



## Short Communication

# Functionally graded ceramics by a new in situ processing route: Residual stress and wear resistance

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## Abstract

A new in situ method of producing ceramic functionally graded materials (FGMs) with continuously changing microstructure by pressureless sintering in combination with chemical reactions and evaporation is reported. Solid solutions of  $\text{Fe}_2\text{O}_3$  in  $\text{Al}_2\text{O}_3$  were first sintered in air and then reduction aged in 4 vol%  $\text{H}_2/\text{N}_2$ . Metallic Fe formed near the surface and was able to evaporate, producing a surface layer with graded composition. The thermal expansion mismatch led to compressive residual stresses in the surface region of  $\sim 500$  MPa and the graded structure avoided delamination. The abrasive wear resistance of the FGM ceramics was improved by a factor of 5 times compared with monolithic alumina. The improvement was attributed to the suppression of crack propagation normal to the surface by the compressive surface stresses.

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## 1. Introduction

Laminated ceramics have been widely used as a method to improve properties of monolithic ceramics such as flaw tolerance and wear resistance.<sup>1–3</sup> However, a major problem in ceramic laminates is that the high thermal residual stresses between the layers which are often responsible for the property improvements can also lead to failure by delamination and edge cracking, especially when the interlaminar bonding is weak.<sup>4</sup> Functionally graded materials (FGMs) have been developed to reduce the residual stress concentrations and enhance the bonding strength of laminates, by using a systematically varied microstructure and/or composition to produce gradual changes in mechanical and thermal properties across the geometry.<sup>5,6</sup> Several studies have been reported about ceramic FGMs, among which hot pressing has been widely used<sup>6,7</sup> because of the difficulty of co-sintering mixtures of powders with different composition owing to the mismatch in shrinkage rates.<sup>4</sup> Hot pressing is

expensive, however, and also limits the layered structure to planar geometries.

In this work, we investigated the residual stresses and wear resistance of new in situ ceramic FGMs with continuously changing microstructure developed by pressureless sintering in combination with chemical reactions and evaporation. This offers a more economical and versatile way of producing ceramic FGMs. This work is based on the precipitation in  $\text{Al}_2\text{O}_3$ –10 wt%  $\text{Fe}_2\text{O}_3$  solid solutions under reducing atmospheres when sintered pressurelessly at high temperatures which has been used previously by Mukhopadhyay and Todd<sup>8</sup> to produce ceramic composites. Although Mukhopadhyay and Todd<sup>8</sup> aimed to produce homogeneous bulk composites, the presence of a graded structure due to the different degrees of chemical reaction near the surface suggests the possibility of making FGMs through pressureless sintering using a related route.

## 2. Experimental methods

During the fabrication, high purity (99.99%), fine (particle size 200 nm)  $\alpha$ - $\text{Al}_2\text{O}_3$  powders (AKP-50, Sumitomo, Japan) were mixed with  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (purity >98%, Sigma Aldrich, UK) dissolved in ethanol to form slurries that would achieve

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Al<sub>2</sub>O<sub>3</sub>–10 wt% Fe<sub>2</sub>O<sub>3</sub> after calcination. 250 ppm (by weight) MgO powder was added to the slurries to prevent abnormal grain growth during sintering<sup>8</sup> and 850 ppm Y<sub>2</sub>O<sub>3</sub> in the form of Y(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O was added to limit the growth of large FeAl<sub>2</sub>O<sub>4</sub> particles at a later stage of the process.<sup>9</sup> The slurry was dispersed using an ultrasonic probe, and then ball milled for 24 h in polyethylene bottles using high purity (99.99%) alumina balls. After ball milling, the slurries were dried in a rotatory evaporator at a temperature of 60 °C in vacuum. The dried powders were heated at 10 °C/min up to 600 °C and held there for 1.5 h to decompose the Fe(NO<sub>3</sub>)<sub>3</sub> to Fe<sub>2</sub>O<sub>3</sub> completely. The calcined powder was then mixed with ethanol and ball milled for another 24 h. The final slurry was dried completely by rotatory evaporator under the same conditions as those used for the first drying step and then gently ground using an alumina mortar and pestle and passed through a 150 μm sieve. Green compacts were fabricated by cold isostatic pressing at a pressure of 150 MPa and sintered pressurelessly in air with an alumina tube furnace at 1450 °C for 5 h. The heat treatment was designed to achieve full dissolution and homogeneous distribution of Fe<sup>3+</sup> in the cation sub-lattice of Al<sub>2</sub>O<sub>3</sub> according to the Fe<sub>2</sub>O<sub>3</sub>–Al<sub>2</sub>O<sub>3</sub> phase diagram.<sup>10</sup> The second step in the heat treatment scheme (aging treatment) was carried out by annealing Al<sub>2</sub>O<sub>3</sub>–10 wt% Fe<sub>2</sub>O<sub>3</sub> solid solutions at 1350 °C for 20 h under 4 vol% H<sub>2</sub>/N<sub>2</sub>. The resultant specimen was 2.5 mm thick and is denoted the FGM sample in what follows. Discs of both pure alumina and solid solution were made for comparison with similar processing except for the omission of the aging step. The sintered densities of all samples were measured in distilled water according to Archimedes' principle, and were all above 98% of the theoretical density.

Back Scattered Electron (BSE, JEOL JSM 840A, Japan) images of a polished cross section were used to examine the microstructure of the FGM sample. Energy Dispersive X-Ray

Spectroscopy (EDX) attached to the SEM was used for elemental analysis.

The residual stresses were measured by piezospectroscopic microscopy using a Renishaw 1000 Series Raman microscope, which relates the R1 Cr<sup>3+</sup> fluorescence peak position to the sample stress.<sup>3,6</sup> Given the uncertainties involved, two distinct methods were used. In Method 1 a line scan (step size 50 μm) was made through a polished cross section of the specimen (scan 1 in Fig. 1). To provide a stress free reference, a second line scan was made on the bulk part of the same polished cross section but with the surface regions on both sides removed by grinding (scan 2 in Fig. 1). The two line scans were made on the same position in the bulk part. The shifts of R1 Cr<sup>3+</sup> fluorescence peak position between the two line scans were measured. The stresses along the lines scanned before removal of the surface regions should be approximately uniaxial with the stress direction lying in the polished cross-section and parallel to the surface regions. However, the surface regions must have approximately the same in-plane strain across the cut surface as in the bulk, where the stress should be biaxial. Thus the biaxial residual stress in the bulk part can be calculated using Eq. (1)

$$\sigma = \frac{3}{\Pi_H(1-\gamma)} \Delta\nu \quad (1)$$

where  $\sigma$  is the biaxial stress in the centre of the bulk,  $\Delta\nu$  is the frequency shift of the R1 peak compared with the stress-free sample,  $\gamma$  is Poisson's ratio and the hydrostatic piezospectroscopic coefficient  $\Pi_H$  is 7.59 cm<sup>-1</sup> GPa<sup>-1</sup> for texture-free polycrystalline alumina.<sup>11</sup>

The main problem with Method 1 is the uncertainty due to the stress relaxation at the cut surface. To give confidence in the stress levels, an alternative Method 2 was therefore used to measure the residual stress avoiding this stress relaxation issue. This method involved two steps (Fig. 1). In step 1, an FGM sample was polished on one large surface until the midpoint of the cross-section was reached. Cr<sup>3+</sup> fluorescence spectra were recorded from at least 100 points on the polished bottom surface of each sample. Spectra were taken from points at least 2 mm from the sample edges to avoid edge related effects. In step 2, the top surface layer was removed and the measurements on the polished bottom surface were repeated to provide a stress free reference. The shift of the average R1 Cr<sup>3+</sup> fluorescence peak position between the two steps was measured. The polished bottom surface should have a biaxial stress given by:<sup>11</sup>

$$\sigma_r = \frac{3}{2\Pi_H} \Delta\nu \quad (2)$$

The hardnesses and wear resistances and of pure alumina, the solid solution and the FGM sample were compared. Vickers hardness indentations (AVK-C1/C2) were made on the lightly polished surfaces (the thickness removed was around 5 μm) with indentation loads ( $P$ ) 5 kg applied for 15 s to evaluate the hardness (Hv) of all three samples. A micro-scale abrasive wear tester (TE 66, Phoenix Tribology, UK) was used to measure the wear resistance of all samples. Samples were loaded by a pre-conditioned chrome steel ball (25 mm in diameter) with a dead weight of 5 N. The abrasive slurry consisted of silicon carbide

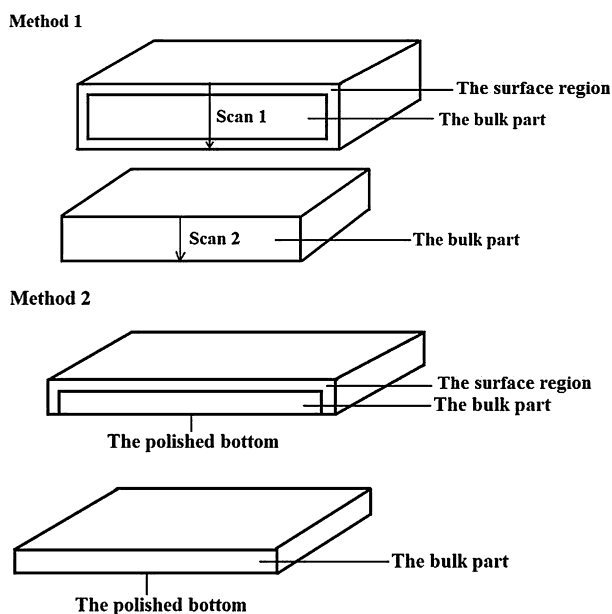


Fig. 1. The geometry of the cross sections and the position of fluorescence measurements for (top) Method 1; (bottom) Method 2.

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