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Interfacial Microstructure of Solid State Diffusion Bonded Silicon Nitride using Niobium Foils[†]

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Abstract

The present work aims to bond silicon nitride using niobium foils by solid state diffusion bonding, to clarify the reaction behavior at the joint interface. Bonding temperature and time ranged from 1573 to 1773 K and 3.6 to 32.4 ks, respectively. The resultant interfacial structure was analyzed by SEM, EPMA and XRD. The phase sequence at the interface appears as $Si_3N_4 / Nb_5Si_3 / Nb_2N+Nb / Nb$ in the case that commercial Nb foils are used. On the other hand, when fine grained films are used, Nb_2N is formed as a layer adjacent to Nb_5Si_3 .

KEY WORDS: (interfacial microstructure), (solid state diffusion bonding), (structural ceramics), (silicon nitride), (niobium), (nano-grained deposition film)

1. Introduction

One of the biggest social demands in the 21st century is focused on the energy efficiency of power plants in order to reduce CO₂ emission. To raise the energy efficiency is to raise the operating temperature of turbine generators. Innovation in raising operating temperature will occur by replacing ordinary metallic components with silicon nitride (Si₃N₄), which provides better heat and corrosion resistivity than metallic materials¹⁾. Bonding technique of Si₃N₄ to each other and/or to metallic materials is required for construction of each component. To achieve strong and reliable bonding, fundamental knowledge on interfacial reaction and the resultant microstructure is required. On the other hand, niobium (Nb) is known as one of the refractory metals, which is used as the key element for heat resistive superalloys. Moreover, some reports concerning the application of niobium sheet as the filler metal for joining alumina suggest that niobium forms strong bond with alumina without the formation of reaction products²⁻⁴⁾. Since alumina is commonly used as one of the sintering aids for Si₃N₄, niobium is expected to form a strong bond also with silicon nitride.

The present work aims to clarify the interfacial reaction and structure of solid state diffusion bonded silicon nitride with niobium and to propose a way to control the structure.

2. Experimental Procedure

Pressureless sintered Si₃N₄, which contains a few mass percent of alumina as the sintering aid, was shaped into cylinders with 6.0 mm and 4.0 mm diameters and lengths, respectively. Two types of niobium with different grain sizes were used as the insert metal. One was 25 μm-thick commercial foil. The other was niobium film deposited directly on the bonding surface of Si₃N₄ using an RF magnetron sputter deposition technique. A niobium foil was inserted between two Si₃N₄ cylinders for each bonding specimen. Then, they were set into an induction-heating vacuum furnace. The bonding temperature and time were set at certain values between 1573 K and 1773 K and between 3.6 ks and 32.4 ks, respectively. The vacuum inside the furnace was kept below 1.3×10⁻³ Pa. Uniaxial pressure of 120 MPa was loaded to the specimens, perpendicular to the bonding interface, during the bonding treatment. The microstructure of the joint interfaces was analyzed by SEM, EPMA and XRD.

3. Results and Discussion

The average grain size of each type of niobium foils was measured at first. The sizes for commercial foils and deposited films were 46 μm and 86 nm, respectively. The deposited niobium foils have significantly fine grained structure. Moreover, no reaction products are formed at the interface during deposition, as shown in Fig. 1.

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Fig. 2 shows the interfacial microstructure of a Si_3N_4 / commercial-Nb joint bonded at 1773 K for 3.6 ks with the result of phase identification. The dark region on the left side is Si_3N_4 . The bright region on the right side is niobium. Nb_5Si_3 is formed as a 1.3 μm -thick layer adjacent to Si_3N_4 . Nb_2N is also formed as grains in contact with niobium and the Nb_5Si_3 layer. The phase sequence is described as Si_3N_4 / Nb_5Si_3 / Nb_2N + Nb / Nb. This result agrees well with that reported by Suganuma *et al.*⁴⁾

Fig. 3 shows the interfacial microstructure of a Si_3N_4 / deposited-Nb joint bonded at 1673 K for 14.4 ks. Nb_2N is formed as grains isolated in Nb and as a layer between Nb_5Si_3 and Nb. Therefore, the phase sequence is described as Si_3N_4 / Nb_5Si_3 / Nb_2N / Nb + Nb_2N . The difference between the niobium foils is only their initial grain size, i.e., their density of grain boundary. Therefore, this result implies that the nucleation frequency of Nb_2N is enhanced in fine-grained deposition films by an increase in density of grain boundary.

4. Conclusion

Interfacial microstructure of Si_3N_4 / Nb solid state diffusion bonded joints using niobium foils with two different grain sizes were analyzed. Nb_5Si_3 is formed as a layer adjacent to Si_3N_4 . Nb_2N is formed as grains with random shape and size when commercial Nb foil is used, while it is formed as grains with uniform size in the Nb matrix and as a layer between Nb_5Si_3 and Nb when a fine-grained deposition film of Nb is used.

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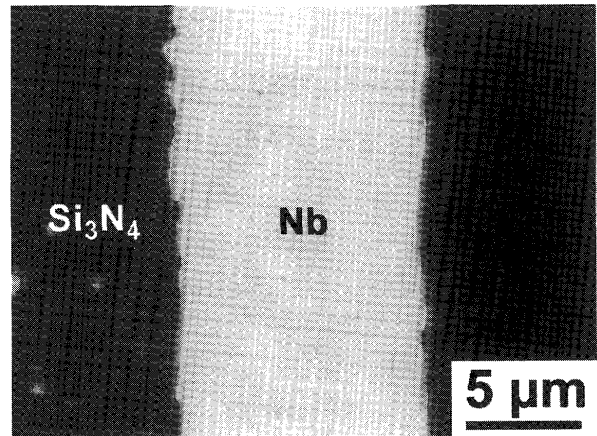


Fig. 1 Initial state of the deposited Nb film.

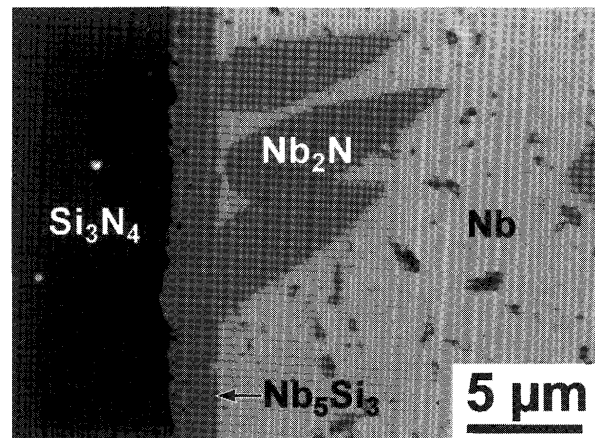


Fig. 2 Interfacial structure of Si_3N_4 / commercial-Nb joint bonded at 1773 K for 3.6 ks.

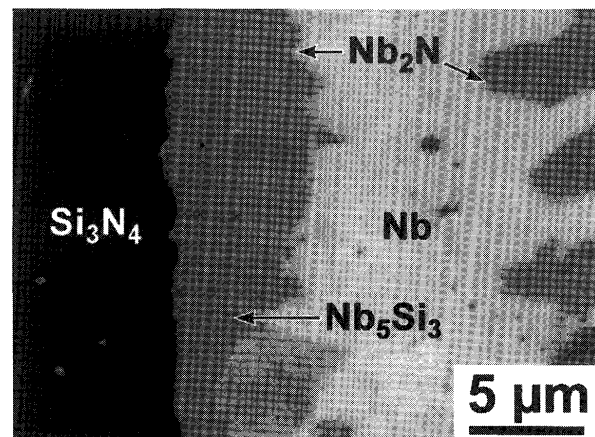


Fig. 3 Interfacial structure of Si_3N_4 / deposited-Nb joint bonded at 1773 K for 3.6 ks.