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Residual stress distributions around indentations and scratches in polycrystalline Al_2O_3 and Al_2O_3/SiC nanocomposites measured using fluorescence probes

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Abstract

We report a study of the residual stress state around indentations and single-point scratches in polycrystalline alumina and alumina/SiC nanocomposites using Cr^{3+} fluorescence piezospectroscopy. The alumina specimens displayed residual stress levels up to 550 MPa, whereas the nanocomposite specimens had maximum stress levels close to 2 GPa. These stress levels are consistent with those obtained using other experimental techniques. The spatial variation of this stress is shown to be consistent with simple elastic/plastic models of indentation. The broadening of the peaks in the fluorescence spectra is used to estimate the density of dislocations in the plastically deformed region below indentations and scratches. Our results indicate a greater depth of deformation around indents and scratches in the nanocomposites when compared with the alumina surfaces. The inferred dislocation densities and the depth of the deformed region beneath the alumina and nanocomposite surfaces are shown to be consistent with those of ground surfaces reported in earlier studies.

Keywords: Raman spectroscopy; Ceramics; Microindentation; Plastic deformation; Residual stresses

1. Introduction

Alumina/silicon carbide nanocomposites (alumina polycrystals containing small fractions of sub-micron-sized SiC particles) can show large increments of strength compared to polycrystalline alumina samples with equivalent grain size; any increase in strength is not accompanied by a significant increase in material toughness [1–4]. A number of workers have proposed that the surface properties of alumina/SiC nanocomposites – in particular the near-surface in-plane compressive stresses introduced by surface finishing operations – are the key to their enhanced strength.

Recent work has found intriguing differences between the surface behaviour of alumina polycrystals and alumina/SiC nanocomposites. Sternitzke et al. [5] first reported a quantitative difference in the sub-surface damage between alumina and alumina/SiC nanocomposites after grinding and polishing. Other studies also reported enhanced strength in these nanocomposites after surface grinding [1] and a much superior polished surface with suppressed grain pull-out [6,7]. Wu et al. showed [8] that grinding the surface of alumina/SiC nanocomposites with a coarse diamond grit wheel resulted in a four-point bend strength of ~400 MPa, whereas similarly ground alumina specimens had a strength of ~300 MPa. The enhanced strength of the nanocomposites was shown to be associated with a large compressive surface stress induced by surface grinding, while such a magnitude of residual stress was not present in the alumina after identical surface treatment. A more detailed study of the surface stress state, and the sub-surface dislocation structure after deformation, found

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that the nanocomposite material showed a significantly greater dislocation density beneath ground and polished surfaces, compared to identically treated polycrystalline alumina [9–11].

In order to investigate further the influence of surface grinding on the mechanical behaviour of alumina and alumina/SiC nanocomposites, we have undertaken a study of the deformation around single-point indentations and simple scratch grooves. Such surface deformation can be taken as a model for the processes occurring around individual grit particles during grinding. The mechanisms of deformation around indentations in ceramics and brittle materials have been the subject of considerable research in the past. It is now generally accepted that even in highly brittle materials there is a region of plastic deformation immediately below the indentation [12]; this is shown schematically in Fig. 1. During indentation an elastic stress field is generated, of radial compression and tangential tension, extending into the material outside the plastic zone, the magnitude of which decays with distance, r, from the centre of the plastic zone as $1/r^2$ [13,14]. The tensile components of this stress field can lead to the nucleation of radial and median cracks on loading. The magnitude of the residual elastic stress field after unloading varies as $1/r^3$ from the contact point [14,15] (though it may be influenced by the presence of the crack systems illustrated in Fig. 1). The residual stress field close to the surface has a tension component normal to the surface, which may lead to the nucleation of lateral cracks. For a sliding indentation, there is as yet no model that can completely describe the elastic/plastic stress fields under the indenter. An extension of Yoffe's model of static indentation to sliding contacts has been reported [16], though further validation is needed.

Conventional methods of residual stress measurement, e.g. X-ray diffraction or surface curvature changes, measure the residual stress averaged over a considerable region of material. In order to obtain a high-resolution local picture of the residual stress state on the surface close to scratches and indentations, we have used the Cr^{3+} fluorescence spectra obtained from natural concentrations of Cr impurities within alumina crystals. Grabner [17] first described the relationship found between the measured line shift in Cr^{3+} fluorescence and the local stress state. Ma and Clarke developed this analysis for a more general case to relate the mean line shift to the local stress tensor [18]. They also presented an analysis of the broadening of the fluorescence peak to determine the mean stress distribution in an illuminated area. Thus, by using an optical microscope of sufficient resolution fitted with an appropriate spectrometer, it is possible to determine the surface stress within alumina specimens with a lateral spatial resolution of about 1 μm and over a sampled depth slightly greater than this [19].

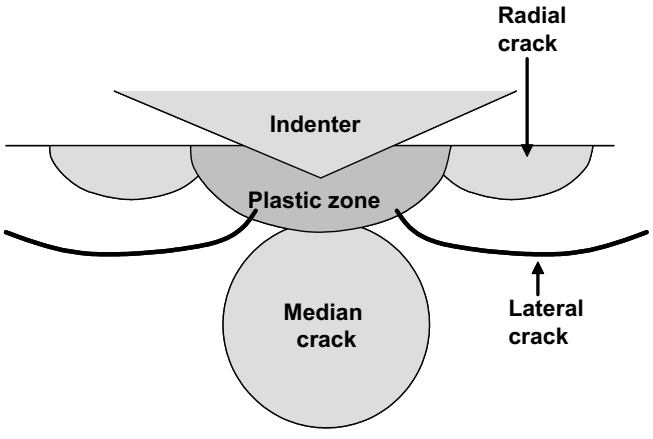

Fig. 1. Schematic showing the plastic zone beneath the indenter and the crack systems observed after unloading.

2. Experimental procedure

The materials used in this study consist of a polycrystalline alumina (AKP53, Sumitomo Chemical Co., Tokyo, Japan) of mean grain size about 3 μm and an alumina/SiC nanocomposite with a matrix of the AKP53 alumina containing 1, 5 and 10 vol.% α-SiC particles (UF 45, Lonza, Waldshut, Germany), with a mean particle diameter of 90 nm. Processing conditions were chosen to ensure that the nanocomposite and polycrystalline alumina had equivalent mean grain size. The processing routes used have been described in detail elsewhere [3,8]. All nanocomposite samples were hot pressed at 1650 °C for 1 h under a pressure of 20 MPa in argon. The alumina samples were hot pressed at 1500 °C for 1 h under the same pressure. All nanocomposites and alumina ceramics achieved full density after fabrication.

The hot pressed ceramic discs, with a thickness of about 5 mm, were ground with an epoxy resin bonded diamond wheel (grit size 150 μm) to remove the top surface on both sides, producing a final specimen thickness of about 3 mm. Specimens for indentation and scratch experiments were cut directly from the ground discs with a diamond saw, and the ground surfaces were lapped and polished using a series of diamond grits of 25, 8, 3 and 1 μm grit size. Each step of the polishing sequence was performed for sufficient time to eliminate all surface damage induced by the previous polishing step. Full details of this surface finishing process have been presented elsewhere [8,9].

2.1. Single-point indentation and scratch tests

All indentation and scratch tests were carried out on surfaces polished to a 1 μm diamond finish. A microindenter (Matsuzawa MHT1, Tokyo, Japan) was used to produce indentations on the surface of the alumina and alumina/SiC nanocomposites using a square pyramid diamond with tip included angle of 136° (Vickers profile). Indentation loads in the range of 0.5–10 N were used with a holding time of 15 s at maximum load for each indentation experiment.