

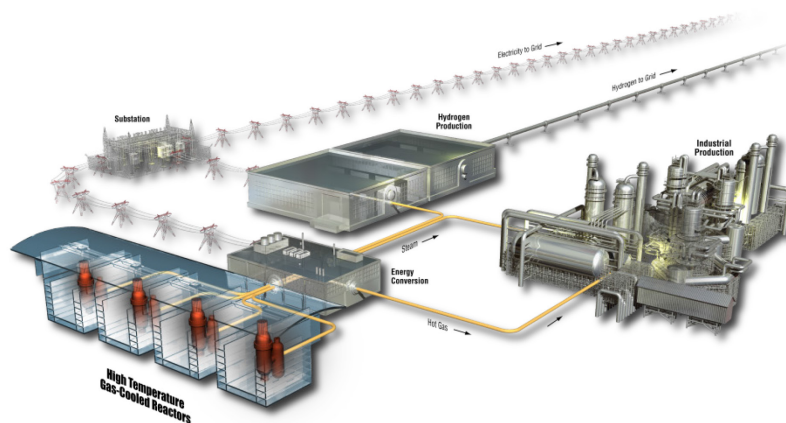
Overview of Alloy 617 Notched Specimen Testing

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Richard Wright

August 2017

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ABSTRACT

Prior to use in high temperature nuclear applications, Alloy 617, which is currently undergoing approval for inclusion in codes and regulations related to high temperature construction, must be approved by the Nuclear Regulatory Commission. Several concerns relating to the effects of geometric discontinuities, such as notches and multi-axial stress states (as opposed to uniaxial stress states typically present in laboratory run creep tests), must first be addressed. This work has shown that at short time intervals (1000–2500 hours), Alloy 617 is notch-strengthening and will fail preferentially at locations away from the notch, provided that diameters are similar (resulting in similar stresses). Additionally, specimens designed with semi-circular notches (or U-notches, as they are referred to in this report) to impose multi-axial stress states on the specimen during creep-testing were found to either maintain similar creep-rupture-lives as uniaxial stress specimens tested at similar conditions, or have creep-rupture-lives that were significantly longer. This work, while not fully completed, shows that notches and multi-axial stress states do not appear to raise concerns with relation to the use of design rules created for Alloy 617 using smooth, straight-gauge test specimens and uniaxial creep tests.

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ACRONYMS

ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Code
EBSD	electron backscatter diffraction
INL	Idaho National Laboratory
LMP	Larson-Miller parameter
NRC	Nuclear Regulatory Committee
SEM	scanning electron microscope
VHTR	very high temperature reactor

Overview of Alloy 617 Notched Specimen Testing

1. INTRODUCTION

Section III, Division 5, of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) contains the design rules that are applicable for the design of components for elevated temperature reactors. This section of the BPVC also specifies those materials that are allowed for nuclear construction, contain the required material properties for design and construction, specifies welding processes that are acceptable, and specifies inspection requirements. There are a very limited number of materials for which sufficient high-temperature properties for design are available. The austenitic materials that are available for elevated temperature design are limited to Type 304 and Type 316 stainless steel and Alloy 800H.

Alloy 617 has been identified as the primary candidate material for the intermediate heat exchanger in very high temperature reactor (VHTR) concepts. This material exhibits good high-temperature mechanical properties that are minimally affected by long-term exposure to elevated temperatures. Recent testing in the United States, in collaboration with Generation IV International Forum partners and an extensive historical database, has been used to develop a draft Code Case for qualification of Alloy 617 in Section III, Division 5, of the BPVC [1]. Code qualification of this material will allow it to be used in nuclear construction up to 950°C and up to 100,000 hours.

The Nuclear Regulatory Commission (NRC) has not officially endorsed the design rules that are contained in Section III, Division 5, for use in VHTR construction. Potential application of these rules to elevated temperature nuclear designs, including the Clinch River Breeder Reactor, has been reviewed by the NRC and comments have been published several times [2]. These reviews have concluded that the impact of notches and structural discontinuities on the service behavior of components at elevated temperature has not been adequately addressed. Resolving these potential issues is a cross-cutting activity that can impact designs using any qualified high-temperature material. Alloy 617 has been chosen as the prototypical material to investigate these issues because of the vast amount of readily available material property data that are available as a result of preparing the Code Case.

Extensive characterization of Alloy 617 high-temperature properties has been carried out under laboratory conditions using uniaxial loading conditions for both base and welded plate material. This testing has included determination of the mechanism responsible for steady-state or minimum creep rate, measurement of creep-rupture behavior over a wide range of stresses and temperatures, and examination of the relationship between onset of tertiary creep and cavity formation. Despite the fact that uniaxial testing is exclusively used in determining time-dependent allowable stresses for ASME design purposes, relevant components operate under multi-axial stress states in service. Thus, it is desirable to examine behavior of high-temperature materials under multi-axial conditions, and compare the behavior to standard uniaxial laboratory tests. Testing of multi-axial stress state effects has begun on base metal, according to Idaho National Laboratory (INL) document PLN-5086 [3]. This report covers the results from fiscal year 2017.

2. EXPERIMENTAL PROCEDURE

Specimens were machined with their longitudinal axes oriented along the rolling direction from a 37 mm thick plate provided by ThyssenKrup VDM (Heat #314626). Composition is given in Table 1. This is the same material used in the Code Case studies and developing the steady-state creep rate equations and the Larson-Miller equations to describe rupture behavior. The average grain size (as measured by the linear intercept method) is 150 μm . Carbide banding and stringers parallel to the rolling direction are also present in the as-received microstructure. The bands are approximately 100-300 μm wide and the carbides appear to form on a prior grain structure, which are finer and unrelated to the present grain boundary structure. Outside of these banded regions, there are few secondary particles, with the exception of a small number of titanium nitrides.

Table 1. Chemical Composition of the Alloy 617 Plate (weight percent).

Ni	Cr	Co	Mo	Al	Ti	Fe	Mn	Cu	Si	C	S	B
54.1	22.2	11.6	8.6	1.1	0.4	1.6	0.1	0.04	0.1	0.05	<0.002	<0.001

Three specimen types were examined in this work. Two were characterized as U-notch specimens, with semi-circular notches, differing only in the radii of the notches. The third type were V-notch specimens with straight-gauge portions (the diameter of the straight-gauge section is the same as the diameter at the root of the notch). The V-notch specimens were used to establish multi-axial creep-rupture properties, while the U-notch was used to examine the multi-axial creep-deformation response [4].

The V-notch specimen design is specified in American Society for Testing and Materials (ASTM) Standard E292-09 [5]. A small modification to the ASTM design was made to allow an extensometer to be connected to the specimen via grooves placed between the V-notch and the threads, the straight-gauge section and the V-notch, and the straight-gauge section and the threads, as seen in Figure 1a. This allows for displacement measures over the straight-gauge section, the V-notch, or the entire length of the specimen. The ASTM standard limits the use of the v-notch specimen to evaluating rupture behavior. This type of specimen was originally developed to describe the behavior of materials at the root of a thread. V-notch specimens are widely used to determine if a particular material at a particular temperature exhibits notch-strengthening or notch-weakening. The presence of both a standard straight-gauge section and a V-notch allow for comparison between a conventional creep test and a notched test. If the specimen fails at the V-notch, it is notch-weakening, if the failure occurs in the straight-gauge, it is notch-strengthening. The diameter at the straight-gauge section and at the root of the V-notch is 6.4 mm, similar to the sizes of straight-gauge creep specimens tested previously at INL, allowing for direct comparisons between the previous and current work.

Two different U-notch specimens, as shown in Figure 1b and Figure 1c, were machined, one with a d_{n0}/r_{n0} ratio of 1 and the other with a ratio of 4.83, as shown in Figure 2. The ratio of the outer and inner diameters of the specimen (e.g., D/d_{n0} in Figure 2) is arbitrary, however, a ratio of $\sqrt{2}$ has been used extensively with this specimen design and is recommended by the Code of Practice [4]. U-notch specimens are commonly used for notch/multi-axial stress effects on creep behavior. The double notch allows for the examination of the failure mechanism for one of the notches and the remaining, unfailed notch may be sectioned for metallographic analysis. The ratio of the diameter across the specimen at the root of the notch and the radius of the notch (e.g., d_{n0}/r_{n0} in Figure 2) defines the severity of the notch and the resulting multi-axial stress state at the root of the notch. A ratio of 4.83 is the maximum that can be obtained, while still maintaining a semi-circular notch.

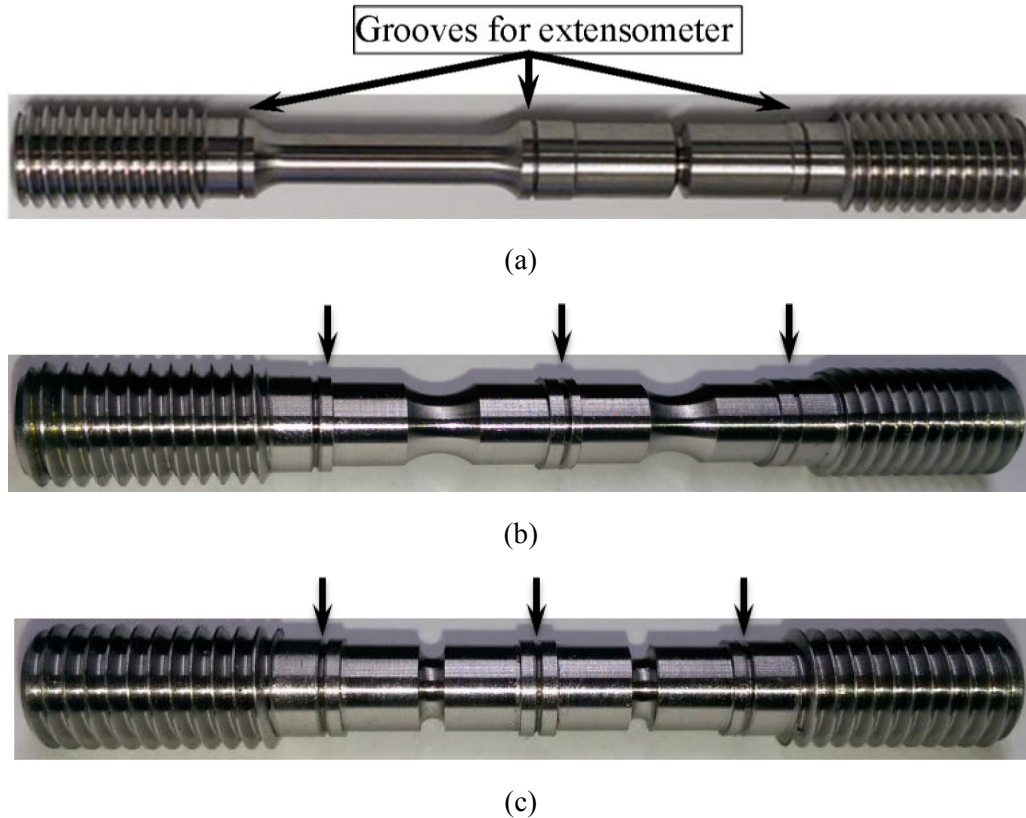


Figure 1. Actual V-notch and U-notch specimens used in testing of notch effects: (a) V-notch specimen, (b) large radius U-notch specimen, (c) small radius U-notch specimen.

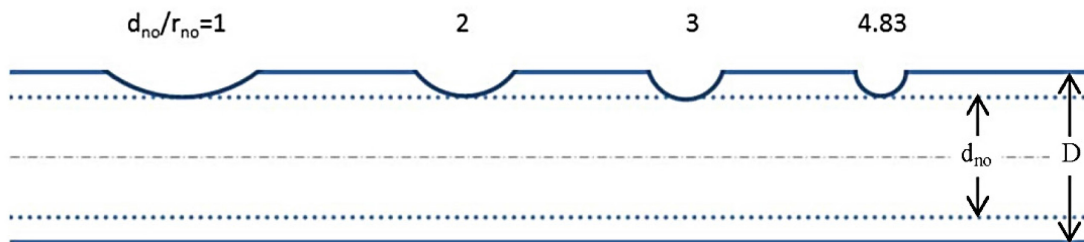


Figure 2. Schematic of the Bridgeman notches with varying d_{n0}/r_{n0} ratios. D is the outer diameter of the gauge, the d_{n0}/r_{n0} ratios, and r_{n0} is the radius of the notch itself. All notches in the schematic have a D/d_{n0} value of $\sqrt{2}$.

Creep-testing has been carried out over a range of temperatures, from 750°C up to 1,000°C. Stresses were chosen such that tests completed within a reasonable amount of time (~1000–2000 hours) and that the tests matched conditions for conventional creep tests run previously at INL. A table of testing conditions is given in Table 2. The testing conditions have been modified since the original test plan [3] in order to match prior work more closely. Following rupture, specimens were cross-sectioned for micrographic analysis at both the straight-gauge section and the V-notch, or both the ruptured and intact U-notch. In addition to the metallographic preparation of cross-sections, some specimens were electrochemically polished and then electrochemically etched to reveal grain boundaries using perchloric acid at 0°C and at a potential of 24V for electropolishing followed by electroetching at 9V using a LetroPol 5 by Struers. Additionally, some specimens were characterized using electron backscatter

diffraction (EBSD) in a Joel 650 scanning electron microscope (SEM). EBSD provides information on grain size/orientation and, by examining small changes to the local misorientation of the crystal lattice, can provide some qualitative information on deformation, highlighting areas of high amounts of deformation.

Table 2. Notched specimens test conditions.

Temperature (°C)	Stress for 1,000h (MPa)
750	145
800	80
900	36
1,000	20

3. RESULTS AND DISCUSSION

Short term testing has largely been completed for V-notch specimens, U-notch testing is still in progress. Overall, results show that Alloy 617, at the conditions tested, is notch strengthening and that there is no life shortening due to the presence of multi-axial stress states. A detailed analysis of the results is presented in the section, with V-notch results first, followed by the U-notch testing results.

4. V-NOTCH SPECIMENS

4.1 Creep Results

Seven creep-rupture tests were completed on V-notch specimens, including replicates of all conditions represented in Table 2, with the exception of the 1000°C test, where the temperature control failed during the replicate test, so the results were not included. All tests resulted in rupture in the straight-gauge portion of the specimen rather than the V-notch, showing that, at these conditions, Alloy 617 is notch-strengthening.

Creep curves for the V-notch specimens, with displacement measured with extensometers over the straight-gauge section, are shown in Figure 3. Also included in each of the graphs in Figure 3 are the curves from prior creep tests with straight-gauge specimens performed at INL with the same conditions. While time variations in time to rupture are observed, the variations are within expectations based on the variability of measured creep properties reported in the ASTM E139 standard for conducting creep tests of metallic materials [6]. Replicate tests were performed with the extensometer over the V-notch portion of the specimen. The results are shown in Figure 4. Note that the results in Figure 4 show displacement vs. time, rather than strain % vs. time, as is the case in Figure 3. This is due to the difficulty in assigning a gauge length for notched specimens, which is needed in order to calculate strain.

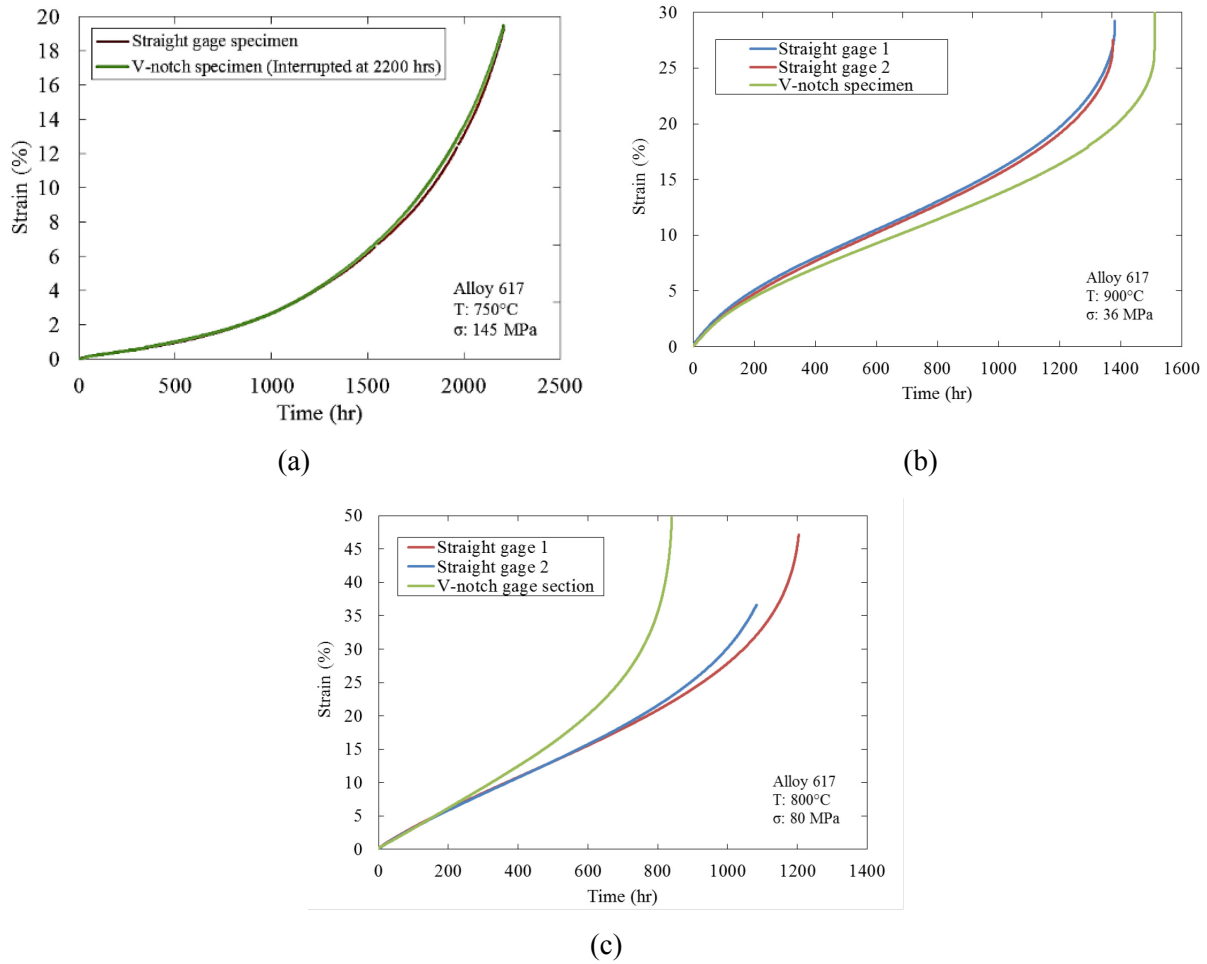


Figure 3. Creep results for V-notch specimens with extensometers over the straight-gauge section and tested at (a) 750°C, (b) 800°C, and (c) 900°C.

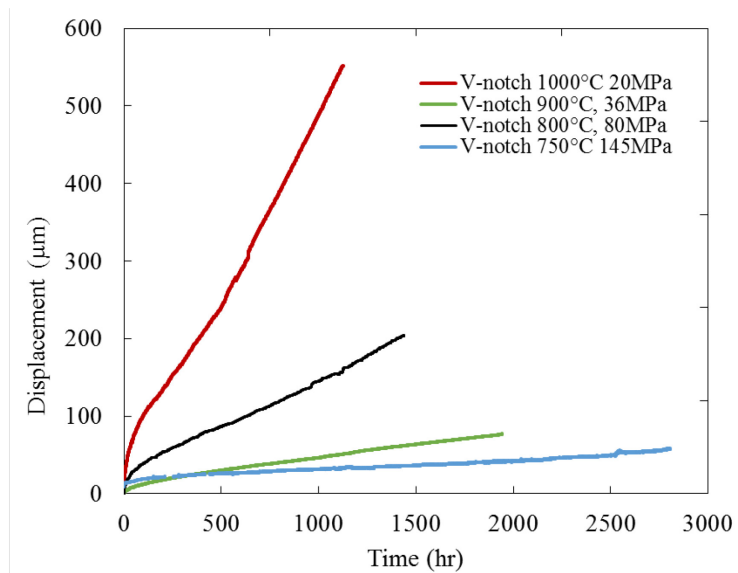


Figure 4. Creep results for V-notch specimens with extensometers across the V-notch.

The Larson-Miller parameter (LMP) is a convenient way to compare creep-rupture data between samples at different loading stresses and temperatures. The LMP is defined as:

$$LMP = T(C + \log_{10}(t)) \quad [1]$$

where T is the absolute temperature, C is the Larson-Miller constant, and t is the time to rupture. Figure 5 shows the results from all of the V-notch tests (U-notch tests are included as well, but will be discussed later in this report) as compared to conventional creep-rupture data collected at INL from a number of sources and reported in the Code Case [1]. The V-notch specimens rupture-lives fall well within the spread of compiled straight gauge data in the Larson-Miller plots, showing that the presence of the V-notch had essentially no impact on the creep-rupture life.

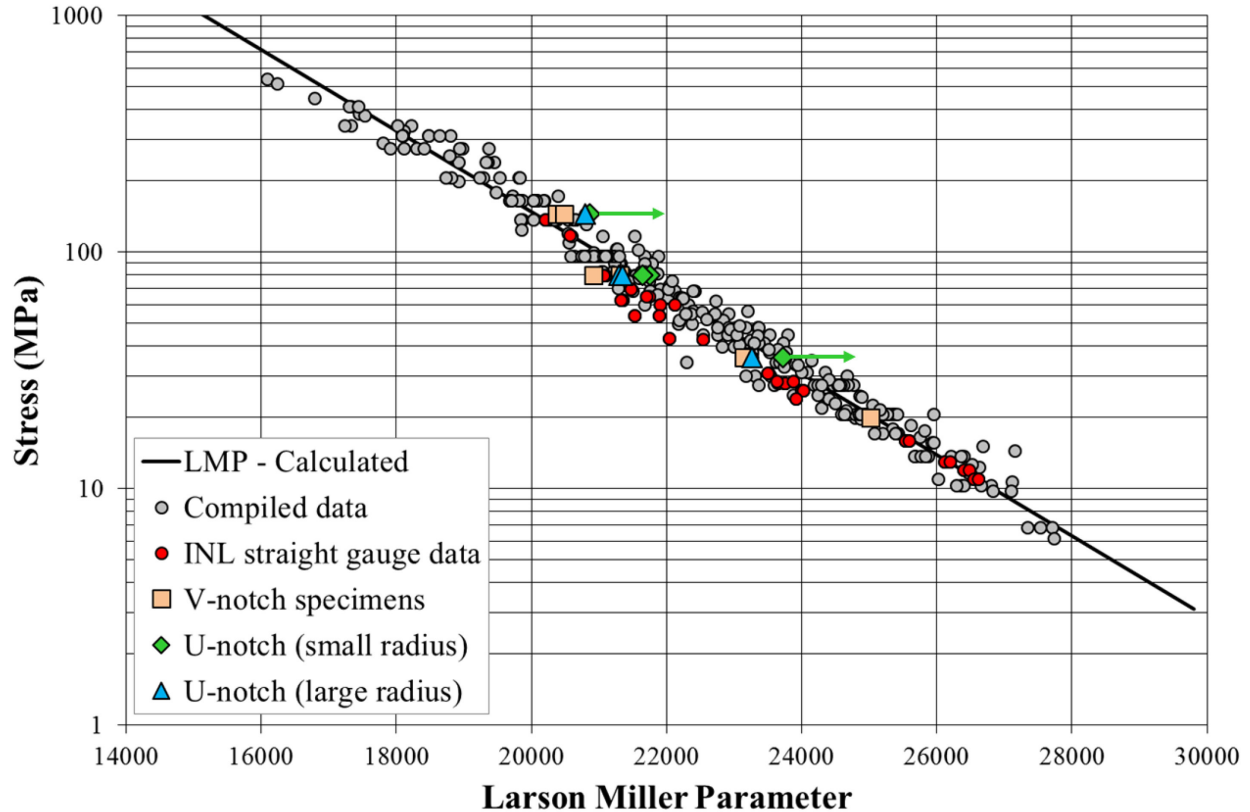


Figure 5. The Larson-Miller plot, combining stress, temperature, and rupture time of the V-notch and U-notch tests for comparison with other 617 creep-rupture data [1]. Note the green arrows indicating that small radius U-notch tests at 145 MPa (700°C) and 36 MPa (900°C) have not yet ruptured.

4.2 Microscopy

Detailed optical micrographs of the first two ruptured V-notch specimens were reported previously [7]. All specimens tested were found to contain creep damage throughout both the entire thickness and length of the straight-gauge portion of the specimen. Only a small amount of damage was observed at the tip of the V-notch, with no additional damage observed elsewhere in the reduced gauge section of the V-notch portion of the specimen. Additional characterization of the damage around the V-notch was performed using EBSD analysis in a SEM.

EBSD results show that along the edge of the specimen, there is a ~20µm thick layer of small grains, which is a result of machining damage. An example of these small grains is shown in Figure 6, showing an area near the V-notch tip; however, these grains were apparent over the entire edge of the specimen, not just the V-notch. A more detailed EBSD analysis was performed to look at local misorientation of the

lattice. Deformation can cause rotation of the crystal lattice (largely due to the presence of certain types of dislocations), which is visible when local misorientation is mapped using EBSD. A V-notch specimen was tested at 750°C, 145 MPa, and interrupted at 2200 hours (prior to rupture). The specimen was cross-sectioned at both the V-notch and the straight-gauge. EBSD local misorientation measurements were taken to use as a baseline for qualitative comparisons of deformation near the V-notch of all the specimens. This was accomplished by taking measurements where no deformation was expected (e.g., the thick shoulder of the specimen) and measurements where significant deformation was known to have occurred, due to overall changes in the specimen measurements (e.g., the straight-gauge section). These were then compared to measurements taken around the V-notch to determine where deformation had occurred. Examples of the measurements that were taken are shown in Figure 7. Note the large amount of green in the measurements taken in the straight-gauge, showing significant local misorientation. Near the shoulder, the EBSD maps show mostly blue, indicating little to no local misorientation. All measurements near the edge (e.g., shoulder, straight-gauge, and V-notch) show a narrow bright green band along the edge, a result of the machining damage already discussed. Note in Figure 7 that the green areas (corresponding to certain grains) seen in the map taken near the V-notch tip are measurement artifacts of the measurement technique. This is why the entire grain appears a near uniform shade of green, rather than bright highlighted portions (generally highlighting grain boundaries and slip planes where deformation occurs) as seen in the high-deformation areas of the straight-gauge.

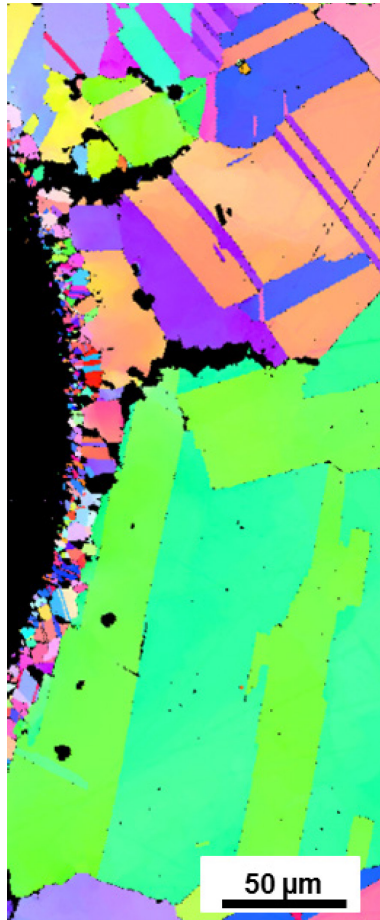


Figure 6. EBSD map showing grains near a V-notch tip.

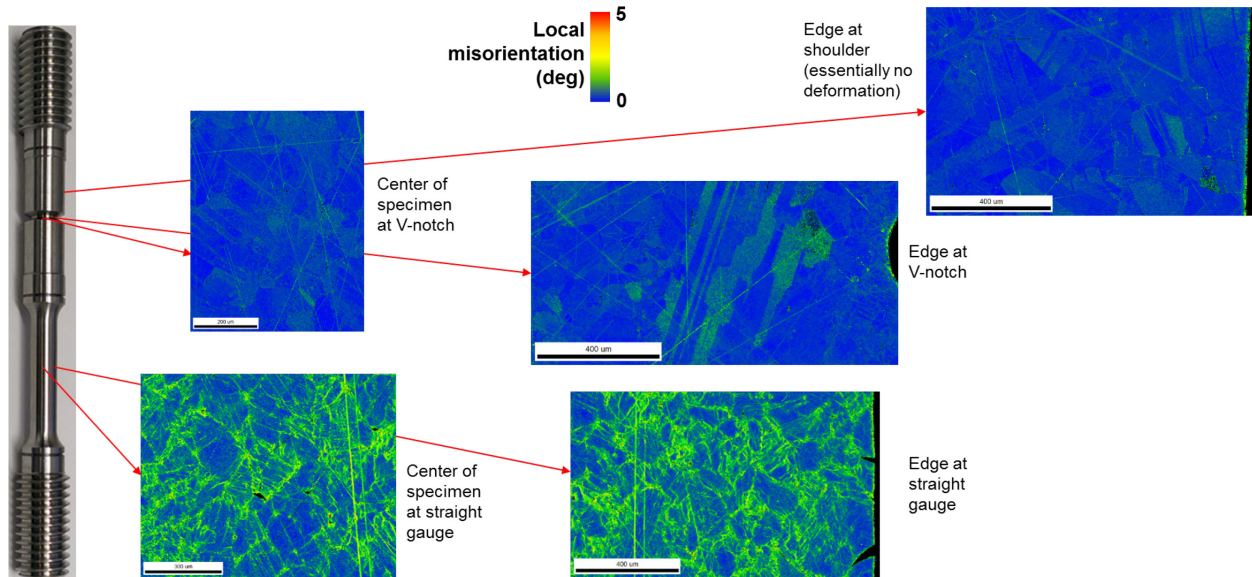


Figure 7. EBSD map showing local misorientation (evidence of deformation) at varying locations of a specimen interrupted just prior to failure (750°C, 145 MPa).

Local misorientation values were extracted from the EBSD maps, starting at the tip of the V notch and traveling towards the center of the specimen. The results for all analyzed specimens are shown in Figure 8. In this figure, distance 0 corresponds to the very tip of the V-notch. All measured specimens show a small, $\sim 20 \mu\text{m}$ peak at 0, which is a measurement of the machining damage already discussed. Measurements taken from the interrupted test, starting at the edge of the specimen and moving inwards from both the shoulder and the straight-gauge give examples how no deformation and high-deformation, respectively, would appear. All specimens, with the exception of the 750°C test, follow a similar trend. Deformation appears to be present up to $\sim 150 \mu\text{m}$, at which point, the lines generally converge into the same area occupied by the “no deformation” (black) line. The 750°C measurements appear to follow the black line (with the exception of two plateaus, which represent the measurement artifacts previously discussed). These results show that there is essentially no deformation occurring in the specimen tested at 750°C, while the higher temperature specimen tests resulted in limited deformation that was constrained to the area within $\sim 150 \mu\text{m}$ of the V-notch tip.

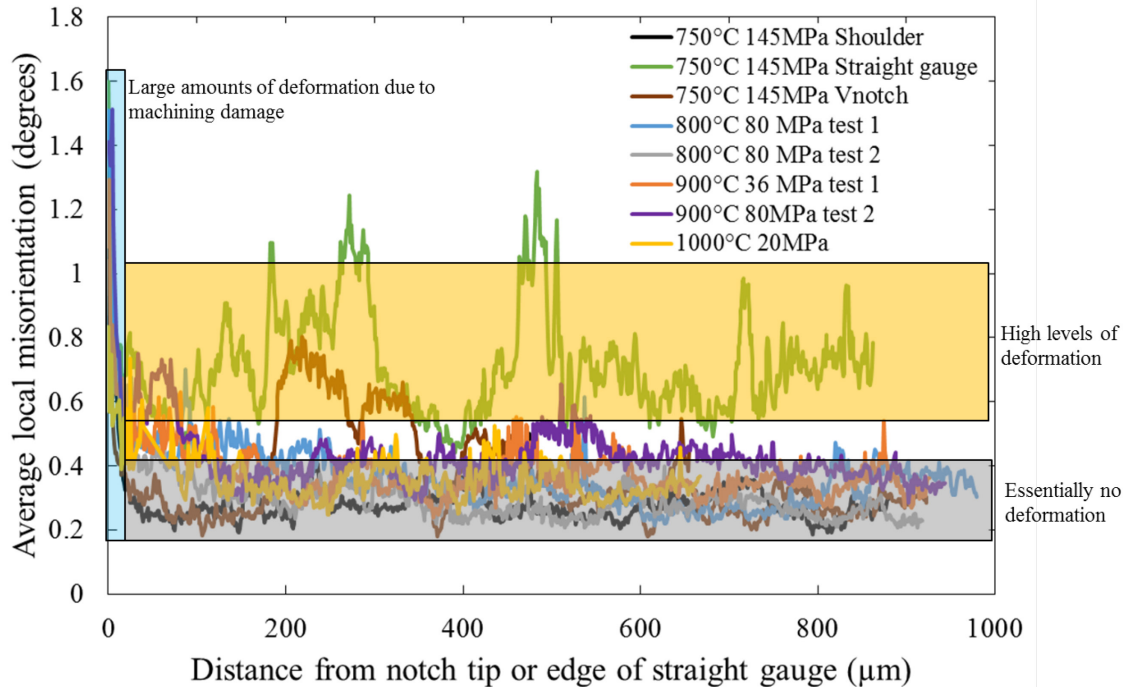


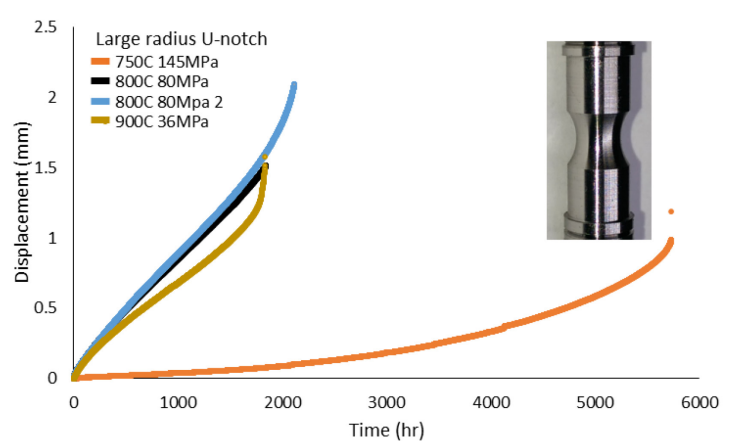
Figure 8. Local misorientation data near the V-notch (distance 0) towards the center of the specimen (~1000 μm into the specimen) from all tests characterized. The first two data sets (black and green) are not taken from the V-notch area, but rather taken from the interrupted test at the shoulder (no deformation) and straight-gauge (high levels of deformation).

The geometric constraints of the notch restrict the radial contraction that would occur in the area near the tip of the notch when the specimen plastically deforms and elongates. The constraints on the deformation are from the wider shoulders adjacent to the notch, which experience significantly lower levels of stress and thus are not inclined to deform and accommodate deformation at the tip notch [8,9]. The straight-gauge section has no such constraints and both elongates along the loading axis as well as radially contracts. As the test progressed, this means that an increased level of stress was experienced by the straight-gauge section, whereas the applied stress at the V-notch remained the same, due to the nature of the constant load test and the changing dimensions of the specimen.

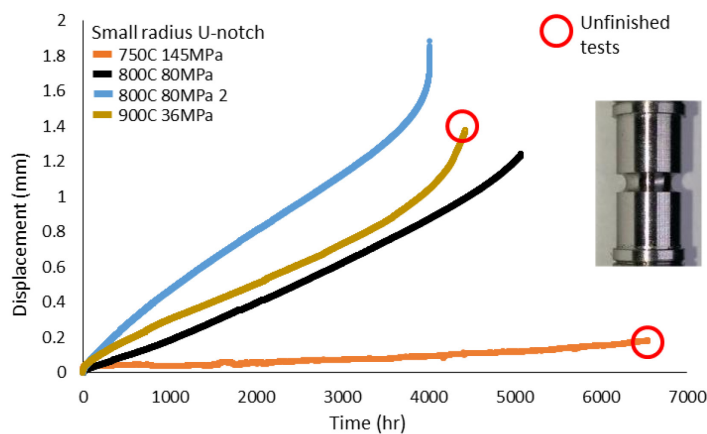
5. U-NOTCH SPECIMENS

5.1 Creep Results

Six U-notch specimen creep-rupture tests have been completed; two are currently running in the creep frames. Four of the completed tests were large radius specimens and the results are shown in Figure 9a. Tests were performed at 750°C, 800°C, and 900°C, with a replicate test being performed at 800°C. The other four tests (two completed, two ongoing) were small radius U-notch specimens. The results of these tests are shown in Figure 9b. The two tests with red circles on the end are the tests that are ongoing.



(a)



(b)

Figure 9. Displacement vs time for creep tests of (a) large and (b) small radius U-notch specimens.

Like the V-notch rupture life measurements, the U-notch results are shown in the Larson-Miller plot in Figure 5. The two diamonds with green arrows attached represent the ongoing tests. It is not clear how far to the right they will be when the test has finished, though the 900°C test is expected to end soon, due to the degree it has entered tertiary creep (i.e., the slope is trending towards vertical). Like the V-notch tests, these results currently all fall within the scatter of data; however, it is important to note that the small radius U-notch tests are all significantly further right than other tests performed at INL. This suggests that though they fall within the scatter of multi-facility/multi-material heat test results, they experience significantly longer creep-rupture-lives than other specimen geometries at similar conditions tested at INL, all tested on the same heat of Alloy 617. The large radius U-notch, which imposes a small, diffuse multi-axial stress state throughout the thickness of the specimen acts very similar to the straight-gauge, uniaxial stress specimens previously tested. The small radius U-notch tests result in longer creep-rupture-lives.

5.2 Microscopy

Cross-sectional analysis of the first three tested specimens are shown in Figure 10. From the analysis of the unbroken notch (see Figure 10a, c, and e), extensive damage is observed near the notch tip of the small radius specimen, while not being present near the center of the specimen. The large radius specimens show near uniform damage across the entire thickness of the gauge. The fractured notches (Figure 10b, d, and f) appear fairly similar between the two geometries.

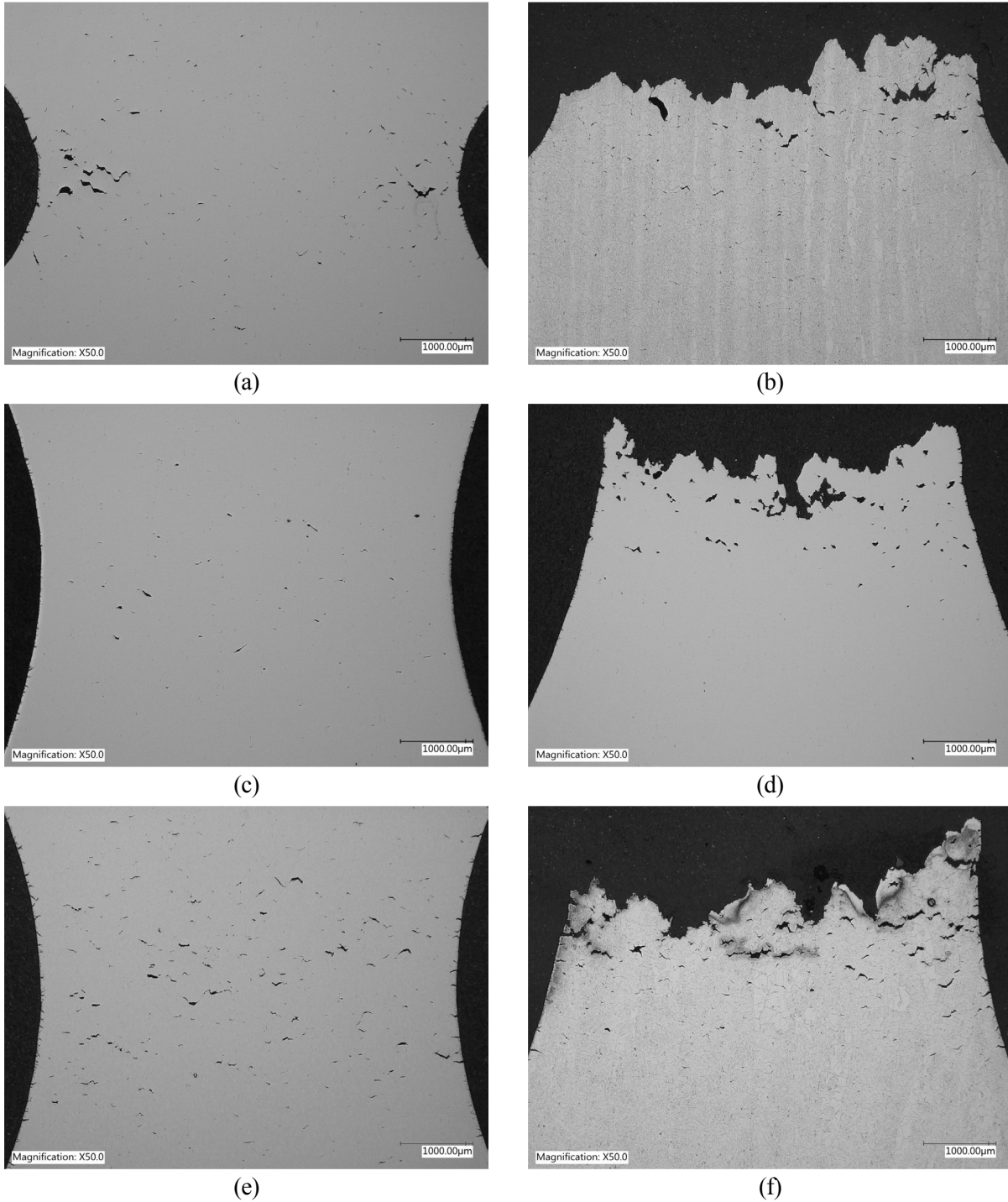


Figure 10. Optical micrographs of cross sections from three U-notch creep-rupture test specimens: (a),(b) small radius specimen tested at 800°C, 80 MPa; (c),(d) large radius specimen tested at 800°C 80 MPa; and (e),(f) large radius specimen tested at 900°C 36 MPa.

Local misorientation maps were created using EBSD analysis for two specimens, a large and a small radius specimen, both tested at 800°C, 80 MPa. The small radius specimen (see Figure 11) showed signs of high deformation near the notch tip; however, in the center of the gauge, very little deformation was

observed. For the large radius specimen (see Figure 12), deformation was observed throughout the thickness of the gauge.

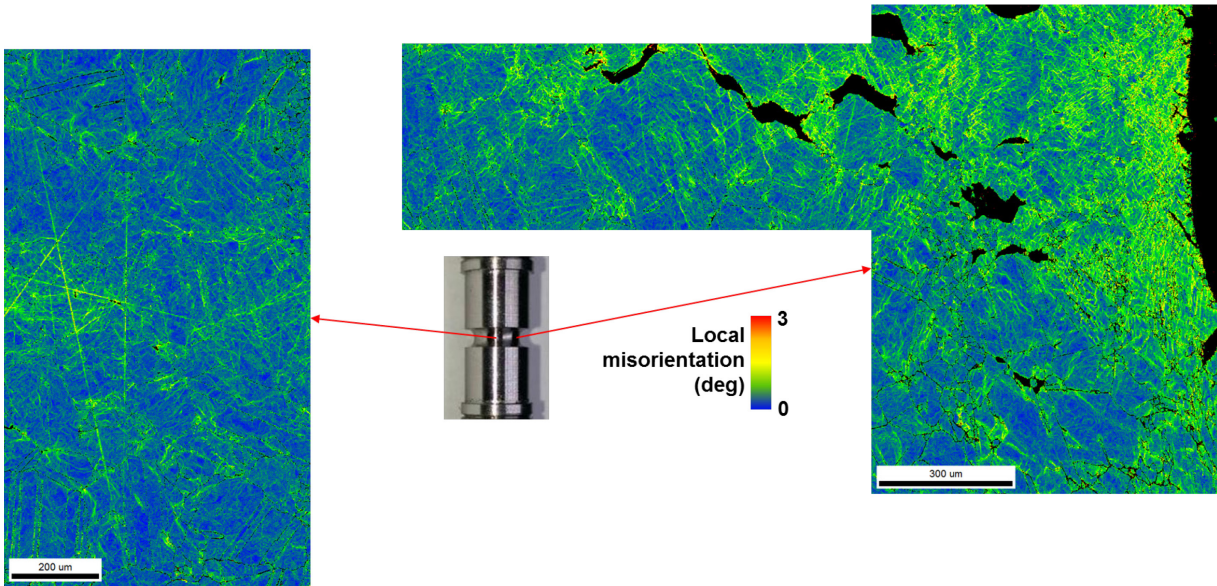


Figure 11. EBSD local misorientation analysis of the small radius U-notch specimen tested at 800°C, 80 MPa.

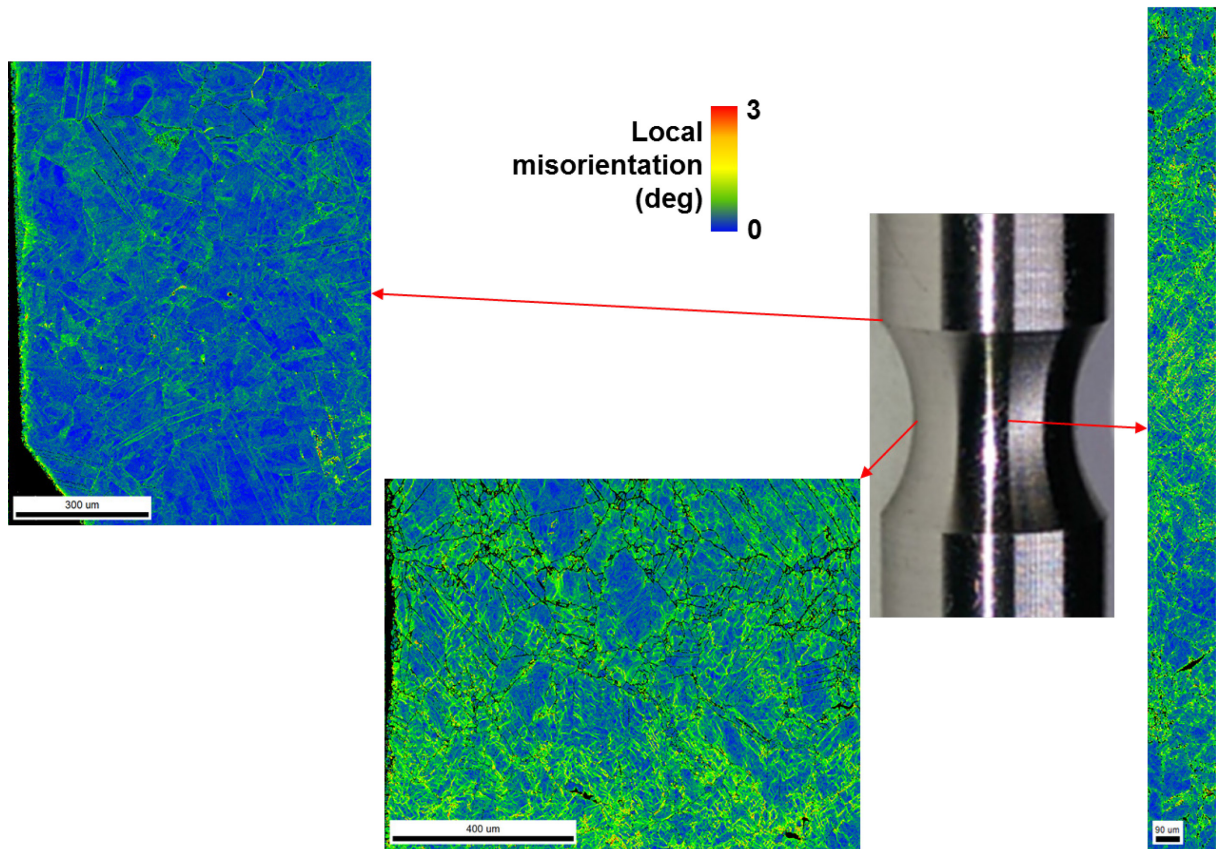


Figure 12. EBSD local misorientation analysis of the large radius U-notch specimen tested at 800°C, 80 MPa.

Analysis of the damage in the cross-section and the local misorientation show that the large radius U-notch specimens, despite having a diffuse multi-axial stress state that extends throughout the thickness of the specimen, behave nearly identically to the straight-gauge, uniaxial stress creep specimens. This is further supported by the similarities in creep-rupture-life of the large radius U-notch specimens and the straight-gauge specimens, as seen in Figure 5. The small radius U-notch specimens, however, show deformation and damage localized in the area near the notch tip, where the multi-axial stress states are strongest. However, despite this damage, the overall rupture life of the small radius U-notch specimen is longer than other tests performed at INL. The multi-axial stress state is not detrimental to overall creep-rupture-life when compared to typical uniaxial stress laboratory tests.

6. CONCLUSIONS

Alloy 617 is a notch-strengthening material for tests performed between 750°C and 1000°C, with creep-life between 1000–2500 hours. No work has currently been performed to determine if the notch-strengthening behavior changes at longer test times; however, this work will be performed going forward. Multi-axial stress states do not detrimentally affect creep-rupture-life when compared to uniaxial stress testing. While damage did occur in areas with the highest degree of multi-axial stress, overall rupture life remained unaffected, or was found to increase, in the notched-testing performed in this work.

7. REFERENCES

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