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Quantitative optical fluorescence microprobe measurements of stresses around indentations in Al_2O_3 and Al_2O_3/SiC nanocomposites: The influence of depth resolution and specimen translucency

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Abstract

Residual stresses around 1 kg Vickers indentations in Al_2O_3 and Al_2O_3/SiC nanocomposites were measured using high-resolution Cr^{3+} fluorescence microscopy. Experiments and modelling showed that the use of non-confocal microscopes can lead to significant underestimation of the surface stress in Al_2O_3 because of the sampling of subsurface regions where the stresses are lower. The nanocomposites were less sensitive to the depth resolution of the microscope because their strong absorption limited the depth from which fluorescent radiation was collected. The use of confocal microscope settings allowed accurate measurements to be made and the indentation stresses were found to be very similar in Al_2O_3 and the Al_2O_3/SiC nanocomposites. The stresses measured were significantly different from the predictions of the Yoffe model for indentation stresses. This was because of indentation cracking, which is not accounted for in the model. Cracking was also considered to be important in determining the plastic zone size in ceramics, which is much smaller relative to the indentation size than in metals.

Keywords: Crystalline oxides; Nanocomposite; Residual stresses; Microindentation; Fracture

1. Introduction

The indentation of ceramics by sharp indenters leads to residual stresses in and around the impression left after removal of the indenter. It is important to develop a quantitative understanding of these stresses. One reason is that the commonly used indentation cracking method of toughness measurement [1–3] relies on a rigorous understanding of these residual stresses because they are responsible for the extension of the indentation cracks, the lengths of which are used to calculate the toughness. As Quinn and Bradt [4] have pointed out, however, the equations used at present are based on expressions for the stresses which have no experimental verification, are based on unrealistic assumptions and contain unknown factors which are supplied by calibration. Progress in making this method more reliable therefore requires better methods of measuring the residual stresses.

A second reason for developing a quantitative understanding of stresses around indentations, and the related single-scratch test, is that these tests can serve as simplified representations of wear, grinding and polishing suitable for the laboratory investigation of the mechanisms involved. The residual stresses drive near-surface cracks that can lead to material removal in these processes. A recent example of such an investigation is provided by the work of Wu et al. [5], who studied indentations and scratches in Al_2O_3 and Al_2O_3/SiC nanocomposites in order to further the understanding of the improved surface finish and wear resistance of the nanocomposites compared with Al_2O_3 [6–11]. The

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investigation centred on residual stress measurements by Cr^{3+} fluorescence microscopy. Their results are discussed below, after a short review of this technique.

In principle, Cr^{3+} fluorescence microscopy [12,13] is an ideal technique to measure the residual stresses induced by indentation in Al_2O_3 ceramics due to its superior spatial resolution (lateral resolution ~2 μm) [14,15] and experimental simplicity compared to diffraction methods [16,17]. There is a problem with the use of this method with translucent ceramics such as Al_2O_3, however, because some of the fluorescent radiation sampled comes from beneath the surface of the specimen [14,18,19] rather than just from the surface as is sometimes assumed. This effect also degrades the lateral resolution. In previous work using Cr^{3+} fluorescence microscopy to measure indentation-induced stresses in Al_2O_3-based materials [5,20], non-confocal microscopes were used with axial (depth) resolution ~10 μm. This is similar in scale to the dimensions of a 1 kg indentation in Al_2O_3 so the rapidly varying stress distribution around the indentation cannot be studied quantitatively by direct measurements using such instruments. If this effect is not taken into account, measurements should be regarded as semiquantitative at best.

This is also a concern for the comparative measurements of indentation-induced stresses in Al_2O_3 and Al_2O_3/SiC nanocomposites made by Wu et al. using this technique [5] as Al_2O_3 is much more translucent than Al_2O_3/SiC nanocomposites [14]. Wu et al. reported that raw measurements of spectral peak positions close to indentations and scratches in Al_2O_3/SiC nanocomposites show significantly greater stress-induced peak shifts than those in Al_2O_3 [5]. The interpretation of this in Ref. [5] is that these ceramics may show genuinely different responses to indentation and scratching that lead to differences in the residual stress fields produced. An alternative interpretation, however, is that much of the sampled volume in the more translucent Al_2O_3 came from further beneath the indentations and scratches, where the stresses are expected to be smaller, and that the apparent difference in behaviour is therefore at least partly an artefact of the technique rather than a real effect.

This paper describes the development of a method enabling fully quantitative comparison of Cr^{3+} fluorescence microscopy measurements with models for indentation stresses and of one material with another. An experimental improvement in this work compared with Refs. [5,20] is the use of a confocal microscope with axial resolution of ~3 μm and lateral resolution of ~1.5 μm. However, even this high-resolution is not necessarily sufficient to make simple point measurements of surface stresses. The measured stress is still the convolution of the actual, spatially distributed stresses with the probe response function (PRF) [14,19,21,22] describing the relative collection efficiency as a function of depth. The PRF depends in turn on the instrument and the translucency of the material. In order to investigate the importance of depth resolution, the residual stresses around 1 kg Vickers indentations in Al_2O_3 and Al_2O_3/x vol.% SiC ($x = 2, 5, 10$) nanocomposites were measured in this study with the microscope in both confocal and non-confocal modes. The experimental results were compared with the well-known analytical model for indentation stresses of Yoffe [23], by convolution of the stress-induced spectral peak shifts predicted from the theoretical stress field with the previously developed PRF for our instrument in combination with the materials used [14,24]. The results are also used to make a quantitative comparison of the indentation stresses in Al_2O_3 with those in Al_2O_3/SiC nanocomposites.

2. Experimental

2.1. Materials and specimen preparation

The starting powders were AKP50 Al_2O_3 (200 nm, Sumitomo, Japan, 99.995% purity) and UF45 SiC (260 nm, Lonza, Germany, containing 0.2% free Si, 0.6% free C and 3.5% oxygen). 0.25 wt.% MgO was added to prevent abnormal grain growth. The powders were attrition milled (Szegvari HD, USA) using yttria-stabilized zirconia milling media at a speed of 300 rpm for 2 h. The ratio of water to powder was 4:1 by volume, and 2.1 wt.% (relative to the dry powder) 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 in an argon atmosphere with a dwell time of 30 min at maximum temperatures between 1550 and 1700 °C, chosen to give materials of similar grain size (5–6 μm, measured by the conventional linear intercept method [25]). Three Al_2O_3/x vol.% SiC nanocomposite specimens ($x = 2, 5, 10$) and one monolithic Al_2O_3 specimen were used in this work.

2.2. Indentation

One kilogram load indentations were made on 1 μm diamond polished surfaces of Al_2O_3 and Al_2O_3/SiC nanocomposites with 10 s loading time using a Vickers hardness tester (Mitutoyo, UK). Several indentations were made on each specimen and the line scan results (see below) across different indentations on each specimen were quite similar [26], with peak shifts showing a scatter $<1\ cm^{-1}$ close to the indentations. The line results presented in this work were therefore from one typical indentation on each individual specimen (Fig. 1). The hardness and indentation crack lengths of the Al_2O_3 and the nanocomposites were similar, with values of ~20 GPa and ~45 μm (measured from the indentation centre), respectively.

2.3. Cr^{3+} fluorescence measurements

The principle of Cr^{3+} fluorescence microscopy is to measure the stress-induced shifts of the characteristic R1 and