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### Fretting wear properties of sinter-HIPed ZrO<sub>2</sub>-ZrB<sub>2</sub> composites

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#### Abstract

In view of the fact ceramic composites have an established niche in tribological applications, research efforts were invested in assessing and understanding the tribological properties of the recently developed  $ZrO_2$ –30 vol.% $ZrB_2$  composites against bearing steel. As part of the tribological investigation, fretting wear tests under varying load (2–10 N) were conducted on  $ZrO_2$  composites, presintered at 1400 °C in argon for 1 h followed by HIPing at 1400 °C under 110 MPa argon pressure for 1 h. Under the investigated fretting conditions, sinter-HIPed  $ZrO_2$  composites/steel couple exhibits higher COF of 0.5–0.8. At higher load of 10 N, lowest COF of 0.5 is commonly measured with all the tribocouples. The abrasion is commonly observed as the dominant wear mechanism in the investigated composites. However, the occurrence of transfer layer formation with occasional spalling is also noted. An important result of our research is that the wear rate decreases with increasing toughness and the sinter-HIPed  $ZrO_2$ – $ZrB_2$  composites exhibit lower wear rate ( $\sim 10^{-7}$ – $10^{-8}$  mm<sup>3</sup>/Nm) against steel.

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#### 1. Introduction

There has always been a major impetus to develop tougher and harder ceramics for the tribological applications and extensive research efforts had been put forward to evaluate and understand the tribological properties [1–12]. In recent times, we have been able to demonstrate that dense TZP–30 vol.%ZrB<sub>2</sub> composites can be obtained after sinter-HIPing at 1400 °C for 1 h in 110 MPa Argon pressure [13]. It was also observed that the addition of finer ZrB<sub>2</sub> reinforcements significantly enhances the mechanical properties of Y-TZP materials and a range of mechanical properties with high hardness (upto 16 GPa) and excellent toughness (upto 18 MPa m<sup>1/2</sup>) is measured in these composites. Because of their potential tribological applications, we examine the fretting wear properties of the ZrO<sub>2</sub>–ZrB<sub>2</sub> composites in the present work.

The friction and wear properties of composites depend on material parameters (matrix, reinforcement chemistry and volume, hardness, toughness), counterbody material, operating parameters (sliding speed, load, humidity etc.) and hence are system dependent. The effect of particulate additions on the tribological performance of composites is also reported [4,5]. The incorporation of a second phase is reported to result in improved wear performance. For example, the incorporation of TiB<sub>2</sub> in Si<sub>3</sub>N<sub>4</sub> matrix enhanced the wear resistance. Also, the addition of either TiC or TiN to a Si<sub>3</sub>N<sub>4</sub> matrix has been shown to improve wear performance substantially under tribological conditions, where tribochemical wear is dominant [5].

The influence of grain size on wear of Y-TZP monoliths was investigated by He and co-workers [3]. In one of our earlier works, the fretting wear properties of a range of Y-TZP monoliths were studied against WC–Co hardmetal [14]. Rainforth [8] investigated the micromechanical and microchemical processes involved in dry sliding of several structural ceramics (Y-TZP, Mg-PSZ and ZTA) against bearing steel and zirconia toughened alumina (ZTA)

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ceramic counterfaces. The wear mechanism in these tribocouples was dominated by the tribochemical reactions at the sliding contacts. The role of zirconia phase transformation was found to be negligible.

Although the detailed tribological study was conducted largely on  $ZrO_2$  monoliths with a major focus to understand the role of microstructure and mechanical properties on wear behaviour, such a study is limited in case of  $ZrO_2$ based composites. In the current research, the influence of the mechanical properties on the wear behavior of the developed Sinter-HIPed materials, when fretted against steel (selected because of its engineering importance) is studied. The tribological results are discussed in terms of friction and wear resistance, and the underlying wear mechanisms for the investigated tribocouples have been elucidated.

### 2. Experimental

#### 2.1. Materials

In the tribology experiments, ZrO<sub>2</sub>–ZrB<sub>2</sub> composites are used as flat materials. ZrO2-based 30 vol%ZrB2 composites are densified to  $\sim 97-100\% \rho_{th}$  using a Sinter HIPing route. In the composites, the stabilization of the ZrO<sub>2</sub> matrix is tailored by changing the overall yttria content between 2 and 3 mol%. The ZrO<sub>2</sub> matrix is obtained from a powder mixture of 3 mol% yttria co-precipitated (Tosoh grade TZ-3Y) and monoclinic ZrO<sub>2</sub> (Tosoh grade TZ-0Y) powders. For some composites, by changing the amount of TZ-0Y addition, Y<sub>2</sub>O<sub>3</sub> stabilisation is varied at 2 mol% and 2.5 mol% in the starting powder mixture. All the composite compacts are presintered at 1400 °C for 1 h followed by Sinter-HIPing at 1400 °C for 1 h under 110 MPa Argon pressure. All the mixed grade powder mixture based materials will be termed 'mixed grade' composites and those based on TZ-3Y powders will be termed 'co-precipitated' powder based composites. Table 1 provides the details of the developed ceramic composites together with mechanical properties. More details of the processing, microstructures and properties of the Sinter-HIPed composites are reported elsewhere [13]. The Vickers hardness was measured using a Vickers hardness tester with an indentation load of 30 kg. The toughness values, based on the radial crack lengths emanating from the indents, are calculated according to the formula of Anstis et al. [15]. The investigated Sinter-HIPed composite has a hardness range of 15– 17 GPa and a toughness in the range of 7–18 MPa m<sup>1/2</sup> [13]. Commercially available, 6 mm diameter bearing (commercial SAE 52100 grade, hardness ~7 GPa, data given by supplier) steel balls (heat treated grade, carbon content ~2.1%) were used as counterbodies (stationary).

#### 2.2. Wear tests and characterization

The fretting experiments were performed using a computer-controlled fretting machine (model TR281-M, DUCOM, Bangalore, India), which produces a linear relative oscillating motion with ball-on-flat configuration. By a stepper motor, the flat sample is made to oscillate with a linear displacement of constant stroke and frequency. An inductive displacement transducer monitors the displacement of the flat sample and a piezoelectric transducer is used to measure the friction force. Variation in tangential force is recorded and the corresponding coefficient of friction is calculated on-line with the help of a computer-based data acquisition system. The use of this fretting machine has been made in our recent research, which are reported elsewhere [9,10].

Prior to the fretting tests, both the flat and ball were ultrasonically cleaned in acetone. The fretting experiments were performed on different grades of Sinter-HIPed ZrO<sub>2</sub>– 30 vol%ZrB<sub>2</sub> composites against steel balls with varying load (*P*) of 2, 5 and 10 N at 8 Hz oscillating frequency and 100 µm linear stroke for 100,000 cycles duration. Also, the combination of testing parameters results in gross slip fretting contacts. All experiments were conducted in ambient atmosphere at room temperature ( $30 \pm 2$  °C) with relative humidity (RH) of  $45 \pm 5\%$ . The schematic of the fretting test configuration is shown in Fig. 1.

The wear volumes of the developed HIPed composites are computed by measuring wear scar diameter in the transverse direction on the worn samples and following the Klaffke's equation [1]:

$$V = \left[\frac{\pi d}{8} + \frac{4A}{3}\right] \left(\frac{d^3}{8R}\right) \tag{1}$$

where V is the wear volume; R the radius of the ball (3 mm), d the average diameter of the wear scar in the transverse direction and 2A the displacement amplitude (100 µm).

Table 1

Relative density (RD) and mechanical properties of the investigated ZrO<sub>2</sub>-ZrB<sub>2</sub> composites presintered at 1400 °C followed by HIPing at 1400 °C for 1 h at 110 MPa argon pressure

Sample designation	mol% yttria in $ZrO_2$	Relative density (% $\rho_{\rm th}$ )	Vickers Hardness, Hv <sub>30</sub> (GPa)	Indentation toughness, <i>K</i> <sub>Ic30</sub> (MPa m <sup>1/2</sup> )
TM2B <sup>a</sup>	2 (mixed grade)	97.4	$16.1 \pm 0.5$	$17.8\pm0.3$
TM2.5B	2.5 (mixed grade)	100	$16.6 \pm 0.6$	$17.3 \pm 2.6$
T3B	3 (co-precipitated grade)	100	$16.2\pm0.5$	$7.1 \pm 1.5$

<sup>a</sup> B stands for 30 vol%ZrB<sub>2</sub>.

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