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Synchrotron X-ray micro-tomography at the Advanced Light Source: Developments in high-temperature in-situ mechanical testing

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Abstract. At the Advanced Light Source (ALS), Beamline 8.3.2 performs hard X-ray micro-tomography under conditions of high temperature, pressure, mechanical loading, and other realistic conditions using environmental test cells. With scan times of 10s–100s of seconds, the microstructural evolution of materials can be directly observed over multiple time steps spanning prescribed changes in the sample environment. This capability enables in-situ quasi-static mechanical testing of materials. We present an overview of our in-situ mechanical testing capabilities and recent hardware developments that enable flexural testing at high temperature and in combination with acoustic emission analysis.

1. Introduction

As the use of advanced structural materials, such as carbon fiber composites and ceramic matrix composites (CMC), expand in aerospace, turbine, nuclear, and other engineering disciplines, there is a growing need for understanding the response of these materials' microstructure to their operational environments through high resolution imaging and in-situ testing. Beamline 8.3.2 at the Advanced Light Source (ALS) specializes in synchrotron hard X-ray micro-computed-tomography (μ CT) [1, 2]. With μ CT, transmission radiographs of an object are taken from multiple angles and are then used to computationally reconstruct a 3D image of the object's internal structure. The high X-ray flux from synchrotron sources like the ALS uniquely enable high-speed micro-scale X-ray imaging and tomography. At 8.3.2, μ CT scans typically require 10s–100s of seconds to acquire high-resolution ($0.6\ \mu\text{m}/\text{pixel}$) data sets. With short acquisition timescales, relative to other lab-based μ CT instruments, synchrotron tomography is well suited for in-situ mechanical testing materials. Analysis of advanced structural materials exposed to incrementally increasing loads can provide detailed knowledge of how material microstructure evolves under realistic loading and failure conditions.



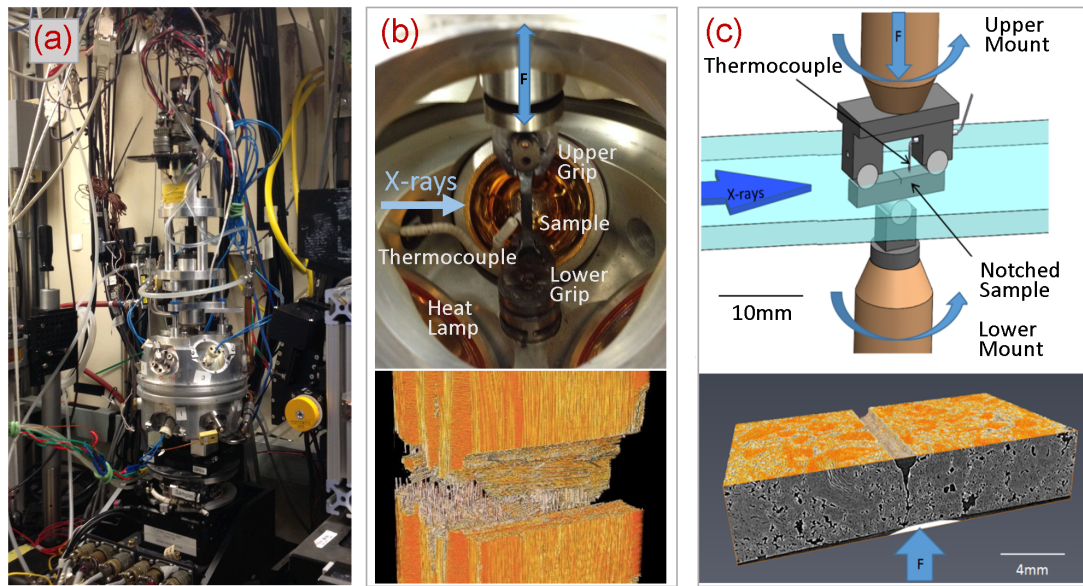


Figure 1. (a) Photo of environmental cell for high temperature mechanical testing. (b) Uniaxial loading grippers and volume rendering of analyzed CMC sample. (c) Three Point bend loading system and volume rendering of analyzed graphite sample.

In-situ environmental cells are a critical part of the beamline 8.3.2. program. Various cells are available for studies spanning fields such as aerospace, plant physiology, geology, and materials science. Each of these cells is used to impose unique controlled environmental conditions on samples. Studying materials under loads, especially at high temperature (800° to $> 1600^{\circ}\text{C}$), is critical for understanding properties and failure modes of advanced structural materials. Uniaxial loading tests of such materials, particularly tensile testing of CMCs has been and continues to be a highly successful part of the 8.3.2. user program [3]. Over the past several years, the majority of mechanical testing on 8.3.2 has been performed using a high temperature loading cell described in detail in reference [4] and shown in Figure 1. To address growing scientific needs, we are developing new in-situ mechanical testing capabilities to include in-situ tensile testing with acoustic emission analysis in addition to flexural testing with both 3-point and 4-point bending.

2. Acoustic Emission Analysis

Acoustic emission (AE) analysis is an established technique used in industry for monitoring how a material changes under load, particularly the formation of fractures and damage. AE analysis refers to the recording of elastic waves produced by damage within a material with high frequency transducers. For loaded samples, analysis of characteristics of the detected waves such as propagation time, amplitude, energy, and characteristic shape can indicate damage location and mechanism. AE has been shown to be beneficial for analysis of fiber-reinforced composites including CMCs [5, 6, 7, 8, 9] and can therefore be an important tool to couple with μCT .

Preliminary AE analysis has been performed on data collected during room temperature in-situ tensile tests. These measurements successfully detected the initiation and propagation of cracks in loaded CMC samples and have therefore demonstrated that AE analysis can be performed with in-situ tomography (Figure 2). AE was monitored using two sensors clamped in place 20 mm apart on either side of the gauge section of CMC sample. The AE system used was composed of Pico HF-1.2 sensors connected to an IL-LP-WS, 26 dB gain preamplifier, and

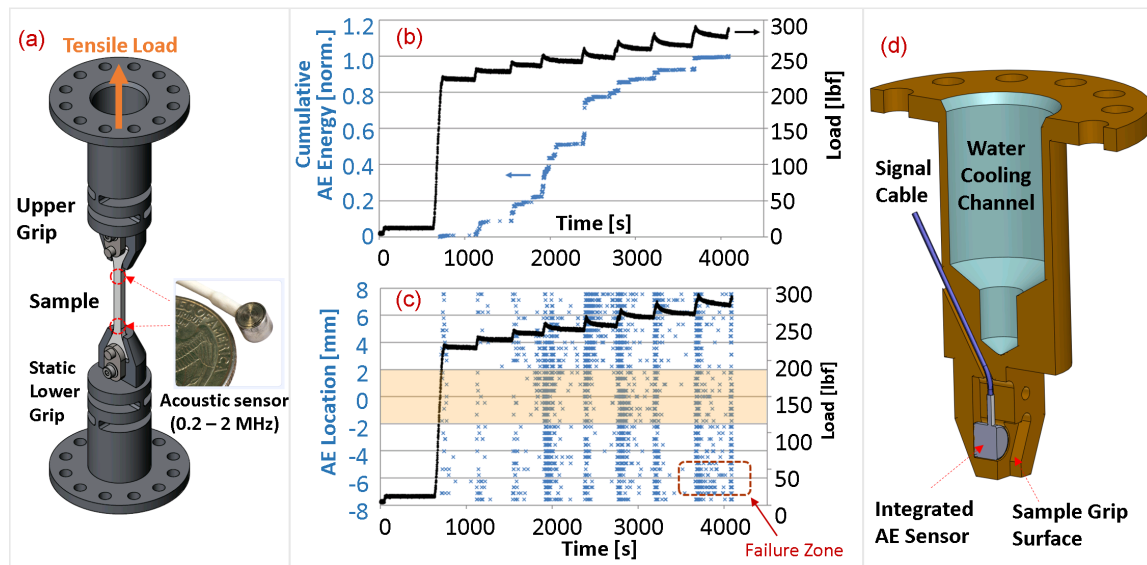


Figure 2. (a) Schematic of tensile loading system: sample, grippers, and AE sensor placement. (b) Cumulative AE energy vs. time and (c) location of AE events vs. time for CMC sample. In (c) the highlighted region corresponds to the scanned length and the dashed-line box indicates the failure zone. (d) Design concept for water-cooled sample gripper with integrated AE sensor.

a 1283 USB AE Node from Mistras Group.

Cumulative AE signal energy and signal amplitudes were monitored continuously during tensile loading until the sample failed, and the AE signals were used to guide the load increase. Load was increased until AE activity became significant. Further load increments were determined based on the amount of increased accumulated AE energy. μ CT scans were taken between each loading step and are thus closely associated with development of cracks in the samples during continued load increase. Figure 2b shows cumulative AE energy as a function of time. Cumulative AE energy is a direct measure of damage progression; the energy of an AE signal being directly related to the surface area created by fracture. From the difference in times of arrival of signals at the top and bottom sensors, the location of each AE event can also be determined. Figure 2c shows that significant damage occurred along the entire gauge and that failure location corresponds to a zone of high AE activity prior to failure.

Further work will focus on correlating the locations and signal characteristics of AE events in the scanned volume with observations from the tomography scans. In addition, since the sensors' thermal limits are typically $\sim 200^{\circ}\text{C}$, water-cooled sample grippers with integrated AE sensors are in development to make high temperature AE analysis possible (Figure 2d).

3. Flexural Testing

A common method for determining mechanical properties of materials is by flexural testing using 3-point or 4-point bend configurations [10, 11, 12], where the number of points refers to the number of contacts points used to impart a bending moments on the sample. Flexure testing enables analysis of the fracture mechanics of materials. Broadly speaking, the 3-point-bend test is best suited for studying initiation and propagation of cracks due to concentration of tensile stresses in the center of sample. The 4-point-bend test is well suited for determining the bulk properties of the sample under pure flexure, which occurs between the two inner loading points. Flexural tests are relatively straightforward in the engineering laboratory setting because

sample preparation is simple and bending instruments are well established. These tests become more challenging for in-situ tomography environments due to geometric constraints but can provide critical understanding of the mechanisms for material failure and fracture resistance. Development of flexure systems at beamline 8.3.2 are described in the following sections.

3.1. Horizontal Three-Point Bending

A horizontally oriented 3-point bending apparatus was developed for the high temperature loading cell used on 8.3.2 [13]. This mechanism, shown in Figure 1c, consists of two 4.8 mm (3/16 inch) diameter rollers that load a horizontal beam-like sample from the top, and a stationary 4.8 mm diameter roller that supports the sample from the bottom. This configuration was chosen because stationary support roller allows the middle of the sample – where crack propagation is typically observed – to remain in the field of view with minimal motion as the sample is loaded. To accommodate high temperature experiments the loading and support rollers are made from alumina (Al_2O_3) and are supported by a stainless steel structure mounted in water cooled supports. A thermocouple is also integrated into the design to directly measure the sample temperature. 10 mm and 16 mm span was chosen for samples of 2–4 mm in width w and height h .

The sample's large aspect ratio L/w – longest dimension L versus shortest dimension w – and its horizontal orientation, can be problematic for X-ray attenuation in L and contrast. Therefore this 3-point bend method works best for samples that have sufficient transmission in L and has been demonstrated with low-Z materials such as graphite [13].

3.2. Vertical Four-Point Bending

A vertically oriented 4-point bend apparatus is currently in development. The prototype for the 4-point bend mechanism that is compatible with our high temperature uniaxial loading cell is shown in Figure 3. This mechanism converts the vertical linear motion of the loading stage into a symmetric bending moment applied to both ends of the sample. The vertical bending configuration is advantageous because it eliminates the contrast issues that arise with large aspect ratio samples mounted horizontally (as in section 3.1).

The bending grips are adjustable to accommodate samples of different thickness and are made from super-alloy MAR M247 because they operate at high temperature with limited conductive cooling. The support arms are made from phosphor bronze and are conductively cooled through the water-cooled support posts.

With 4-point bend measurements, it is desirable to load the sample purely with bending moments applied to the sample through the four contact points. With this mechanism, however, axial compressive loads cannot entirely be avoided. To minimize compression, the bending arms are made to the maximum length allowed by the internal geometry of the environmental cell. Based on the geometry of the current design, the axial compression force should be fairly minimal, 20 times less than the bend force applied at the contact points.

Preliminary 4-point bend measurements were performed on CMC samples demonstrating that samples can be loaded to failure in-situ between tomography scans. Digital image correlation (DIC) [14] analysis was also performed ex-situ to characterize the performance of the 4-point bend mechanism, as shown in Figure 3b. In this analysis, the motion of the load stage, grippers, and sample were tracked to determine the relationship between the strain distribution across the sample and the motion of the mechanism. Figure 3d shows the strain distribution as a function of applied displacement Δ_{applied} by the chamber's vertical uniaxial loading stage. The strain measurements on both tensile and compressive sides of the sample were within $\sim 5\%$ of each other, indicating relatively symmetric loading and minimal influence of the compressive load. Figure 3e shows the motion of the sample's centroid – typically near the region of interest – versus the applied displacement Δ_{applied} . As expected, this shows that the centroid $C_{\text{vert,horiz}}$

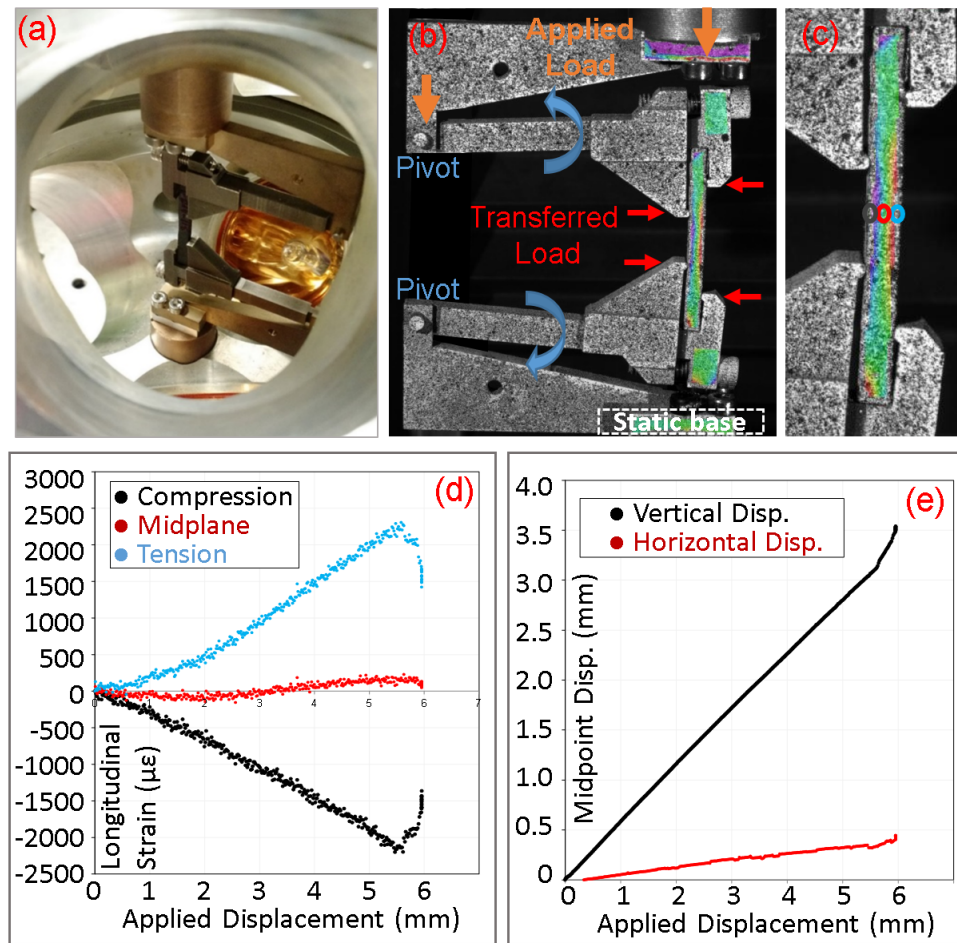


Figure 3. (a) Photo of 4 point bend mechanism. (b) Digital image correlation (DIC) analysis of 4-point bend system. Arrows indicate motion of and forces generated by components. (c) Zoomed-in view of DIC analysis showing strain distribution in the sample regions highlighted by circles correspond to (d) the plot measured strain vs applied displacement. (e) Shows the vertical and horizontal displacement of the midpoint of the sample.

moves substantially in the vertical direction with $\Delta C_{\text{vert}} \approx \Delta_{\text{applied}}/2$. Motion of the sample between μCT scans can be problematic however it can be managed with a > 5 mm tall x-ray window, appropriate positioning of the sample and registration algorithms to correct for the sample motion in the analysis. Development of this system is ongoing and looks promising for in-situ 4-point bending.

4. Conclusions

Coupling μCT with high temperature mechanical testing provides powerful tools for understanding the microstructural behavior of advanced materials. Due to increasing scientific demand, at beamline 8.3.2, we have expanded our capabilities to include acoustic emission (AE) and flexural testing. Preliminary analysis with AE has been performed, with high temperature AE experiments planned. Three-point bending has been successfully implemented and used for brittle materials such as graphite. Four-point bending has been demonstrated with CMCs and continues to be developed.

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