



## Regular article

## Reversible flash-bonding of zirconia and nickel alloys

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## ABSTRACT

A electric field-assisted flash-joining technique of ceramic and metal was shown here. It was demonstrated that when the voltage and current density were higher than critical values, the zirconia ceramic and Ni based superalloy were strongly bonded in as fast as 1 s at 800 °C. Another important advantage of the technique is that the resultant joint can be readily de-bonded by reversing the electric field, which provides significant manufacturing flexibility. The underlying bonding mechanism follows an electric field-induced internal reaction, and the novel method could be extended to a wide range of ceramic-metal systems in principle.

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The continuous demand for rapid and low-temperature bonding of ceramics and metals has led to the development of various bonding techniques that are classified into liquid-state bonding (LSB) and solid-state bonding (SSB) [1]. LSB involves the use of low-melting-point fillers (or in-situ formed eutectic phases) that form a liquid phase and fill the gaps between the materials to be bonded [1–4]. While LSB allows rapid bonding at low temperatures, the working temperature of the resultant component is severely constrained by the softening of the joining media at low temperatures [5–8]. Solid-state bonding involves direct inter-diffusion (and/or reaction) of the materials to be bonded, and can greatly increase the working temperature of the component. However, SSB must be performed at high temperatures and prolonged times, often resulting in severe degradation of both the parent materials and the joints [9–13].

A electric field has been employed to assist ceramic-to-metal bonding, wherein electric field can greatly reduce the bonding temperature and bonding time [14–19]. Such reductions are attributed to the enhanced diffusion and/or wettability of ceramics by the metals. However, the joint formed between the ceramics and metals has always contained defects such as micro-voids and micro-cracks. Moreover, a reversible bonding is needed in some cases.

Herein, we demonstrate a novel electric field-induced flash- and reversible bonding technique for the joining of ceramics and metals. ZrO<sub>2</sub> ceramic can be rapidly bonded to Ni alloy in 1 s at 800 °C. When a reversed electric field is applied to the bonded couple, the ceramic-metal joint can be easily debonded.

The starting materials were commercial 3 mol%-Y<sub>2</sub>O<sub>3</sub> stabilized zirconia (3YSZ) ceramics (Dongguan Mingrui Ceramic Co., Dongguan,

China, 99% theoretical density, shear strength ~322 MPa) and Ni-based superalloy GH710. The joining surfaces were polished to a roughness of 1 μm and ultrasonically cleaned for 30 min.

The specimens to be bonded were assembled and placed between two graphite blocks acted as electrodes (Fig. 1a). A pressure of ~5 MPa was applied to the assembly that was then placed in a vacuum furnace at ca. 10<sup>-2</sup> Pa and heated to required temperatures at heating rate of 15 °C/min. After reached to the goal temperature, an electric field was applied to the sample. The voltage and current in the circuit were output by digital multimeter (UT61E). Typical variations in the voltage, current, and power dissipation during flash-bonding are shown in Fig. 1b as a function of time. Upon the application of voltage, the electric current increased gradually and surges occurred to a preset limitation. From then onwards, the current remained the same as the pre-set value, while the voltage decreased to a lower level. The joining time was defined as the dwelling period after the current reached the pre-set value. Such variations in voltage, electrical current, and power dissipation (defining flash-bonding), only occurred when the applied initial voltage was higher than a critical value. In this study, all bonding experiments were performed at the electric field strength of 100 V/cm on the ceramic block. After bonding, the system was cooled naturally to room temperature.

The morphologies of the bonded and debonded samples were observed by using scanning electron microscopy (SEM, HITACHI S-4700) equipped with energy dispersive X-ray spectroscopy. The microstructures of the joints were investigated with transmission electron microscopy (TEM, Tecnai G2 F20 S-Twin). The strength of the bonded samples was assessed by the shear testing model (Fig. 1c) on a universal tester (SANS CMT-4304), and the loading rate used was 0.05 mm/min.

Fig. 2a plots the shear strength of the bonded samples as a function of current density for the ZrO<sub>2</sub>-Ni joint formed at 800 °C for 30s. The

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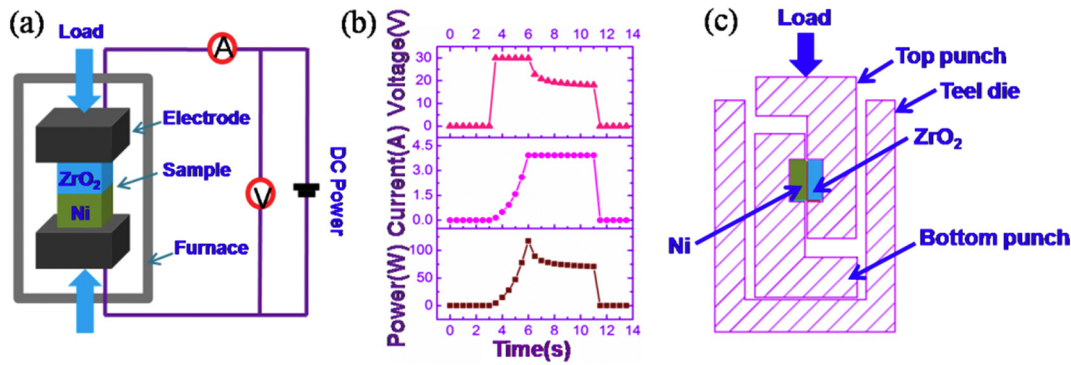


Fig. 1. (a) The schematic of the joining apparatus, (b) the variations of electric current, voltage, and power dissipation with time, and (c) the apparatus for the shear testing.

result clearly reveals that there is a current density threshold (80–90 mA/mm<sup>2</sup>) above which the strength of the joint is abruptly increased. Fracturing in samples bonded at low current density occurred within the bonding layer, while that in samples bonded at high current density occurred in the ZrO<sub>2</sub> ceramics close to the bonding layer.

The effect of bonding time on the shear strength was studied for the samples joined at 800 °C and 100 mA/mm<sup>2</sup> (Fig. 2b). The shear strength was observed to decrease with increasing joining time. The maximum strength of ca. 133 Mpa is reached at a bonding time of approximately 1 s. The fractures were observed located in the ceramics, close to the bonding layer, suggesting that the electric field led to ceramic degradation.

Fig. 2c compares the effect of bonding temperature on the shear strength of the joints formed at 100 mA/mm<sup>2</sup> and 1 s. The shear strength was observed to increase with increasing temperature up to 800 °C, then subsequently decreasing with further increases in temperature. The bonding layer formed at lower temperatures (700 and 750 °C) did not fully cover the gap between the two materials, and thus the fracture was observed in the bonding layer. On the other hand, the bonding layer formed at higher temperatures (800–900 °C) was fully dense and defect free, and the fracture was observed in the ZrO<sub>2</sub> ceramics close to the bonding layer.

The fracture surfaces of the samples bonded at 800 °C and the original ZrO<sub>2</sub> ceramic were examined by SEM. The original ZrO<sub>2</sub> showed a

transgranular fracture with rather smooth fracture surface (Fig. 2d). However, the fracture of the bonded samples gradually transitioned from transgranular mode to intergranular mode with increasing bonding time (Fig. 2e and f), indicating that the electric field weakened the grain boundaries of the ceramic. This weakening effect became more obvious through the formation of pores within the ceramic when the bonding time was extended to 15 min (Fig. 2g).

The microstructures of the bonding layers formed at 800 °C and 100 mA/mm<sup>2</sup> for 1 and 60s were examined. In both samples, the 3YSZ and Ni-alloy were well bonded with the formation of a dense bonding layer (Fig. 3a and b). The layer thickness was approximately 1.3 and 2.9 μm for the samples bonded for 1 and 60s, respectively. The linear elemental distribution (Fig. 3c and d) indicated that the bonding layers were formed via diffusion of Ni into the ZrO<sub>2</sub>. High resolution TEM (HRTEM) and selected area electron diffraction (SAED) showed the bonding layer in the 1-s bonded sample to be tetragonal ZrO<sub>2</sub> (Fig. 3e), while that in the 1-min bonded sample consisted of tetragonal ZrO<sub>2</sub> and a small fraction of Ni metal (Fig. 3f). The precipitation of Ni metal within ZrO<sub>2</sub> was confirmed by scanning TEM (STEM, Fig. 3g), which revealed that the Ni metal was distributed along ZrO<sub>2</sub> grain boundaries. A thin layer of approximately 0.4 μm was formed on the Ni-alloy side for the 1 min bonded sample (Fig. 3b). Elemental analysis revealed that the bonding layer contained Zr and elements from the starting Ni-alloy (Fig. 3h), indicating that the thin layer was formed by

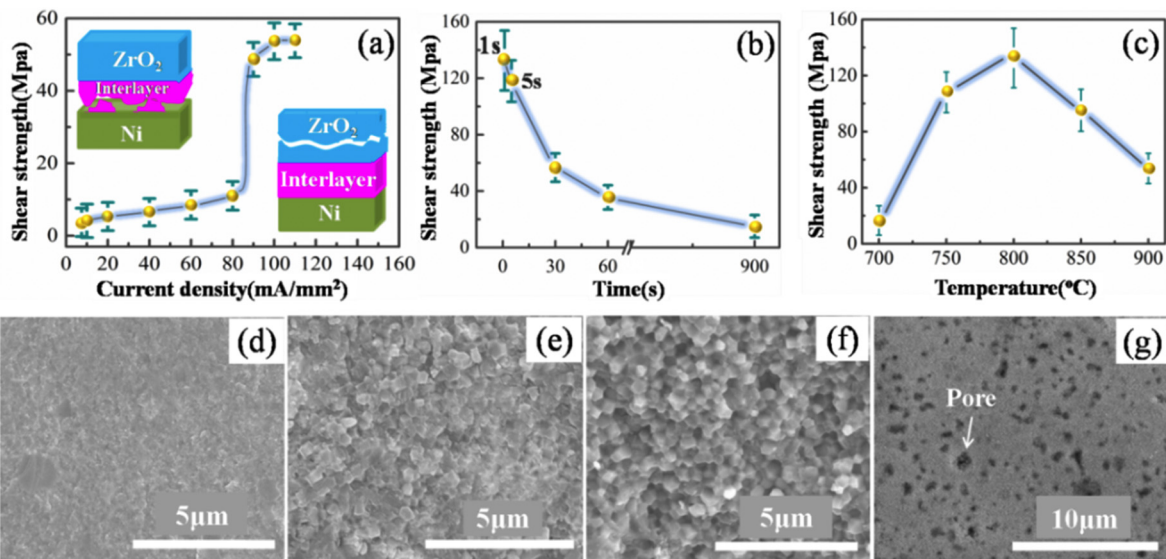


Fig. 2. The shear strength of the samples joined at: (a) different current densities at 800 °C for 30s, (b) 800 °C, current density 100 mA/mm<sup>2</sup> for different times and (c) current density 100 mA/mm<sup>2</sup> for 1 s at different temperatures. The SEM images of the fractured samples of: (d) the as-received ceramic, (e) the 1 s joined sample, (f) the 1 min joined sample and (g) the cross-section of the 15 min joined sample.

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