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A multi-scale correlative investigation of ductile fracture

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ABSTRACT

The use of novel multi-scale correlative methods, which involve the coordinated characterisation of matter across a range of length scales, are becoming of increasing value to materials scientists. Here, we describe for the first time how a multi-scale correlative approach can be used to investigate the nature of ductile fracture in metals. Specimens of a nuclear pressure vessel steel, SA508 Grade 3, are examined following ductile fracture using medium and high-resolution 3D X-ray computed tomography (CT) analyses, and a site-specific analysis using a dual beam plasma focused ion beam scanning electron microscope (PFIB-SEM). The methods are employed sequentially to characterise damage by void nucleation and growth in one volume of interest, allowing for the imaging of voids that ranged in size from less than 100 nm to over 100 μm. This enables the examination of voids initiated at carbide particles to be detected, as well as the large voids initiated at inclusions. We demonstrate that this multi-scale correlative approach is a powerful tool, which not only enhances our understanding of ductile failure through detailed characterisation of microstructure, but also provides quantitative information about the size, volume fractions and spatial distributions of voids that can be used to inform models of failure. It is found that the vast majority of large voids nucleated at MnS inclusions, and that the volume of a void varied according to the volume of its initiating inclusion raised to the power 3/2. The most severe voiding was concentrated within 500 μm of the fracture surface, but measurable damage was found to extend to a depth of at least 3 mm. Microvoids associated with carbides (carbide-initiated voids) were found to be concentrated around larger inclusion-initiated voids at depths of at least 400 μm. Methods for quantifying X-ray CT void data are discussed, and a procedure for using this data to calibrate parameters in the Gurson-Tvergaard Needleman (GTN) model for ductile failure is also introduced.

1. Introduction

The nucleation and growth of void damage in metals is critical to our understanding of many types of failure, for example in ductile fracture or high temperature creep. In such cases, the conventional approach has been to use destructive serial sectioning to quantify damage levels at various times on the path to ultimate failure. More recently, X-ray computed tomography (CT) has opened up the opportunity of studying void growth on a single sample over time [1,2], for example during simple tensile tests [3]. Unfortunately, the spatial resolution of X-ray CT means that either the defects must be large or the samples small (the highest resolution achievable is approximately 1000th of the field of view using present 2000 pixel wide cameras [2]). This makes nucleation and growth difficult to study. In some cases, small cylinders of material have been extracted from specimens post-fracture and examined by CT to quantifying void shapes, sizes, volume fractions, etc, at higher resolution [4,5]. Others have utilised laminography [6] or special sample geometries [7] to help control stress conditions in smaller samples subject to CT examinations. However, the use of X-ray CT on its own is limited, in that while it can image some defects, it is often of too poor a resolution to image smaller inclusions from which damage nucleates, or to capture the nucleation. Furthermore, grain boundaries and other microstructural features that may play an important role, for example in creep cavitation [8], are invisible to CT. In order to gain a fuller picture of failure correlative

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approaches are required.

Multi-scale correlative imaging methods involve the characterisation of the features contained within a single volume of material across a wide range of length scales, using numerous complementary analytical techniques. They have been used, primarily in 2D, to great effect in the examination of structures in soft-tissue biological systems [9], and interest in their use in other areas of materials science is increasing rapidly. Recently, 3D correlative tomography methods have emerged [2], so that it is now possible to obtain multi-scale datasets even from challenging scenarios in which the volume of interest is hidden beneath the surface of a metallic sample [8,10]. In essence, high spatial resolution information from a volume of interest identified at lower resolution by X-ray computed tomography can be gathered using techniques such as focussed-ion beam serial sectioning, electron backscatter diffraction and transmission-electron microscopy. This information across all scales can be threaded together to generate a multiscale and multifaceted picture. Here, for the first time, we examine how the results of a 3D correlative approach can be used to provide greater insight into the mechanisms of ductile fracture.

The toughnesses of steels used in the construction of pressure vessels are of paramount importance, particularly when the components are critical to the safe operation of power plants. Historically, much attention has been devoted to the understanding of brittle fracture initiation and propagation in steels, and the associated ductile-to-brittle transition. However, there is now increasing interest in the ductile upper-shelf behaviour of pressure-vessel steels, since modern grades demonstrate such excellent start-of-life toughness that the onset of the brittle regime is less of a concern. It is well known that ductile tearing in steels proceeds through void nucleation, growth and coalescence. Large voids tend to nucleate and grow from inclusions in the microstructure (typically MnS, SiO_2 or Al_2O_3), and are joined together by sheets of smaller voids ('void sheets') nucleated at small particles such as carbides [11–14].[1] It is thought that the size and spatial distributions of voids, and their rate of growth, play a critical role in tearing behaviour. Equally, because it is assumed that the inclusions have no significant bond strength, they are often taken to represent the initial void population, meaning that their orientation and shape is also important [14,15].

In order to predict the ductile fracture behaviour of alloys, local mechanistic approaches like the Gurson-Tvergaard-Needleman (GTN) model are often utilised [16–19]. Derived from the original work of Gurson [16], with modifications by Tvergaard [17,18] and Needleman [19], the GTN model assumes that the material is homogeneous and behaves as a continuum. The voids are accounted for by influencing the global flow behaviour of the material and their effects on behaviour are averaged. Crucially, the model takes into consideration the dependence of yielding on the plastic strain and hydrostatic stress exerted on the material by introducing a strain softening term. This strain softening term accounts for the initiation, growth and coalescence of voids and is used in conjunction with the hardening of the matrix material which follows the Von Mises yield criterion. The model is defined by the following semi-empirical yield function, Φ:

$$\Phi(\sigma_e,\sigma_m,\overline{\sigma},f^*)=\left(\frac{\sigma_e}{\overline{\sigma}}\right)^2+2q_1f^*\cosh\left(\frac{3q_2\sigma_m}{2\overline{\sigma}}\right)-\left(1+q_3f^{*2}\right)=0 \tag{1}$$

where σ_e is the macroscopic von Mises stress, σ_m the macroscopic mean stress, and $\overline{\sigma}$ the flow stress for the matrix material. The constants q_1, q_2 and q_3 were introduced by Tvergaard [17,18] to better reproduce experimental observations. The function f^* was introduced by Tvergaard and Needleman [19] to account for the rapid loss of stress carrying capacity (and failure) that accompanies void coalescence, which the original model by Gurson did not account for. f^* is defined in terms of the void volume fraction f as follows:

$$f^*=\begin{cases} f & \text{for } f\le f_c \\ f_c+K(f-f_c) & \text{for } f>f_c \end{cases} \tag{2}$$

$$K=\frac{f_u^*-f_c}{f_F-f_c} \tag{3}$$

where f_c is the critical void volume fraction for void coalescence, f_F is the void volume fraction at final fracture, and f_u^* is ultimate value of f^* at final fracture, $f_u^*=1/q_1$. The function for f^* when $f\le f_c$ ensures that there is an acceleration towards the final volume fraction when f_c is exceeded. The value of f at the start of deformation is set as f_0, the initial void volume fraction of the material.The change in f with an increment of deformation, denoted $\dot{f}$, is the sum of the volume fraction increase due to growth of existing voids and the increase due to the nucleation of new voids. The rate of void growth is assumed to be related to the plastic part of the strain rate tensor $\dot{\varepsilon}_{ij}^p$, assuming the material is incompressible, whilst the void nucleation rate is related to the equivalent plastic strain rate $\dot{\varepsilon}_{eq}^p$ [20]:

$$\dot{f}=\dot{f}_{growth}+\dot{f}_{nucleation}=(1-f)\dot{\varepsilon}_{ij}^p+\Lambda\dot{\varepsilon}_{eq}^p \tag{4}$$

$$\Lambda=\frac{f_N}{s_N\sqrt{2\pi}}\exp\left[-\frac{1}{2}\left(\frac{\varepsilon_{eq}^p-\varepsilon_N}{s_N}\right)^2\right] \tag{5}$$

where f_N is the volume fraction of void-nucleating particles, ε_N the mean value of strain, and s_N the standard deviation. Hence, the nucleation of voids follows a normal distribution in which 50% of void-nucleating particles are assumed to have nucleated a void at the mean strain of ε_N [20].In summary, the primary parameters that require calibration in order to run a GTN simulation are the following: f_0, f_c, f_F (that describe void growth and coalescence to failure), ε_N, s_N, f_N (that describe void nucleation), q_1, q_2, and q_3 (that characterise material plasticity). Since GTN models are solved using finite-element method (FEM) approaches, another parameter, the crack-tip mesh size L_C, must also be determined – this is essentially the dimension of which behaviour is averaged. The adequate calibration of all these parameters is not an easy task. Typically it is achieved iteratively, by estimating values using the results of previous studies, running a finite element analysis using these parameters, comparing the simulation results to experiment, and adjusting the parameters accordingly before repeating. As a result, 'optimised' parameter values often bear little relation to the physical quantities that they are supposed to represent [21–26].In this paper, we describe an experimental multi-scale correlative method that is capable of quantifying the damage induced by ductile fracture. The approach enables the detailed characterisation of the

[1] In this paper, we adopt the following naming convention for voids, which helps to avoid potential confusion arising from only referring to their size: inclusion-initiated voids (IIVs), and carbide-initiated voids (CIVs), depending on their initiation site.