Synchrotron radiation imaging and characterization of creep cracks in 2219 aluminum alloy

F M Xu¹, Q Guo¹, Y X Xu¹, W Dong¹,³, T T Li¹ and Y N Fu²

¹School of Materials Science and Engineering, Dalian University of Technology, Dalian, 116024, China
²Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, 201204, China

Email: w-dong@dlut.edu.cn

Abstract. Aluminum alloys are lucrative aero craft materials due to their high-temperature performance, which involves creep crack propagation. However, it is difficult to acquire three-dimensional morphology, volume, and quantity through traditional imaging and characterization techniques, as metallic alloys are optically opaque. The development of synchrotron radiation X-ray tomography has facilitated the study of creep crack propagation. In this study, samples obtained by an interruption experiment were subjected to X-ray tomography observation. Based on reconstruction and visualization software analysis, cracks were first formed inside the material and then propagated via the growth and connection of creep voids. While propagating forward, the cracks also propagated toward the material surface, with the inside of the material propagating more than the surface of the material. When the main crack propagated, irregularly shaped microcracks and large independent branch cracks were generated, promoting the propagation of the main crack.

1. Introduction
In recent years, there have been a number of studies on two-dimensional (2D) creep crack propagation [1]. The study of three-dimensional (3D) creep crack propagation, however, is limited by observation and analysis methods. In the studies, the crack tip is simulated by finite element simulation to obtain the tip stress field and crack tip parameters [2]. However, the actual 3D morphology of the creep crack tip and other information differ from the simulation results [3]. To deepen the study of creep crack propagation and establish a more accurate 3D model, an effective 3D observation characterization method is necessary. Absorption contrast imaging can be utilized to perform imaging through different X-ray absorption due to the different densities, compositions, and thicknesses of materials. However, absorption contrast imaging is not ideal for imaging light alloys. In recent years, with the development of high-brightness synchrotron radiation sources, phase-contrast imaging has become a popular research method. In light alloy imaging, phase-contrast imaging is 2–3 orders of magnitude higher than absorption contrast imaging [4, 5]. Owing to the excellent spatial coherence characteristics of synchrotron radiation, phase-contrast imaging provides an advanced method for the research of medicine and materials.

In this study, synchrotron radiation tomography is used to obtain 2D projections of a creep crack by scanning a 2219 aluminum alloy including a creep crack. And then a visualization software is utilized to reconstruct the 3D morphology of the crack. In this way, we are able to obtain the 3D information of
the creep crack, including the morphology, volume, and distribution, to reveal the microscopic mechanism of creep crack propagation.

2. Experimental and imaging methods

2.1. Sample preparation
To ensure the stability of the sample structure during the experiment, microstructure adjustment was required for the sample, as the creep crack propagation experiment took a long time to be completed. A heat-treated sample was processed as a compact tension (CT) specimen [6], and creep crack propagation experiments were conducted on a tensile creep machine. The experimental temperature was 300 °C and the load was 833 N. After the creep crack propagated for 20 hours, the experiment was interrupted and the sample was removed. In order to meet the Shanghai Synchrotron Radiation Facility (SSRF) requirements for the sample size and penetration rate of the X-ray energy for the sample, and to observe the shape of the crack, the rectangular parallelepiped bar sized thickness (X) 1.7 mm, length (Y) 1.6 mm, and height (Z) 15 mm was cut from the CT sample using wire cutting, as illustrated in figure 1, and processed as a synchrotron radiation tomography (SRT) sample. Care was taken to ensure that the crack tip was kept in the acquired sample [7].

![Crack growth direction](image)

**Figure 1.** Outline of SRT sample preparation from the CT specimen with a creep crack.

2.2. Tomographic imaging
The synchrotron radiation experiment was conducted at BL13W1 beamline station of the SSRF. The beamline required that the selected ray energy should penetrate the sample by no less than 20%; thus, the final tomographic parameters were determined as listed in table 1. The X-ray tomography offers a set of tomographic projections that contain internal information about a material. Only by reconstructing these projections can the true structure of a CT specimen be reflected in 2D and 3D directions.

| Set item                  | Value   |
|---------------------------|---------|
| X-ray energy              | 23 keV  |
| Sample-to-detector distance| 0.25 m  |
| Pixel                     | 3.25e-6 m |

**Table 1.** Tomographic parameters.

3. Imaging and characterization
The SRT sample was rotated 180° on the sample stage to take a total of 900 pictures. The imaging method was in-line phase-contrast imaging (IL-PCI), and the phase recovery marker background was performed using the reconstruction algorithm. Then, 2D images reconstruction were performed and the reconstructed slices were imported into the visualization software for 3D reconstruction.

3.1. Volume rendering

Volume rendering of SRT sample was completed after 2D slices were incorporated into the visualization software. The resulting 3D rendering is presented in figure 2.

Figure 2. Reconstructed 3D images of SRT sample.

(a) Volume rendering; (b) YZ plane; (c) XZ plane; (d) opposite side to YZ plane of SRT sample.

Figure 2(a) presents a 3D rendering of the SRT specimen after crack propagation. It can be seen that the crack propagates in the direction perpendicular to the stress inside the material. Figure 2(b) illustrates the YZ plane of the 3D rendering of the crack tip and we find that the crack tip exhibits discontinuity; however, additional visualization of the crack tip is required to determine whether it is connected in the 3D direction. Nevertheless, it can be concluded that the local propagation of the crack inside the material is not always perpendicular to the stress direction. A crack may expand in other directions during propagation; however, under the action of stress, it will eventually return to the direction perpendicular to the stress.

Figure 2(c) presents the XZ plane of the 3D rendering image. It can be seen that the crack tip exhibits irregularity on the surface perpendicular to the propagation direction, which further indicates that the direction of the local propagation of the crack tip is uncertain. This is because the direction is not entirely subject to the influence of stress. Figure 2(d) presents the opposite surface to figure 2(b), which is also the outer surface of the SRT specimen. The cracks have a small opening displacement on the material surface, which suggests that a creep crack has a longer propagation path inside the material than on the material surface. This finding is of great significance in engineering applications. When a microcrack appears on the surface, it indicates greater internal damage.

3.2. Crack visualization

Reconstructed slice maps were imported into the visualization software for thresholding segmentation after median filtering. A crack with a lower gray was extracted from the matrix materials, and cracks were volume-marked. The final volume interval was divided into 34.3281/1e+5/1e+7/1e+9 (μm³),
corresponding to the cavity (marked by red color), microcrack (blue color), independent branch crack (green color), and main crack (yellow color), respectively. There are numerous cavities of different sizes around the main crack. Some of them are very larger and not connected to the main crack. They are referred to as independent branch cracks. The morphology of the resulting 3D crack tip is presented in figure 3.

Figure 3. Reconstructed 3D images of creep crack tip.

Figure 3(a) indicates that the main crack propagates forward in the direction perpendicular to the stress. The number of cavities is significantly larger than the number of microcracks and independent branch cracks. Independent branch cracks, microcracks, and cavities are distributed around the crack tip. The independent branch crack volume is less than the volume of the main crack, and the independent branch crack may be linked with the main crack during the crack propagation process, which is one of the causes of crack propagation instability. Figure 3(b) indicates that cavities have the most concentrated density at the forefront of the crack; this is because stress is the driving force for the formation of cavities. The stress is most concentrated at the crack tip; thus, cavities have the highest forming ability here. The creep crack has a larger opening displacement in the interior of the material than on its surface. This suggests that the creep crack is initially formed inside the material and then simultaneously propagates toward the material surface and in the direction perpendicular to the stress. The crack growth rate is the highest in the direction perpendicular to the stress.

Using the volume filtering function of the software, main cracks with microcracks and main cracks with cavities were extracted. Figure 3(c) indicates that the microcracks are radially distributed from the main crack to the material surface. In subsequent propagations, these microcracks may be interconnected to form independent branch cracks, which may also be connected to the main cracks. A comparison between figures 3(c) and (d) demonstrates that there are more cavities than microcracks and the cavities have a greater distribution range than microcracks and main cracks. This is because stress acts as a driving force for creep crack propagation, and pores are preferentially generated inside the material. The growth and connection of cavities prepares for the second stage of creep crack propagation.
Figure 4. (a) Main crack; (b) independent branch crack; (c) partial enlargement of creep crack.

The main crack and independent branch crack were extracted separately. Figures 4(a) and (b) demonstrate that main cracks and independent branch cracks have irregular shapes. To demonstrate this phenomenon, independent branch cracks were selected and locally amplified to obtain the morphology, as illustrated in figure 4(c). It can be seen that there are several fine connection bridges marked by black arrows, just like sintering neck in powder metallurgy, which are caused by the coalescence of cracks. Therefore, creep crack propagation is a process, in which cracks are coalesced with such defects as cavities or microcracks under the action of stress. This is consistent with traditional creep crack propagation mechanism.

4. Conclusions
Synchrotron radiation provides an effective method for establishing an accurate creep damage model and accessing quantitative information, such as volume, distribution, and the quantity of creep cracks. We can draw some conclusions via the present study. The propagation of creep cracks depends on the connection with cavities and microcracks. Under the action of stress, creep cracks are preferentially formed inside a material while propagating toward the surface of the material and in the direction perpendicular to the stress. However, the crack opening displacement inside the material is much larger than that on the material surface. Larger independent branch cracks are formed during creep crack propagation, and the existence of independent branch cracks provides conditions for unstable crack propagation.

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