Investigation on Joining of AlN Ceramics
Effects of TiH₂ Coating and Insertion of Ti and Cu Foils

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We have conducted joining between AlN ceramics by spark plasma sintering (SPS) process and examined the effects of TiH₂ coating on AlN and the insertion of Ti and Cu foils. Titanium hydride was decomposed and a joint was formed by heating at 500°C for 10 min. The resultant reaction layer has a shear strength of 24.8 MPa. The interface layer was characterized using scanning electron microscope (SEM), electron-probe microanalyzer (EPMA) and x-ray diffraction (XRD).

[Received August 5, 2003; Accepted January 14, 2004]

Key-words: AlN, TiH₂, Joining, SPS, Ti, Cu

1. Introduction
Aluminum nitride (AlN) ceramics have been widely used as a substrate material for high-power and high speed semiconductors due to their high thermal conductivity, and low thermal expansion and large electrical resistivity. Joining techniques between ceramic/ceramics and ceramic/metal have been expected to develop these electrical and electronic applications. In numerous studies for metallizing technique, processes utilizing TiH₂ activation are attractive because TiH₂ forms a wettable titanium coating by decomposition at 350–550°C and the resultant Ti has strong adhesion with AlN. Norton et al. reported brazing of AlN ceramics to Cu and Fe–Ni alloy at 850°C using powders of TiH₂.

A spark plasma sintering (SPS) process, also referred to as pulse electric current sintering (PECS), in which a high pulsed electric current is applied to powder samples, has been developed recently as a rapid sintering method. The SPS process is effective for difficult-to-sinter ceramics, and metal/ceramic composites. We reported previously on the densification of AlN with no additives by the SPS at 1200°C, which is 530°C lower than the previous reported temperature. Furthermore, the simultaneous metallization of AlN with Ti, Mo and W foils was achieved at 1200°C by SPS. In the sample, a thin reaction layer of TiN was detected at the Ti/AlN interface, which seemed to contribute to the high adhesion strength of the interface. Therefore, if SPS is applied to the joining of AlN/AlN and AlN/metal together with the TiH₂ activation technique, it is expected to form a strong adhesion of the interface at low temperatures.

In this work, we joined AlN/AlN, AlN/Ti/AlN and AlN/Cu/AlN using TiH₂ powders by SPS. The joining of these samples was achieved at 500°C, and the interfaces were investigated using SEM, EPMA and XRD.

2. Experimental
The 3.6 mm thick AlN ceramic substrates were supplied by Tokuyama Co., Japan. The primary impurities of the AlN substrate were Y (3.5 wt%) and O (1.8 wt%) added as a sintering aid, and Y₂O₃. The samples used for joining were cut to 10 × 10 mm, and their joining surfaces were rough, as-sintered, without grinding. Coating of TiH₂ on the samples was prepared using the following procedure. The samples were soaked in acetone dispersed TiH₂ powders to be deposited on these samples and then air-dried to produce a coating of about 10 μm/cm². Three kinds of joint assembly were prepared using the TiH₂-coating sample: (1) AlN/AlN, (2) AlN/Ti/AlN and (3) AlN/Cu/AlN. The thickness of the metal insertion was 0.05 mm for Ti (purity 99.5%, Nirasco Co.) and 0.05 mm for Cu (purity 99.5%, Nirasco Co.).

The joint couple was put into a carbon die and secured by two carbon punches. This assembly was set between electrodes in the SPS chamber, which was then evacuated. The joint couple was uniaxially pressed at 24 MPa through punches and heated by a pulsed electric current in the SPS system. The temperature was monitored by a K-type thermocouple inserted into the hole of the carbon die, and controlled by a programmable proportional integral differential controller. The joint couple was heated at 50°C/min up to 500°C, held at that temperature for 10 min, and then cooled down to room temperature in the chamber.

The bond strength of the joints was measured using a die shear strength test after the samples were cut to 2.0 × 2.0 × 2.0 mm. The microstructures of the joint interfaces were examined along the cross-section of the joints using SEM and the interactions of the joints were analyzed using EPMA and XRD. Samples for EPMA were prepared using a standard technique that included grinding and polishing the samples.

3. Results and discussion
The shear strength was 25 MPa for the AlN/AlN joint. In the case of the AlN/Cu foil and AlN/Ti foil, the shear strengths were estimated to be higher than 25 MPa because the joint samples were not destroyed under this load which is the maximum applied in these experiments. Figures 1 (a) and (b) show SEM images of the AlN/AlN joint. The Ti rich region formed by decomposition of TiH₂ was clearly observed between the AlN ceramics. The Ti rich region was approximately 20 μm in width. The interface between the Ti rich region and the AlN ceramic meandered and combined tightly, though partial cracking was occurred. Figures 1 (c) and (d) show SEM images of the AlN/Cu/AlN specimen. The interface between the Cu and Ti rich regions is obscure in comparison with that between the AlN and the Ti rich regions. A narrow gap a few μm in width and less than 1 μm in depth partially existed. Since the gap is extremely shallow and the interface has a strong adhesion, it is assumed that the gap was absent in the as-joined specimens. The gap suggests that a brittle phase may be formed in the boundary layer between Ti and AlN. The joint of the AlN/Ti/AlN specimen exhibited similar characteristics as that of the AlN/Cu/AlN specimen in SEM morphology.

Figure 2 (a) shows the distribution of Al and Ti in the inter-
It is known that Ti reacts with AlN at the surface and forms reaction phases depending on the temperature. In the range of 600–800°C, TiN, Ti₅N₃, and Ti₃N and TiAl₃ were observed at the Ti/AlN interface. Since Ti and TiAl₃ are the most stable phases, and the overall reaction proposed is 4Ti + 3AlN = 3TiN + TiAl₃, on the other hand, TiH₂ was assumed to react with AlN at the surface above 600°C after decomposition at 340°C and the brazing temperature of AlN ceramics to Cu using TiH₂ is reported at 850°C. However, this work achieved successful joining between AlN ceramics with TiH₂ at 500°C and 50°C/min by SPS. Furthermore, in our preliminary data, joining at a heating rate lower than 25°C/min exhibited weak adhesion. These results can be explained by the decomposition of TiH₂ and the diffusion of the resultant Ti. When hydrogen is released from the TiH₂, the resultant Ti is active, and the reaction with AlN may be facilitated. As soon as the active Ti forms, it must diffuse into AlN to form the stable reaction layer responsible for strong adhesion. When the temperature is low, the active Ti must react with each other. If this assumption is correct, it is essential to steeply raise the temperature because the active Ti can diffuse into AlN before being bound to the neighboring Ti. This model is in good agreement with the experimental results. The SPS process may induce additional effects, e.g., enhancement of atom diffusion and decomposition of TiH₂ through a pulsed electric current. We will discuss the effects of the SPS in a separate paper.

Acknowledgements We would like to thank Tokuyama Co., Japan for supplying AlN powder and for measuring the shear strength.

References