

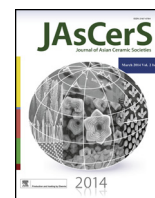
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## Full Length Article

## Accelerating the oxidation rate of AlN substrate through the addition of water vapor

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## ABSTRACT

To apply a pre-oxidation treatment on aluminum nitride (AlN) substrate is a common practice before its metallization. In the present study, the microstructure of AlN after oxidation either in dry air or in wet air is characterized. The resulting thermal conductivity is measured. With or without the presence of water vapor, the oxidation of AlN is a reaction-dominating process. The addition of water vapor speeds up the oxidation rate by one order of magnitude. The surface oxide layer is full of nano-sized pores. The presence of surface oxide reduces the thermal conductivity by ~15% when the thickness of oxide layer is only 3  $\mu\text{m}$ .

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## 1. Introduction

Aluminum nitride (AlN) is an electrical insulator [1]. Before applying AlN as the substrate for electronic components, a metallization treatment is usually needed [2,3]. For example, a recent study demonstrated that copper could be bonded to AlN substrate only after a pre-oxidation treatment was applied [4,5].

The electronegativity difference between Al and N is smaller than that between Al and O [6]. The bonding between Al and N tends to be covalent in nature. Such covalent bonding characteristic induces many problems, such as high sintering temperature and difficulty of bonding to metals [7]. Nevertheless, the transportation of phonons within a rigid AlN structure is fast, resulting in a high thermal conductivity. Depending on the oxygen content and sintering additive, the thermal conductivity of AlN substrate can reach >100 W/mK [8].

To solve the difficulty of bonding AlN to metals, a surface oxide layer is usually helpful. The formation of Al–O bonds may facilitate the bonding between AlN and metals. In the present study, a pre-oxidation treatment is applied to AlN substrate. The microstructure of the surface oxide layer is carefully characterized. The influence of the oxide layer on the thermal conductivity is then investigated.

## 2. Experimental

The polycrystalline aluminum nitride (AlN, Maruwa Co., Aichi, Japan) substrates with a thickness of 380  $\mu\text{m}$  were used in the present study. The surface roughness ( $R_a$ ), as measured by a surface profiler (SJ-201P, Mitutoyo Co., Japan), was 0.3  $\mu\text{m}$ . The substrates were cleaned in ultrasonic bath of ethyl alcohol for 5 min, then in acetone for 3 min.

The oxidation was carried out in a flowing air at a rate of 50  $\text{cm}^3/\text{min}$ . The water content in air was 35 Pa, as reported by the manufacturer (Air Products Inc., Taipei, Taiwan). In order to remove the water, the air was bubbled through a concentrated sulfuric acid (97.0%  $\text{H}_2\text{SO}_4$ , Sigma-Aldrich, USA). The water content could be reduced to  $1 \times 10^{-3}$  Pa after this treatment [9]. In order to investigate the effect of water vapor on the oxidation of AlN, the wet air was prepared by bubbling through a water tank at 25 °C. The water vapor in wet air was approximately  $3 \times 10^3$  Pa [10].

The crystalline phases of the oxidized substrate were identified using an X-ray diffraction technique (XRD, TTRAX-III, Rigaku Co., Tokyo, Japan). To determine the extent of oxidation, the thickness of the surface oxide layer was evaluated using electron probe X-ray microanalyzer (EPMA, JXA-8200, JEOL Co., Tokyo, Japan). This technique could locate oxygen near the surface. The thickness of the oxide layer was then estimated using the region with high oxygen concentration [4]. The resolution of the technique was around 1  $\mu\text{m}$ . A transmission electron microscope (TEM, FEI Tecnai G<sup>2</sup> F20, Philips Co., Oregon, USA) was used for microstructure characterization. The TEM foil was prepared using an environmental dual-beam

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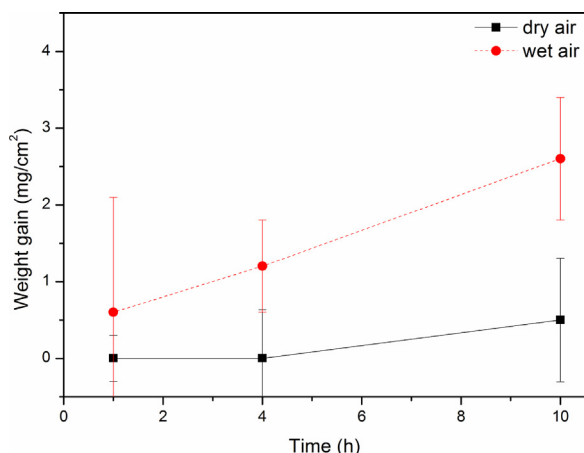


Fig. 1. Weight gain as a function of oxidation time at 1150 °C in dry air and wet air.

focus-ion-beam system (FIB, FEI Helios 600i, Philips Co., Oregon, USA).

A flash method (LFA447, Netzsch Co., Selb, Germany) was used to determine thermal diffusivity ( $\alpha$ ) of oxidized AlN plates [11,12]. Ten discs were used for each oxidation treatment. Specific heat capacity ( $C_p$ ) was measured using a differential scanning calorimeter (DSC, PerkinElmer Instruments Co., Connecticut, USA). The density of specimens was measured with the water displacement method.

### 3. Results

The weight before and after the oxidation treatment was monitored; the weight gain is shown in Fig. 1. The weight gain for the AlN substrate oxidized in wet air is higher than that in dry air. Furthermore, the weight gain shows a linear relationship with the oxidation time at 1150 °C. Fig. 2 shows the cross-section of oxidized AlN substrate. Along with the back-scattered micrograph, its corresponding EPMA mapping on oxygen is also shown. From

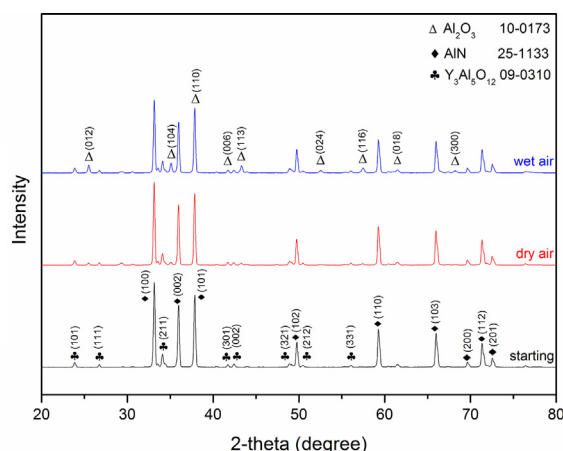


Fig. 3. XRD patterns for AlN substrates oxidized at 1150 °C for 1 h in dry air and wet air. The XRD pattern for starting AlN substrate is shown for comparison.

the mapping on oxygen, the thickness of the oxide layer can be estimated. The oxide layer on the AlN substrate after oxidation in wet air for 1 h is a continuous layer; its thickness is approximately 3  $\mu\text{m}$ . The oxide layer on the substrate oxidized in dry air is not continuous.

Fig. 3 shows the XRD patterns for the substrates after oxidation at 1150 °C for 1 h. The XRD pattern for the starting AlN substrate is also shown for comparison. A small amount of  $\text{Y}_3\text{Al}_5\text{O}_{12}$  (YAG) is found in the starting substrate, indicating that yttria ( $\text{Y}_2\text{O}_3$ ) is used as the sintering additive for the preparation of AlN substrate [13]. After oxidation in both dry and wet air, a small amount of  $\alpha\text{-Al}_2\text{O}_3$  is found on the surface of oxidized substrate. The intensity of  $\text{Al}_2\text{O}_3$  peaks on the AlN substrate oxidized in wet air is higher than that on the AlN substrate oxidized in dry air.

The TEM micrographs for the oxidized layer are shown in Fig. 4. Many small pores are found within the oxidized layer. The size of pores in the oxide layer formed in dry air varies in the range from

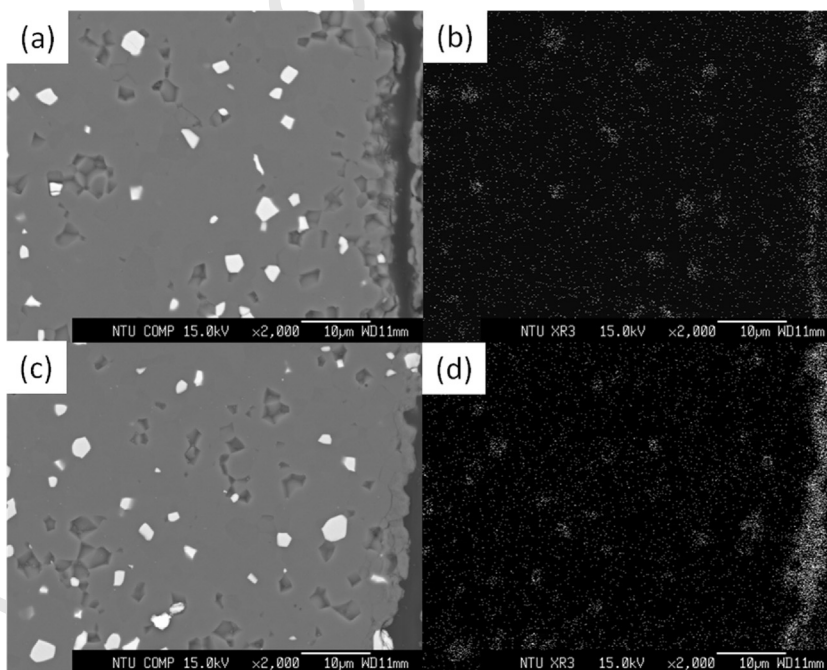


Fig. 2. Cross sections of the specimens oxidized at 1150 °C for 1 h in (a), (b) dry air and (c), (d) wet air. (a), (c) are the backscatter images; (b), (d) are their corresponding EPMA maps on oxygen.

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