponds to metallic Nb and the other (207·15 eV and 209·75 eV) corresponds to the oxidation state of Nb due to surface oxidation. The binding energy of Nb(3d) at the interface has only one group of peaks which does not contain metallic Nb peaks. The result shows Nb at the interface is oxidized due to the existance of Al₂O₃. Nb donates electrons to the O atoms with an Nb-bond forming at the interface,

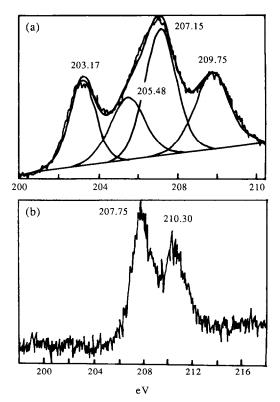


Fig. 2. Nb(3d) binding energy: (a) Nb; (b) Nb/Al₂O₃ interface.

which means that all Nb(3d) in the interface and Nb has an Nb⁵⁺ state. The present study clearly shows that there is a chemical reaction at the Nb/Al₂O₃ interface. This conclusion agrees with Ohuchi⁶ and will be further supported by the theoretical method. Calculations on the cluster models of the interface by DV-X α method will be performed in a future study.

ACKNOWLEDGEMENTS

This project was supported by the National Natural Science Foundation of China and the National Advanced Materials Committee of China.

REFERENCES

- SHIEU, F. S. & SASS, S. L., Acta Metall. Mater., 38 (1990) 1653.
- 2. LU, P. & COSANDEY, F., Ultramicroscopy, **40** (1992) 271.
- RÜHLE, M., EVANS, A. G., ASHBY, M. F. & HIRTH, J. P. (eds), *Metal-Ceramic Interfaces*. Pergamon Press, New York, 1990.
- MAYER, J., FLYNN, C. P. & RÜHLE, M., Ultramicroscopy, 33 (1990) 51.
- 5. MADER, W. & RÜHLE, M., Acta Metall., 37 (1989)
- OHUCHI, F. S. & KOHYAMA, M., J. Am. Ceram. Soc., 74 (1991) 1163.