# Transient liquid phase bonding of an ODS ferritic steel to silicon nitride

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The primary objective of this research was to assess transient liquid phase (TLP) bonding as a method of fabricating ceramic to metal bonds for high temperature applications (900–1000 °C) using amorphous brazing foils based on the Fe—B—Si composition. These interlayers are capable of reducing the temperature at which bonding occurs, but gives a joint region with a high melting point. Although some work has been undertaken by researchers to develop high temperature interlayers, these have been used to fabricate ceramic to ceramic bonds, and need a joining temperature of 1300 °C or greater [1].

Hitherto, transient liquid phase bonding has been used successfully for joining superalloys [2–4], but has not been used for fabricating ceramic to metal bonds. The TLP bonding process involves bond formation by isothermal solidification of the joint region as melting point depressants are lost from the interlayer at the bonding temperature. The diffusion away of these melting point depressants has a two-fold effect. First, the joint formed has a re-melt temperature similar to that of the base alloy of the interlayer (i.e., greater than 1200 °C) and secondly, the joint region is free from brittle intermetallic compounds.

A ferritic oxide dispersion strengthened (ODS) alloy was used with a composition in wt% of Cr-20, Ti-0.5, Al-4.5 and $Y_2O_3$-0.5 and hot pressed $Si_3N_4$ ceramic samples. The ODS alloy was bonded to the ceramic in a vacuum of $\sim 10^{-4}$ mbar at a temperature of 1200 °C using an Fe-based interlayer with a composition in wt% of Fe-78, B-13 and Si-9. Rapid heating using r.f. induction coils was used to heat the bond region up to the bonding temperature. The bonding time was varied between 2–20 min after which the joint region was allowed to slow cool to room temperature in vacuum. The ceramic-metal bonds were prepared for metallographic examination using light and scanning electron microscopy.

The light micrograph shown in Fig. 1 is for a bond made using the Fe—B—Si interlayer. Fine precipitates were visible in the vicinity of the bond-line and within the ODS alloy. These have been identified as metal borides and silicides in earlier work [2]. Increasing the bonding time reduced the number of precipitates formed within the joint region and the application of some light pressure (0.1 MPa) during bonding further improved bond quality. The use of pressure reduced the volume of liquid interlayer and this resulted in a greater diffusion away of melting point depressants and a reduction in precipitate formation. In general, good ceramic to metal bonds were produced free from porosity and intrinsic defects. However, in some joints microcracks were visible in the $Si_3N_4$ ceramic parallel to the bond-line and adjacent to a Ti-rich region near the ceramic interface. Energy dispersive spectroscopic analysis (EDS) from the ceramic/braze interface indicated that the migration of Fe towards the ceramic had not taken place although a high concentration of Al (9 wt%) and Ti ($\sim$4 wt%) was detected. Both of these elements are present within the ODS alloy and appear to have been released into the liquid layer and diffused to the ceramic owing to the high affinity of these elements to nitrogen.

X-ray diffraction taken from fracture surfaces of ceramic-metal bonds made using the Fe—B—Si interlayer showed peaks for TiN and CrB. The variations

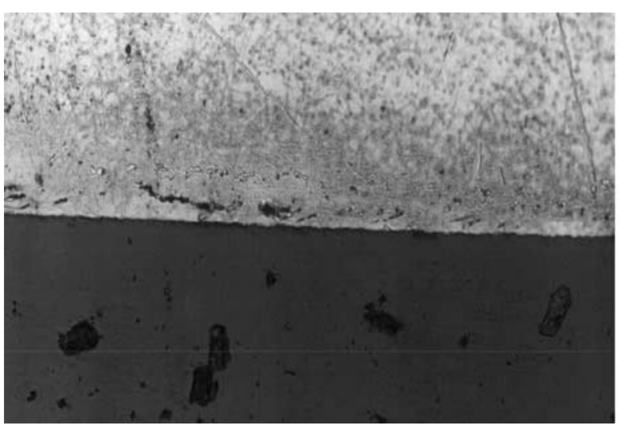


*Figure 1* Light micrograph showing a transverse section through the TLP bonded ceramic-metal joint.

in their compositions and lattices suggested that these were formed as hypostoichiometric compounds. The formation of titanium silicides was also expected due to the reaction of Ti with free Si from the ceramic, but this was not detected.

EDS analysis indicated that there was a significant movement of Cr towards the ceramic interface. It is suggested that the diffusion of Cr to the ceramic interface will result in the formation of chromium nitrides, and was could be responsible for encouraging wettability and for holding the bond together, but more work is necessary to confirm this. A few joints were shear tested in order to examine the point of fracture and the bonding surfaces. A fracture surface of the steel side of a joint produced using the Fe—B—Si interlayer is shown in Fig. 2. Failure was at the bond interface and through the interlayer. Part of the interlayer (A) was seen on the steel surface (B). In the same way, the ceramic fracture surface (see Fig. 3) also indicated the presence of "islands" of the interlayer (A) bonded to the ceramic surface (B). Interestingly, the fractographs showed that the problems of wettability of the ceramic surface were not observed and this corresponded to transverse sections taken of the ceramic-metal bonds which showed good contact between the liquid interlayer and ceramic and metal interfaces. This could be attributed to the diffusion of Cr and B to the ceramic surface where the formation of a reaction layer would promote bonding. Although a significant concentration of Cr was detected at the ceramic/interlayer interface (13 wt%), B being a light element could not be detected at the interface, see Table I.

TABLE I EDS analysis showing the change joint composition (wt%) as a function of distance from the $Si_3N_4$/braze interface

| Distance from $Si_3N_4$/braze interface ($\mu$m) | Fe | Cr | Ti | Al | Si |
|---|---|---|---|---|---|
| 0 | 66.8 | 13.4 | 3.6 | 8.9 | 7.3 |
| 10 | 80.0 | 16.6 | 0.1 | 2.1 | 1.1 |
| 20 | 78.0 | 16.7 | 0.9 | 2.6 | 0.8 |
| 30 | 79.6 | 16.8 | 0.3 | 2.3 | 0.7 |
| 40 | 69.7 | 26.9 | 0.2 | 2.0 | 0.8 |
| 50 | 79.2 | 17.2 | 0.2 | 2.4 | 0.7 |
| 250 | 76.2 | 20.6 | 0.2 | 2.9 | 0.1 |

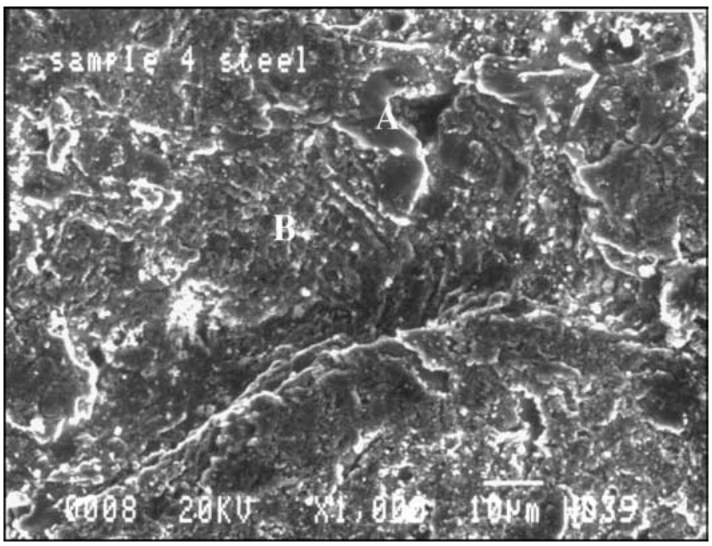


*Figure 2* Fractograph showing steel surface of the TLP bonded joint.

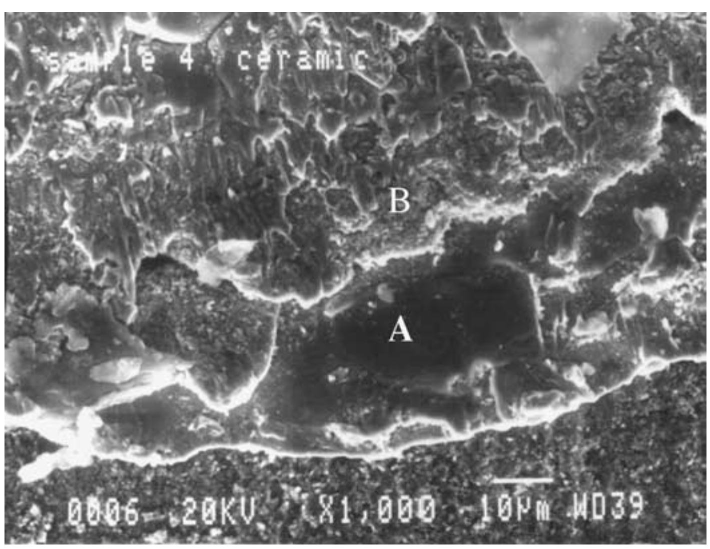


*Figure 3* Fractograph showing ceramic surface of the TLP bonded joint.

These preliminary results show that TLP bonding can be used as a technique for fabricating ceramic-ODS alloy bonds for high temperature use. However, the success of the bonding process will depend upon the concentrations of alloying elements and melting point depressants within the interlayer and further work in this area is necessary.

## References


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