Letters

Joining of dense silicon carbide by hot-pressing

Two dense SiC sintered bodies were joined with sinterable submicron SiC powder by hot-pressing at a temperature as low as 1650° C, and the bond strength of the joined specimen was measured by four-point bending at high temperatures. The dense SiC sintered body* was hot-pressed, with dimensions of $10 \times 10 \times 15 \text{ mm}^3$, the impurities were 2.5 wt % W, 1.5 wt % Fe, 0.5 wt % Cr, 0.5 wt % Ni, 0.5 wt % Co and a small amount of B and C, and the bulk density was $3.39 \,\mathrm{g}\,\mathrm{cm}^{-3}$. The submicron powder of SiC was prepared by heating a mixture of SiO and carbon black with metallic aluminium [1]; the resulting powder consisted of 2H and 3C polytypes of SiC and it further contained 0.03 wt % Fe and 6 wt % Al. 1 wt % of B and C were added to the powder and mixed. The mixture was placed between the two bodies and prepressed in a graphite die, and then hotpressed in Ar at 1650° C for 0.5 h with a pressure of $100 \, \text{kg} \, \text{cm}^{-2}$.

The joined specimen was sawed into $3 \times 5 \times 30 \text{ mm}^3$, and the surfaces were ground and polished with a diamond to a surface roughness, R_{max} , of less than $1 \mu \text{m}$. The four-point bending test was carried out in vacuum (~ 10^{-4} mm Hg) at high temperatures, with an upper span of 6 mm and lower span of 14 mm and at a cross-head speed of 0.1 mm min⁻¹. The temperature was measured using a platinum thermocouple (PR6/30) placed near the specimen.

The results are shown in Fig. 1, for a bending strength of the original sintered body of 60 kg mm^{-2} . The microstructure of the joined part is shown in Fig. 2. The lower part of the picture shows the original dense SiC in which the white spots indicate impurities, and the black spots indicate sites from which an impurity has been removed. On the other hand, the upper part shows the adhesive SiC, about $150 \,\mu\text{m}$ thick, where the grey spots a few μm wide are not pores. Lange [2] obtained a dense SiC of density 99% theoretical by hot-pressing SiC powder with 2% A1₂O₃ of 1950° C for 1 h at a pressure of 280 kg



Figure 1 Bond strength in four-point bending at high temperatures.

cm⁻². He found $A1_2O_3$ phase in the microstructure. In the present study, electron microprobe analysis showed that impurities did not significantly diffuse from the original to the adhesive SiC and that the adhesive was studded with Al. The state of Al is not as yet determined, although the wavelength of AlK α corresponds to that of a compound in which the Al atom has a co-ordination number of 6, as α -Al₂O₃ does [3]. It seemed that the decrease in strength above 1500° C was due to effect of the Al.

Acknowledgements

The authors thank Mr T. Hase for the many helpful suggestions. The research was supported in part by a grant from the Ministry of Education, no. 343030.



Figure 2 Microstructures of the joined area. Upper: the adhesive SiC; lower: the original SiC.

References

- 1. T. HASE, H. SUZUKI and T. ISEKI, Yogyo-Kyokaishi 87 (1979) 576.
- 2. F. F. LANGE, J. Mater. Sci. 10 (1975) 314.
- 3. T. ISEKI and H. TAGAI, J. Amer. Ceram. Soc. 53 (1970) 582.

Received 26 July and accepted 13 September 1979

Microstructure of the weld region in resistance-welded zircaloy 4

An important factor determining the properties of zircaloys is microstructure. This is affected by heat treatment and cold work. The zircaloys owe their good mechanical properties to a large extent to their fine α -grain size. During fabrication, zircaloy tubes are subjected to certain stages for developing these mechanical properties. The different welding and brazing processes used to attach some parts to the fuel sheathing involve heating of these parts (or some localized areas of them) into the β -phase field (above 975° C). This can be harmful to some of the properties which have been achieved during the fabrication process. But there are some welding processes which are so rapid that only very fine β -grains are produced: one of these is the resistance welding process. This process takes place in a time shorter than 1 sec and to this time corresponds the heating and cooling rates in the range of $10^{3^{\circ}} \text{Csec}^{-1}$ [1]. The resistance welding process leaves the thermomechanical properties of the tubes unaffected practically right up to the joint [2]. The rapid heating and cooling cycles, together with the intimate contact of the parts to be welded, avoid the formation of brittle or rapidly corroding surface layers during welding of the zircaloys.

Zircaloy, when welded, is locally heated above 975° C. This induces phase transformations during heating and cooling cycles. The result of the temperature cycle is a structure dependent on cooling rates. On cooling from the β -phase field at moderate rates (oil quenching to furnace cooling) zircaloys transform to give widmanstätten-type structure, but increasing the cooling rate produces finer widmanstätten plates to a quenched martensitic structure [3–5].

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An attempt was made to characterize the microstructure obtained in resistance (projection) welded zircaloy 4. The samples chosen for this study came from a production zircaloy 4 tubing. The specimens were prepared for metallographic examination by mechanically polishing on progressively finer emery papers finishing on 600-grit paper. This was followed by chemical polishing using the following solution: H₂O:HNO₃:HF (50:47:3). Fig. 1 is an optical micrograph showing the transition of the welded zone, heat-affected zone and the base material. The heat-affected zone of a zircaloy spot (or projection) weld is very narrow because of its low thermal conductivity. The non-random grain orientation of the base material is due to the plastic deformation during fabrication of zircaloy fuel sheathing. Higher magnifications of the welded zone gave a similar structure to that of a specimen cooled at $2 \times 10^{3^{\circ}} \text{ C sec}^{-1}$, which was named a marensitic-type structure [3]. Fig. 2 is a scanning electron micrograph at higher magnifications of the welded zone. Electron



Figure 1 Optical micrograph of the transition zone between the welded part, heat-affected zone and base material. Polarized light, \times 245.