

## Abrasive wear in ceramic laminated composites

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Received 26 January 2005; received in revised form 14 July 2005; accepted 28 July 2005

Available online 29 August 2005

### Abstract

Laminated ceramic structures in the system  $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3 + 3\text{Y-TZP}$  (A/AZ) were prepared using a tape casting technique in order to obtain ceramic layers with different compositions and thicknesses. Piezo-spectroscopy was used to evaluate the residual stresses arisen from a calibrated mismatch in thermal expansion coefficients of the layers during the sintering process of the composite. The dependence of the residual stresses in the A and AZ layers on their thickness ratio was established. A microscale ball cratering method was used to investigate the influence that the surface compressive stress can play on the abrasive wear resistance of the composite structures. The results were compared with those obtained with an unstressed reference material prepared either by lamination of pure alumina green-sheets or by cold isostatic pressing of alumina powder. The experimental results have shown that the abrasive wear resistance is higher for samples with compressive residual stresses within the surface regions.

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**Keywords:** Laminated composites;  $\text{Al}_2\text{O}_3$ ; Abrasive wear; Residual stresses

### 1. Introduction

Due to high hardness, high modulus and chemical inertness, ceramic materials are good candidates for tribological applications. It has been shown that the toughness also plays an important role in improving the wear resistance [1]. Consequently, an increase in toughness can lead to an improvement in the tribological behaviour of ceramics. Phase transformations induced at the surface by thermochemical treatments have been explored with the aim to increase the surface toughness of 3Y-TZP [2,3]. In this way, high fracture toughness at the surface was obtained but a worse tribological behaviour was observed [4].

On the other hand, it has been shown that laminated hybrid structures constituted by alternate layers of different materi-

als can be properly designed in order to induce a surface compressive stress leading to an improved surface toughness [5–8] and wear resistance [9]. Residual stresses arise from a mismatch between the coefficients of thermal expansion (CTE), sintering rates and elastic constants of the constituent phases and neighbouring layers [10,11]. Compressive residual stresses are induced in layers with lower CTE, while tensile stresses arise in those with higher CTE. However, it is worth noting that the residual stress field also depends on the geometry of the layered structure and in particular on the thickness ratio among layers [12–16].

The effectiveness of laminated hybrid structures in improving the sliding wear resistance of alumina has been already reported by Toschi et al. [9]. The aim of this work is to verify the influence that a surface compressive stress and then an increase in toughness can play on the resistance to abrasive wear of tape cast  $\text{Al}_2\text{O}_3$  in laminated structures, by means of a comparison with the resistance of unstressed alumina. For this purpose, three laminated composites were produced with ceramic layers differing in the thickness ratio, together with

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layered structures made of pure alumina and monolithic samples produced by powder sintering. The residual stresses were measured using a piezo-spectroscopic technique applied to the chromophoric fluorescence of  $\text{Al}_2\text{O}_3$  [15,16], while the abrasive wear resistance was determined using a microscale ball cratering method [17,18].

## 2. Experimental procedures

### 2.1. Specimen preparation

Thin ceramic sheets were prepared by tape casting. Raw ceramic powders were  $\text{Al}_2\text{O}_3$  (Alcoa A16, Alcoa Aluminum Co., New York, USA) and  $\text{ZrO}_2$  (TZ3Y-S, Tosoh Corp., Japan), both with a mean particle size of  $0.3\text{ }\mu\text{m}$ . Sheets of pure alumina (hereinafter designated as A) as well as of the alumina–zirconia composite in the volume ratio 60/40 (hereinafter AZ) were produced. The CTE values are  $9.0 \times 10^{-6}\text{ K}^{-1}$  for alumina and  $10.0 \times 10^{-6}\text{ K}^{-1}$  for the alumina–zirconia composite [9]; this limited mismatch allows residual stresses to arise in the hybrid laminates in absence of microcracking or delamination effects [9].

The ceramic powders were ball mixed with solvents (ethanol and methyl-ethylketone), binders (polyvinylbutyral and dibutylphtalate) and a surfactant (triolein), to prepare slurries suitable for tape casting. The green tapes were dried and punched into  $34\text{ mm} \times 50\text{ mm}$  components of rectangular shape. The stacking sequence of the components in the composite was designed in order to obtain three kinds of laminates with different AZ/A thickness ratio.

The green-sheets were warm pressed at  $80^\circ\text{C}$  and at a pressure of 30 MPa for 30 min. Before sintering, an extremely careful burn out up to  $600^\circ\text{C}$  was carried out at low heating rate. Finally, the laminates were sintered at  $1550^\circ\text{C}$  for 1 h with heating and cooling rates of  $30^\circ\text{C/h}$ . In this way, hybrid laminates were produced with AZ/A thickness ratio equal to 1.12, 1.36 and 2.82 (S2, S3 and S4 samples, respectively). The composite S4 was prepared by stacking two AZ layers for each A layer. To obtain a symmetrical structure, two A layers were applied on each external side of the laminated structure. This also allowed one layer to be removed by grinding from each side of the sintered composite for a proper machining and surface finishing.

As unstressed reference materials both a laminated structure (S1) and monolithic alumina (S0) were used. S1 contained 11 layers of pure alumina and were prepared with the same procedure and sintering conditions used for the hybrid composites, while S0 was prepared by cold isostatic pressing at 300 MPa using the same alumina powder used in tape casting. Sintering was carried out at the same temperature used for laminated composites.

The density of the sintered samples was measured by the Archimede's method. Both surfaces and cross-sections of both laminates and reference samples were ground and polished up to  $1\text{ }\mu\text{m}$  diamond paste in order to obtain optically

flat surfaces, suitable for both abrasive wear tests and spectroscopic measurements.

### 2.2. Surface stress measurements

The stress distribution along cross-sections of the multilayered samples was determined by a piezo-spectroscopic technique related to the characteristic R1, R2 doublet produced by the chromophoric fluorescence of  $\text{Cr}^{3+}$  impurities in  $\text{Al}_2\text{O}_3$ . The method of relating a line shift in a fluorescence spectrum to the stress state has been described by Grabner [19]. In polycrystalline, fine grained and untextured  $\text{Al}_2\text{O}_3$  samples subjected to a normal stress  $\sigma$ , a luminescence line undergoes a change in frequency  $\Delta\nu$  given by the tensorial relationship:

$$\Delta\nu = \frac{1}{3} \Pi_{ii} \sigma_{jj} \quad (1)$$

where  $\Pi_{ii}$  (the trace of piezo-spectroscopic matrix) is the piezo-spectroscopic coefficient relating frequency to stress.

The spectrometric apparatus (T 64000 Horiba/Jovin-Yvon) used had a 400 mW argon-ion laser beam operating at a wavelength of 488 nm as the excitation source. An optical microscope lens was used both to focus the laser beam on the sample and to collect the scattered signal, and a micron-scale magnification was obtained. The scattered frequencies were analysed with a triple monochromator equipped with a charge coupled device camera. When focussed by means of the optical microscope using a  $20\times$  optical lens, the laser spot on the sample was  $5\text{ }\mu\text{m}$  in size. Thermal and instrumental fluctuations were compensated by monitoring the spectrum from a Hg/Ne discharge lamp. The recorded spectra were analysed with a commercial software (LabSpec 4.02, Horiba/Jobin-Ivon). The frequency shifts were obtained by subtracting the centre frequency of the peak, measured for the reference material in the unstressed state, from the centre frequency of the peak recorded under stress. A standard value of frequency for zero external stress was obtained acquiring an array of 100 spectra measured on the surface of sample S1 (produced by stacking only A layers) and averaging all the values of the peak centre.

An important characteristic of the piezo-spectroscopic technique is that the average uniaxial piezo-spectroscopic coefficient  $\Pi_{\text{uni}}$ , which characterises the linear dependence between peak shift and stress, strongly depends on several parameters which are specific of the material, and particularly on processing derived parameters such as grain size, presence of other phases, porosity, etc. Hence, a preliminar calibration procedure is required to determine the  $\Pi_{\text{uni}}$  value pertinent to each material. For this purpose, bending bars were obtained from laminated structures prepared with layers of the same composition (A and AZ, respectively), mounted on a 4-points bending jig under the laser beam focussed using the optical microscope and loaded below the fracture stress. After loading, the jig was moved under the microscope and spectra were recorded every  $40\text{ }\mu\text{m}$  on going from the side in compression

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