The effect of focused ion beam machining on residual stress and crack morphologies in alumina

B J Inkson1, D Leclere2, F Elfallagh1, B Derby2

1Dept of Engineering Materials, Univ. of Sheffield, Mappin Street, Sheffield, S1 3JD
2School of Materials, University of Manchester, Manchester, M1 7HS, UK

beverley.inkson@shef.ac.uk

Abstract. Focused ion beams (FIB) are widely used to locally sputter away material from surfaces at the nanoscale, but the effect of localised geometry changes and surface damage generated by FIB processing on material stress states are poorly understood. Evolution of stress states has been investigated in alumina samples with high local residual stress concentrations around nanoindents and scratches. Crack morphologies under the nanoindents and scratches have been investigated with respect to the location and geometry of the ‘cross-sectional’ surface trenches machined by FIB. It is found that the density of cracks observed around the nanoindentation sites depends on the location and milling sequence of the cross-sectional FIB trenches which alter local stress states. Cr3+ fluorescence spectroscopy has additionally been used to map stresses around alumina scratch and FIB-machined surface trenches.

1. Introduction

Focused ion beams (FIB) are now widely used as a multi-purpose nanotechnology tool for surface nanofabrication and nanoanalysis. One side-effect of using an ion beam to machine 3D nanostructures can be changes in the residual stresses within the material being processed. This can be due to both surface damage/amorphisation caused by the ion implantation/sputtering process, and an alteration of the original stress state of the material due to the changes in surface geometry.

Changes in residual stress may have important implications for FIB-processed materials, for example surface damage and relaxations in stress at new free surfaces may cause changes in geometry of components (e.g. undesirable warping of FIB machined TEM specimens), alterations in crack density/morphology, and changes in the mechanical properties of nanostructures such as cantilevers.

There has been much recent interest in the use of FIB for 3D tomographic analysis, systematically cutting materials into many parallel 2D sections, from which a 3D analysis of microstructure can be obtained [e.g. [1,2]]. The use of FIB for tomography, and indeed the FIB preparation of TEM and electron tomography specimens, requires minimal change in microstructure during FIB milling of the substrate material. In this paper we investigate if crack morphologies observed under nanoindents and scratches in alumina can be affected by the invasive method of FIB cross-sectioning.

2. Surface damage of alumina

Alumina is an important structural ceramic, used widely as an abrasive and for wear-resistant components. Most studies of alumina wear have involved sectioning wear surfaces post-mortem into 100-1000nm thick slices and examining the microstructure and retained dislocation distribution by TEM [e.g [3]]. FIB has recently enabled the sectioning of alumina wear surfaces with a lateral
accuracy of <50nm, enabling ‘2D’ site-specific cross-sectioning and 3D tomographic reconstruction of crack zones under surface damage in a volume of the order of 20 µm$^3$ to be carried out [e.g. [1,2,4]].

Here the crack distribution under nanoindents and scratches have been examined. Fully dense Al$_2$O$_3$ samples with 3µm average grain size were polished to a 1µm finish and then indented in arrays with a Vickers diamond tip using a 1N load. This load is sufficient to initiate significant sub-surface cracking and dislocation activity, with some radial and lateral cracking visible at the corners of the nanoindentations and along the scratches. The polycrystalline Al$_2$O$_3$, and additional single crystal Al$_2$O$_3$ samples, were also scratch tested using a Vickers diamond tip and a 1N normal load.

3. Focused ion beam sectioning of scratches and nanoindents
The indented and scratched samples were coated in conductive Au or C to inhibit charging, and Pt was locally deposited by FIB over the indents and scratch sections to be analysed. Cross-sections through the worn surfaces were then obtained by standard FIB methods, that is deep trenches were sputtered into the surface with a 30kV Ga$^+$ 50-7000pA focused ion beam impinging normal to the surface. One side of the trench was polished with a low current 100pA beam enabling an image to be taken of the near-normal 2D cross-section through the surface using in-situ SEM or by tilting the specimen and imaging with ion induced secondary electrons (ISE).

3.1. FIB cross-sections through scratch tracks
Figure 1 shows a typical FIB cross-section through a 1N load scratch track in alumina. As found previously, the crack distribution observable by FIB can be described by 3 zones [2]: (I) minimal cracking in the compressive zone directly under the scratch track, (II) substantial microcracking under the compressive zone and extending up to the edge of the surface track, and (III) deep radials and lateral cracks penetrating into the tensile zone below and around the crack. Multiple parallel cross-sections in both single crystal (with no crystallographic variation or grain boundaries) and polycrystalline samples show scatter in the precise locations of the observed cracks in zones I-III along the scratches due to local variations in loading stress, crack initiation and crack propagation.

3.2. FIB cross-sections through residual deformation at nanoindentation sites
Figure 2 shows typical FIB cross-sections through a 1N load nanoindentation in polycrystalline alumina. The indent in Fig. 2 was sectioned into 58 sequential 2D x-y sections along the +ve z axis direction. It was observed that the measured crack density was considerably higher for cross-sections on the first side of the indent to be cut (e.g. Fig. 2(b)) than on ‘symmetrical’ cross-sections the same linear distance from the original indent centre but on the second side of the indent to be sectioned (e.g. Fig. 2(c)). This asymmetry in the crack distribution was observed for a number of indents in an array generated sequentially with exactly the same indenter tip and loading conditions, and was independent of the sectioning direction chosen (e.g. bottom to top or left to right in Fig. 2). This indicates that it

![Figure 1](image1.png)

![Figure 2](image2.png)
was not the result of an asymmetric indenter tip, asymmetric loading or local crystallography. The same effect was also seen in the FIB sectioning of a 1N indent in an Al₂O₃-SiC nanocomposite [1].

![Figure 2. FIB cross-sectioning of a 1N load nanoindentation in alumina. (a) Indentation site covered in protective Pt, subsequently sectioned into 58 sequential 2D x-y slices along +ve z axis direction. (b) Section A marked in (a). (c) Section B marked in (a), ∆z=10.2µm from A. A and B are approximately equidistant from the indent centre (∆z=5.0 and 5.2µm respectively), but only A exhibits identifiable cracking.](image)

The asymmetry of the crack observations around Al₂O₃ indents must be related to the asymmetrical changes in the local residual stress during the invasive FIB cross-sectioning of the residual indent impression. In a unidirectional sequential FIB sectioning of an indent [Fig. 2, [1]], the first side of the indent examined has the new observational free surface cut close to the severe damage/high stress zone at the indent centre. Many cracks can be observed, and it is likely that, with the adjacent high residual stress concentration at the indent centre, existing/new cracks may open towards the new free surface. In contrast, the second side of an indent analysed in a unidirectional sectioning sequence has had the severe damage/high stress zone at the indent centre removed (Fig. 2(c)). This will undoubtedly significantly reduce the local residual stress, and with a reduced driving force for opening/widening at the cut FIB surface many existing cracks are likely to be below the threshold for observation using the current experimental imaging conditions. Provisional studies show that if an indent has FIB trenches dug simultaneously on both sides (either side of the severely stressed indent centre), then the crack density observed on ‘symmetrical’ cross-sections is comparable.

For the case of a scratch, the FIB sectioning direction seems to have no great influence on the average crack density observed (Fig. 1). Local concentrations of residual stress occur all along the scratch track. Unless there is a directionality to the crack propagation, it will be random for a given observed crack whether a FIB cross-section trench is located so as to remove (or retain) the nearby local stress concentration centres driving crack initiation, propagation and opening.

4. Stress analysis of surface damage by Cr³⁺ fluorescence spectroscopy

The residual stress around surface damage features in Al₂O₃ was investigated by optical fluorescence spectroscopy [5,6]. For this study, residual stress was measured in single crystal samples to ensure that there was no influence of grain boundaries and variable crystallography on the stress measurements. Figure 3 shows an optical/SEM image of a 1N load scratch with a 3 µm deep FIB trench machined into the scratch track with a 30kV 7000pA Ga⁺ beam. From previous 3D analysis of the deformation microstructure below 1N load damage [2], this trench should be located in the zone of maximum plastic deformation (dislocations and cracks) and peak compressive stress. 105 Cr³⁺ fluorescence spectra were taken at distances 0-50µm from the scratch centre using a ~2µm wide ~20µm deep probe (Fig. 3). Spectra positions relative to the FIB milled trench were recorded. The shift and broadening of
the R₁ and R₂ Cr³⁺ fluorescence lines were measured. The average local residual stress was calculated from the R₁ frequency shift using the calibration of Ma and Clarke [6], that is the total sum of the stress acting along the 3 cartesian axes being associated with a R₁ shift of 7.7 cm⁻¹/GPa.

Figure 3. Optical image showing the location of fluorescence spectra around a 1N scratch with FIB milled trench (dotted box).

Figure 4. 2D Contour plot of the R₁ fluorescence peak shift measured across the specimen and extrapolated to a 20 x 20 grid. The shift is associated with the total sum of the stress acting along the 3 cartesian axes with a shift of 7.7 cm⁻¹/GPa.

Figure 4 shows a 2D contour map of the R₁ fluorescence peak shift corresponding to the average local residual stress around the FIB trench and scratch shown in Fig. 3. The main features of the local residual stress map are those expected from the 1N scratch, that is net compressive stress under and close to the scratch, and a long range tensile stress field at increasing distance from the scratch [2]. The local stress measured on and close to the FIB trench cannot be distinguished from the stress away from the FIB trench at the same linear distance from the centre of the scratch. This means that any reduction in local residual stress for this shallow FIB trench is within the scatter of stress values which occur naturally due to the local variations in residual damage (dislocations/cracks) along the scratch.

5. Conclusions
(a) FIB cross-sections through scratch tracks: Multiple parallel cross-sections in both single crystal and polycrystalline Al₂O₃ show scatter in precise positions of observed cracks under scratch tracks due to local variations in loading stress, crack initiation and propagation, but the average observed crack density is not noticeably influenced by FIB trench position or milling procedure. An analysis of residual stress around a 1N load scratch with a shallow 3 µm deep FIB trench by Cr³⁺ fluorescence spectroscopy did not resolve FIB-induced stress changes.
(b) FIB cross-sections through indentation sites: FIB cross-sections through 1N load indentation damage sites in Al₂O₃ show that the density of cracks observed depends on the location and milling procedure of the cross-sectional FIB trenches. Crack densities, for a given linear distance from the indent centre, are measured to be higher if the FIB-milled cross-sectional surface is adjacent to the (retained) severe damage/residual stress zone, than if the cross-sectional surface has been cut from the opposite direction removing the severe damage/residual stress zone.

References
[1] B.J. Inkson, H.Z. Wu, T. Steer and G. Möbus, Fundamentals of Nanoindentation and Nanotribology II, MRS Proceedings, 649, 7.7.1-7.7.6 (2000).
[2] H.Z. Wu, S.G. Roberts, G. Möbus and B.J. Inkson, Acta Mater., 51, 149-163 (2003).
[3] B.J. Hockey, J. Am. Ceram. Soc., 54, 5, 223 (1971). Proc. Br. Ceram. Soc., 20, 95 (1972).
[4] S. Chaiwan, M Hoffman, P. Munroe and U. Stiefel, Wear, 252[7-8], 531-676 (2002).
[5] Q. Ma and D.R. Clarke, J. Amer. Ceram. Soc., 76, 1433 (1993).
[6] Q. Ma and D.R. Clarke, J. Amer. Ceram. Soc. 77, 298-302 (1994).