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Strength of Si_3N_4/Ni–Cr–Fe alloy joints with test methods: shear, tension, three-point and four-point bending

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The experiments were carried out in order to examine the relationship between shear, tension, three-point and four-point bending strengths of Si_3N_4/Inconel 600 alloy joints. Average values of the shear, tension, and three and four point bending strengths were 178, 233, 321, and 344 MPa, respectively. From the results the strength ratio was 1 : 1.32 : 1.81 : 1.95 in the order of shear, tension, three-point and four-point bend tests. Based on Weibull moduli three-point bend and tension tests showed smaller strength scattering than did the shear and four-point bend tests. As opposed to the behaviour observed for the monolithic ceramic, the average bending strength of Si_3N_4/Inconel 600 alloy joints decreased as the effective volume of the bend test specimen increased.

1. Introduction

A ceramic/metal joint is a system consisting of materials that are both physically and mechanically different to each other. Therefore, the strength of a joint is governed by a number of variables that include (i) the elastic mismatch between the two materials, (ii) the plastic flow of the metal, (iii) the thermal residual stress, (iv) the heterostructure of joint, and (v) defects introduced into the joint and interface [1–3]. In addition to these factors, the size and geometric shape of the specimen and the test methods have an effect on the measured strength of a ceramic/metal joint. Despite this complexity, the mechanical strength of joints has been conventionally evaluated by either shear, tension, three-point bend or four-point bend tests. This is because no standardized method for the strength evaluation of such joints has been defined. In fact no relationship between the test methods has been clarified. Suganuma, *et al.* [4] have examined the relationship between a tensile test and a three-point bend test for an Si_3N_4/Invar joint bonded with aluminium. However this work has not been expanded to include shear and four-point bending tests. In order to provide a relationship between the four types of test methods it is very important to measure other joint systems and also to compare the engineering designs. The purpose of this paper is to investigate the relationship between shear, tension, three-point bending, and four-point bending strengths for an Si_3N_4/Inconel 600 alloy joint bonded with a Ag–27Cu–3Ti filler metal.

2. Experimental procedures

2.1. Materials and bonding

The base materials used in these experiments are a pressureless sintered, hot-isostatically-pressed silicon nitride (sintering additives: $Al_2O_3 + Y_2O_3 < 10\%$; one piece had the dimensions of 12.7 mm × 12.7 mm × 4.8 mm whilst others were of 9.5 mm diameter and 4.6 mm thickness) and a cold-worked Inconel 600 alloy (Inco Alloys International Inc., Huntingtions, WVa USA). The chemical composition of the Inconel 600 alloy is 75.09Ni–16.07Cr–7.11Fe–0.32Ti–0.28Si–0.25Al –0.07C at %. Brazing alloys were produced by vacuum induction melting, multistage annealing, and rolling to an approximate thickness of 200 μm. They were used in the form of a thin disc (11.0 mm diameter × 0.12–0.15 mm thickness). Their chemical composition is Ag–27.0Cu–3.0Ti in wt% (Ag–37.0Cu–5.5Ti in at %).

Prior to joining, the surface of the silicon nitrides and metals were polished using a 15 μm metal-bonded diamond disc and emery paper (no. 1200), respectively, then ultrasonically cleaned in acetone, followed by drying in an hot oven. Joining was performed in a quartz tube using an infrared heating vacuum furnace ($< 2.64 \times 10^{-3}$ Pa) at 1063 K for 0.42 ks. The heating and cooling time was 3.6 and 14.4 ks, respectively. This is the joining condition that has been reported to show the highest strength of a Si_3N_4/Inconel 600 alloy joint [5].

2.2. Evaluation of the strength

Fig. 1(a–c) shows the dimensions of the specimens for the shear, tension, three-point bending and four-point bending tests. After joining, the surface of the tensile and four-point bending specimens were carefully polished using emery paper (no. 1000 to 2000) and a 15 μm metal-bonded diamond disc, respectively. The joint strength was evaluated with an Instron testing machine. Special jigs, which are shown in Fig. 2(a–c),

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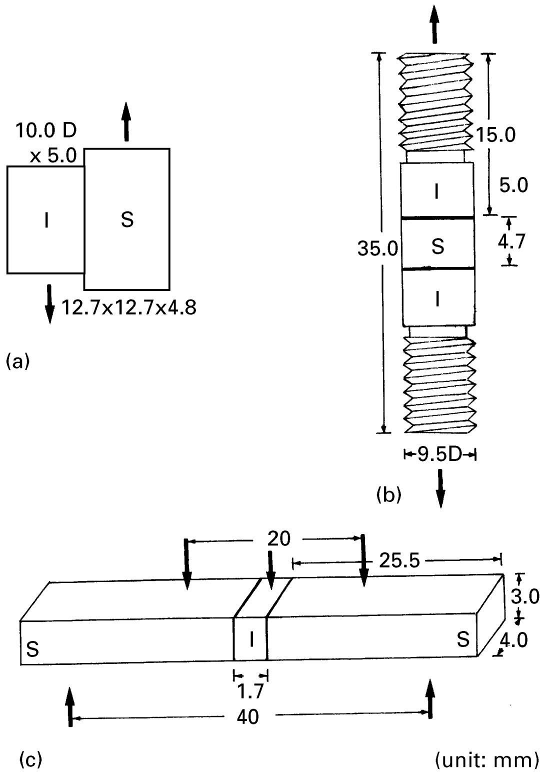


Figure 1 Dimensions of (a) shear, (b) tension, and (c) three-point and four-point bending test specimens (S, I, and D stand for silicon nitride, Inconel 600 alloy, and diameter, respectively).

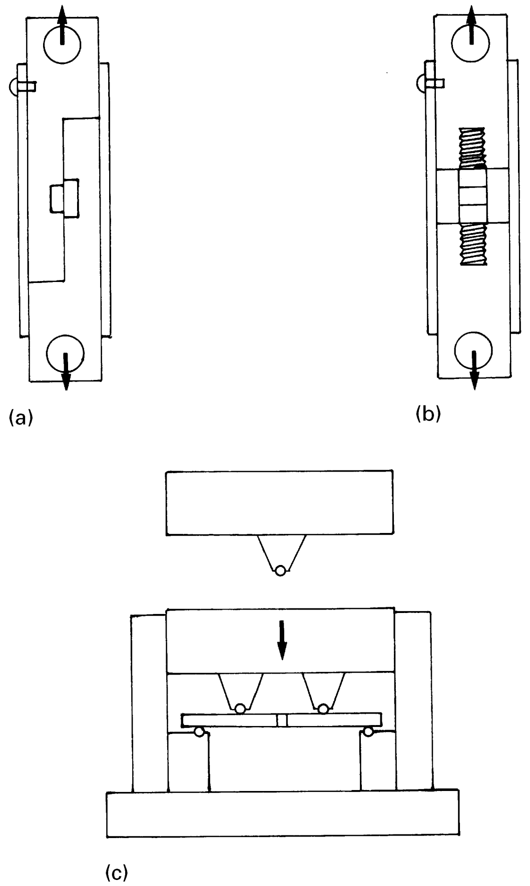


Figure 2 Jigs for (a) shear, (b) tension, and (c) bending strength tests.

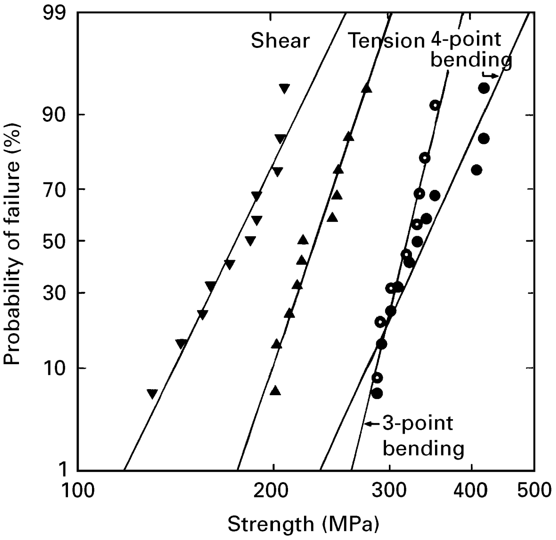


Figure 3 Weibull plot of shear, tension, three-point and four-point bending strength data for the Si_3N_4/Inconel 600 alloy joints which were brazed with the Ag–37Cu–5.5Ti (in at %) alloy at 1063 K for 0.42 ks.

were used in order to minimize any possible bending moment that could be encountered during the shear and tension tests. Eight to eleven specimens were measured for each test. The cross head speed was fixed at 8.33×10^{-5} for all tests. In order to obtain a Weibull plot, the strength data was treated using median rank and least square methods.

3. Results and discussion

3.1. Weibull strength distribution of Si_3N_4/Inconel 600 alloy joints

Fig. 3 and Table 1 show the Weibull plot and list the data, for the shear, tension, three-point and four-point bending strength of the Si_3N_4/Inconel 600 alloy joints that were brazed with Ag–27Cu–3Ti alloys at 1063 K for 0.42 ks. The average shear, tension, three-point and four-point bending strengths of the Si_3N_4/Inconel 600 alloy joints were 177, 233, 321, and 344 MPa respectively. From these results, the strength ratio is 1 : 1.32 : 1.81 : 1.95 in the order of shear, tension, three-point and four-point bend tests. Suganuma *et al.* [4], have reported that the average value of the three-point bending strength was 2.5 times higher than that of the tension strength for Si_3N_4/Invar alloy joints bonded with aluminium. They also reported that this experimental value was higher than the calculated value. The present results indicate that the average value of the three-point bending strength was 1.74 times higher than that of the tension strength. This value is very similar to that calculated by Suganuma *et al.* However, it is considered that the strength ratio varies with the type of materials in the joint, the size and the geometric shape of the specimen, and the bonding variables.

From Fig. 3 and Table 1, the Weibull moduli of shear, tension, three-point and four-point bending strength of the Si_3N_4/Inconel 600 alloy joint were 7.29, 9.97, 13.95, and 7.36, respectively. The Weibull moduli have different values depending on the strength test method used to obtain it. The higher a Weibull modulus is, the smaller is the scatter on the

TABLE I Weibull data of shear, tension, three-point and four-point bending strengths for the Si_3N_4/Inconel 600 alloy joints that were brazed with the Ag–37Cu–5.5Ti (in at %) alloy at 1063 K for 0.42 ks

Test method	Average strength σ_A (MPa)	Maximum strength (MPa)	Minimum strength (MPa)	Weibull modulus m	Scale parameter ξ	Standard deviation (MPa)	Number of specimens measured
Shear	177.6	208.7	131.2	7.29	187.9	25.9	11
Tension	233.0	277.7	201.5	9.97	244.3	25.1	11
4-point bending	344.0	419.6	287.0	7.36	366.1	49.8	11
3-point bending	321.4	355.5	288.5	13.95	332.7	24.3	8

strength measurement. Based on the above Weibull moduli, three-point bend and tension tests showed smaller strength scattering than that of shear and four-point bend tests.

In the bending tests, the Weibull modulus of the three-point bending strength was higher than that of the four-point bending strength. However the average value of the three-point bending strength was lower than that of the four-point bending strength. In order to clarify this apparent contradiction, three-point and four-point bend tests were carried out on Si_3N_4/Si_3N_4 joints, that had been brazed with the Ag–27Cu–3Ti alloys, as is shown in Fig. 4. In case of the Si_3N_4/Si_3N_4 joints, the three-point bending strength had nearly the same Weibull modulus as the four-point bending strength. However, the average strength value from the four-point bending test was slightly higher than that measured using the three-point bending test. In addition the fracture showed the same modes for the two tests.

Fig. 5 shows the Weibull plots for bending strength for Si_3N_4/304 stainless steel joints that had been brazed with the Ag–27Cu–3Ti alloys. The Weibull modulus obtained from the four-point bending test was slightly higher than that of the three-point bend test. However, the average strength value measured by the three-point bend test was slightly lower than that obtained from the four-point bend test.

It can be seen from Figs 3–5 that the strength measured using the four-point bend test on ceramic/metal joints was slightly higher than that obtained from the use of the three-point bend test, even if the Weibull moduli were different to each other. These results are contrary to those observed for a monolithic ceramic, and must be discussed in comparison with an effective volume or area.

The results of Katayama and Hattori [6] have clearly shown that an effective volume or an effective area is a main factor that determines the strength of a monolithic ceramic. That is, the strength of a monolithic ceramic increases as the effective volume or effective area decreases. The effective volume in a four-point bend test specimen is larger than that of a three-point bend test specimen. However, this result for monolithic ceramics did not reflect the observed behaviour for the ceramic/ceramic and ceramic/metal joints. From Figs 3–5, as was discussed previously, the four-point bending strength of ceramic/metal joints was slightly higher than that obtained from a three-point bend test. Whereas the effective volume or effective area in a four-point bend test specimen is larger than that for a three-point bend test specimen. Thus, the bending strength of a ceramic/metal joint decreased as the effective volume or effective area of the bend test specimen decreased.

On the other hand, the effective volume of a tension test specimen is much larger than that for a bend test

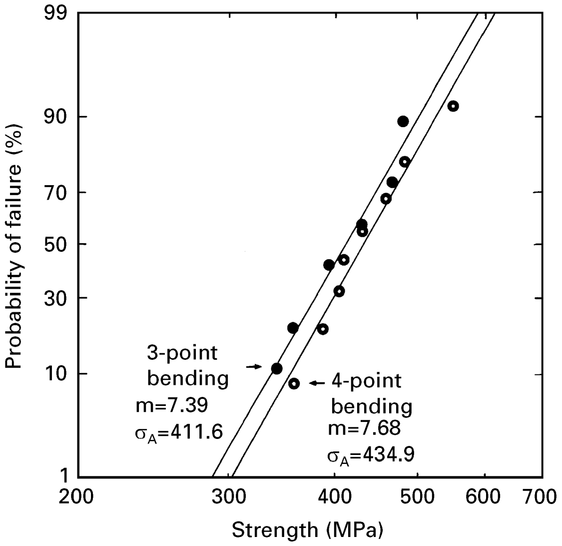


Figure 4 Weibull plot of three-point and four-point bending strength data for Si_3N_4/Si_3N_4 joints that were brazed with Ag–37Cu–5.5Ti (in at %) alloy at 1063 K for 0.42 ks.

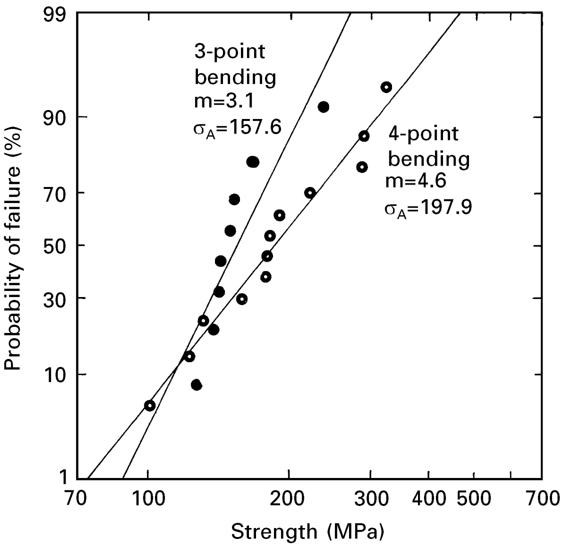
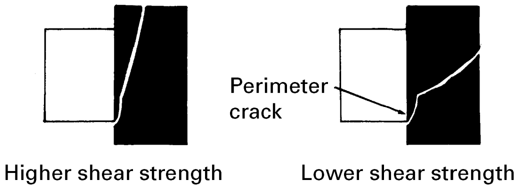


Figure 5 Weibull plot of three-point and four-point bending strength data for the Si_3N_4/304 stainless steel joints that were brazed with Ag–37Cu–5.5Ti (in at %) alloy at 1133 K for 2.1 ks.

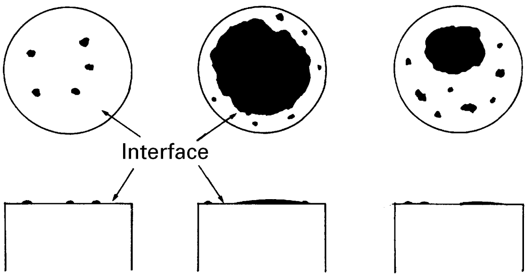
specimen. For the present ceramic/metal joint, the tension strength showed a much smaller value than bending strength. A comparison between the two test methods shows that the results for the Si_3N_4/Inconel 600 alloy joint closely agree with the conclusions of Katayama and Hattori [6] for monolithic ceramics.

3.2. Analyses of the fracture path

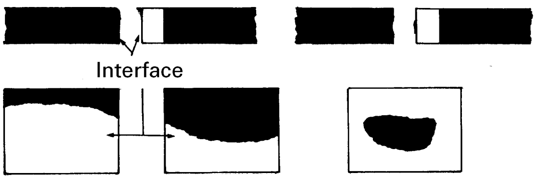
Fig. 6(a–c) shows schematic diagrams of the fracture path in the specimens used to obtain Weibull data from the various strength test methods. From Fig. 6a, the fracture path observed after the shear test shows that a crack propagated from the perimeter [10] of the bond interface into the silicon nitride. In addition, a crack in the silicon nitride propagated parallel to the bond interface in higher shear strength specimens and at a large angle to the bond interface in the lower shear strength samples. Fig. 6a shows that tension failure occurred along the bond interface and in the silicon nitride near to the interface (this crack corresponds to a concave edge crack [10] towards the silicon nitride). This means that the crack propagated by varying its path through the bond interface and the edge of the silicon nitride. In the case of the tension tests, three types of fracture paths were, observed regardless of the strength values, as is shown in Fig. 6b. In three-point and four-point bend tests, a fracture occurred which changed its path along the bond interface and through the silicon nitride near to the interface. In the case of the four-point bend tests, three types of fracture paths also were observed regardless of the strength values, as is shown in Fig. 6c.



(a) Side-view



(b) Top-view and side-view



(c) Top-view and side-view

Figure 6 Schematics of fracture path observed after (a) shear, (b) tension, and (c) three-point and four-point bending strength tests for the joint which was brazed at 1063 K for 0.42 ks (white region: Inconel 600 alloy; black region: silicon nitride).

From these results, it can be seen that the fracture path depends on the strength test methods. That is, the fracture path observed in shear tests was different from that of tension and four-point bend tests. However, the fracture path after all four types of strength tests is found to be caused by differences in the stress distribution generated in the test pieces as well as the stress concentration at the bond interface and it's perimeter.

4. Conclusion

The experiments reported in this paper were performed in order to examine the relationship between the shear, tension, three-point and four-point bending strengths of Si_3N_4/Inconel 600 alloy joints. The Si_3N_4/Inconel 600 alloy joints were produced using an Ag–27Cu–3Ti alloy at 1063 K for 0.42 ks.

Average values of the shear, tension, and three and four point bending strengths were 178, 233, 321, and 344 MPa, respectively. From these results the strength ratio was 1 : 1.32 : 1.81 : 1.95 in the order of shear, tension, three-point and four-point bending tests. Weibull moduli of the shear, the tension, the three-point and the four-point bending strengths of the Si_3N_4/Inconel 600 alloy joint were 7.29, 9.97, 13.95, and 7.36, respectively. Based on the above Weibull moduli, it appears that the three-point and tension tests showed smaller strength scattering than did the shear and four-point bending tests.

From the results of Katayama and Hattori [6], the strength of a monolithic ceramic increased as an effective volume or an effective area decreased. Conversely from the above result for the monoclinic ceramic, the average bending strength of ceramic/ceramic and ceramic/metal joints decreased as the effective volume of the bend test specimen increased. These results were obtained from bending strength tests of the Si_3N_4/Inconel 600 alloy joints.

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