3D tomographic analysis of crack morphologies in alumina and glass using FIB microscopy

F A Elfallagh and B J Inkson

Department of Engineering Materials, The University of Sheffield, Mappin Street, Sheffield, S1 3JD, UK

E-mail: f.elfallagh@sheffield.ac.uk

Abstract. 3D Focused Ion Beam tomography is increasingly being used for 3D characterisation of microstructures in the 50 nm –20 μm range. Here FIB tomography has been used to study crack morphologies under Vickers microindentations in R-cut alumina and in soda-lime-silicate glass samples. 3D tomographic reconstruction of crack distribution around 100g microindentation sites shows that the crack density in alumina is very much greater than glass. In addition the cracks observed are influenced by the location of the FIB milled surface trenches due to localised stress changes.

1.Introduction

3D characterization of microstructure provides quantitative information about the properties of the material which in turn helps to predict the behaviour of the material in certain environment, i.e. characterization of grains, pore size and crack morphology provides good information about the properties and behaviour of the material [1]. Microindentation of brittle materials generates a significant number of cracks which nucleate and propagate to relieve the high local stresses generated during indentation process. 3D FIB tomography can be used to examine the 3D crack distribution in samples, and is especially useful for the analysis of very localised damage such as from indents and scratches [2,3,4]. The system of cracks formed under Vickers microindentation differs from one material to another depending on the mechanical and crystallographic properties of the material. A wide range of cracks can be generated in the surface of the brittle material by indentation but the there are major crack types (ex. radials, median and lateral) which may be shared by most of these materials depending on the load and process of indentation[5]. Alumina is expected to show higher density of cracks than glass for the same load since it has low value of fracture hardness. In this work 3D crack maps are constructed for alumina and soda-lime-silicate glass at the same load 100g and compared in density and morphology in relation to crystallography of the material.

2. Experimental procedures

The samples used for these studies were (a) an optically polished single crystal sapphire wafer 0.55 mm thick with surface orientation (1 1 0 2) and (b) standard soda-lime-silicate glass 0.5 mm thick. Single crystal alumina was chosen to avoid any influence of local grain crystallography and grain boundaries on the crack analyses. A microhardness tester (Mitutoyo) was used to indent the (1 1 0 2) alumina and glass sample using a diamond Vickers indenter tip loaded to 100g, keeping a constant tip and sample orientation.
Samples were imaged and processed using a JSM 6500F SEM fitted with an Orsay Physics FIB column. The FIB-SEM samples were pre-coated with < 20 nm of carbon to prevent charging and protect from ion beam damage.

FIB trenches through indentation sites, in alumina and glass samples, were sputtered using 30kV Ga⁺ ions and beam currents of 50 pA-200 pA. For 3D FIB tomography, the gallium focused ion beam was used at normal incidence to locally cross-section the indentation sites, and SEM secondary electron (SE) images were taken of each sequential x-y 2D slice at a tilt angle of 55°. The dimensions of the 3D volumes analysed (for the 100g indent site on alumina) were approximately $x = 20 \, \mu m$, $y = 15 \, \mu m$, $z = 10 \, \mu m$, total volume $\sim 3000 \, \mu m^3$. And for the 100g indent site on glass was $x = 25 \, \mu m$, $y = 22 \, \mu m$, $z = 8 \, \mu m$, total volume $\sim 4400 \, \mu m^3$. To analyse these 3D volumes in a reasonable time, a microscope magnification of 3700–5000 x was used, and the separation, z, of the 2D FIB sections was $\sim 0.25 \, \mu m$ for the alumina and $0.8 \, \mu m$ for the glass. The 2D SE images were aligned and corrected for drift using cross-correlation of fiducial markers milled into the sample surfaces [4,6,7]. Cracks were identified and traced in each 2D section, and a mesh was generated to interpolate between sequential sections and display the analysed 3D volume using IMOD software [8,9].

3. Microindentation of alumina and glass

The microindentation tests of (1 0 2) orientation alumina gave a Vickers Hardness of $HV = 20.5 \, GPa \pm 2 \, %$ averaged over four indents, which is within the expected range e.g. the reported value of 20.8 GPa for (2 1 0) sapphire [10]. The residual damage at the Vickers indentation sites, 100g loaded with identical diamond tip and crystal orientation, was imaged by SEM (no coating). Fig. 1(a) shows the two types of cracks visible on the surface around these indentation sites (i) radial cracks, and (ii) circumferential cracks. The maximum length of the radial cracks was 12 $\mu m$ from the corners of the residual indent impression. In brittle materials radial cracks typically initiate near the edges of the Vickers indenter tips where there is the highest stress concentration [5,10]. The radial cracks in this study were not located exactly on the corners of the residual indent impression. This offset can be due to the local crystallography of the sample [5,11]. Some linear surface traces, probably twins or slip bands, were also observed around the residual indent impression (Fig.1a, [10,12]. The surface...
observations of the indentations are consistent with those previously reported in other studies of microindentation of alumina [10-12].

On the other hand Vickers hardness of the glass sample was found to be 5.55 GPa ± 2 % averaged over four indent which is within the expected range e.g. the reported value of 5.9 GPa [5]. The 100g indent sites show radial and circumferential cracks but in fewer number than in the alumina sample. The radials also show small offset from the corners of the residual indent impression. The maximum length of the radial cracks at 100g indent site was 12 μm from the corners of the residual indent impression Fig.1(b) which is the same as in the alumina sample, depth of residual damage crater was measured to be ~ 3.7 μm whereas for the 100g indent site in alumina was ~ 1.9 μm.

4. 3D tomographic reconstructions of crack distribution under indents

4.1. Alumina

3D maps of the subsurface cracks were generated from multiple 2D cross-sections using IMOD software [9]. Figure 2a and 2b shows the 3D reconstruction from 34 sections through a 100g indent site. At least 20 major cracks can be clearly identified under the 100g indent (Fig. 2a and 2b). The main features of the observed 3D crack distributions are:

i. A relatively crack-free zone directly under the centre of the indent < 1.5 μm from surface

ii. Radial cracks occurring at a steep angle to the surface and close to the four diagonals of the Vickers indenter

iii. Circumferential cracks running between the radials, with the most outermost having an angle of ~ 30° to surface. The density of circumferential cracks identified in the 2D sections was lower that that observed from the surface (Fig. 2), indicating that either there is a size threshold for crack observation, possibly due to FIB sputtering of material into the finest cracks masking their presence, or that the FIB sectioning procedure is resulting in some crack closure due to stress changes.

iv. Deep lateral cracks, linking the radials.

For the 100g indent the observed crack lengths ranged up to ~ 14 μm. Cracks were observed propagating along, and connecting together, the planar fault traces, possibly following twin interfaces which are known to act as preferential crack planes [11, 13]. The 3D reconstructions enable quantitative data on individual crack sizes and shapes to be obtained. One of the largest cracks under a sectioned 100g indent, a deep lateral oriented close to (1 1 0 2) in the first half of the indent to be sectioned, had an approximate surface area of 120 μm² (see Fig.2b).

4.2. Glass

The mechanisms by which the system of underneath Vickers indentation propagate have been extensively discussed in [14]. 3D map of subsurface cracks in glass due to 100g Vickers indentation is shown in Fig.2c and 2d. only two major cracks were observed i.e. circumferential crack and median crack. The circumferential crack has maximum depth ~ 2 μm and inclined to the surface by ~ 30°, the median crack connects between two opposite radial cracks and runs under the plastic damage zone which is underneath the centre of the indentation site (See Fig.2c and 2d) forming half-penny shape[5]. The maximum depth of this crack is > 9 μm and has surface area >160 μm² and crack opening >1 μm in 2D sections near impression corners of the indent site where maximum stress is expected see Fig.1d.
5. Conclusions

Crack morphologies around Vickers microindentations in single crystal alumina and glass samples have been analysed using Focused Ion Beam (FIB) tomography. It is shown that:

- Microindentation of single crystal alumina generates a complex central plastic deformation zone consisting of interconnecting cracks (radial, median, circumferential, lateral).
- 3D FIB tomographic analysis of crack population around indent sites shows a significant increase in measured crack density in alumina sample when compared to soda-lime-silicate glass at 100g load.
- Half-penny crack shape was observed beneath indentation site in soda-lime-silicate glass.

It is concluded that FIB tomographic analysis is a useful technique to investigate the major features of the crack distribution around indents, however FIB sputtering causes changes in microstructure and residual stress, and hence changes the crack distribution from that existing prior to FIB processing.

References

[1] Holzer L, Indutnyi F, Grasser Ph, Munch B and Wegmann M 2004 J. Microsc. 216 84-95
[2] Inkson B J, Leclere D, El fallagh F and Derby B 2006 J. of Phys. Conf. Series 26 219-222
[3] Inkson B J, Wu H Z, Steer T J and Möbus G 2001 MRS Proc. 649 7,7
[4] Wu H Z, Roberts S G, Möbus G and Inkson B J 2003 Acta Materialia 51 149-163
[5] Cook R F and Pharr G M 1990 J. Am. Ceram. Soc., 73 787-817
[6] Inkson B J, Steer T, Möbus G and Wagner T 2001 J. Microsc. 201 256-269
[7] Steer T J, Möbus G, Wagner T, Kraft O and Inkson B J 2002 Thin Solid Films 413 147-154
[8] Uchic M D, Groeber M A, Dimiduk D M and Simmons J P 2006 Scripta Mater 55 23-28
[9] Kremer J R, Mastronarde D N and McIntosh J R 1996 J. Struct. Biol. 116 71-76
[10] Guillou M O, Henshall J L and Hooper R M 1998 Int. J. of Refractory Metals and Hard Materials 16 323-329
[11] Chan H M and Lawn B R 1988 J. Am. Ceram. Soc. 71 29-35
[12] Farber B Ya, Yoon S, Peter K, Lagerlof D and Heuer A H 1993 Z. Metallkd. 84 6
[13] Inkson B J 2000 Acta mater. 48 1883-1895
[14] Whittle B and Hand R 2001 J. am. Ceram. Soc. 84 2361-65