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Piezospectroscopic measurement of the stress field around an indentation crack tip in ruby using SEM cathodoluminescence

R.I. Todd [a,*], D. Stowe [a,b], S. Galloway [b], D. Barnes [a], P.R. Wilshaw [a]

[a] *University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH, UK*
[b] *Gatan UK, 25 Nuffield Way, Abingdon, Oxon OX14 1RL, UK*

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Abstract

Piezospectroscopy using stress-sensitive lines in the cathodoluminescence (CL) spectrum generated in response to excitation by the electron beam of an SEM has recently been shown to be a promising technique for submicron resolution stress measurements in alumina and other ceramics. This paper develops and applies the technique by mapping the wavelength shifts of the R_1 CL line around the tip of an indentation crack in a ruby single crystal. Accounting for crystallographic anisotropy, the shifts observed were quantitatively consistent with the classical crack tip stress field for all polar angles ahead of the crack tip and indicated a crack tip stress intensity factor of 1.0 MPa m$^{1/2}$. This is significantly lower than the fracture toughness of the crack plane (4.5 MPa m$^{1/2}$), and indicates the post-indentation development of lateral cracks and slow crack growth. The spatial resolution of the stress measurements was measured as 550 nm and the effects of specimen heating by the electron beam were shown to be negligible.

Keywords: Spectroscopy; Fracture; Toughness and Toughening; Al_2O_3; Cathodoluminescence

1. Introduction

Ceramic components often contain residual stresses. These have many sources, including plastic deformation due to indentation or abrasion, phase transformations and thermal expansion mismatches. The residual stresses may be beneficial or detrimental to the mechanical properties. Either way, it is important for the stresses to be investigated and understood.

This task is made difficult by the fine scale of many sources of stress and the lack of measurement techniques with sufficiently high spatial resolution that are suitable for bulk specimens. One technique that has been applied successfully to a wide range of ceramics and glasses is optical microprobe piezospectroscopy (OMPS).[1,2] In this technique a laser is focused to a small spot through the objective lens of an optical microscope. Any photoluminescence produced in response to this excitation is collected by the same lens and analysed using a spectrometer. Lines in the luminescence spectrum can be produced by several phenomena and their wavelength is often sensitive to stress. By suitable calibration this method can therefore be used for the measurement of the stresses within the sampling volume.

The spatial resolution of OMPS is restricted to ~2 μm by the diffraction-limited optics of the microscope used. Clearly, this could be improved if either the luminescence generation volume or the collection volume were released from this constraint. A practical way of achieving this is to retain the diffraction-limited collection optics, but to excite cathodoluminescence (CL) using the potentially much smaller generation volume of a focused electron beam in an SEM.

SEM-CL is widely used to investigate the properties of semiconductors and related materials and there are many reports of the sensitivity of CL wavelength to stress in such studies (e.g. see Refs. [3,4]). In many cases this is peripheral to the aims of the investigation but Rudloff et al.[5] have presented a detailed investigation of the stresses in microcracked $Al_xGa_{1-x}N$ films. Maps of the stress-induced shift in luminescence wavelength were obtained using computer control of the SEM electron beam and compared favourably with the predictions of a FE model for the films.

* Corresponding author. Fax: +44 1865 273783.
E-mail address: richard.todd@materials.ox.ac.uk (R.I. Todd).

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It is also possible to make SEM-CL stress measurements on metal oxides. Pezzotti et al. have reported SEM-CL stress measurements from several glass compositions [6] with accelerating voltages as low as 1 kV, which leads to a potentially very small luminescence generation volume and correspondingly improved spatial resolution. Stress maps based on measured wavelength shifts near imperfections in clad optical fibres are presented.

The ability to make high resolution stress measurements on the surface of bulk specimens of structural ceramics, such as alumina, would be very useful. The first systematic attempt to measure stresses in alumina using SEM-CL was by Ostertag et al. in 1991[7] who measured the stresses around a hardness indentation in a single crystal using the shift in the luminescence associated with Cr impurities, the so-called ruby R_1 and R_2 lines. The accelerating voltage used was 20 kV resulting in a spatial resolution of around 5 μm, i.e. slightly worse than the OMPS technique described above. A further problem was in obtaining sufficient signal, and noise in the spectrum led to considerable uncertainty in the stress measurements. Despite these limitations, systematic variations in stress around the indentations were convincingly observed.

Recently, Pezzotti et al.[8] have shown that the ruby R_1 and R_2 lines can be collected using the lower accelerating voltages required for high spatial resolution measurements. The R line wavelength shifts directly ahead of an indentation crack were measured and used in conjunction with the crack tip stress intensity, measured independently, to estimate the relationship between the line shift and stress. A hydrostatic stress map around the indentation is presented based on this calibration.

The work of Pezzotti et al.[8] is encouraging, but raises several issues needing clarification. Firstly, the plot of R_1 line shift against $1/\sqrt{r}$ presented, where r is distance ahead of the crack tip, does not extrapolate to the origin and therefore does not have the simple proportionality expected from the classical crack tip stress field. Secondly, the stress mapping should be seen as semi-quantitative, both because of the above uncertainty over the form of the stress field used in the calibration and because this calibration procedure does not account for the considerable crystallographic anisotropy of the piezospectroscopic coefficients[9]. Finally, the resolution of the technique is not measured, although the wavelength variation behind the crack tip in the plot of line shift against r presented suggests a resolution ∼1 μm.

This paper investigates these issues by mapping the stress-induced shifts of the R lines in the CL spectrum around the tip of an indentation crack in single crystal ruby taking into account the piezospectroscopic anisotropy of the crystal. This provides a comprehensive comparison between the line shifts and the classical stress field in all directions from the crack tip, not just the straight ahead direction. The crack tip singularity provides a convenient method of quantifying the resolution of the experimental technique as used in the present work.

2. Experimental

All measurements were carried out on a 0.5 mm thick $\{11\bar{2}0\}$ (a-plane) slice of a ruby single crystal containing 0.05–0.1% Cr (Rubicon Technology, IL, USA, manufacturer's data for Cr content). One surface of the crystal was polished using 0.25 μm diamond paste and then annealed in air for 1 h at 1 550 °C to remove surface residual stresses. Vickers hardness indentations were made on the surface of the ruby using a load of 1 kg. The indentations were orientated so that the diagonals were parallel to the m and c axes of the crystal. The indented specimen spent approximately 20 h stored under vacuum and 2 h open to the atmosphere prior to CL spectra being taken.

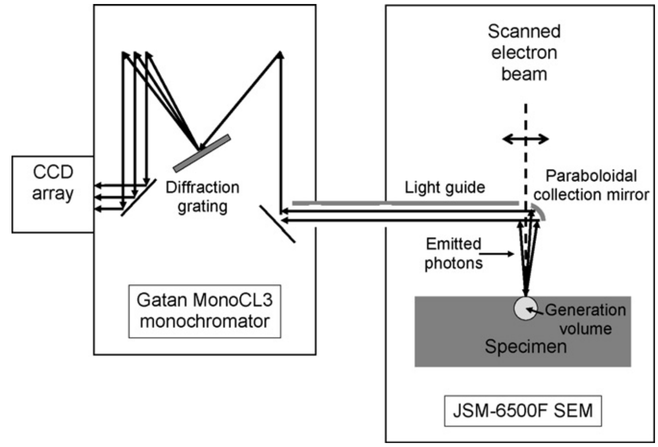

Fig. 1. Schematic of the CL system showing light paths.

The specimen was coated with a layer of carbon to minimise charging. The coating was sufficiently thin to allow most of the CL emission to pass through. Indentation cracks were examined at room temperature in a JSM-6500F SEM equipped with a Gatan XiCL*one* system. The experimental setup is shown schematically in Fig. 1. The CL emissions in response to the electron beam are collected by a paraboloidal mirror which, in conjunction with a system of lenses and mirrors, transmits the light collected to a diffraction grating. The diffracted spectrum is collected directly using a Princeton Instruments Spec-10 CCD camera. The system includes digital beam control software which enables the automated collection of a spectrum for each pixel within a user-defined region of interest, thus creating a three-dimensional data set with spectral information as a function of position (spectrum imaging).

Maps of the CL spectrum were collected around indentation crack tips with an accelerating voltage of 10 kV and absorbed specimen currents of ∼1 nA using a pixel size of 0.22 μm × 0.22 μm. Several crack tips were mapped in this way, but the detailed analysis presented here is confined to results from the tip of a well developed indentation radial crack on the basal plane of the ruby and intersecting the surface along the m-axis of the crystal.

Fig. 2 shows a typical R line doublet collected using this system. Only the stronger R_1 line was used in the present work and its wavelength was defined for each pixel by fitting a Gaussian profile to the central portion of the line.