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## High strength alumina joints via transient liquid phase bonding

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#### Abstract

Low melting boron oxide, instead of metallic materials in other methods of transient liquid phase bonding, was taken as braze in joining alumina in this paper. Pure boron oxide melts at low temperature and reacts with alumina matrix to form a stable high melting compound. This transient liquid phase bonding has the advantage of producing a ceramic joint for high temperature applications at low processing temperature. In this study, alumina pieces coated with boron oxide layers in various thicknesses were bonded at  $800\,^{\circ}\text{C}$  for various times in air under minor loading. The average flexural strength of joints were measured by means of four point bending, while the microstructure of the cross-section and fractured surface was observed by means of scanning electron microscopy. Phases at joints were identified by low angle X-ray diffraction. The maximum flexural strength reaches a value of 155 MPa after joining at  $800\,^{\circ}\text{C}$  for 15 h with a  $21\,\mu\text{m}$  interlayer. Three compounds,  $3Al_2O_3$ – $B_3O_3$ ,  $2Al_2O_3$ – $3O_3$  and  $3Al_2O_3$ – $3O_3$  have been found at the joint. It is also found that  $2Al_2O_3$ – $3O_3$  whiskers dominate at the joint with the maximum strength.

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#### 1. Introduction

Many industrial processes need parts lasting under severe conditions including high temperature, high stress, corrosive solutions, biochemical reactions, etc. Undoubtedly, fine ceramics meet the need due to their special properties, including high melting/decomposition temperature, excellent wear/corrosion resistance, biocompatibility, high strength and hardness, etc. However, these properties make ceramic objects hard to form with complicated shape or large size. To join several ceramic parts into a large, complicated assemble is a promising way. It inspires the improvement of ceramic joining techniques aiming that mechanical properties of joined objects are influenced by the joint as little as possible.

The methods for joining ceramics include solid-state diffusion bonding [1], friction joining [2], laser welding [3], ultrasonic joining [4] and brazing [5]. Except brazing, ceramic joining is usually carried out at elevated temperature which results in the degradation of joint and hinders its application. Transient liquid phase bonding (TLPB), also known as

"isothermal solidification" academically or as "diffusion brazing" technically, is a joining technique in which a low melting interlayer is introduced, melts at joining temperature and either reacts to form solid compounds or diffuse into matrix isothermally. Due to the formation of high melting compound(s) the operational temperature of the joined object can be higher than the joining temperature. This method has been successfully applied for joining some metallic and ceramic systems [6-10]. For joining alumina, different metallic materials as interlayer have been experimented and fine results have already been obtained. Using the Cu/Pt, Cu/Ni/Cu, Cu/Nb/ Cu multilayer combinations, the joining temperature must be above 1000 °C and a flexural strength above 200 MPa has been reached. However, metallic brazes restrict the joining and usage environments of a joined component and reduces its practicability. Using metals with high temperature and oxidation resistance, e.g. Pt, Nb [11] or Au [12], is a way to improve the joining property in expense of high material costs.

Recently, a new attempt was proposed, instead of metallic interlayer, boron oxide interlayer was used for joining alumina pieces [13]. Glassy boron oxide has a very low melting point of 540 °C. The eutectic point in the  $B_2O_3/Al_2O_3$  system is 470 °C. Two equilibrium incongruent compounds  $2Al_2O_3-B_2O_3$  and  $9Al_2O_3-2B_2O_3$  with melting point of 1035 and 1950 °C are

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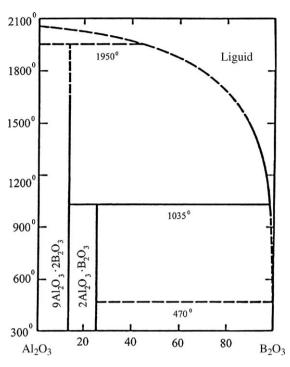


Fig. 1. Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub> phase diagram [14].

seen in the phase diagram (Fig. 1) [14]. These high melting compounds formed at the joint of the  $Al_2O_3/B_2O_3/Al_2O_3$  assembly after joining, but the maximum strength reported was merely 71 MPa with an interlayer thickness of 3  $\mu$ m at a joining temperature of 800 °C. However, some advantages of this technique like moderate joining temperature, no precious metal and no protective atmosphere were needed to reduce the overall cost of joining. Besides, the ceramic interlayer may cause a galvanic effect much lower than that expected in a ceramics/ metal heterogeneous system. The purpose of this work is to improve the flexural strength of alumina joint by optimizing the joining time and interlayer thickness of transient liquid phase bonding and to explain it with the formation and morphology of compounds at the interface.

#### 2. Experimental

In this study, boron oxide was sandwiched in two alumina pieces. The raw materials are 99.6%  $\alpha$ -alumina powder (ALCOA A16SG). Alumina slurry with 50% solid content and 0.05 wt.% dispersant (Darvan 7) was ball mixed for 24 h. Green cakes of alumina were formed by pressure slip casting with an air pressure of 7 atm, dried at 40 °C for 24 h, 60 °C for 24 h and at 200 °C for 6 h to completely remove water and dispersant. Finally, dried cakes were sintered at 1550 °C for 6 h to get alumina bulks with a density of 3.91 g/cm² and four-point flexural strength of 332 MPa.

Alumina bulks were slowly cut into pieces with the size of  $15 \text{ mm} \times 20 \text{ mm} \times 10 \text{ mm}$  by diamond saw. The joined surface was polished to 1  $\mu$ m diamond paste and ultrasonically de-contaminated in ethanol. 99.6% boron oxide powder (STREM CHEMICALS) was heated to 300 °C on a hot plate (HP-46825) and boron oxide films in various thicknesses were

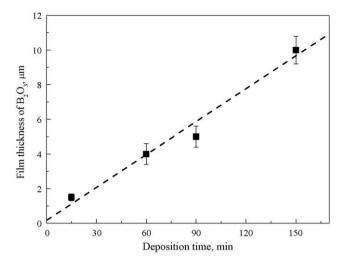


Fig. 2. Relation between the film thickness of boron oxide and deposition time.

deposited on water-cooled alumina surfaces in air for deposition times of 15, 60, 90 and 150 min. The relation between the film thickness and deposition time is plotted in Fig. 2. Two alumina pieces were vertically joined at the coated sides, put into an electric furnace (THERMOLYNE FB-1300) and dwelt at 800 °C for different times. A small load of 15 kPa was applied for keeping the interface in good contact.

Joined specimens were slowly cut-off in the direction perpendicular to the joint plane. The cross-section of the alumina joints was observed by SEM (JEOL JSM-5400). Six pieces of 4 mm  $\times$  4 mm  $\times$  25 mm specimens for each joining condition were four-point bended with MTS (HT-8116). In order to see the morphology and phases formed, the fractured surface of specimens was analyzed by using SEM and low-angle XRD (MAC SCIENCE MXP3), respectively.

#### 3. Results and discussion

The average flexural strength of the alumina joints TLP bonded at 800 °C for different joining times and with various

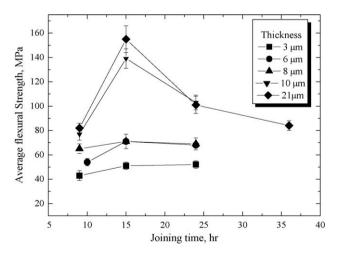


Fig. 3. Average flexural strength of the alumina joints TLP bonded for different joining times and with various interlayer thicknesses from the four-point bending test.

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