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Joining and machining of (ZrB_2-SiC) and (Cf-SiC) based composites

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ABSTRACT

Filler materials of (ZrB_2-SiC-B_4C-YAG) composite were developed for gas tungsten arc welding (GTAW) of the ZrB_2-SiC and Cf-SiC based composites to themselves and to each other. Reaction with filler material, porosity and cracks were not observed at weld interfaces of all the joints. Penetration of filler material in to voids and pores existing in the Cf-SiC composites was observed. Average shear strength of 25.7 MPa was achieved for joints of Cf-SiC composites. By incorporation of Cf-SiC (CVD) ground short fibre reinforcement the (ZrB_2-SiC-B_4C-YAG) composite was machinable with tungsten carbide tool. The joint and machined composites were resistance to oxidation and thermal shock when exposed to the oxy-propane flame at 2300 °C for 300 s. The combination of (ZrB_2-SiC-B_4C-YAG) and Cf-SiC based composites can be used for making parts like thermal protection system or nozzles for high temperature applications.

1. Introduction

Zirconium diboride (ZrB_2) is well known for its oxidation property since 1960s. It has very attractive combination of properties such as high melting point, high chemical stability, high hardness and strength, and high thermal and electrical conductivities suitable for thermal and chemical environments in hypersonic flight, rocket propulsion, and atmospheric re-entry [1–3]. It is a promising material for structural parts in high temperature environments such as high speed air craft leading edges and nose cone. Due to its covalent bonding, and low self diffusion coefficient, ZrB_2 based composites can be sintered to high density by hot-pressing (HP) [4–6]. Through HP process, simple geometries and moderate sizes can only be made. Fabricating complex shapes by diamond machining is an expensive and time taking process. Pressure less sintering (PS) offers a cheap and near-net shaping of complex parts by minimizing machining. Large size or complicated shape components of ceramics can be fabricated at low cost by joining.

Carbon fiber reinforced silicon carbide (Cf-SiC) composites are well known for high strength and modulus, moderate fracture toughness, and high resistance to oxidation at high temperatures. They are considered promising materials for hypersonic aircraft applications particularly for heat shields and advanced propulsion structural components [7]. Uniform and complete densification is difficult to achieve in large and complex shaped components. Joining is inevitable to develop Cf-SiC components for high temperature applications. There exist several techniques to join monolithic ceramics or ceramic matrix composites: brazing [8], diffusion welding [9], reaction joining process [10,11], precursor infiltration and pyrolysis [12], reactive melt infiltration [13] and chemical vapor deposition online joining process [14,15]. But the joining strengths are still not enough for engineering application. Mechanical joining with screws, nails, bolts, and adhesives (glues and epoxies) is not suitable for high temperature environments [16]. Welding and brazing of Cf-SiC composites is not easy because commonly used filler materials have little or no wetting. Brazing with suitable filler normally requires special surface treatment and equipment [17,18]. Different layers of refractory borides (TiB_2 and ZrB_2), carbides (SiC, B_4C, and WC), or their mixtures (TiB_2 + SiC + B_4C), were used for solid state diffusion bonding [19,20]. Long exposures to high temperatures (about 2000 K) under relatively high loads are used to obtain a pore-free layer and strong joint. But long heat treatment at high temperature deteriorates the properties of parent composites.

Recently Cf-SiC composites were joined by spark plasma sintering (SPS) using (SiC + 5 wt% B_4C) powder mixture [21], Ti foil and calcia-alumina glass ceramic [22]. All these techniques require rapid heating by SPS under a pressure of 60 MPa to a temperature of 1480–1900 °C for 3–10 min. In spite of heating to such a high temperature the shear strength of the resulting joint was

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17.3 ± 7.8 MPa only. Ti_3SiC_2 max phase produced by self propagating high temperature synthesis (SHS) was also employed for diffusion brazing in high vacuum hot pressing at 1600 °C under 25 MPa pressure [23].

Different braze alloys are used to join ZrB_2-SiC based composites to themselves and to commercially pure Ti [24,25]. Chemical interaction with braze alloy and interfacial cracking due to residual stresses were observed. Due to melting of these solders the joints cannot be useful at temperature above 1000 °C. Parts up to 3 mm thick of ZrB_2-20 vol% SiC and ZrB_2-SiC-B_4C composites were joined by gas tungsten arc welding (GTAW) or plasma arc welding. Porosity at weld interface is unavoidable [26,27].

In an earlier work [28] a filler with high vol.% of B_4C and YAG (ZrB_2-25 vol.% SiC-25 vol.% B_4C - 16 vol.% YAG) was used to join hot pressed (ZrB_2-20 vol.% SiC), and pressure less sintered (ZrB_2-SiC-B_4C-YAG) composites to themselves. In this work a modified filler material of (ZrB_2-SiC-B_4C-YAG) with low quantities of (Y_2O_3, Al_2O_3, and B_4C) [29] has been used to join Cf-SiC composites to themselves and to (ZrB_2-SiC) based composites. The joint facing ZrB_2-SiC composite was exposed to the oxy-propane flame (2300 °C) in 30 s interval to examine the resistance of the joint to thermal cycling, ablation and oxidation.

Some degree of machining is always required to make complex and precision components. Even with diamond tools, laser machining and ultrasonic machining the machining cost usually accounts for 70–90% of the total cost [30]. Electrical discharge machining (EDM) requires a material resistance and can only machine components of small size [31]. Traditional mechanical machining is of both cost-effective and time-efficient. Mechanical machining of strong and hard ZrB_2-SiC is very difficult. By introducing mica, h-BN, graphite, pores, rare-earth phosphates and Ti_3SiC_2 the machinability of ceramic materials can be improved [32]. The fabrication of a machinable ZrB_2-SiC-BN composite by hot pressing at 1800 °C and 23 MPa was reported [33]. Though machinability increased the fracture toughness and hardness of the composite decreased due to the formation of large agglomerates or platelets of BN. Dense ZrB_2-SiC-BN composites with fine grain size and homogeneous microstructure were fabricated via reactive spark plasma sintering of a mixture of ZrH_2, Si_3N_4 and B_4C powders at 1900 °C in vacuum [34].

A ceramic throat insert for a nozzle was machined by EDM of a composite prepared by hot pressing a mixture of 46 vol % ZrB_2 + 8 vol % Si_3N_4 and 46 vol % C chopped fibers at 2100 K and 30 MPa [35]. Fiber degradation was effectively inhibited in ZrB_2-SiC-Cf composites containing 20–50 vol % carbon short fibers using nanosized ZrB_2 powders and hot pressing at low sintering temperature (1450 °C) [36]. In the present work a machinable ZrB_2-SiC based composite was pressure less sintered at relatively low temperature of 1580–1650 °C. Scrap pieces of Cf-SiC composites were ground and the (−200, +325) mesh size powder obtained after sieving was used to reinforce the ZrB_2-SiC based composite. The resultant composite was subjected to drilling and exposure to the oxy-propane flame at 2300 °C.

2. Experimental

2.1. Materials

The ZrB_2 powder was synthesized via B_4C reduction of ZrO_2 reaction (1). When B_4C and ZrO_2 react in the stoichiometric wt. ratio of $ZrO_2/B_4C \approx 3.0$, the formation of ZrB_2 with impurities like ZrO_2, B_4C, and C occur due to the loss of boron as B_2O_3. To compensate the loss of boron as B_2O_3 and to obtain a single phase ZrB_2 without impurities, excess of B_4C was taken in a wt. ratio of $ZrO_2/B_4C = 2.5$ [37]. ZrO_2 powder was supplied by Nuclear Fuel Complex, Hyderabad, India. B_4C powder was purchased from China Abrasives, Zingzhou, China. Fine SiC powder with particle size (d_{50} ~ 0.8 μm) was received from H.C. Starck, Germany. Super fine size (d_{50} ~ 0.7 μm) Al_2O_3 from Alcan and sub micron sized Y_2O_3 were used. Cf-SiC composites processed by chemical vapor deposition (CVD) and reaction bonded silicon carbide (RBSC) were manufactured by CSIR, National aerospace laboratories, Bengaluru and DRDO, Advanced systems laboratory, Hyderabad respectively. The waste or scrape pieces of Cf-SiC (CVD) retained after fabricating test samples for flexural strength and oxidation evaluation are ground in a mortar with pestle. After sieving the ground powder, the (−200 and + 325 #) fraction was used for reinforcing (ZrB_2-SiC-B_4C) composites.

$$7ZrO_2 + 5B_4C \rightarrow 7ZrB_2 + 5CO + 3B_2O_3 \quad (1)$$

2.2. Pressure less sintering of ZrB_2-SiC based composites and filler welding rods

Y_2O_3 and Al_2O_3 were added as sintering additives for PS of (ZrB_2-SiC-B_4C) composite. The typical composition used was (ZrB_2: SiC: B_4C: Y_2O_3: Al_2O_3 = 65: 20: 8: 3: 4) vol. %. The mixing of dry powders was done for 24 h with alumina balls in a polythene bottle. Compacts of 60 and 30 mm diameter were made by uni-axial compaction with a load of 9 Tons and 3 Tons respectively. PS of ZrB_2-SiC based composites at 1650 °C in argon atmosphere for 1.0 h was carried out in a carbon furnace (ASTRO, USA, Model 1000-3060-FP20) [38]. The bulk density of the PS composite is 4.52 $g \cdot cm^{-3}$. The Vickers micro hardness with 200 g load was about 12.53 GPa and its flextural strength was 213 MPa. Further, a 20 vol % of Cf-SiC (CVD) powder of (−200 and + 325 #) size was added to above (ZrB_2: SiC: B_4C: Y_2O_3: Al_2O_3 = 65: 20: 8: 3: 4) vol. % composite and pressure less sintered.

For GTAW of ZrB_2-SiC based composites a filler with high vol.% of B_4C and YAG (ZrB_2 - 25 vol.% SiC - 25 vol.% B_4C - 16 vol.% YAG) was used [28]. The vol. % of Y_2O_3, Al_2O_3 and B_4C in the filler was decreased to (ZrB_2: SiC: B_4C: Y_2O_3: Al_2O_3 = 65: 20: 8: 3: 4) to allow rapid deposition of high volume of molten filler to flow into the pores existing in the Cf-SiC based ceramics. Dry powders of ZrB_2, SiC, B_4C, Y_2O_3 and Al_2O_3 were mixed for a minimum of 8 h. PVA binder in water solution is used to make a thick paste. The paste obtained is extruded through a nozzle or medical syringe to obtain a wire/rod of about 3 mm diameter. Alternately green compacts of filler material were made by uniaxial pressing in a steel die. The extrusions or compacts after drying in a oven at 110 °C for 1 h were pressure less sintered at 1650 °C. Using diamond cut off wheel or electro discharge wire cut machine, bar samples of size 3 × 3 and 55–75 mm long were cut from sintered compact. The sintered extrusions or cut bars were used for GTAW of Cf-SiC composites to themselves and to ZrB_2-SiC based composites.

2.3. Joining of (ZrB_2-SiC) and (Cf-SiC) based composites

The Cf-SiC composite and ZrB_2-SiC based composite of size 4 × 5 × 50 mm long were used for joining by GTAW. The coupons to be joined are kept on steel platform. Keeping a Cf-SiC composite piece and another Cf-SiC composite piece or ZrB_2-SiC based composite piece at a distance or gap around 1 mm. A square butt weld configuration was employed in the present study. Welding parameter employed are: 90–120 A current, and speed of 3 $mm \cdot min^{-1}$. After welding the argon flow was continued till the joint was cooled to a temperature below 800 °C. The joining was repeated on the opposite side. A Cf-SiC (CVD) composite piece of

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