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A statistical assessment of ductile damage in 304L stainless steel resolved using X-ray computed tomography

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ABSTRACT

X-ray computed tomography (XCT) has been shown to reveal the true extent of ductile damage below the fracture surface of failed test specimens, which is often significantly underestimated when probed using 2D serial sectioning techniques and a microscope, since a single plane of material may only exhibit only a handful of resolvable voids. In contrast XCT offers the capability to generate large datasets consisting of hundreds, if not thousands, of individually resolvable voids, where each void can be characterised in terms of its distance to nearest neighbour, distance from the fracture surface, volume, orientation, amongst other parameters, or collectively in the form of void volume fraction. In order to probe the various stages of ductile failure in greater detail, where it may be possible to directly assign voids to the various stages of ductile failure using their individual geometric and location characteristics, it is essential that we determine methodologies to analyse such datasets. In order to give this study a material basis, we have employed the use of statistical methods to characterise differences in ductile void characteristics between two forms of stainless steel of the same grade (304L): one manufactured by hot isostatic pressing and the other by forging, since it has been shown previously that these materials display appreciably different fracture toughness properties. Through statistical sampling of the voids within these materials we aim to shed some light on the differences in mechanistic ductile fracture and the capabilities of XCT for probing mechanistic failure of materials.

1. Introduction

Fracture of metals can occur *via* a number of failure modes including: catastrophic failure (termed brittle) or *via* comparatively controlled tearing (termed ductile). The failure mechanisms are governed by its metallurgical and microstructural makeup, temperature, stress state, and loading conditions [1]. Ductile failure occurs *via* the nucleation, growth, and coalescence of voids in the material microstructure, which nucleate either at the interface between the matrix and foreign secondary phase particles or *via* particle cracking [2,3]. It is generally accepted that these voids grow under the influence of increasing plastic strain and hydrostatic stress [4–7] until their respective plastic zones interact with those of neighbouring voids; the ligament of material connecting the two voids fails once it reaches sufficient levels of strain. During ductile failure ahead of a pre-cracked tip, thousands of voids form in close proximity to the extending crack. Voids that are located directly ahead of the crack tip will be subjected to such high levels of plastic strain that coalescence occurs; voids that experience coalescence in close proximity to the fracture path through the material may become part of the fracture surface. In contrast, voids located out of the direct line of crack propagation will be subjected to lower levels of plastic strain and as such may only grow a relatively small amount. While the global fracture path is governed by the plastic zone and applied stress, the local fracture path is mediated by the statistical distribution of particles through the microstructure. On average, as distance increases from the crack tip (or below the fracture surface), one might encounter almost all forms of void growth, from voids partially exposed to the fracture surface to voids typically displaying 'just nucleated' characteristics at distances far remote from the fracture path, and this forms the basis for our investigations as we aim to explore the applicability of X-ray computed tomography (XCT) to probe ductile failure mechanisms in greater detail.

XCT is an increasingly powerful imaging technique that allows investigation of the internal structure of both naturally occurring and engineering materials. XCT is particularly well suited to the study of ductile damage during material failure, since it allows for the visualization (and crucially, the measurement) of the voids formed during the failure process [8–10]. There are few viable methods to characterise

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Table 1
Elemental composition of HIP (both powder and consolidated form) and Forged 304L.

		Grain size	Cr	Ni	Mo	Mn	Si	C	O/ppm	N/ ppm
304L	Spec. (wt%)	–	18.5–20.00	9.00–10.00	–	< 2.00	< 1.00	< 0.035	200	–
	Forged	94 μm	19.40	9.648	0.345	1.654	0.573	0.027	**15**	817
	Powder (wt%)	–	19.2	9.44	–	1.37	0.74	0.022	110	–
	HIP (wt%)	27 μm	19.5	9.45	0.01	1.33	0.72	0.022	**120**	840

voids formed during ductile failure in terms of their spatial distribution; one method is to section failed test specimens and image using a high-resolution microscope such as a scanning electron microscope (SEM). 2D representation of voids can misleading however, since sectioning of material may typically only reveal a handful of voids per slice of material and in addition to this, it is impossible to ascertain an accurate measure of void size, with voids appearing smaller than they actually are since material sectioning will rarely, if ever, split a void directly through its mid-point.

3D representation is possible by serial sectioning, either mechanically (*e.g.* Robo-Met.3D) or through the use of laser ablation [11] or Plasma focussed ion beam (FIB) [12,13]. Whilst the use of FIB is quite widespread, it is limited to small volumes on the order of 50 μm^3, this route offers the capability to resolve extremely small features and can generate crystallographic contrast to see grain structures. Plasma FIB [14,15] allows the access of increasingly large volumes of material up to 300 μm^3. In contrast, XCT allows for the fast scanning of comparatively large volumes of material on the order of mm, whilst providing microscale resolution, and is most appropriate for this study. XCT allows for the scanning of comparatively large volumes of material, where determination of void size and location relative to the fracture surface is governed by the detection limits of the XCT scanner and detector.

We have recently employed the use of XCT to probe the ductile void characteristics, and by extension the ductile failure mechanism, of stainless steel manufactured by forging and hot isostatic pressing (HIP) [16]. Because there has been a substantial amount of work already completed looking at comparisons in material behaviour between HIP and forged steels, the reader is directed to [17–19] for mechanistic insight (microstructures, chemistry, tensile and fracture toughness testing) into the differences between these two materials' fracture behaviour and we focus here instead on differences in ductile damage characteristics resolvable using XCT.

HIP is an advanced manufacturing technique which utilizes the production and consolidation of metal alloy powder of required chemistry, subjected to high temperature and isostatically controlled pressure, to produce bulk metal that typically displays isotropic microstructure and uniform material properties throughout the bulk component [20,21]. HIP is extremely well suited to the manufacture of complex geometric components without the need for additional machining and welding stages and offers the capability to join dissimilar materials in a more favourable fashion to conventional arc welding. While HIP is clearly a more advanced form of manufacture over conventional forging and casting, there remain fundamental questions surrounding microstructure [22], material properties, and fracture behaviour [17] that require thorough mechanistic exploration if HIP is to make an impact in future nuclear manufacturing, which is rightly governed by safety case regulation. This means that new manufacturing approaches, regardless of their recognized improved performance and reduced costs, must be met with rigorous fundamental metallurgical and material assessment if they are to replace more conventional 'tried and tested' routes to manufacture.

Herein we have extracted material from the ductile tearing region of failed fracture toughness test pieces and imaged the cores using XCT so as to characterise and quantify the level of ductile damage in HIP and forged variants of 304L stainless steel, in order to shed light on the materials' differing fracture toughness properties. Due to the large number of resolvable voids in these test pieces, which all play a part in the failure mechanism to a certain degree, we have employed the use of statistical models in order to establish relationships between void characteristics, namely void size, distance to nearest neighbour, and distance from the fracture surface.

2. Experimental

2.1. Material and microstructures

The material examined was HIP 304L material, supplied by Areva, and forged 304L stainless steel by Creosote Forge et Creusot Mécanique, Areva (Le Creusot, France). For HIP304L, stainless steel grade 304L argon gas atomised powder was heated from ambient temperature to 1423 K (1150 °C) at a rate of 633 K (360 °C) h^{-1} and held at 1423 K (1150 °C) and 104 MPa for a period of 180 min under an inert argon atmosphere. Post-HIP heat treatment of HIP304L was performed by heating from room temperature to 1343 K (1070 °C) at 633 K (360 °C) h^{-1}, held for 280 min, and water quenched. Forged 304L pipe was subjected to similar heat treatment as the HIP materials (1343 K (1070 °C), for *ca.* 250 min) and water quenched.

The materials' elemental compositions (wt%) and grain sizes are tabulated in Table 1; elemental analysis was performed by The Welding Institute (TWI, UK) by inert gas fusion technique on an ELTRA ONH-2000. Grain size measurements were conducted in accordance with ASTM E112-96.[23]

The microstructures of the materials studied are shown in Fig. 1. HIP 304L displays an appreciable number of fine oxide inclusions in the microstructure, whereas F304L displayed a relatively cleaner microstructure. It is these oxide inclusions which are thought to act as initiation sites for the formation of voids during ductile failure. The origin and mechanistic effects of these inclusions on fracture have been previously reported on in great detail [17,18], and the microstructures are only included here to place some context on the results presented.

J-R fracture toughness testing was performed on F304L and HIP304L, the experimental details and results of which are presented in a previous study [24]. Specimens selected from the fracture toughness testing for analysis were loaded to an applied J and crack growth of 742 kJ m^{-2}/0.454 mm and 672 kJ m^{-2}/0.367 mm, for HIP304L and F304L, respectively. These specimens were selected as they exhibited the smallest degree of crack growth and loaded to the smallest applied J, in an attempt to assess specimens which were loaded to within the J_{max} validity limit of 738 kJ m^{-2}, as determined using ASTM E1820 [25].

Specimens prepared for metallurgical analysis were sectioned, mounted, ground, and polished in accordance with the recommended procedures in ASTM practice E3-01 [12]. Electron microscopy was performed using an FEI Quanta 650 ESEM equipped with a field emission gun. The SEM was performed under vacuum using a 20 kV accelerating voltage and a spot size of 4.0 nm, at a working distance of *ca.* 10 mm.

2.2. Tomography specimen preparation

Cylindrical core specimens were extracted from failed compact