3D cellular automata finite element (CAFE) modelling and experimental observation of damage in quasi-brittle nuclear materials: Indentation of a SiC-SiC$_{fibre}$ ceramic matrix composite

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Abstract

Cellular automata integrated with finite elements (CAFE) have been used to develop a method to account for the effect of microstructure on quasi-brittle damage development. The microstructure is simulated explicitly by subdividing a finite element into smaller cells. A heterogeneous structure is created from key cells (seeds) using defined characteristics; the influence of the initial finite element mesh is effectively removed during the development of the microstructure. Graded microstructures, textures, particle anisotropy and multiple phases can be readily simulated, such as those in composites and porous materials. A mesh-free framework has been developed to compute the damage development through the microstructure, using cellular automata. With this method, we can study the development of discontinuous cracking and damage coalescence, and its sensitivity to microstructure. Experiments have been carried out to observe the three-dimensional development of damage, using high-resolution synchrotron X-ray computed tomography and digital volume correlation to observe Hertzian indentation of a SiC-SiC fibre composite, quantifying damage by measurement of the displacement fields within the material. The results demonstrate the applicability of the modelling strategy to damage development, and show how model input data may be obtained from small specimen tests, which could be performed at elevated temperatures with irradiated materials.

Introduction

Quasi-brittle materials have heterogeneous structures, typically with brittle constituents. This important class of structural materials includes SiC-SiC$_{fibre}$ ceramic-matrix composites, as well as concrete, nuclear graphite, zirconia toughened alumina, and geological structures like rocks and tectonically faulted formations and also biomedical materials such as bone and bone replacements. Their damage or defect tolerance is much less than engineering metal alloys, but can be quite significant compared to fully brittle materials such as monolithic ceramics. They differ in their length-scales of both their structures and the distributions of damage, and have varying degrees of brittleness.

Quasi-brittle fracture is an emergent characteristic, and this cannot be treated satisfactorily with numerical methods based on macromechanics. Because of their complex microstructure, the continuum approach can be too simple for these materials, and needs a finer discretisation to obtain satisfactory results. In numerical terms, this means that the computational cost of advanced methods, such as cohesive elements or embedded cracks, is often too high for engineering scale problems. However, including the role of the microstructure is critical to reproduce structural behaviour in these materials.

In this paper we apply the cellular automata finite element (CAFE) method to model Hertzian indentation in a SiC-SiC_{fibre} composite. Such composites are candidate materials for high temperature fuel cladding in several Generation IV nuclear reactor concepts. The influence of the microstructure on the development of the damage and strain fields is significant in these materials, and for this reason the microstructure needs to be introduced into the model. The ultimate goal of this research is to be able to predict the influence on fuel-cladding integrity of the microstructure and the degradation of its properties under normal and extreme conditions, in order to better select materials and optimise the fabrication of fuel components.

The paper is structured as follows: first we describe the mechanical properties and fabrication process of the SiC-SiC composite; after this, the experimental programme and the analysis of the data are explained. From those results we propose a numerical modelling method that can simulate the interaction between the microstructure and the Hertzian indentation, validating this against the experimental results.

Material

The SiC/SiC_{fibre} composite was fabricated in three steps: preparation of SiC fibre performs, deposition of fibre coating and fabrication of SiC/SiC composites. The 3-dimensional 4-directional (3D4d) fibre preforms were woven by Bolong Co. Ltd (China), with a fibre fraction volume of approximately 40 %. The KD-I SiC fibre used, made by the National University of Defence Technology, China [1] has a diameter of 12.5 μm, tensile strength of 1.6-2.0 GPa and a density of 2.4 g cm^{-3}. This is a polymer-derived multiphase fibre consisting of β-SiC crystalline, Si-C-O amorphous and free carbon phases. A pyrolytic carbon (PyC) coating was deposited onto the surface of the fibres to form the fibre/matrix interphase through a CVD (Chemical Vapor Deposition) process. The SiC/SiC_{fibre} composite was fabricated via the PIP (Polymer Infiltration and Pyrolysis) process; the polycarbosilane precursor (PCS) was infiltrated into the preform and heated up to 1100°C in an inert atmosphere, with the infiltration and pyrolysis process repeated for about 10 cycles until weight increase was less than 1%. The final heat treatment was at 1400°C for 1 hour in an inert atmosphere. Table 1 shows the measured mechanical properties of the material [1].

Table 1: Mechanical properties of the SiC/SiC_{fibre} composite [1]

Modulus (GPa)	Hardness (GPa)	Modulus (GPa)	Hardness (GPa)	Flexural Strength (MPa)	Modulus (GPa)
fibre		matrix		composite	
115	14	188	20	514	82

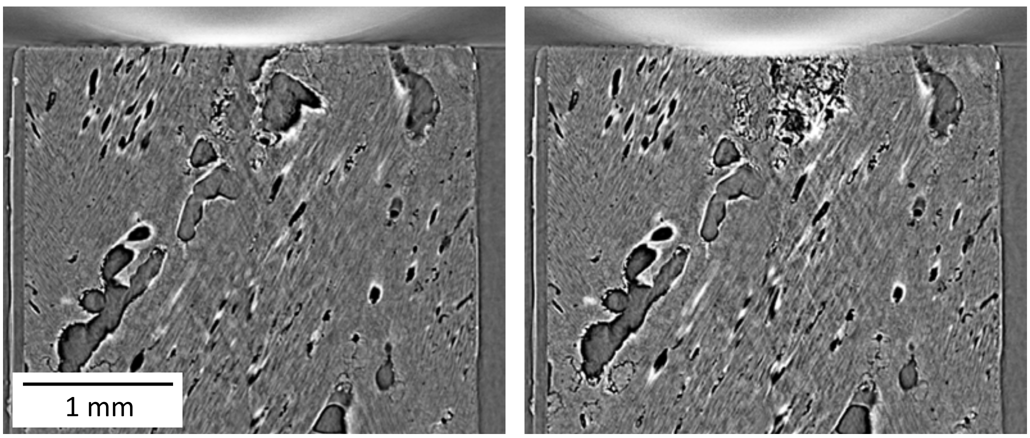
Experimental programme

The three-dimensional microstructure of the composite sample was characterised by X-ray computed tomography, with the development of damage from indentation studied using three-dimensional digital image correlation (digital volume correlation, DVC) [2,3] of the tomography images. The high-resolution synchrotron X-ray computed tomography was performed at the Diamond Light Source, Joint Engineering, Environmental and Processing beam line (I12 – JEEP), using radiographic projections obtained at an X-ray beam energy of 53 keV with a nominal voxel size of 0.9 μm. The exposure time was 2 seconds per radiograph with projections at increments of 0.03 degrees over 180° rotation. The specimen, a rectangular prism (3×3 mm square, 3 mm high), was indented by a 5 mm radius ZrO_2 ball under displacement control using a loading stage that had been modified to accommodate the indenter.

A reference tomograph was recorded before indentation under a small pre-load (10 N), applied to reduce rigid body movement between successive scans, then a scan was recorded in situ under a maximum applied load of 275 N (indentation depth 43 μm, measured from radiographs); this dropped to 245 N during the tomography scan. A combined Fourier-wavelet ring artefact removal algorithm [4] was used to suppress ring artefacts in the reconstructed tomographs. These arise from instrument features such as defective or inefficient pixels in the scintillator; if not adequately supressed they can significantly increase noise in the DVC analysis as they do not displace with the material.

Vertical slices through the tomography data are shown in Figure 1, before and during indentation. The complexity of the microstructure is clear; large macro-pores, fine micro-pores and oriented bundles of fibres with elongated inter-fibre pores are observed. The damage induced by the indentation occurs over a length scale (~0.5 mm) that is comparable to the length scale of the microstructure.

Figure 1: Vertical slice through the tomographic image of the SiC-SiC_{fibre} sample



a) before indentation **b) in situ under indentation**

Analysis

The tomographic data of the scanned specimen were analysed using the Avizo Fire software to obtain a statistical description of the porosity, which would be used subsequently to create the CAFE simulation. To reduce noise, 3-D median image filtering was applied to prepare the data sets before segmentation, which was done by contrast thresholding to identify the groups of inter-fibre pores (Figure 2a); each group was assigned a unique label field material, with fifteen groups observed in the analysis volume. The 3-D material statistics collected for each fibre pore including 3-D volume, length, width, orientation and the spatial coordinates of the pore's origin. Binary thresholding was performed to define a bounding volume for each pore group, with centreline trees evaluated for each pore group to obtain the mean radius and tree length. A similar analysis was carried out for the small micro and larger macropores in the matrix.

The DVC analyses were carried out using the Davis StrainMaster 8.1 software [5], correlating the loaded 3D tomographic dataset against the unloaded reference to map the relative displacements. Each dataset, which originally measured 4 016×4 008×2 672 voxels (160 GB as 32 bit data) was cropped to 3 500×3 500×2 000 voxels and converted to 8 bit data (reduced to 24 GB) for the analysis. The vertical (z) rigid body movement (~ 50 μm) between datasets was corrected prior to image correlation by visual matching of image slices in a horizontal *xy* plane close to the indented surface; translations of 32 μm in *x* and 49 μm in y were similarly applied. These displacements arise from the compliance of the loading jig. The following image correlation parameters were judged to be optimal; 256×256×256 voxel interrogation window, 50% overlap and 1 pass, followed by 64×64×64 interrogation window, 50% overlap and 2 passes. Reducing the final interrogation window size in image correlation increases the displacement map spatial resolution, though excessive noise arises with smaller window size [6]. Overlapping interrogation windows may improve the displacement map spatial resolution in smoothly changing fields, allowing the use of larger interrogation windows to reduce measurement noise. Increasing the number of passes may also reduce noise, with a diminishing effect with increasing passes. Finally, minor (i.e. sub-voxel) rigid body translations and rotations were corrected [7], such that the relative displacements remote from the indentation, i.e. close to the bottom surface of the sample, were zero.

Numerical modelling

To simulate the indentation we used the CAFE method (Cellular Automata integrated with Finite Element) [8] to account for the effect of microstructure on quasi-brittle properties within a finite element simulation. In the CAFE method the microstructure is modelled explicitly by subdividing a finite element into smaller elements called cells. The heterogeneous microstructure is created from key cells, called seeds, from which particle-like regions may be grown with defined characteristics. By this topological approach we obtain sets of cells with variable properties to model the microstructure (rules are enforced during the selection of the seeds to avoid overlap between particles). Graded microstructures, textures and particle anisotropy can be readily simulated in microstructures with multiple phases. The influence of the initial finite element mesh is erased during the development of the microstructure.

A mesh-free framework has been developed to compute the fracture development through the microstructure, using cellular automata to calculate the damage to the microstructure. With this method, we can study the development of discontinuous cracking and fracture, and its sensitivity to microstructure, by using two sets of elements representing the finite element model and the microstructure. The first is used to link the engineering scale problem with the microstructure, obtaining the stress and strain fields of the macromechanical problem. With those, we compute the micro-mechanical fields using the second set of elements, which describes explicitly the microstructure. The material properties of the finite elements are recomputed according to the microstructure damage; hence the redistribution of strain and stress with crack propagation and damage is computed. Factors such as the effects of size on crack path and crack stability are therefore addressed. The fracture path is completely free with respect to the finite element mesh. Consequently, very complex fracture behaviour can be modelled, such as multiple or discontinuous cracks.

Reproduction of the microstructure in the model

A key aspect of the CAFE method is its capability to reproduce the microstructure, and thereby to insert discontinuities into the continuum model. To investigate its application to damage development in a ceramic-matrix composites we need to reproduce numerically the main significant microstructure features that are observed experimentally, which are the inter-fibre pores and also the significant macro pores. The elongated inter-fibre pores were therefore distributed randomly in the simulation of the specimen within the identified bounds of the 15 groups of fibre bundles, with their dimensions, orientation and position defined randomly using parameters from their measured statistical distributions (Figure 2b). The macro and micro-pores were similarly distributed randomly in the matrix (Figure 3). A number of simulations were developed; for consistency with the experiment a simulation was selected that had a large pore close to the indentation point (Figure 4). The model therefore consisted of SiC matrix and SiC fibres within the 15 fibre bundles with mechanical properties given in Table 1, and empty pores.

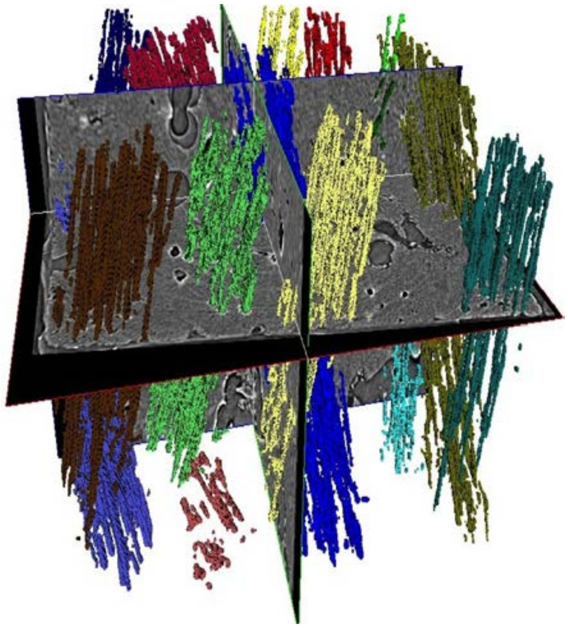
Simulation of hertzian indentation damage

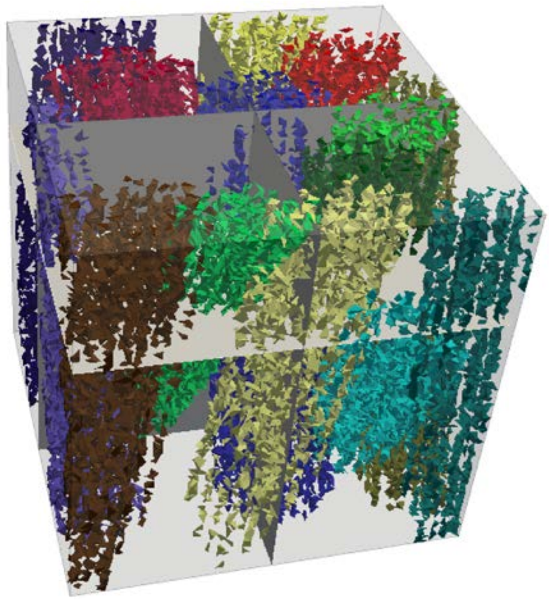
The CAFE simulation aims to describe the heterogeneous development of damage at the microstructure scale, whilst being consistent with the deformation at the continuum macroscale. To simulate the continuum effects of damage, the material properties of the finite elements are recomputed according to a damage criterion imposed in the FEM. This is done in an iterative loop that relaxes the stresses of the FEM in order to accommodate the displacement of the indenter while ensuring that the critical strength of the material is not exceeded. Once this is achieved, the nodal displacements of the relaxed FEM are used as an input of the mesh-free framework that deals with the microstructure. Again a relaxation is carried out in the microstructure to fit the nodal displacements of the FEM, introducing damage into cells in which a critical strain is exceeded [8]. The procedure is explained in more detail below.

The objective was to investigate whether the general characteristics of the observed damage were developed in the simulation of an indentation, so as a first approximation a simple 2-parameter damage model was used in the FEM simulations. The damage model was initially applied to the FEM as follows: first the FEM was computed without damage and the Von Mises stresses evaluated (the imposed boundary conditions are null displacements at the bottom of the sample and the measured 43 µm z-displacement of the rigid indenter ball into the top surface), second we reduced by an arbitrary factor the Young's modulus of any element in which the Von Mises stress exceeded the critical stress; we then recomputed the same problem with updated Young's moduli. This relaxation was done until all the elements had a Von Misses stress lower than the critical strength; in this analysis a critical stress of 500 MPa was chosen, which is very close to the flexural strength of the composite [1]. The factor by which the modulus of damaged elements was reduced was adjusted to obtain a reaction force close to the experimentally applied force of 275 N; for a modulus reduction factor of 30, the calculated reaction force was 274 N. Once the continuum behaviour of the sample was properly simulated, the nodal displacements of the FE mesh were used as an input of the mesh-free calculation in order to develop the damage in the microstructure. For this, the failure strain of the cells around the inter-fibre pores was chosen to be 0.005 (~ critical stress/Young's modulus); the critical strain for the stronger matrix was arbitrarily chosen to be 0.01. The relaxation from damage in the microstructure was again determined iteratively; at every step we identified cells with a strain coefficient above unity, (i.e. strain/critical strain); we treated these as damaged and recomputed the mesh-free model by reducing the Young's modulus in the same way as previously. This was repeated until all cells had a strain lower than their critical strain. The obtained simulation was one in which all the cells and elements were consistent with the physical model of the material and the global model provided the same response (i.e. the same reaction force) to the same boundary condition (i.e. displacement of the indenter) as its experimental counterpart. The CAFE simulation contained 800 pores (1 105 950 cells and 4 800 nodes, i.e. 6 nodes per pore); its computational cost was 7.5 hours running as a serial code as a single thread, compared to 550 seconds for the continuum FEM simulation (343 nodes and 1 080 tetrahedral elements), both running on a Intel Core i7-3930K, 3.20 GHz machine. Further studies are being carried out to compare the computational cost of the CAFE method with an FEM of equivalent discretisation; the CAFE method is amenable to parallel computing.

The observed compressive strain field in a vertical section under the indentation is shown in Figure 5, compared with the CAFE simulation result for the same section. The experimental data show a region of large displacements under the indentation, with some areas of poor correlation, which are due to the effects of damage; significant changes in the image will prevent DVC from converging (the areas of high deformation around the sample edge are image correlation errors due to the interface). The CAFE simulation predicts a similar sized zone of high strain, with some local strain concentrations due to the macropores. The slight asymmetry in the experimental data may be due to either to anisotropy of the microstructure or a component of shear loading from misalignment of the loading rig, and will be investigated in future simulations.

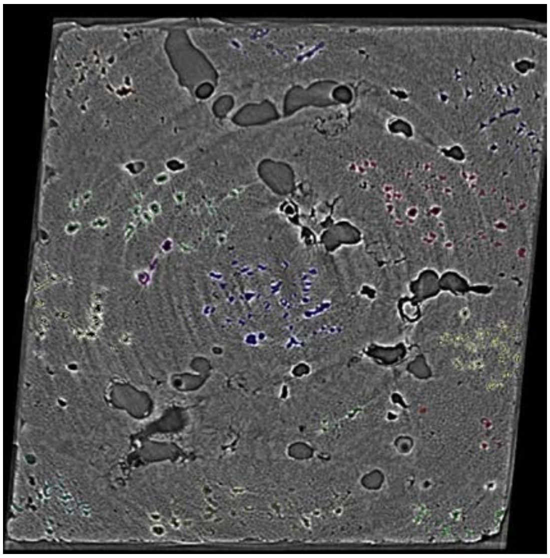
Figure 2: Inter-fibre pore distributions within fibre groups: a) experimentally observed structure after segmentation; b) numerical simulation

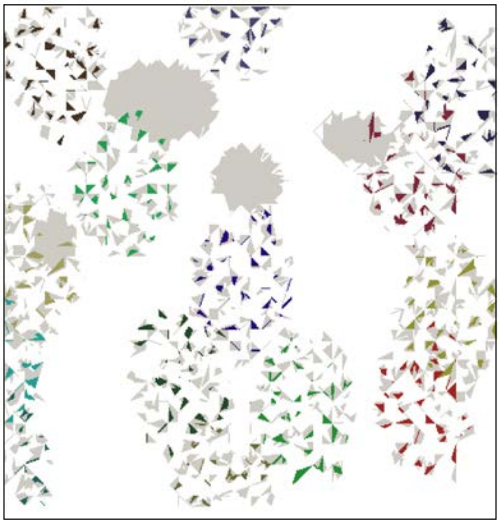




a) **b)**

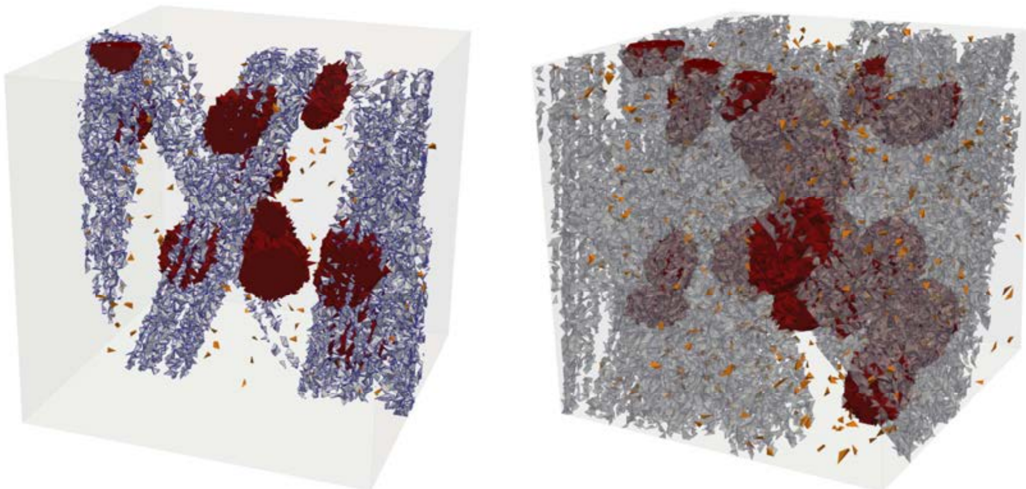
Figure 3: Macro and micropore distributions in the matrix: a) experimentally observed structure after segmentation; b) numerical simulation





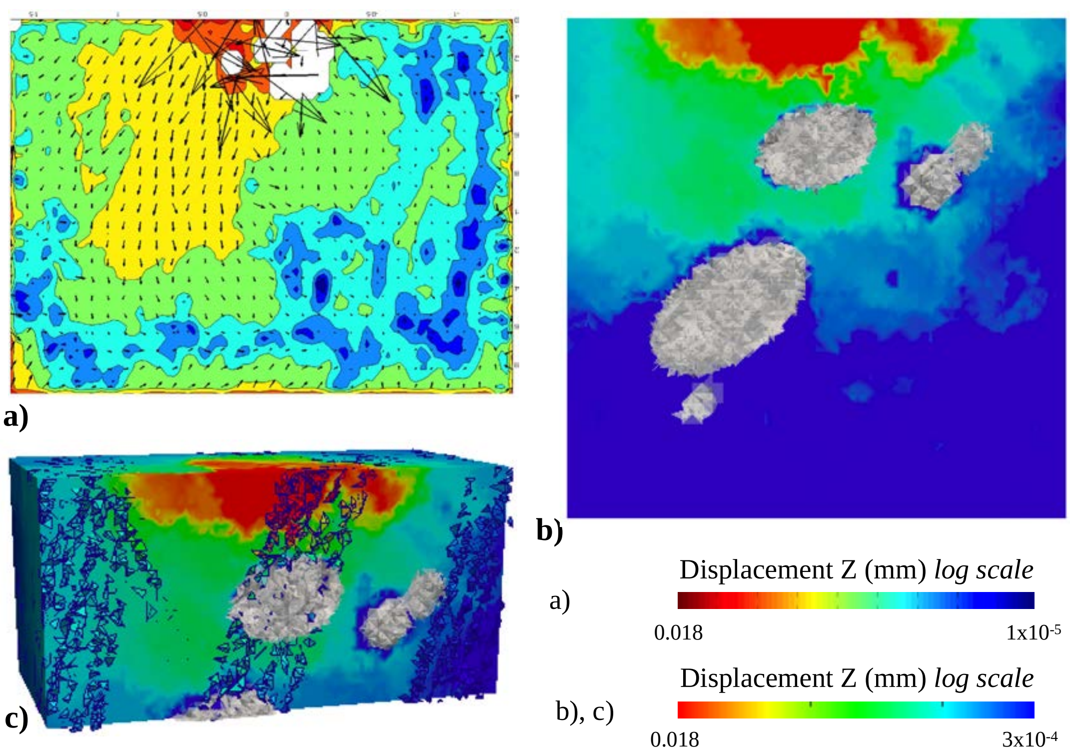
a) **b)**

Figure 4: Overall view of the structure in the numerical simulation: fibres (white), macro-pores (red) and micro-pores (orange) a) cross-section, b) surface view



a) b)

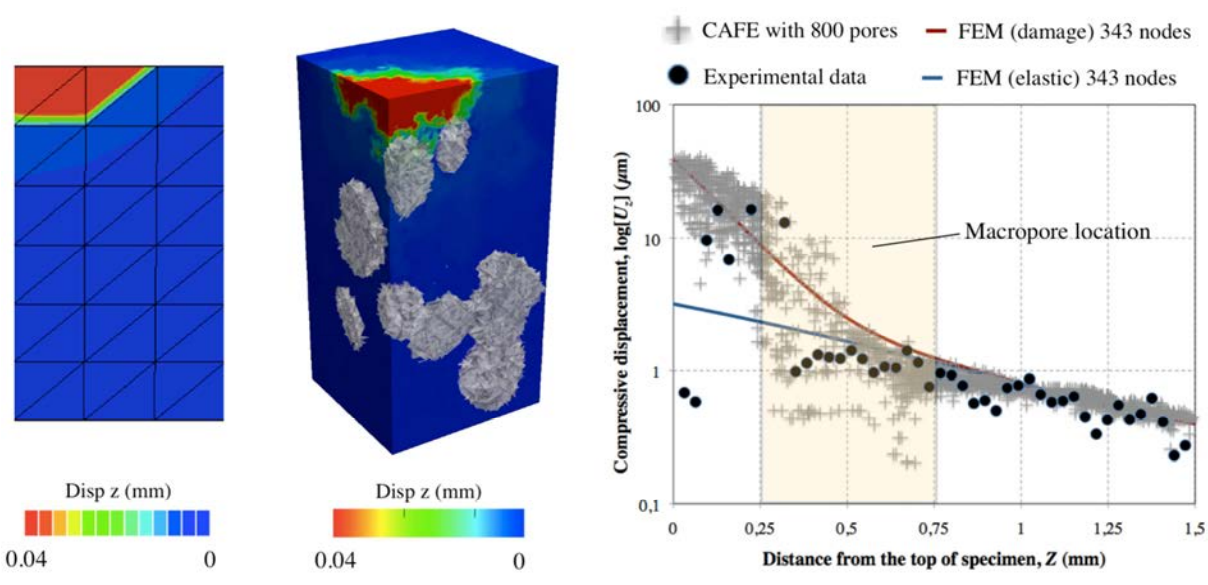
Figure 5: Comparison between experiment and CAFE simulation in a vertical section (3 mm × 2 mm) under the indentation: a) Experimental data with displacement vectors overlaid (white areas have poor correlation), CAFE simulation of displacements showing b) macro-pores and c) inter-fibre pores and macro-pores.



Further comparison is made using the compressive displacements and strains along the z-axis under the indentation (i.e. at x=1.5 mm and y=1.5 mm), which are shown in Figures 6 and 7 together with quarter slices of the CAFE numerical simulation in which the macro-pores are shown and the FEM that describes the continuum. As the CAFE simulation cells are not necessarily located exactly on the z-axis, the displacement data for all the cells within 1/30 of the side length (i.e. 100 µm) from the z-axis are shown. For the strain data in Figure 7c, due to the variations in the CAFE displacements, only representative strain values from cells on the z-axis are plotted. The agreement between the experiment, CAFE simulation and FEM-damage simulation is generally good, particularly remote from the indentation where the deformation is elastic. The data for a simple elastic FEM simulation for a reaction force of 275 N are also shown; agreement is good only in the elastic region. The variability in the CAFE displacements is due to the local effects of pores on the development of damage; there is a significant influence from the large pore that is beneath the indentation, which is generally consistent with the observed effect of a similar sized pore in the experimental data. The continuum FEM-damage simulation does not capture this behaviour.

The sensitivity of the CAFE simulation to the microstructure components is shown in Figure 8, which shows the effects of including either the inter-fibre pores or macro-pores, compared with a full simulation of both and the continuum FEM simulation with damage (all simulations contain micro-pores in the matrix). In the CAFE simulations, the dark gray colour denotes damaged cells in which the critical strain was reached. Comparing the simulations with and without macro-pores (Figures 9a and 9b), it can be seen that there is increased deformation and damage where a macro-pore is close to a bundle of inter-fibre pores. This is due to the stress concentrating effect of the macro-pores, which is also seen in the absence of fibres (Figure 8c), where larger displacements are observed in the vicinity of the macro-pores. The global pattern of deformation is similar in the CAFE and FEM simulations, but the inclusion of microstructure provides a higher fidelity description of the localisation of damage. This is a necessary ingredient of a model that can capture the effects of microstructure in damage propagation and failure of the composite material.

Figure 6: Comparison between the numerical and experimental vertical compressive displacement: a) FEM simulation with damage, b) CAFE simulation, c) displacements along z-axis



a) **b)** **c)**