JOURNAL OF MATERIALS SCIENCE 40 (2005) 2477–2479

High-strength oxide ceramics joints obtained by diffusion bonding with the use of platinum and palladium gaskets

YU. NAIDICH, I. GAB*, T. STETSYUK, D. KURKOVA
Frantsevich Institute for Materials Science Problems, National Academy Sciences of Ukraine, 3 Krzhyzhanovsky st., 03680 Kyiv, Ukraine
E-mail: naidich@ipms.kiev.ua

Alumina and zirconia ceramics joints were made by diffusion bonding through platinum and palladium gaskets in air at temperatures between 1300 and 1550°C. The instrumentation and the procedure for these joints manufacturing are described. The effects of temperature, pressure and exposition time on 3-pt bending strength of resulting joints were established. The maximum bending strength of the samples reached 500 MPa. During thermal resistance testing the samples withstood several hundreds of thermal cycles at temperatures up to 800°C in air.

1. Introduction

Permanent joints of two oxide ceramics parts, and of ceramics with metals, obtained by metal solders brazing [1], or by diffusion bonding using metal gaskets [2] have been used successfully for a long time in various areas of engineering in a broad range of temperatures from cryogenic [3] up to 800°C [4, 5]. However, in order to satisfy the requirements for applications in extreme conditions, there is more often necessity to obtain strong joints between ceramics, capable to work at temperatures up to 1200–1400°C in various conditions including the atmospheric environment. At present, ceramic materials based on alumina and zirconia are suitable for long-term operation in air atmosphere at high temperatures up to 1600°C. But manufacturing of joints able to work under the above mentioned environmental conditions is a complicated problem: solders able to work in such conditions can be materials of the platinum group, resistant to the oxygen action at high temperature. For the activity reported in the present paper we selected palladium and platinum as solders to join alumina and zirconia. Besides great technological difficulties (very high melting temperatures, especially in the case of platinum, evaporation of these metals in vacuo, etc.), active brazing by platinum and palladium solders is also not acceptable because of bad wetting of the chosen ceramics by these melts [6]. Therefore our selection of a method to join alumina and zirconia was based on the diffusion bonding technique, carried out in air atmosphere.

Articles devoted to manufacturing of alumina/alumina [7] and alumina/zirconia [8] joints through platinum gasket in air by the diffusion bonding method are known from the literature. In both papers bending strength values of joints did not exceed 250 MPa. The purpose of the present work was to develop stronger joints between Al_2O_3 and ZrO_2 ceramics, by diffusion bonding in air using platinum and palladium gaskets.

2. Experimental procedure

For the development and characterisation of joints high purity (99.5%) alumina ceramics and partially stabilised zirconia (8% wt. Y_2O_3) were produced. Sample bars 2 × 4 mm in cross-section and 10 mm high were prepared. Samples for diffusion bonding were ground with diamond powders of 14 and 5 microns sizes and then polished with diamond powder of 2–3 microns sizes to produce a smooth surface. Samples were then degreased with acetone and alcohol in ultrasonic bath. Gaskets 1.5 × 2.5 mm in size were cut from a platinum foil 100–150 micron thick and were also degreased in ultrasonic bath of acetone and alcohol. The initial gaskets sizes was smaller then the size of the ceramic part in view of the increase of the gasket area due to deformation by the applied pressure. Samples assembling for joint manufacturing was carried out by inserting the platinum gaskets between the parts to be joined in a specific ceramic equipment made of zirconia in which grooves 2 × 4 mm in size (Fig. 1) were made. Three samples pairs were put simultaneously in the equipment. Samples joining by pressure was carried out in a SVK 8163 furnace with lantanum chromite heaters able to heat samples up to 1700°C in air. This furnace was equipped with a special device allowing to transfer the pressure (25 MPa) to the samples (Fig. 2). Pressure was applied to lever 4 by loading it by graded weights 9. This lever presses on the ceramic rod 3 through the ceramic rotating joint 2. In turn, this rod is in contact

*Author to whom all correspondence should be addressed.

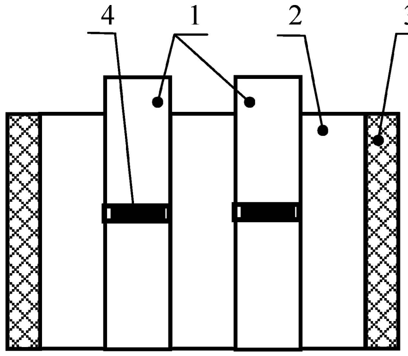

Figure 1 Scheme of ceramic samples assembly for pressure bonding in ceramic equipment: 1—ceramic samples to be joined; 2—demountable ceramic insert made from two halves; 3—ceramic holder; 4—platinum gasket.

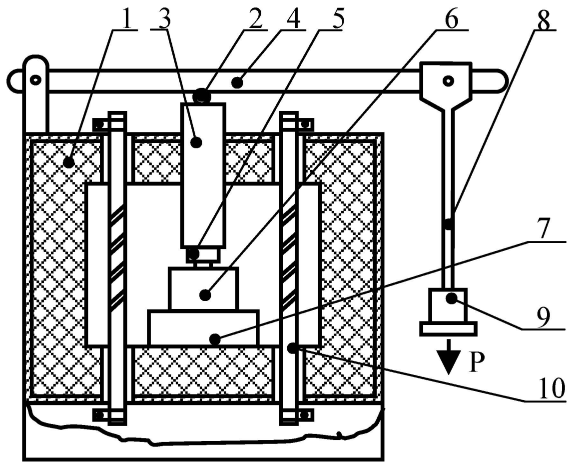

Figure 2 Scheme of installation for materials pressure joining on air: 1—furnace SVK 5163; 2—rotating joint; 3—ceramic rod; 4—lever; 5—ceramic insert; 6—samples in ceramic equipment; 7—ceramic support; 8—shackle; 9—load; 10—lanthanum chromite heaters.

TABLE I Strength of ceramic samples diffusion bonded in air

No	Bonding characteristics	Bonding creation conditions	Bonding bend strength (MPa)
1	ZrO_2/Pd/ZrO_2	$T = 1200°C$ $p = 25$ MPa $\tau = 30$ min	280
2	ZrO_2/Pd/ZrO_2	$T = 1300°C$ $p = 25$ MPa $\tau = 120$ min	400
3	ZrO_2/Pd/ZrO_2	$T = 1300°C$ $p = 25$ MPa $\tau = 240$ min	420
4	ZrO_2/Pd/ZrO_2	$T = 1450°C$ $p = 15$ MPa $\tau = 30$ min	470
5	ZrO_2/Pt/ZrO_2	$T = 1300°C$ $p = 25$ MPa $\tau = 30$ min	210
6	ZrO_2/Pt/ZrO_2	$T = 1300°C$ $p = 25$ MPa $\tau = 120$ min	430
7	ZrO_2/Pt/ZrO_2	$T = 1300°C$ $p = 25$ MPa $\tau = 240$ min	460
8	ZrO_2/Pt/ZrO_2	$T = 1550°C$ $p = 15$ MPa $\tau = 30$ min	490
9	Al_2O_3/Pt/Al_2O_3	$T = 1300°C$ $p = 25$ MPa $\tau = 30$ min	180
10	Al_2O_3/Pt/Al_2O_3	$T = 1300°C$ $p = 25$ MPa $\tau = 120$ min	320
11	Al_2O_3/Pt/Al_2O_3	$T = 1300°C$ $p = 25$ MPa $\tau = 240$ min	340
12	Al_2O_3/Pt/Al_2O_3	$T = 1450°C$ $p = 25$ MPa $\tau = 30$ min	360

with the samples to be joined. Rotating joint 2, rod 3, insertion 5, and the support 7 were made of high-strength zirconia ceramic which is the most stable to softening and deformation at temperatures up to 1550°C. Heating up was performed at 5°C/s, samples cooling was carried out together with the furnace within 24 h down to 40°C. The samples joined in such a way were submitted to mechanical tests in a 3-pt bending apparatus.

3. Results and discussion

Test data are presented in Table I. Flexural strength values are the result of 4–5 testing in every regime. Bending strength of ZrO_2 ceramics was 600 MPa and that of Al_2O_3 ceramics was 450 MPa. As it is evident from data of Table I, the strength of joints is high enough and results 75–80% of the ceramics strength. Samples examination after the tests has shown that fracture of samples made at high temperatures (1400–1550°C) and also with long terms of exposure to pressure (2 and 4 h) at 1300°C occurred in the ceramic near the joining zone. In other cases, the fracture took place in part in the bulk ceramic and in part along the metal/ceramic interface. Similar joints strength values can be obtained by different combinations of the key parameters of the joining process: temperature, pressure, and time of exposure to pressure. Indeed, ZrO_2/Pt/ZrO_2 joint with bending strength 490 MPa can be made at 1550°C under high pressure (15 MPa) during 30 min. Identical joint with bending strength of 460 MPa can be produced at lower temperature (1300°C) but under higher pressure (25 MPa) and during longer time (240 min.). However, because it is rather difficult to reach the high temperatures in air (more than 1200°C) and, besides, at these temperatures both the ceramics to be joined and all other ceramic parts (rod, gaskets, etc.) start deformation under load, it seems more favourable to make joints at temperatures up to 1300°C with an exposure time of 120 min. Longer exposure times at this temperature (up to 240 min) yield only a slight increase of the joint strength.

Thermal resistance tests were carried out in a tubular furnace with nickel-chrome heater, where the samples were heated and cooled from 50°C up to 800°C for one hour. All samples have sustained 300 thermal cycles without fracture.

4. Conclusion

The diffusion bonding technique using platinum or palladium gaskets in air is suitable to produce high-strength thermally resistant joints of alumina and zirconia. The strength of the joints reaches 80% of the initial strength of the ceramic parts. Depending on the size and the final application of the joined ceramic product, it is possible to recommend the following processing conditions:

1. For platinum gasket: $T = 1550°C$, $P = 15$ MPa, $\tau = 30$ min.
2. For palladium gasket: $T = 1450°C$, $P = 15$ MPa, $\tau = 30$ min.
3. For both types of gaskets: $T = 1300°C$, $P = 25$ MPa, $\tau = 120$ min.

Using the conditions $T = 1300°C$, $P = 25$ MPa, $\tau = 120$ min, diffusion bonded sapphire-alumina optical windows for observation and photographing of high-temperature processes in physical and chemical reactors at temperatures up to 1500°C were produced. These windows consist of optically clear leucosapphire disk 20 mm in diameter and 4 mm wide joined through platinum gasket to an alumina tube 20 mm in diameter and 100 mm long. These products have shown good characteristics during their use.

The particular joints presented here can find application in mechanical and chemical instrumentation, in particular in the design and creation of alumina/zirconia fuel and oxygen cells.

References

1. M. A. RUBASHEV, G. I. BERDOV, V. N. GAVRILOV, et al., "Heat-Resistant Dielectrics and Their Junctions with Metals in New Engineering" (Atomizdat, Moskow, 1980) p. 246 (Russian).
2. N. F. KAZAKOV, "Diffusion Bonding of Materials" (Engineering, Moskow, 1976) p. 312 (Russian).
3. YU. V. NAIDICH, V. A. KONDRATSKY, V. S. ZHURAVLEV, et al., *Adhesiya rasplavov i paika materialov* **1** (1976) 74 (Russian).
4. YU. V. NAIDICH, I. I. GAB, V. S. ZHURAVLEV, *et al.*, *Avtomaticheskaya svarka* **11** (1996) 33 (Russian).
5. YU. V. NAIDICH, V. S. ZHURAVLEV and N. I. FRUMINA, *Adhesiya rasplavov i paika materialov* **3** (1978) 99 (Russian).
6. N. F. GRIGORENKO, A. I. STEGNY, I. E. KASICH-PILIPENKO, *et al.*, in "HTC'94," edited by N. Eustathopoulos (Institute of Inorganic Chemistry Slovak Academy of Science, Bratislava, 1994) p. 123.
7. R. V. ALLEN and W. E. BORBIDGE, *J. Mater. Sci.* **18**(9) (1983) 2835.
8. J. CHRISTENSEN and K. GOTTHJAEIP, *DVS-Ber.* **148** (1992) 187.

Received 31 March
and accepted 18 July 2004