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High resolution optical microprobe investigation of surface grinding stresses in Al_2O_3 and Al_2O_3/SiC nanocomposites

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Abstract

It has previously been suggested that Al_2O_3/SiC nanocomposites develop higher surface residual stresses than Al_2O_3 on grinding and polishing. In this work, high spatial resolution measurements of residual stresses in ground surfaces of alumina and nanocomposites were made by Cr^{3+} fluorescence microspectroscopy. The residual stresses from grinding were highly inhomogeneous in alumina and 2 vol.% SiC nanocomposites, with stresses ranging from ~ -2 GPa within the plastically deformed surface layers to $\sim +0.8$ GPa in the material beneath them. Out of plane tensile stresses were also present. The stresses were much more uniform in 5 and 10 vol% SiC nanocomposites; no significant tensile stresses were present and the compressive stresses in the surface were ~ -2.7 GPa. The depth and extent of plastic deformation were similar in all the materials (depth $\sim$ 0.7–0.85 μm); the greater uniformity and compressive stress in the nanocomposites with 5 and 10 vol% SiC was primarily a consequence of the lack of surface fracture and pullout during grinding. The results help to explain the improved strength and resistance to severe wear of the nanocomposites.

Keywords: Al_2O_3; Nanocomposites; Residual stresses; Spectroscopy; Fluorescence microscopy

1. Introduction

Al_2O_3/SiC nanocomposites combine polycrystalline alumina and small amounts of sub-micron SiC particles [1–3]. The typical microstructure of Al_2O_3/SiC nanocomposites is composed of a polycrystalline matrix with an average size of 1–5 μm and SiC particles with size ranging from 100 to 200 nm. The addition of a small amount of sub-micron sized SiC to the alumina matrix can significantly improve the surface finish after machining, the resistance to severe wear, and the strength [1–11]. The nanocomposites have better surface finish and wear resistance both because the mean size of the individual pieces of material removed by brittle fracture at the surface is reduced and because the initiation of fracture is itself suppressed by the SiC additions [12,13]. The strengthening mechanism of nanocomposites, however, is still controversial and a number of possible mechanisms have been proposed. One obvious explanation is simply the improved surface finish and reduction in cracking during specimen preparation mentioned above. Another related suggestion is that the compressive surface residual stress after machining is increased [9,14,15]. In this work, the grinding induced surface residual stresses in Al_2O_3 and Al_2O_3/SiC nanocomposites are measured and compared, in order to investigate the validity of the proposed residual stress strengthening mechanism.

Previously, grinding induced surface residual stresses in Al_2O_3 and Al_2O_3/SiC materials have been measured by X-ray diffraction [14,16,17], curvature measurement [15] and Hertzian indentation [18,19]. The disadvantage of these techniques is that they all have poor spatial resolution compared with the scale of the microstructure [20] and as a result the measured stress is volume averaged rather than reflecting the local stress at the surface and its spatial distribution. Furthermore, the mean stress deduced depends on estimating a thickness for the compressive surface layer and often there is little information about what value this should take.

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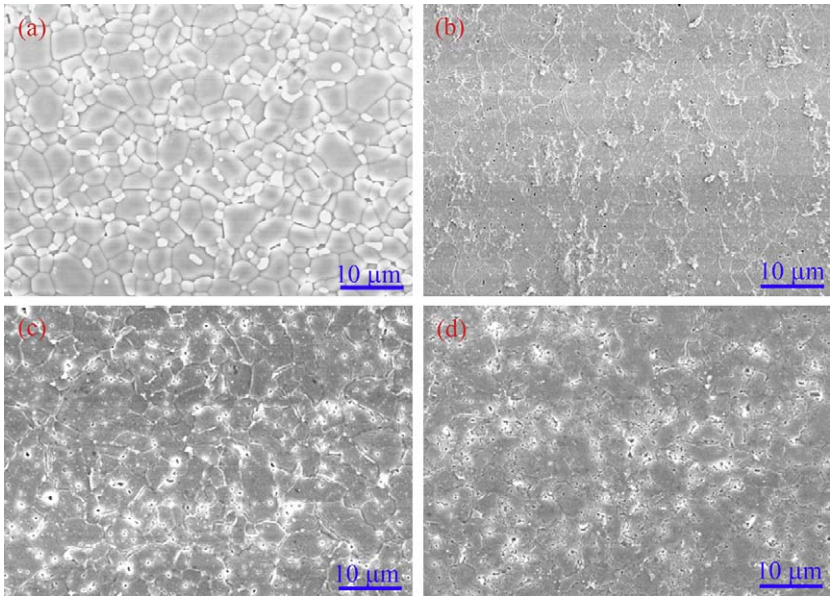

Fig. 1. Microstructure of the Al_2O_3 and Al_2O_3/SiC nanocomposites used in this work. (a) Al_2O_3, (b) 2 vol.% SiC, (c) 5 vol.% SiC and (d) 10 vol.% SiC. The specimens were thermally etched at 50 °C below the sintering temperature for 30 min in vacuum to reveal the grain boundaries.

To probe the local stress variation in the ground surfaces more directly, a higher spatial resolution technique is required. In this work, confocal Cr^{3+} fluorescence microscopy was used, with lateral and axial (depth) resolutions of ~1.5 μm and ~3 μm, respectively [21,22]. Previous work on alumina based materials using Cr^{3+} fluorescence microscopy investigated only residual stresses induced by indentation or scratching [23,24]; in addition, it used weakly confocal microscopes with depth resolution of ~10 μm. From both TEM observations [18] and results in our previous work [25], it is known that grinding stresses are expected to be found at depths of ~1 μm for monolithic alumina. Considering the translucency of alumina materials, therefore, the conclusion in Ref. [24] that the residual stresses around indentations and scratches in alumina were lower than in alumina/SiC nanocomposites may be an artefact of lower transparency in the nanocomposites, which would confine the sampled volume more closely to the stressed region.

The confocal microscope used in this work alleviates this problem but does not entirely remove it because the axial resolution is still not sufficient to make simple point measurements of surface stress. The experimentally measured stress is actually the convolution of the real stress with the axial probe response function (PRF) [26] which describes the relative collection efficiency as a function of depth and depends on the instrument and the translucency of the material. In our previous work, on ground surfaces of alumina, residual stress distributions were estimated by modelling the plastic displacement of material resulting from grinding as an array of continuously distributed edge dislocations [21,25], and established the PRF of our instrument when used with Al_2O_3 and Al_2O_3/SiC [21,22]. The convolution of the fluorescence response predicted by the model with the PRF allowed the local residual stress variation for polycrystalline alumina after grinding and polishing to be estimated by adjusting the physical parameters in the model to fit the experimental results. In the current work, the same method will be used to compare the local stress distributions in surface ground monolithic Al_2O_3 and Al_2O_3/SiC nanocomposites.

2. Experimental

2.1. Materials and specimen preparation

The starting powders were AKP50 alumina (200 nm, Sumitomo, Japan, 99.995% purity) and UF45 SiC (260 nm, Lonza, Germany, contains 0.2% free Si, 0.6% free C and 3.5% oxygen) respectively. 0.25 wt% MgO was added to all materials to prevent abnormal grain growth. Mechanical mixing by attrition milling (Szegvari HD, USA) using yttria stabilized zirconia milling media was performed at a speed of 300 rpm for 2 h. The ratio of water to powder was 4:1 by volume and 2.1 wt.% of Dispex A40 (Allied Colloids, UK) was used as a dispersant. The mixture was freeze dried (Edwards Micromodulyo, UK) for 24 h. The powder was passed through a 150 μm sieve and then calcined at 600 °C for 1 h. Hot pressing was used to produce dense specimens. A pressure of 25 MPa was applied for 30 min in an argon atmosphere with a graphite die at maximum temperatures between 1550 and 1700 °C, to give materials of similar grain size (5–6 μm (Fig. 1), measured by the conventional liner intercept method [27]). Three Al_2O_3/x vol.% SiC nanocomposite (x = 2, 5, 10) specimens were used in this work, and a monolithic alumina specimen was used as a comparison. The grain sizes of the materials are given in Table 1.

2.2. Grinding

The procedure for grinding of the specimens followed our previous work [25]. Specimens were sequentially polished down to a 1 μm diamond finish first to start from smooth surfaces, and they were then ground on a resin bonded alumina wheel for