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A Nitriding Treatment of Aluminum Films by Atmospheric Pressure Discharge (2) Effects of Gas Pressure for Helium/Nitrogen Mixture Gases

大気圧プラズマを用いたアルミニウム膜の窒化処理
(2) ヘリウム／窒素混合ガスによるガス圧効果

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Characteristic of aluminum films exposed to helium/nitrogen (He/N_2) mixture plasma was investigated using an X-ray photoelectron spectroscopy (XPS) and a powder X-ray diffraction (XRD). The aluminum films exposed to the He/N_2 plasma was progressed nitridation and contained dominantly an aluminum-nitrogen (Al-N) component. For the nitridation under relatively low nitrogen concentration, microcrystalline aluminum nitride (AlN) would grow. On the other hand, for relatively high nitrogen concentration, large AlN crystals started to grow.

1. Introduction

Nondestructive inspection with an electromagnetic acoustic transducer (EMAT) employing electric drive system is promising to monitor the crack and wastage caused at pipes of the fast-breeder reactor under the high temperature of 550˚C during the operation. The EMAT is required to cover electronic circuits with an insulating material in order to provide superior performance. Aluminum nitride (AlN) is known as one of good insulating coating materials because of their excellent properties such as the resistivity and the high thermal conductivity. In order to operate the EMAT at 550˚C, it is necessary to coat the electron circuits with the AlN film of the thickness more than 10 μm. Therefore, the AlN films were fabricated by the nitridation of aluminum film using atmospheric pressure plasma with a reaction rate because of high plasma density. Characteristic of their nitrided films was investigated using an X-ray photoelectron spectroscopy (XPS) and a powder X-ray diffraction (XRD).

2. Experimental

Aluminum films deposited onto a copper-tungsten substrate with the rf wave magnetron sputtering in the argon gas pressure of 6.7×10^{-1} Pa were nitrided by helium/nitrogen (He/N_2) mixture plasma under atmospheric pressure. Using gas mass flow meter, the He and N_2 gases with flow rates of 10 slm and 50 sccm, respectively, were accurately introduced into a reaction chamber and the gas pressure in the chamber was kept at the pressure from 2.7×10^4 Pa to 9.3×10^4 Pa. The XPS measurement of the nitrided aluminum films was performed using X-ray photoelectron spectrometer (Shimadzu Co. Ltd./Kratos Analytical Ltd.; AXIS-165). The magnesium target as an X-ray source was employed. An X-ray beam from a magnesium film was irradiated on the nitrided film. The pass energy was employed the value of 40 eV. In this system, the total resolution has been estimated to be ~850 meV. The vacuum of an analysis chamber was below 1.0×10^{-7} Pa. Photoelectron spectra in an Al 2p region of the nitrided aluminum films were measured by detecting the photoelectrons emitted from the surface region. The XRD measurement of the nitrided aluminum films was performed using powder X-ray diffractometer (Rigaku Co. Ltd.; RINT-2000). The copper target as an X-ray source was employed. An X-ray beam from a cupper film was monochromatized by means of a graphite monochromator and was irradiated on the nitrided film. Diffracted X-ray beam intensity was recorded using a NaI scintillation counter mounted on the 2θ arm. Diffraction patterns were obtained by scanning the 2θ in the angular range from 31˚ to 42˚.

3. Results and discussion

Figure 1 shows a typical core-level photoelectron spectrum in the Al 2p region for an aluminum film exposed to He/N_2 mixture plasma at the pressure of 8.0×10^4 Pa. The spectrum is divided into four components with consideration of the chemical shift. Peaks centered at 71.8 eV, 73.5 eV and 74.7 eV are assignable to Al-Al, Al-N and Al-O bondings, respectively.[1,2] This spectrum is dominantly composed of the Al-N component. It is noted that a typical spectrum for native aluminum oxide/aluminum is observed before nitridation. This phenomenon indicates that the Al-N component is generated in the aluminum film by expose of the He/N_2 plasma.

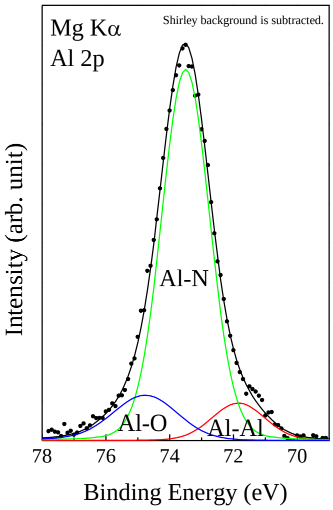

Fig.1. A typical Al 2p photoelectron spectrum for an aluminum film exposed to He/N_2 mixture plasma

Gaussian width of the Al-N component derived from the Al 2p spectra is plotted as a function of gas pressure in Fig. 2. It is noted that red circle indicates the Gaussian width estimated from a photoelectron spectrum for a sample, where an AlN0002 reflection peak is observed in an X-ray diffraction pattern. (not shown) The Gaussian width seems to be close to the value expressed by the red circle over the pressure. This implies that crystalline structure of these samples would be similar.

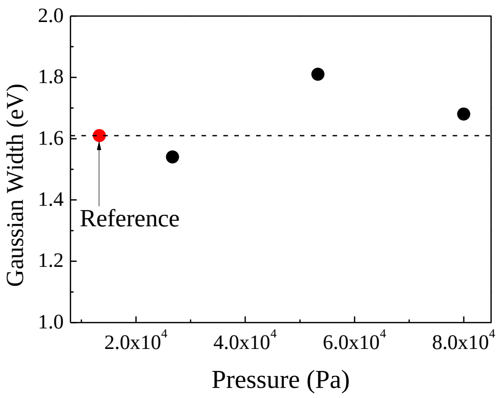

Fig. 2 Gaussian width of the Al-N component.

Figure 3 shows X-ray diffraction patterns for the nitrided films with respect to different He flow rate under the pressure of 9.3×10^4 Pa. It is noted that nitrogen concentration increases with decrease of the He flow rate. A weak AlN0002 reflection peak starts to be observed when the He flow rate is reduced and the nitrogen concentration is relatively elevated. It is inferred that a large AlN crystal starts to grow. For relatively low nitrogen concentration, the reflection peak is not visible although the Gaussian width would be close to that of the sample for relatively high concentration. It is considered that collection of microcrystalline AlN would grow.

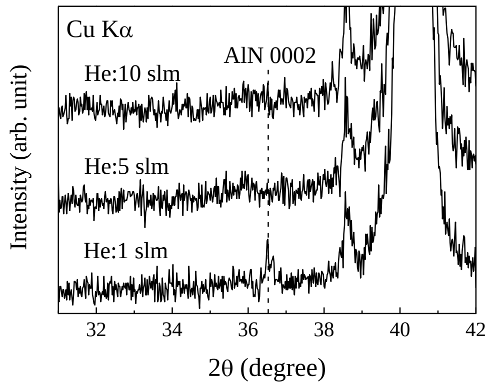

Fig. 3 X-ray diffraction patterns for the nitrided films with respect to different He flow rate.

4. Conclusion

The characteristic of the aluminum films exposed to the He/N_2 mixture plasma was investigated using XPS and XRD. The films exposed to the He/N_2 mixture plasma are composed of the Al-Al, Al-N and Al-O components. It is found that the microcrystalline AlN would grow for the relatively low nitrogen concentration and the large AlN crystal starts to grow.

Acknowledgments

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