STRENGTH AND FRACTURE TOUGHNESS OF Si_3N_4-METAL JOINTS WITH PHASE TRANSFORMING INTERLAYER METAL

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Ceramic-metal bonding techniques are most important for practical applications involving ceramics. In the present study, silicon nitride-molybdenum joints were produced with Fe-Ni alloy interlayers, which show phase-transformation with volumetric expansion at low temperatures during cooling process of joining. The phase-transforming Fe-Ni alloy interlayer significantly relieved the residual stresses of the joints. Consequently, higher bending strength and fracture toughness were attained compared to those for the case of common soft metal interlayers.

Introduction

In ceramics-metal joints, most serious problem is considered to be the tensile residual stress induced near bonding interface, which significantly degrades strength of the joint. Soft metal interlayers between ceramic and metal joining materials are commonly used to relieve the residual stress due to their plastic deformation (1,2). Since soft metal generally has low yield strength but also has high thermal expansion coefficient, sufficient reduction of residual stress is not always assured. Furthermore, the soft metal interlayer often results in the degradation of strength of joints due to its low yield strength (3).

The phase-transforming metal will be one of most promising interlay metals to reduce residual stress of joints. The low temperature phase of phase-transforming metals is generally hard, so that it does not give the cause for the degradation of strength of joints like the case of soft

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metals.

In the present study, silicon nitride-molybdenum joints were produced with phase transforming Fe-Ni alloy interlayers having different amounts of nickel content. Bonding strength and fracture toughness experiments as well as residual stress measurements of the joints were carried out to investigate the effect of the phase- transforming metal interlayer.

Thermal expansion behavior of Fe-Ni alloys

Thermal expansion properties of the materials used in this study was investigated. The joining materials used were pressureless sintered Si_3N_4 with additives of 5mol%Al_2O_3 and 5mol%Y_2O_3 and Mo. The interlayer materials were Fe-Ni system alloys that produced in a high-frequency induction heating centrifugal cast machine. Four Fe-Ni alloys with nickel contents of 4, 10, 20 and 30 wt% were prepared. The silver-based brazing foil (71wt%Ag, 27wt%Cu and 5wt%Ti, 50 μ m in thickness) was used for joining.

From the measurements of thermal expansion under the same thermal cycle as the joining process, the thermal shrinkage strain during cooling process, where the starting point is taken at the solidus temperature of the brazing material (780 ℃) , is shown in Fig.1. The significant phase-transformation was observed for Fe-4%Ni, Fe-10%Ni and Fe-20%Ni alloys. The transforming temperature decreased with increasing nickel content. The amount of volumetric expansion along with the phase-transformation also increased with increasing nickel content. Since the high temperature phase (γ -phase) of Fe-Ni alloys is soft, the effect of reduction in residual stress similar to the soft metal is presumed at high temperatures in addition to that due to phase-transformation. Therefore, the significant effect of reduction in residual stress is expected by use of phase-transforming Fe-Ni alloy interlayer for ceramic-metal joining. No phase-transformation was found in Fe-30%Ni alloy, whose phase-transforming temperature seemed to be lower than room temperature. Since the crystal structure of Fe-30%Ni alloy was γ -phase during cooling to room temperature, the effect of reduction in residual stress similar to the soft metal is expected.

Bonding strength and fracture toughness

Experimental procedure

The joining materials were combined as shown in Fig.2, where brazing foils were inserted between joining surfaces. For fracture toughness specimens, the brazing foil was attached only on the half area of joining surfaces to introduce an artificial precrack, which was sufficiently sharp (the width between crack surfaces was less than 8 μ m). Brazing was carried out in a vacuum furnace at 850 °C for 10min under less than 5×10^{-5} Torr. The heating and cooling rates were controlled to be 20 °C /min and 10 °C /min, respectively. The bonding pressure of 0.01MPa was applied to attain the complete and thin brazing.

Room temperature bending strength and fracture toughness tests were carried out in four point bending with supporting span length of 40mm and loading span length of 20mm under a crosshead speed of 0.5mm/min.

Results and discussion

From the test results, relationships between interlayer thickness and bending strength and fracture toughness are shown in Fig.3 and 4, respectively. The results for soft metal (Cu, Ni) interlayer in the figures are referred to the previous work (3). The fracture toughness values in Fig.4 were apparent values estimated using the fracture load and the equation for evaluating stress intensity factor of homogeneous elastic material (4). It was found from Fig.3 that the bending strength of the joints with soft metal interlayer decreased with increasing interlayer thickness, and that the bending strength of the joints with phase-transforming Fe-Ni alloy interlayer was higher than those with soft metal interlayer. It was also found from Fig.4 that the fracture toughness of the joints with soft metal interlayer increased with increasing interlayer thickness, and that the fracture toughness of the joints with phase-transforming interlayer was much higher than those with soft metal interlayer.

The relationship between bending strength and fracture toughness is shown in Fig.5. For the case of soft metal interlayer, the fracture toughness decreased with increasing strength. It was impossible to improve both the fracture toughness and strength at the same time by using the soft metal interlayer. On the other hand, both the fracture toughness and strength were improved by using the phase-transforming Fe-Ni alloy interlayer.

Residual stress measurements

The residual stress distribution was obtained using a small spot X-ray diffraction apparatus with spot size of 100 μm in diameter. The residual stresses measured in the direction normal to the interface are shown in Fig.6. The measured residual stresses in the Si_3N_4/Mo joints without interlayer were compressive. On the basis of the equilibrium for force and the previous reports (5,6), the existence of high tensile residual stress near the end region is presumed, as shown by the broken line in Fig.6. On the other hand, the measured residual stresses in the Si_3N_4/Fe-Ni/Mo joint were tensile: The absolute values were far smaller than those in the Si_3N_4/Mo joint without interlayer. On the basis of the equilibrium for force, the residual stress near the end region are compressive. as shown by the broken line in Fig.6.

Concluding remarks

In the present study, we aimed to reduce the residual stress produced in ceramic-metal joints by using the volumetric expansion induced by the phase-transformation. The residual stress in the Si_3N_4-Mo joint with phase-transforming Fe-Ni alloy interlayer was significantly reduced, and consequently both the strength and fracture toughness (adhesive strength of interface) were successfully improved .

Acknowledgments

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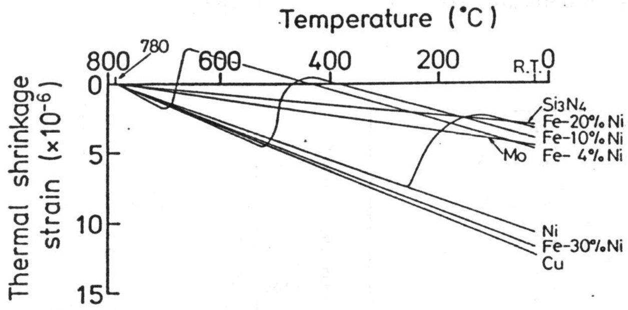
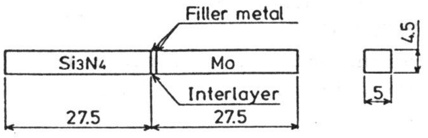
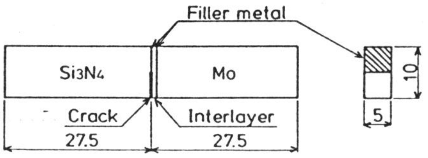


Figure 1 Thermal shrinkage strain of the materials used.



(a) 4-point bend specimen



(b) Fracture toughness specimen

Figure 2 Specimens.

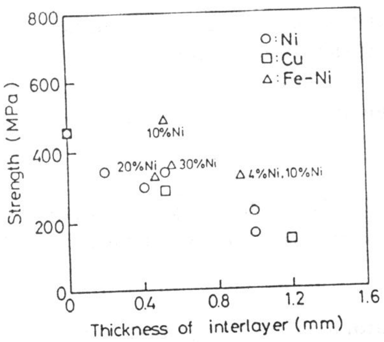


Figure 3 Effect of interlayer thickness on bending strength.

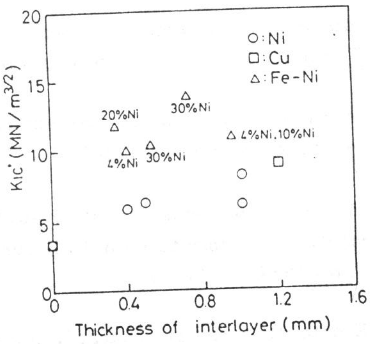


Figure 4 Effect of interlayer thickness on K_{IC}'.

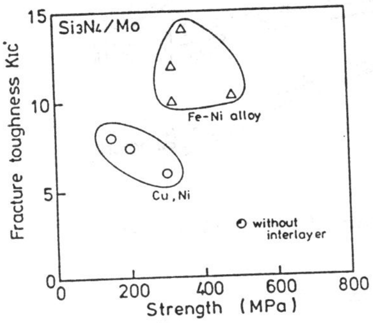


Figure 5 Relationship between bending strength and K_{IC}'.

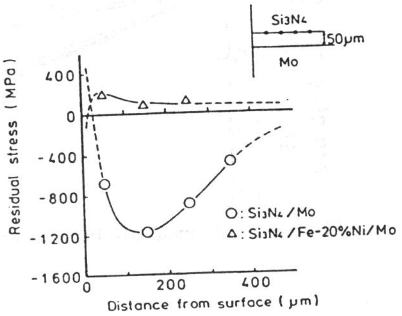


Figure 6 Residual stress measured by XRD.