In Situ Diagnostics of Damage Accumulation in Ni-Based Superalloys Using High-Temperature Computed Tomography

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The design, operation, and performance of a laboratory-scale X-ray computed tomography arrangement that is capable of elevated-temperature deformation studies of superalloys to 800 °C and possibly beyond are reported. The system is optimized for acquisition of three-dimensional (3D) backprojection images recorded sequentially during tensile deformation at strain rates between 10^{-4} and 10^{-2} s^{-1} , captured *in situ*. It is used to characterize the evolution of damage—for example, void formation and microcracking—in Nimonic 80A and Inconel 718 superalloys, which are studied as exemplar polycrystalline alloys with lesser and greater ductility, respectively. The results indicate that such damage can be resolved to within 30 to 50 μm . Collection of temporally and spatially resolved data for the damage evolution during deformation is proven. Hence, the processes leading to creep fracture initiation and final rupture can be quantified in a novel way.

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I. INTRODUCTION

NICKEL-BASED superalloys are ubiquitous in development of high-temperature systems for use in jet propulsion and power generation more broadly. The overarching reason for this is their superior properties—both mechanical and chemical—at operating conditions which can approach 800 °C or beyond. There are significant technical, commercial, and legislative pressures requiring enhancement of their high-temperature performance. Improvements in fuel economy and thus CO₂ emissions are a significant driver for this.

Due to the nature of these applications, great emphasis is placed on characterization of the behavior of superalloys under tensile, fatigue, as well as creep loading conditions. Traditionally, such testing has emphasized measurements of force and displacement to deduce the stress and strain fields; this can be regarded as a form of macroscopic characterization. Microscopic analysis is

usually carried out *post mortem* on, for example, failed^[1–5] or interrupted test pieces^[6–11] and most commonly at ambient temperature. However, a significant difficulty with this approach is that one can never be totally sure that damage deduced *post mortem* is truly representative of that driving deformation under operating conditions at much higher temperature. Ideally, better measurements of damage accumulation are needed, measured *in situ*. A significant advantage of this would be direct determination of the dynamics of damage accumulation, which would lead to better understanding of the constitutive response and indeed more accurate data for calibration of modeling activities.

The research reported in this paper was carried out with these ideas in mind. The operation and performance of a laboratory-scale X-ray computed tomography (XCT) arrangement are reported. It is shown that the system is capable of *in situ* characterization of damage during loading at temperatures up to at least 800 °C. We describe the design and arrangement of the apparatus, its operational characteristics, and its performance during the high-temperature testing of two common superalloys, viz. Nimonic 80A and Inconel 718. The advantages and limitations of the new approach are discussed.

II. EXPERIMENTAL METHODS

A laboratory-scale X-ray computed tomography (XCT) system, developed specifically for *in situ*

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observations and integrated with a thermomechanical testing instrument, was employed. It has been optimized for acquisition of 3D backprojection images during tensile deformation at temperatures of up to 800 °C and strain rates between 10^{-4} and 10^{-2} s^{-1} . The system, depicted in Figure 1, consists of (1) an X-ray source (a) and detector (b), (2) an all-electric thermomechanical testing instrument (c) including a rotary table (d) and a furnace (e), and (3) integration algorithms for reconstruction of sequential 3D, or so-called four-dimensional (4D), images by implementing the inverse Radon transformation procedure.

A. *In Situ* XCT System: Source and Detector

To capture backprojection images, a microfocus X-ray source by Hamamatsu Photonics—with capacity of 150 kV and 500 μA —is paired with a scientific complementary metal-oxide semiconductor (sCMOS) X-ray detector by Photonic Science. These were aligned precisely at right angles to the tensile axis of the testing specimen. Specimens with circular cross-section were chosen and gripped at both ends by connecting rods mounted onto a rotary rotor system. The rotor casing was bolted to the lower crosshead and controlled using a closed-loop feedback system to maintain a torque-free condition in the specimen during the test. The X-ray source and detector, as well as the testing instrument, were fixed, allowing the design of a relatively compact system to capture uninterrupted multiangle backprojection images. To the best of the authors' knowledge, this newly proposed system represents an improvement over

the conventional method, which calls for the testing instrument to be mounted onto and rotated by a rotating floor;^[12,13] such systems are probably impractical for a laboratory setup.

The primary goal of the present study was testing at elevated temperatures. A furnace was chosen for this purpose, since temperature stability is very important; this was installed and attached to the testing instrument. The power and size of the furnace were designed to maintain the controlled temperature with error of less than 1 °C. The furnace was designed with a set of quartz windows to secure an unobstructed path for the X-ray beam passing from the source through the specimen onto the detector screen. To ensure that the system could acquire high-quality backprojection images, the source was positioned as close as practically possible to the specimen, while maintaining a sufficient distance to avoid any direct exposure of the X-ray source to heat radiated from the furnace. In addition, a water cooling system has been specifically designed to cool the clamping flanges of the connecting rods to protect the crossheads from excessive conductive heat transfer. The furnace and the cooling system were designed so as to obtain a homogeneous temperature profile within the sample, from 795 °C to 805 °C, during tensile tests and XCT imaging at 800 °C.

B. Rotary and Displacement Control

Figure 2 illustrates the operating workflow needed for *in situ* XCT observations. Two main steps are executed: (i) capture of backprojection images by rotating the

Fig. 1—*In situ* XCT setup for high-temperature mechanical experiments; (a) X-ray source, (b) X-ray detector, (c) testing machine, (d) rotary table, (e) furnace, (f) specimen (inside the furnace).

Fig. 2—Experimental procedure for *in situ* XCT observation.

Fig. 3—XCT image examples: (a) raw projection image, (b) cropped image of the gauge area after flat-field correction, and (c) calculated sliced image.

sample and (ii) application of tensile load to the sample without rotation. In this procedure, the sample has to be rotated prior to and whilst imposing a displacement-controlled signal. To optimize the rotary setup with the embedded water-flow cooling system, the rotation was controlled to reach 180 deg angular rotation, then directed back and forth alternately. To avoid specimen damage by the torque generated by the rotating offset between the top and bottom rods, the rotation was operated in stepwise fashion, during which backprojection images of each angle were captured sequentially. The rotation for an XCT step required 30 to 60 minutes. After capturing the backprojection images, the displacement was then applied incrementally. This working sequence was continued until the specimen fractured. In the work reported herein, tensile strain rates of 10^{-4} , 10^{-3} , and 10^{-2} s^{-1} were applied at 800 °C.

C. XCT Analysis

Each raw backprojection image was corrected by the flat-field correction method (*e.g.*, Reference 14) prior to the 3D reconstruction process. To eliminate noise, these images were corrected by averaging 3×3 pixels. From the corrected images, sliced images were generated by employing the inverse Radon transformation procedure. In this case, to produce the sinogram, we used the filtered backprojection method^[15] coded in MATLAB. ImageJ^[16] was used to render the calculated images, and Amira software to reconstruct 3D XCT voxels from those data. Figure 3 illustrates some examples of the raw projection, the flat-field corrected, and the calculated sliced images. The flat-field corrected images were used to observe the 3D profile changes—including the necking behavior—during high-temperature experiments; this

Table I. Composition of Studied Alloys in Weight Percent (Ni Base)

Name	Cr	Al	Ti	Fe	Nb	Co	Mn	Cu	Si	C	B	S
Nimonic 80A	20.0	1.5	2.5	0.2	—	0.14	0.01	0.01	0.03	0.07	0.003	0.002
Inconel 718	18.3	0.6	0.9	18.7	5.2	0.26	0.11	0.08	0.07	0.04	0.04	—

Fig. 4—Microstructure of Nimonic 80A (a) and Inconel 718 (b) employed in this work.

method is in line with the optical system as reported in References 17, 18 and 19. Furthermore, the backprojection images were cropped to focus on the gauge area and applied for the 3D reconstruction process. The data acquired in this way were examined critically for evidence of void formation and damage evolution. These *in situ* XCT results were also then compared with further data acquired by *ex situ* XCT observations and scanning electron microscopy (SEM) fractography studies of the traditional type. *Ex situ* XCT analyses were carried out using a North Star Imaging system at 150 kV and current of 27 μ A, which produced a voxel size of 4.9 μ m.

D. Testing Materials

For *in situ* XCT observation, high-temperature tensile experiments were conducted for polycrystalline Ni-based superalloys, viz. Inconel 718 and Nimonic 80A, with the compositions presented in Table I. Inconel 718 was solution treated at 955 °C for 1 hour followed by water quenching (AMS 5662), whilst Nimonic 80A was solution treated at 1080 °C for 15 minutes then water quenched (BS 4HR 601:2010). As these samples were not aged after heat treatment, minor phases were not significantly grown. These materials were chosen to demonstrate the capabilities of the arrangement, on account of their widespread use but also due to their anticipated greater and less creep ductility, respectively. As shown in Figure 4, the grain size in Nimonic 80A and Inconel 718 was 100 to 200 and 30 to 50 μ m, respectively. The specimens were machined from bar samples. The gauge length was chosen to be 2 mm, and after some experimentation, the diameter was fixed at 1.5 mm (Figure 1(f)). The dimension was selected as for the XCT detector to allow the

Fig. 5—Comparison of stress-strain measurement when rotating the sample while keeping the displacement (displacement-controlled mode) or load (load-controlled mode).

X-ray beam to fully penetrate the specimen for optimum capture of damage accumulation.

III. RESULTS

A. Rotary Control for XCT Imaging

Rotation of the specimen is a critical aspect of any CT system. For this reason, experiments were first carried out to prove that the control for this was adequate. A preliminary tensile testing program was carried out in both load- and displacement-controlled modes. Figure 5 illustrates some different features which appeared in the

Fig. 6—True stress–true strain curves during tensile tests at 800 °C for (a) Inconel 718 and (b) Nimonic 80A. For both cases, tensile experiments were carried out at strain rates from 10^{-4} to 10^{-2} s^{-1} .

corresponding stress–strain curves. As shown in this figure, the stress relaxed in the displacement-controlled mode at the temperature of 800 °C, but in comparison, the strain increased drastically in the load-controlled mode. In the latter case, the strain increments were abrupt, and this made the sample unstable with regard to deformation in the tensile axis direction and also resulted in blurred backprojection images, unsuitable for the 3D reconstruction process. Furthermore, as the temperature reached 800 °C, the applied constant load was clearly too excessive, leading to creep fracture in relatively short times. Considering these results, the displacement-controlled mode is thought to be more suitable for the *in situ* XCT observations intended in this study. In what follows, displacement control was used.

B. True Stress–True Strain Curves, and Mapping of Local Thinning of Cross-Sections

The stress–strain curves obtained at 800 °C for the three strain rates employed are illustrated in Figure 6. The fracture strain and strength values obtained are similar to those in literature (References 20, 21 and 22). The stress relaxation observed was caused by the rotation step needed in order to capture the XCT images sequentially. Stress fluctuations were observed for both Inconel 718 and Nimonic 80A, with a clear tendency for strain rate dependence.

Figures 7 and 8 illustrate the cross-section profiles of Inconel 718 and Nimonic 80A during tensile tests at strain rates ranging from 10^{-4} to 10^{-2} s^{-1} . The indicated values—denoted on each profile line—describe the true strain. As indicated, Inconel 718 exhibited a prolonged necking contour prior to the onset of fracture, whilst Nimonic 80A experienced relatively sudden fracture behavior, indicative of more brittle behavior. This was identified consistently for all the applied strain rates. Furthermore, the necking curvature of Inconel 718 was smallest when the system was tested at the lower strain

rate of 10^{-4} s^{-1} , becoming shallower as the strain rate was increased.

From Figures 7 and 8, the evolution of the cross-sectional area of Inconel 718 and Nimonic 80A demonstrated distinctive necking profiles. To take into account the necking profile, the true stress and strain were calculated using the actual diameters from the profile data (Figures 7 and 8). This is because the values plotted in Figures 7 and 8 are the averaged strain within the gauge area, whilst the data calculated using the minimum diameter from the profile reflect the actual strain for the tensile direction in the thinned or necked region. The results are then plotted in Figure 9. For Inconel 718 in the 10^{-4} s^{-1} and 10^{-3} s^{-1} cases, the stress decreased against the increment of strain prior to the onset of fracture. For Nimonic 80A, the stress values were generally more constant for the strain rates of 10^{-3} s^{-1} and 10^{-2} s^{-1} , but at 10^{-4} s^{-1} , stress relaxation was still observed to a degree. The ductility behavior is summarized and plotted in Figure 10. Nimonic 80A was generally less ductile with strain at fracture of around 0.3 at strain rates from 10^{-4} s^{-1} to 10^{-2} s^{-1} . Inconel 718 exhibited more ductile behavior, and the strain at failure increased from less than 0.8 to around 1.0 when the strain rate was decreased from 10^{-2} s^{-1} to 10^{-4} s^{-1} .

C. XCT Results

During the high-temperature tensile experiments, backprojection images were captured to allow for subsequent 3D XCT reconstruction so that the damage accumulation behavior that affects strength could be better understood. To distinguish voids or cracks from artifacts, a number of sequential sliced images were compared. If black patterns could be observed at the same location within more than four sequential images, they were defined as defects, and *vice versa*.

Figure 11 shows the reconstructed 3D XCT images of both materials just before fracture when tested at strain rates between 10^{-4} and 10^{-2} s^{-1} . Overall, Inconel 718

Fig. 7—Sample profile curves against the vertical position, measured at each step of strain (denoted in the upper-right figure legends) during tensile tests at 800 °C for Inconel 718 at strain rate of (a) 10^{-4} s^{-1} , (b) 10^{-3} s^{-1} , and (c) 10^{-2} s^{-1} . Sample profiles are presented as the diameter at each vertical point, as illustrated in the XCT image example (right).

showed more prolonged necking behavior than Nimonic 80A, whilst Nimonic 80A exhibited more surface cracks than Inconel 718. Surface cracks were observed for all three strain rates for Nimonic 80A, while a large crack was observed only at 10^{-4} s^{-1} for Inconel 718. As illustrated by the arrows, at the highest strain rate, Nimonic 80A exhibited more cracks than other cases. As the size of the cracks observed at 10^{-4} s^{-1} was as small as 30 to 50 μm , the authors believe that this represents the limit of the spatial resolution which can be achieved for nickel-based superalloys. For both grades of superalloy and at 10^{-4} s^{-1} , the surface cracks were larger than at slower strain rate, being above 500 μm .

More detailed features including the damage evolution were revealed by comparing the *in situ* XCT results obtained in several strain steps before the onset of fracture. Figures 12 and 13 show the 3D reconstructed and two-dimensional (2D) sliced images of XCT data for Inconel 718 during tensile loading at 10^{-4} and 10^{-2} s^{-1} . In both cases, the results are shown for three

steps of the strain before fracture. For both strain rates, necking behavior was observed before cracks or voids were observed. At 10^{-4} s^{-1} , at $\varepsilon = 0.81$, a surface crack with size of around 300 μm as well as void growth were observed only in the necked region. Those defects grew and coalesced at $\varepsilon = 0.94$, just before the onset of fracture. On the other hand, at 10^{-2} s^{-1} , void formation was observed at many slice positions along the tensile direction. Those voids started to nucleate at $\varepsilon = 0.64$ and grew up to 50 to 100 μm in size. The voids observed in different sliced images enlarged separately without merging into each other. Compared with the slower strain rate, the growth of voids occurred in a rather dispersed fashion and they were not concentrated within the necked region.

Similar analyses were conducted also for Nimonic 80A. Figures 14 and 15 show the 3D and 2D images of *in situ* XCT results for Nimonic 80A during tensile loading at 10^{-4} and 10^{-2} s^{-1} at the three strain stages prior to the onset of fracture. Contrary to Inconel 718, Nimonic 80A exhibited no apparent necking behavior

Nimonic 80A

Fig. 8—Sample profile curves against the vertical position, measured at each step of strain (denoted in the upper-right figure legends) during tensile tests at 800 °C for Nimonic 80A at strain rate of (a) 10^{-4} s^{-1} , (b) 10^{-3} s^{-1} , and (c) 10^{-2} s^{-1} . Sample profiles are presented as the diameter at each vertical point, as illustrated in the XCT image example (right).

or void growth before fracture. Instead, formation and growth of surface cracks were observed. At 10^{-4} s^{-1} , only two surface cracks with size of 100 to 200 μm had nucleated at $\epsilon = 0.29$. They propagated inwards and on the surface at 40 to 50 deg to the tensile axis. The grown cracks were as large as 500 to 700 μm at $\epsilon = 0.37$. However, at 10^{-2} s^{-1} , as many as 7 crack nucleation events were observed over a wider range of the surface. They enlarged separately and grew to be 50 to 100 μm . The direction of those cracks was perpendicular to the tensile axis.

More detailed features in fractured specimens were examined using *ex situ* XCT observation. Figure 16 illustrates fractured Inconel 718 and Nimonic 80A samples after tensile testing at strain rate of 10^{-3} s^{-1} . As

illustrated by arrows in this figure, fractured samples also showed some internal voids near the fracture surface. Although some voids were observed in Inconel 718 also for other strain rates, Nimonic 80A did not include any internal voids near the fracture surface.

These fracture surfaces were also examined using conventional SEM fractography analyses. Figure 17 illustrates the fracture surfaces of the same samples. In Inconel 718, voids on fractured surfaces with similar features to other works^[23] were observed. At the highest strain rate, more voids were observed and the fracture surfaces were rougher. Comparing these fractography analyses with the *ex situ* XCT results mentioned above, at the strain rate of 10^{-2} s^{-1} , internal voids generated

Fig. 9—True stress–true strain plots obtained from Fig. 6 and recalculated true stress–true strain plots from the necked profile for Inconel 718 and Nimonic 80A. Plots denoted with “profile” indicate the recalculated values from necked profile data.

near the necked region during the tensile tests were rapidly grown to coalesce and created the rougher fracture surface. For Nimonic 80A, intergranular cracks were observed on the fracture surface as for other works.^[22] Many of the intergranular cracks were observed near the rim rather than the center of the fractured surface.

IV. DISCUSSION

A. On the Spatial Resolution Achieved in the High-Temperature XCT System

In this study, a new *in situ* XCT system has been shown to be capable of revealing the time dependence of the morphological evolution of both internal and

Fig. 10—Fractured strain values obtained from Fig. 9.

Fig. 11—Reconstructed 3D XCT images of Inconel 718 and Nimonic 80A samples just before fracture at 800 °C under 10^{-4} to 10^{-2} s^{-1} . Arrows indicate detected surface cracks.

external features during specimen testing. Particular novelty arises from the use of high-temperature testing using a furnace arrangement; although some high-temperature tests using a furnace have been reported,^[12] to the best of the authors' knowledge, there have been very few attempts at high-temperature mechanical tests for *in situ* XCT using such a method. This feature makes it particularly suitable for study of nickel-based superalloys, for which deformation over significant periods of time is needed. Two distinct grades (IN718 and Nimonic 80A) were used here, being tested at strain rates between 10^{-4} and 10^{-2} s^{-1} . Evidently, although alternative techniques such as the 2-dimensional digital image correlation (DIC) method are capable of

providing surface information (References 24, 25), these are not suitable for 3-dimensional measurements of the type reported here, or high-temperature experiments over extended periods of time.

Tensile testing at 800 °C has been used for our verification studies. Evidence has been presented to confirm that external features such as localized thinning/necking and surface cracking can be detected and quantified. But, furthermore, the XCT system made it possible to observe the growth of internal defects during the high-temperature experiment. One can confirm this succinctly by comparing images obtained in the *in situ* arrangement and those obtained *ex situ*, thus taking advantage of the absence of the furnace arrangement which—one might think—would compromise the resolution. Figure 18 illustrates several sliced images obtained by *ex situ* and *in situ* XCT of a sample fractured at 10^{-2} s^{-1} under 800 °C. *Ex situ* XCT images were obtained at room temperature using a distinct North Star ImagiX system which is capable of producing 3D images at resolution of 5 to 10 μm , since the current was as low as 27 μA . As is apparent, although the *in situ* XCT images obtained during the high-temperature tensile test exhibit blurring when compared with the *ex situ* XCT images, the *in situ* XCT system is nonetheless capable of revealing internal defects at spatial resolution of 30 to 50 μm .

In practice, the resolution of backprojection XCT images is affected by factors including the image unsharpness (U_g) induced by the focal spot, the movement of the object, and the quality of the detector (Reference 26). As discussed in the previous section, during the *in situ* XCT observation, the sample was fixed in displacement-controlled mode, which thus avoided any effects of creep deformation. Therefore, in this study, any influence of object movement can be confidently ignored as a factor influencing the lack of image sharpness. Assuming that the detector effect could also be eliminated, the major factor limiting resolution is then the focus unsharpness. This value depends upon the distance between the source and the object D , the distance between the detector and the object d , and the focal spot size of the X-ray source F via the following equation: $U_g = F \times \frac{d}{D}$. Since the focal spot size in the present X-ray source with 150 kV and 500 μA is around 50 μm , considering that the distance from the object to the source was longer than that to the detector, the unsharpness is expected to be less than 50 μm , consistent with the observed results in Figure 18. In high-temperature XCT using the present system, even though the X-rays pass through the furnace window with a longer distance from the source to the detector, sufficient X-ray energy penetrating through the sample must be captured to obtain clear XCT images or to distinguish defects. Although the resolution might be improved in principle by capturing backprojection images with lower current for longer time, that might affect the object movement due to the creep fracture which might occur while imaging for longer times at high temperatures.

Fig. 12—*In situ* XCT results for Inconel 718 at 10^{-4} s^{-1} with true strain obtained from profile data (Fig. 7). Circles indicate voids or cracks.

Fig. 13—*In situ* XCT results for Inconel 718 at 10^{-2} s^{-1} with true strain obtained from profile data (Fig. 7). Circles indicate voids.

B. On Internal Voids in Inconel 718

As observed in the high-temperature tensile tests on IN718, the growth of internal voids was imaged differently depending upon the strain rate employed. To quantify the size and distribution of the voids, Amira software was used to visualize differences between the strain rates. Figure 19 illustrates this quantitative

information about voids as distinguished by considering a range of intensity thresholds. As indicated from the sliced images, in the higher strain rate regime, voids were generated along the tensile direction in a rather dispersed fashion. From these 3D maps of voids, the void size distribution can be evaluated as a function of distance from the necked region, as shown in Figure 20.

Fig. 14—*In situ* XCT results for Nimonic 80A at 10^{-4} s^{-1} with true strain obtained from profile data (Fig. 8). Circles indicate voids.

Fig. 15—*In situ* XCT results for Nimonic 80A at 10^{-2} s^{-1} with true strain obtained from profile data (Fig. 8). Circles indicate voids.

While at 10^{-4} s^{-1} voids were found only in the necking region, at 10^{-2} s^{-1} voids were in general smaller and distributed over a wider volume of the gauge. It seems very likely that these internal defects induced the differences in true stress–true strain plots calculated from the necked profiles, as shown in Figure 9. The larger voids at the slower strain rate made the actual cross-section area less, thus contributing to the stress drop before fracture in the figure.

According to Hull and Rimmer,^[27] the stress applied to the void surface increases the diffusion flux needed for void growth. From the profile results in the present study, the necking curve was more apparent and deeper at slower strain rate. These profile differences can affect the void growth behavior in the necking part, as introduced in other work on ductile materials with a different gauge shape.^[28] Hayhurst *et al.* mentioned that, in a high triaxial stress field, when the levels of applied load are high, softening due to continuum cavity

growth predominates over cavity nucleation.^[29] At the slower strain rate, the necked region in the gauge is then likely to promote cavity growth more than at the higher strain rate case, due to the necked curvature created.

The stress was concentrated in the necked region at the slower strain rate, which caused growth of voids to be observed only in that region. On the other hand, at 10^{-2} s^{-1} , the necking curve was comparatively more shallow and the tensile stress affected both the cavity nucleation and the cavity growth over a wider range. Dyson illustrated that the ductility is inversely related to the increase in cavity number per unit strain.^[30] Our results are consistent with Dyson's work, since at the higher strain rate in this study, the ductility exhibited lower values than for the slower strain rate. Considering that the higher strain rate induces higher stress during the tensile tests, these cavities were grown rapidly over a wider range at the high strain rate.

C. On Surface Cracks in Nimonic 80A

In case of Nimonic 80A, intergranular cracks were observed on the fractured surface rather than internally. According to the fractography analysis and *ex situ* XCT images of fractured sample obtained at room temperature, surface cracks originated in an intergranular manner from the rim of the sample gauge. At similar temperature conditions, Sharma *et al.*^[31] mentioned similarly that cracking tended to initiate around γ' -phases and created intergranular fractured surface, which is consistent with our results. Since Nimonic 80A exhibited less ductility than Inconel 718, one can say that cavity nucleation occurred more favorably than for Inconel 718. Such cavitation can occur preferentially at grain boundaries lying transverse to the tensile direction.^[32] Since our results showed that the initiated cracks were largely perpendicular to the tensile direction, tensile cracks were produced by such void coalescence

Fig. 16—XCT images of Inconel 718 and Nimonic 80A after high-temperature tensile tests (at strain rate of 10^{-3} s^{-1}). Arrows indicate internal voids.

Fig. 17—SEM images of fractured surfaces after high-temperature tensile tests.

Fig. 18—*Ex situ* and *in situ* XCT sliced images of a fractured sample (Inconel 718, 10^{-2} s^{-1}).

at the grain boundaries. For damage creation with cavity nucleation dominating, other works have also shown that cracks were initially preferentially at the sample surfaces.^[29]

Thus Nimonic 80A also exhibited damage accumulation which depended upon the strain rate conditions. At slower strain rate, a smaller number of surface cracks were formed and they grew to much larger size, whilst higher strain rates generated a larger number of smaller cracks on the surface. Necking behavior was not as apparent as for Inconel 718, but the slower strain rate in Nimonic 80A also caused a deeper necking profile than in the higher strain rate case. Due to the comparatively more necked profile, at slower strain rate, the tensile stress was concentrated in the necked part, which resulted in the cavity growth and eventually significant cracking in this region.

D. On the Advantages and Limitations of the Devised System

This paper has presented the details of and performance of an XCT system which is capable of observing void growth and crack generation even at temperatures as high as 800 °C. Such capability was achieved using a radiative furnace and not using forms of inductive heating as emphasized in other works (*e.g.*, References 33, 34). Furthermore, the loading capacity of this system is up to 10 kN even at such high-temperature conditions. This is versatile compared with other reported works—for example, a maximum load of 2 kN at temperatures up to 1750 °C^[35] or 5 kN tensile loading capacity with the temperature limited to 250 °C.^[36] The devised *in situ* XCT system is applicable to a much

Fig. 19—XCT analyses of voids detected in Inconel 718 before fracture at strain rate of (a) 10^{-4} s^{-1} , (b) 10^{-3} s^{-1} , and (c) 10^{-2} s^{-1} .

Fig. 20—Void size distribution before fracture for Inconel 718. Horizontal axis shows the distance from the necked region. (a) 10^{-4} s^{-1} , (b) 10^{-3} s^{-1} , and (c) 10^{-2} s^{-1} .

wider range of experiments and materials than hitherto possible. The new technique brings with it some significant advantages, which enable our understanding of the strain rate effects on the growth of voids/cracks to be enhanced. The damage accumulation process or

mechanical responses to such damage might be clarified in more detail *via* further investigations. Within the resolution of 30 to 50 μm , it is possible to pursue the development of defects slower than the present study if the applied step displacement was smaller than 0.1 mm. This could also include real-time understanding of the damage effects on the creep behavior, which is impractical by the *ex situ* technique because one cannot predict when the creep curve might start changing.^[11]

Finally, it is fair to identify some limitations associated with the XCT procedure. Figure 21 compares the obtained stress-strain curves with and without *in situ* XCT—*i.e.*, interrupted *vs* monotonic loading. Independently of the material employed, when deforming at fast strain rates (10^{-2} s^{-1}), the maximum stress post-interruption was lower than the stress values obtained under monotonic loading for the same values of strain. This indicates that some damage occurs during the interruption necessary to rotate the sample for XCT purposes—*i.e.*, when deformed without interruption, there was not enough time for damage to occur. In fact, according to some literature,^[29,37,38] 600 to 700 MPa at 800 °C for Inconel 718 and Nimonic 80A which was measured at 10^{-2} s^{-1} could induce creep fracture in a few hours, while under 400 to 500 MPa, applied stress values at 10^{-4} s^{-1} before XCT steps, the creep fracture would take more than 10 or 100 hours. It seems likely that this effect is responsible for the lower values of strain to failure measured under interrupted experimentation. At slow strain rates (10^{-4} s^{-1}), the behavior was different. For both materials, the values of stress pre- and post-interruption match those obtained under monotonic loading—in this case, the strain rate was low enough that damage occurs both monotonically and with the interruption needed for XCT. In terms of ductility, Inconel 718 showed similar strain to failure independently of the mode of testing. However, for Nimonic 80A, the sample tended to fracture immediately after surface cracks were observed. In those cases for which fracture was caused by cracks in the surface, the rotation necessary for XCT might conceivably enhance the damage. This is believed to be responsible for the lower ductility observed when deforming uninterruptedly at low strain rates.

V. SUMMARY AND CONCLUSIONS

The design, operation, and performance of a laboratory-scale X-ray computed tomography system, capable of quantifying damage processes arising during the high-temperature deformation of nickel-based superalloys, are described. The following specific conclusions can be drawn from this study:

1. The new *in situ* XCT system which uses a laboratory-scale X-ray source has been applied to high-temperature tests with duration of up to 36 hours. The displacement-fixed control for the rotation needed to capture backprojection images has been successfully applied for temperatures of at

Fig. 21—Stress–strain curve in case of with and without XCT procedure. (a) Inconel 718, 10^{-2} s^{-1} , (b) Inconel 718, 10^{-4} s^{-1} , (c) Nimonic 80A, 10^{-2} s^{-1} , and (d) Nimonic 80A, 10^{-4} s^{-1} .

least 800 °C and for strain rates in the range of 10^{-4} to 10^{-2} s^{-1} .

2. The apparatus allows collection of backprojection images with resolution sufficient to detect plastic deformation, void growth, and crack initiation. Void growth and crack initiation were detected if they exceeded $30 \text{ }\mu\text{m}$, and this gives an indication of the spatial resolution which can be achieved.
3. Studies on the grades Nimonic 80A and Inconel 718 have been carried out, with the rationale that these are widely used alloys of lesser and greater ductility, respectively. For testing at 800 °C, Nimonic 80A exhibited surface cracks which are the sites of fracture initiation; for Inconel 718, internal void growth after necking was preferred. In both cases, the XCT system proved to be capable of detecting these failure events.
4. For Inconel 718 at 800 °C and higher strain rates, void growth did not occur uniformly; nevertheless, its temporal and spatial distribution could be detected and quantified. For slower strain rates, void coalescence was more concentrated within the necked region. The surface cracks in Nimonic 80A

were generated widely over a wide range of the gauge volume at higher strain rates, whilst the slower strain rate generated a smaller number of larger cracks.

5. At faster strain rates (10^{-2} s^{-1}) at 800 °C, interrupted tests showed an important degradation of the maximum stress value in each step when compared with monotonic loading conditions—when deformed monotonically, there is not enough time for damage to occur. At slower deformation rates (10^{-4} s^{-1}) at the same temperature, the stress values pre- and post-interruption overlap those obtained monotonically. This proves that the nature of the damage process is time dependent, and thus should be linked to diffusion-assisted forms of damage such as cavitation.
6. Future application of the system will allow for more detailed quantification assessment of the damage accumulation occurring in these materials, including the statistical distribution and evolution with strain and time.

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