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3D Studies of damage by combined X-ray tomography and digital volume correlation

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Abstract

The combined use of high resolution X-ray computed tomography with digital image correlation allows quantitative observations of the three-dimensional deformations that occur within a material when it is strained. In suitable microstructures, the displacement resolution is sub-voxel (a voxel is the three-dimensional equivalent of a pixel), and both elastic and plastic deformations can be studied. This paper reviews recent work in which three-dimensional in situ observations of deformation have provided unique insights that support both continuum and heterogeneous microstructure-dependent models of damage development in a range of materials. The examples presented include; crack propagation in a quasi-brittle porous material (polygranular graphite), sub-indentation radial and lateral cracking in a brittle polycrystalline ceramic (alumina); plastic deformation and damage development underneath indentations in a ductile metal (Al-SiC composite) and a ceramic matrix composite (SiC-SiC$_{\text{fibre}}$). These examples show how material properties can be obtained by analysis of the displacement fields, how such measurements can be used to better define the applied loading on small test specimens and how crack opening magnitude and mode may be extracted also. Some new directions for research are outlined, including the combined use of diffraction and imaging techniques on synchrotron X-ray facilities to map both elastic and inelastic strains.

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Keywords: Computed Tomography; Digital Image Correlation; Damage; Deformation

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1. Introduction

Three-dimensional in situ observation of damage within materials has become possible through high-resolution X-ray computed micro-tomography (µXCT) [e.g. Stock (1999), Maire et al (2001) and Marrow et al (2004)], aided by the brilliance of synchrotron sources. Digital volume correlation (DVC) can map relative changes in displacement between tomographic datasets [Bay et al (1999)], allowing quantitative observations of the three-dimensional deformations that occur within materials when they respond to loads. In suitable microstructures, the displacement resolution is sub-voxel (a voxel is the three-dimensional equivalent of a pixel), and both elastic and plastic deformations can be studied [e.g. Forsberg and Siviour (2009) and Barranger et al (2009)]. This paper describes recent work conducted on the Joint Engineering, Environmental and Processing (I12 – JEEP) beam line at the Diamond Light Source. The examples presented include; crack propagation in a quasi-brittle porous material (polygranular graphite), sub-indentation radial and lateral cracking in a brittle polycrystalline ceramic (alumina); and plastic deformation and damage development underneath indentations in a ductile metal (Al-SiC composite) and a ceramic matrix composite ($\text{SiC-SiC}_{\text{fibre}}$). The objective is to extract material properties, or validate predictive simulations of damage development, by analysis of the three-dimensional displacement fields.

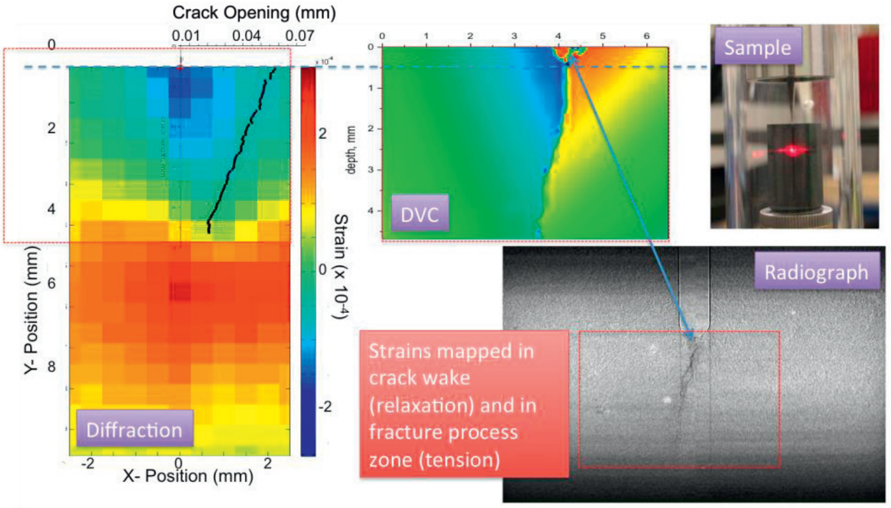
1.1. Crack Propagation in Quasi-Brittle Polygranular Graphite

Crack propagation in polygranular graphite, a model quasi-brittle material, is accompanied by the development of a micro-cracked fracture process zone (FPZ) [Mostafavi et al (2013a), (2013b)]. Through the combined use of µXCT and DVC, the deformation of the fracture process zone has been measured, and described by cohesive zone modelling. A very recent experiment has made novel measurements of elastic strains in the FPZ. This required synchrotron X-ray diffraction mapping, supported by DVC applied to µXCT. Prior to loading, the region ahead of a notch was mapped by diffraction with the beam reduced via slits to 1.5 x 1.5 mm (area mapped 9.5 mm x 5 mm, with step size 0.75 mm). The (00.2) diffraction ring was observed at a distance of 2.55 m (beam energy 80 keV). The same region was mapped by overlapping tomographs (1.8 µm voxel). Radiography during loading observed crack initiation and propagation, to develop a 4 mm crack length. Diffraction mapping and µXCT were repeated with the crack under load. Unfortunately, due to a power failure that terminated the experiment, only the crack wake could be tomographed; beam time has been obtained for a repeat study during 2014. DVC analysis of the tomographs measured the crack opening displacements in the crack wake; the displacement gradients can be used to visualise the crack shape. The crack opening profile is superposed on a map of the [00.1] strains (Figure 1), calculated via a pseudo-Voigt fit to the diffraction peaks; its position in the loaded and unloaded states provides the (00.2) plane spacing via Bragg's law: the crystal strain is their relative change. The preliminary data, presented here only for strains perpendicular to the crack plane, show the crack tip elastic strain concentration has been measured for the first time. The compressive strains in the crack wake are interpreted as relaxation via microcracking of the significant thermal strains that exist after graphitization at >2000°C. The influence of microcracking on the elastic properties and the work of fracture is important in quasi-brittle fracture models. The future experiment will compare the total and elastic strains within the FPZ.

1.2. Plastic Deformation in Ductile Al-SiC Metal Matrix Composite

Hardness testing has long been used to interrogate materials to understand their deformation behavior and to infer the processes of deformation that have occurred underneath the indenters [e.g. Giannakopoulos and Suresh (1999), Mesarovic and Fleck (1999)]. With appropriate assumptions or understanding of deformation processes, hardness tests on small samples can be used to evaluate the effects of changes in microstructure on the mechanical properties of engineering components. Hardness testing is a surface characterisation technique and, except in transparent materials, it provides no direct observation of the assumed damage and deformation processes. Indentation in a model ductile metal, an aluminium-silicon carbide composite (Al-SiC, 15% volume fraction reinforcement), has been studied using DVC of µXCT data to measure the sub-surface displacement field in three-dimensions. Brief details and some results are presented here; further information is provided in Mostafavi et al (2014). The sample was examined by high-resolution µXCT (0.9 µm voxel) at 53 keV: (i) before and (ii) in situ during indentation.

Absorption contrast between Al and SiC is very low, so the distance between the imaging scintillator and specimen was increased to enhance phase contrast. The specimen was indented with a 5 mm radius ZrO_2 ball with an in situ loading rig. The reference tomograph was recorded under a small pre-load to reduce rigid body movement, with the second tomograph at 500 N. Following DVC analysis, fine rigid body movements and rotations were corrected using a novel and efficient algorithm. This is particularly important in indentation experiments due to the small displacements within the sample and the high loads that can cause significant sample movements, even with a stiff loading rig. A vertical μXCT slice shows the composite's texture (Figure 2) and although individual particles are not resolved, DVC operates successfully on the contrast from microstructure heterogeneity. To simplify the displacement data presentation, they are transformed to polar coordinates, with the axis of the indentation as origin, to obtain average radial displacements. The displacement field and the reaction force were found to agree well with an elastic-plastic FE simulation of the indentation, using the measured indentation depth and properties from tensile tests of the same material. This experiment demonstrates that, even in microstructures that produce apparently poor quality μXCT images, DVC can yield sufficiently accurate data to quantify plastic strains. Future work will examine whether "reverse-engineering" via finite element simulation may extract material properties from such observations with sufficient precision to investigate the elastic-plastic behavior of ductile materials, including graded microstructures.



Strains mapped in crack wake (relaxation) and in fracture process zone (tension)

Figure 1: A notched polygranular graphite sample (top-right) is wedge loaded until a crack is observed via radiography of the notch (bottom-left). DVC of μXCT data (centre) measures the crack opening displacement profile, which is compared with the diffraction-measured crystal strain map obtained from the change in basal (00.2) interplanar distance (left). Tensile strain is observed ahead of the crack tip. Microcracking is deduced to relax the internal strains in the crack wake. The rectangle indicates the same region in each image (laser illuminated in top-right).

1.3. Indentation Cracking in Brittle Alumina Ceramic

During indentation of brittle materials a system of sub-surface radial cracks is generated to accommodate the indentation strain. Lateral cracks, which are approximately parallel to the surface, arise from the residual strain field surrounding the indentation plastic zone as the sample is unloaded. Understanding indentation cracking in hard, brittle materials is important, since indentation damage contributes to certain forms of wear [Chauhan et al (1993), Lawn (1998)]. Whilst it is routine to study the surfaces of indented materials to characterise surface cracking, there are very few observations of indentation cracking below the surface [e.g. Cook and Pharr (1990), Elfallagh and Inkson (2008)]. Such studies are usually destructive, and may modify the residual stress state during observation [Guo and Todd (2011)]. A precise, non-destructive method is required to study the shape and orientation of indentation cracks. The combined μXCT/DVC methodology has been applied to investigate its suitability to study the cracks that develop in a model brittle material, polycrystalline alumina, during indentation. Brief details and some of the results are described here; a more full analysis is provided in Vertyagina et al (2014). The polycrystalline Al_2O_3 sample was indented under displacement control using a standard Vickers pyramidal diamond; the sample was imaged by µXCT (0.9 µm voxel, 53 keV): (i) before indentation, with small pre-load to fix the sample position; (ii) in situ at the maximum indentation load of 330 N, and (iii) after removal of the load. The first scan provided the reference for the DVC analyses, and fine rigid body movements and rotations were corrected as before. The µXCT images provide a limited description of the cracking; Figure 3a shows a vertical section (peak load and after unloading) in which one of the four radial cracks is visible by phase contrast. The horizontal displacement component for the same section is below, showing the relative opening of the radial crack decreases on load removal. The DVC data therefore provide a means to study crack opening. Vertical displacements close to the top surface (Figure 3b) reveal the uplift from the development of lateral cracking after radial cracking, such that lateral cracks are bounded by the radial cracks. The experiment reveals complex patterns of shear and opening as the cracks interact with the indentation's residual stress field. Such observations may support models that predict the stability and development of contact-induced cracks during wear.

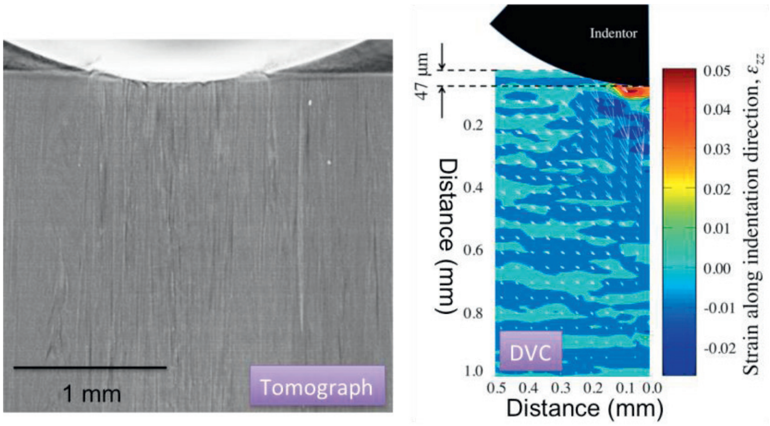


Figure 2: Vertical section of the µXCT data of Herzian indention in Al-15% SiC composite (left). The DVC-measured vertical strain is shown as a contour map (right) with vectors of the displacement field superposed.

1.4. Indentation Damage in a Quasi-Brittle SiC-SiC$_{fibre}$ Ceramic Matrix Composite

Ceramic matrix composites are heat-resistant structural materials with potential aerospace and nuclear applications. The influence of the microstructure on damage development is significant in these materials, so microstructure must be introduced into predictive structural integrity models, in order to optimise the fabrication of materials and components. To validate such models, the simulated deformation fields must be compared with experimental observations. This was done recently, using indentation to introduce mechanical damage; brief details of the experiment are presented here and further information is provided in Saucedo Mora et al (2013). The SiC-SiC$_{fibre}$ composite, supplied by Chinese National University of Defense Technology [Zhao et al (2013)] was fabricated via the PIP (Polymer Infiltration and Pyrolysis) process; the sample was indented using a 5 mm radius ZrO_2 ball, as in the Al-SiC composite study. The reference tomograph (0.9 µm voxel, 53 keV) was recorded under a small pre-load to reduce rigid body movement and a second tomograph was obtained at the maximum applied load of 275 N. Following DVC of the µXCT data, the small rigid body rotations were corrected. The aim of the experiment was to test simulations of the damage behavior in heterogeneous materials, obtained via the Cellular Automata Finite Element (CAFE) method [Saucedo and Marrow 2012]. In the CAFE method the microstructure is modeled explicitly using cells with variable properties. A meshfree framework computes the fracture development in response to deformation, using cellular automata to calculate the damage; the material properties are recomputed according to the microstructure damage, hence the redistribution of stress from damage is considered. The DVC-obtained strain field in a vertical section under the indentation (Figure 4) shows the material displacements have been quantified. There is quite good agreement with the CAFE simulation, which captures the heterogeneity of deformation that is missing in elastic-plastic FE analysis; the only fitting parameter was the materials' strain-softening characteristic (i.e. effective elastic modulus change due to non-resolved fine-scale damage), other properties being obtained from independent data on flexural strength and nano-indentation. The simulations used the measured indentation depth, and the CAFE model used data of pore and fibre distributions obtained by µXCT. These methods are now being applied to crack initiation and propagation in a ceramic matrix composite; the objective is extract model parameters such as the critical strain to failure and the strain-softening characteristics, to thereby develop stochastic models for component strength.

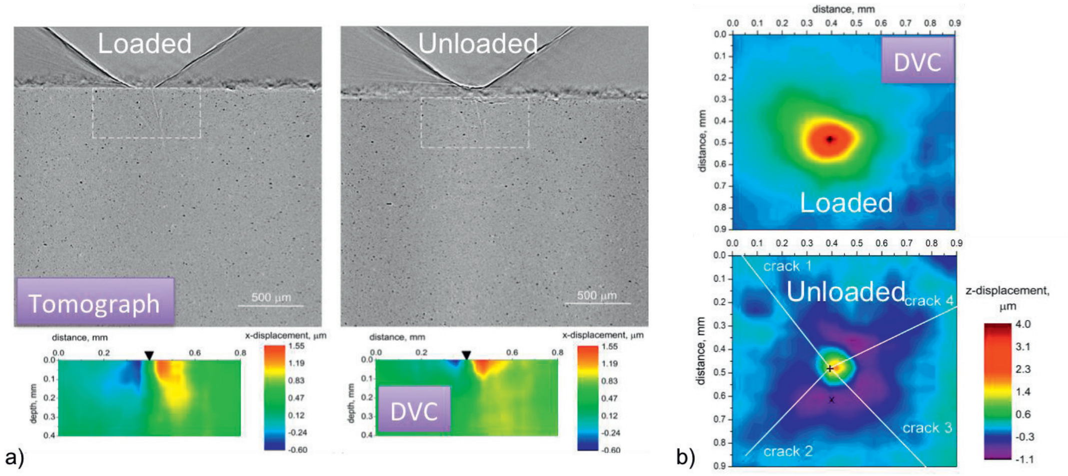


Figure 3: Vicker's indentation of Al_2O_3 ceramic; (a) tomographic vertical cross-sections of sample under load and unloaded. The corresponding maps of the horizontal displacement in the area presented by a dashed rectangle are shown below; (b) maps of vertical displacements in a horizontal plane under the indentation, showing the indentation deformation and the uplift from lateral cracking. The radial cracks are indicated.

2. Conclusion

Combining µXCT and DVC allows one to internally 'strain-gauge' a material, if it has a suitable microstructure; this provides unique opportunities to study deformation, in situ. As image and camera quality continue to improve, higher resolution measurements will become possible in the future.

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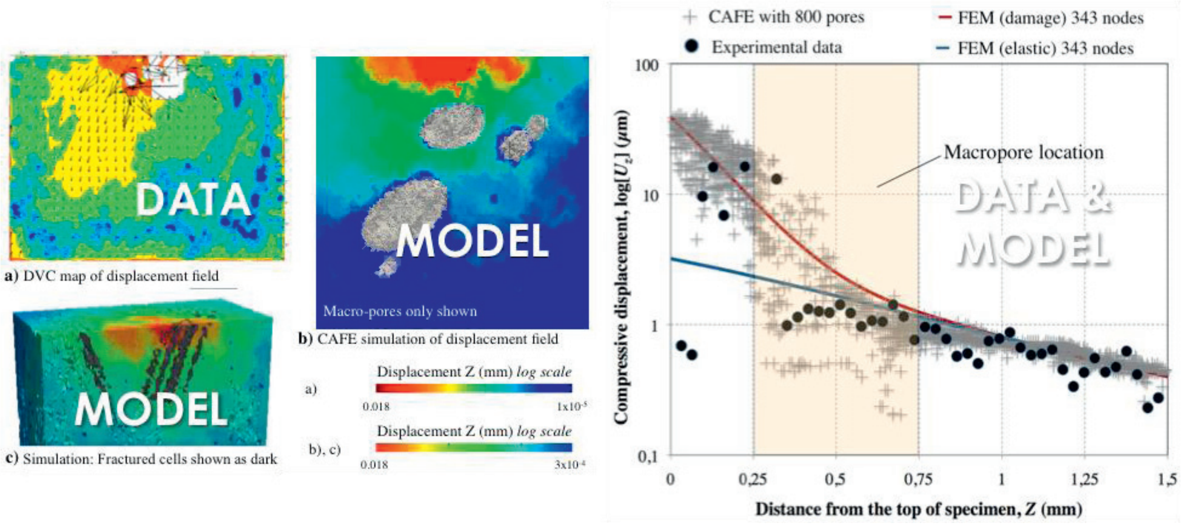

a) DVC map of displacement field

b) CAFE simulation of displacement field

c) Simulation: Fractured cells shown as dark

Figure 4: Comparison of observed and simulated displacements beneath a Herzian indentation in a SiC-SiC_{fibre} composite; the DVC measured displacement field (~3 mm field of view) (a) is compared with a visualisation of the CAFE-simulated damage (b and c). The displacements beneath the indentation are compared with FE simulations with elastic and elastic-plastic damage (d).