

## Densification and Mechanical Properties of Y-TZP/25 wt. % ZrB<sub>2</sub> Composite

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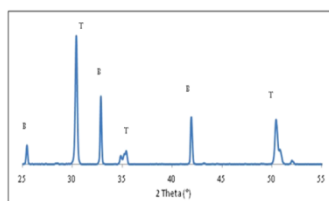
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### Graphical abstract



### Abstract

Over the last three decades, most of the efforts in mechanics of materials science have been focused to develop tougher and stronger ceramics via cost effective processing techniques. In the present work, 3 mol % Ytria-stabilized tetragonal zirconia (3Y-TZP) composites with 25 wt. % of ZrB<sub>2</sub> was prepared by pressureless sintering method in argon atmosphere over the temperature range of 1350-1550°C for one hour holding time. The influences of zirconium diboride addition in the zirconia matrix, as well as the sintering temperature, on the densification, phase stability and electrical properties of sintered samples have been studied. The results revealed that electrical resistivity values is very low (high electrical conductivity) when 25 wt. % of ZrB<sub>2</sub> is incorporated to pure 3Y-TZP.

*Keywords:* Sintering; mechanical properties; Y-TZP composite; zirconium diboride

### Abstrak

Sejak tiga dekad yang lalu, kebanyakan kerja dalam mekanik sains bahan fokus untuk mencipta dan menghasilkan seramik yang lebih kuat dan tahan melalui kaedah pemrosesan yang lebih kos efektif. Di dalam kerja ini, 3 mol% Yttria-stabil tetragonal Zirkonia (3Y-TZP) komposit dengan 25 wt% ZrB<sub>2</sub> telah disediakan melalui pensintiran tanpa tekanan di dalam atmosfera argon di dalam suhu 1350-1550°C selama satu jam. Kesan penambahan ZrB<sub>2</sub> kepada matriks Zirkonia, termasuk suhu pensintiran, densifikasi, stability fasa dan ciri-ciri elektrik sampel yang disinter telah dikaji. Keputusan dari kajian menunjukkan kadar rintangan elektrik yang amat rendah (kadar konduksi elektrik yang tinggi) apabila 25 wt% ZrB<sub>2</sub> ditambah kepada 3Y-TZP

*Kata kunci:* Pensintiran; mekanikal; Y-TZP komposit; ZrB<sub>2</sub>

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### 1.0 INTRODUCTION

Yttria Stabilized Zirconia (Y-TZP) offers many advantages over conventional ceramics. 3Y-TZP (zirconia doped with 3 mol% yttrium oxide) has excellent mechanical properties such as high fracture toughness due to the famous mechanism known as transformation toughening [1, 2]. Because of this unique property, zirconia has been extensively used in many engineering applications and has become appropriate to produce engine parts, valves, cutting tools and moulds, sandblasting nozzles and biomedical implants.

In the last three decades, ceramics industrial fields have been extensively trying to reduce the price of the ceramic parts. If a ceramic part cannot meet the cost requirements, it cannot be employed. Major portion of the price of a ceramic part consists of machining cost. Grinding of ceramics using diamond wheels,

which is one of the machining methods of ceramics, is often very expensive and time-consuming. In addition, producing of very complicated shape ceramics by diamond grinding route is almost impossible. Development of electro-conductive zirconia-based ceramics would be advantageous as components could be fabricated to near-net shape with WEDM machining [3].

Various electro-conductive phases such as WC[4], TiC[5], TiCN[5], ZrB<sub>2</sub>[6] and TiB<sub>2</sub>[7] have been studied to prepare electro-conductive zirconia. One of the most interesting combinations is Y-TZP with zirconium diboride. ZrB<sub>2</sub> is a non-oxide ceramic with special properties such as high electrical conductivity and high Vickers hardness [6]. The objective of the present work is to evaluate the effects of adding 25 wt. % of ZrB<sub>2</sub>, on density, mechanical properties and electrical resistivity (electrical conductivity) of Y-TZP ceramics.

## 2.0 EXPERIMENTAL PROCEDURE

The 3 mol% yttria-stabilized zirconia (3Y-TZP) starting powder used in this work was manufactured by Kyoritsu Ltd., Japan under the code name of KZ-3YF according to the hydrolysis method and spray dried to obtain free-flowing ready-to press powder. The zirconium diboride used in this work was obtained from a commercial available  $ZrB_2$  powder (Wako, 99% purity) and amount of that used was 25 wt. %. The 3Y-TZP and  $ZrB_2$  were mixed in 150 ml. of ethanol via ultrasonification followed by ball milling for 1 hour. After the mixing, the wet slurry was dried, crushed and sieved into powder form. Then obtained composite powder was uniaxial pressed at 2.5-3.0 MPa into circular discs (20 mm diameter and 3mm thickness) and rectangular bars ( $4 \times 13 \times 32$  mm) and subsequently cold isostatic pressed at 200 MPa (Riken Seiki, Japan). Compacted green samples were sintered at temperature ranging from 1350-1550°C, in a tube furnace with ramp rate of 5°C/min and soaking time of 1 hour in Argon atmosphere. Prior to testing, all sintered samples were polished to 1µm surface finishing.

The phase stability studies of the all sintered samples were carried out by using X-Ray diffraction (XRD) (Geiger-Flex, Rigaku Japan). The bulk densities were obtained by water immersion technique (Mettler Toledo, Switzerland). The Young's modulus (E) by sonic resonance was determined for rectangular samples using a commercial testing instrument (GrindoSonic: MK5 "Industrial", Belgium) [8]. The Vickers hardness ( $H_v$ ) and fracture toughness ( $K_{Ic}$ ) of sintered discs was determined using a pyramidal diamond indenter (Future Tech, Japan) with an applied load of 10 kg and for 10 seconds to the polished surface of the sample. The physical quality of the indenter and the accuracy of the applied load as defined clearly in ASTM E384-99 [9] and ISO 14705 [10] must be controlled to get the correct results. After the load is removed, the average diagonal length of the indent was measured 3 times for each sample and the average value was taken [11].

## 3.0 RESULTS AND DISCUSSION

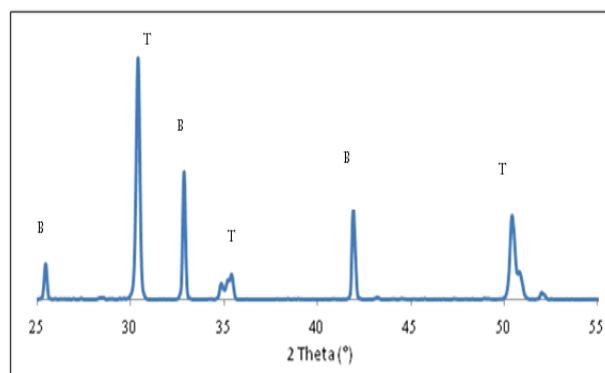
Pressureless sintering experiments with the 3Y-TZP/25 wt. %  $ZrB_2$  composites were carried out in the temperature range of 1350–1550°C in a tube furnace and under argon flow. Table 1 indicates some physical and electrical properties of the 3Y-TZP/25 wt. %  $ZrB_2$  ceramics.

**Table 1.** Properties of 3Y-TZP/ $ZrB_2$  composite sintered at various temperatures

Sintering Temperature (°C)	Relative Density (%)	Young's Modulus (GPa)	Electrical Resistivity (Ω.cm)
1350	87.5	181	25.5
1400	93.3	233	23
1450	96	234	24.2
1500	96	236	25
1550	97.5	246	25

XRD pattern, obtained from the polished surfaces of the 3Y-TZP/25 wt. %  $ZrB_2$  composite is presented in Fig. 1. Here it should be mentioned that sintering temperature did not affect the

phase stability of the samples since XRD plots for all the composites sintered between 1350-1550°C revealed the same signatures. These XRD patterns indicated that the major crystalline phases in the composites are tetragonal- $ZrO_2$  and hexagonal- $ZrB_2$ .



**Figure 1** X-Ray diffraction patterns of 3Y-TZP/25 wt. %  $ZrB_2$  composite. Key: T = tetragonal  $ZrO_2$  and B = hexagonal  $ZrB_2$

It can be seen in the Table 1 that the sample, sintered at 1550°C shows comparatively higher relative density i.e. 97%. Relative density was calculated by taking the theoretical density of 3Y-TZP and  $ZrB_2$  as 6.09 and 6.10 g/cm<sup>3</sup> respectively. On the other hand relative density of the pressureless sintered 3Y-TZP/25 wt. %  $ZrB_2$  composite at 1350°C is considerably lower than the other samples. It should be stated here that relative density of the sample sintered at 1300°C is much lower than samples prepared via other sintering methods [12–14].

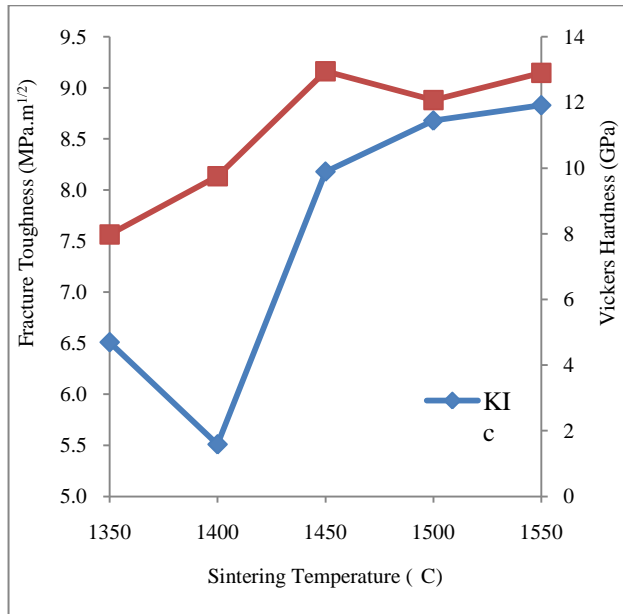
It is well stated that monolithic  $ZrO_2$ , with varying  $Y_2O_3$  content between 2 and 3 mol% could be densified to near theoretical density using pressureless sintering methods [12]. With comparison with the densification of  $ZrO_2$  monoliths, it can be concluded that there are two major reasons for hindering the densification of 3Y-TZP/25 wt. %  $ZrB_2$  composites; significant difference in initial powder particle sizes (nanometer for  $ZrO_2$  and micrometer for  $ZrB_2$ ) and lower self-diffusion co-efficient of  $ZrB_2$  particles. In addition, fully dense for some compositions of  $ZrO_2$ - $ZrB_2$  system is reported for via hot isostatic pressing (HIP) [13, 14].

As far as electrical resistivity is concerned, the sintered composites have low electrical resistivity for efficient electrical discharge machining application. In general, electrical discharge machining requires the material to have the electrical resistivity lower than 100 Ω.cm for efficient machining.

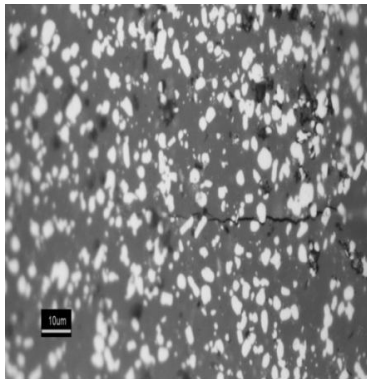
The Vickers hardness and fracture toughness of the samples sintered between 1350-1550°C is shown in Fig. 2. The low Vickers hardness for the 3Y-TZP/25 wt. %  $ZrB_2$  composite sintered at below 1400 °C might be due to lower density of the sample. On the other hand, increasing Vickers hardness values in the samples sintered at above 1450 °C is due to high relative density as well as the effect of  $ZrB_2$  incorporation.

In general, additions of 25 wt. %  $ZrB_2$  had significant effect on the fracture toughness of Y-TZP matrix. The fracture toughness of these samples was found to increase from 7.5 MPa.m<sup>1/2</sup> to 13 MPa.m<sup>1/2</sup> for the composites sintered at 1350°C and 1550°C respectively ( $K_{Ic}$  value for monolithic Y-TZP is ~6 MPa.m<sup>1/2</sup>). The fact that the  $K_{Ic}$  was changed significantly indicates that the additions of  $ZrB_2$  affect the toughening mechanism in the composite. Fig. 3 is the optical micrographic images of crack propagation in the composite sintered at 1500°C. The  $ZrB_2$  grains (bright grains) are expected to deflect the

propagating crack and consequently resulting in a reduction of the stress at the crack tip. High fracture toughness of the 3Y-TZP/ZrB<sub>2</sub> composites could be as a result of crack deflection mechanism thanks to the presence of ZrB<sub>2</sub> phase.



**Figure 2** Variation of Vickers hardness and fracture toughness of Y-TZP/25 wt. % ZrB<sub>2</sub> composite as a function of sintering temperature



**Figure 3** Presence of deflected crack in the 3Y-TZP/25 wt. % ZrB<sub>2</sub> composite sintered at 1550 °C

#### 4.0 CONCLUSIONS

In the present research 3Y-TZP/25 wt. % ZrB<sub>2</sub> composites are sintered at 1350-1550 °C for 1 hour under argon flow to achieve more than 97% of theoretical density. XRD analysis of polished surface of composites shows that fully tetragonal ZrO<sub>2</sub>-based structure can be obtained using pressureless sintering method. More specifically, a high Vickers hardness value (>13 GPa) and high elastic modulus (>230 GPa) were measured for YTZP/25 wt. % ZrB<sub>2</sub> composite sintered at 1550°C. The research also found that the addition of ZrB<sub>2</sub> had a significant effect on the fracture toughness value through crack deflection mechanism.

#### Acknowledgement

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