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Abstract. There is increasing demand for 3D micro-scale time-resolved imaging of samples in realistic - and in many cases extreme environments. The data is used to understand material response, validate and refine computational models which, in turn, can be used to reduce development time for new materials and processes. Here we present the results of high temperature experiments carried out at the x-ray micro-tomography beamline 8.3.2 at the Advanced Light Source. The themes involve material failure and processing at temperatures up to 1750°C. The experimental configurations required to achieve the requisite conditions for imaging are described, with examples of ceramic matrix composites, spacecraft ablative heat shields and nuclear reactor core Gilsocarbon graphite.

INTRODUCTION

Material properties are often dictated by the three-dimensional (3D) nature of material microstructure. Therefore, imaging microstructures and modeling material behavior in 3D has become increasing important in the development and utilization of emerging high-performance materials [1]. But only in the past 10-15 years have the computing capabilities required to exploit 3D imaging and 3D computational models become available at low cost. The two tasks – imaging and computational modeling are highly synergistic :- imaging pristine microstructures and their evolution during failure allows for the development of high-fidelity representations of microstructure as well as validation of computational models for prediction of damage and failure. The result is a reduction in development time and a more rapid delivery of new materials to market. This paper describes progress on the development of high temperature cells for x-ray micro-tomography of various materials being developed for high temperature applications.

Synchrotron micro-tomography measurements require the illumination of a sample by an x-ray beam, typically 5 mm high by the width of the sample [2]. The sample is required to rotate 180°, usually about a vertical axis, and to be viewed continuously by the x-rays during rotation. The cell is thus required to have a 360° x-ray view in the plane of interest. All cell components in the imaging plane must be essentially transparent to the x-rays and the imaging camera is preferred to be in close proximity to the sample to reduce coherent imaging artifacts. These requirements have led to novel cell designs that are capable of operating at temperatures up to 2000°C while tensile

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or compressive loads are applied to the sample, either in vacuum or in inert gas at a specified pressure. The nature of the cell developed at the Advanced Light Source and four specific applications of its use are described here.

High Temperature Cell with Mechanical Sample Loading

Carbon fiber-reinforced ceramic-matrix composites (CMCs) are promising materials that offer the possibility of maintaining high strength at temperatures approaching 2000°C. They are envisioned for use in rocket propulsion systems and in future hypersonic flight vehicles. The long-term structural performance of these materials remains largely unknown. A study of 3D imaging of defects and crack evolution under loading at elevated temperatures is underway to provide insight into the operative failure mechanisms and to assess and validate computational models that are being developed concurrently. A hot cell was therefore developed that can apply tension or compression to samples while heated to ~2000 C whilst also being imaged by x-rays [3].

The cell [4] is shown schematically in Figure 1. Briefly, it consists of a ~150 mm diameter water-cooled aluminum sphere that supports six 150 watt radiant lamps with elliptical reflectors that illuminate a ~5 mm diameter uniform hot zone region in the chamber center. The sample is required to fit within this hot zone. Typically the sample size used was 25x2x2mm and is held vertically with its center in the hot zone by top and bottom water cooled grippers. Figure 1b shows the cell cross-section with sample and grippers. The top gripper is motorized for sample tension or compression up to 2 kN. A cylindrical aluminum window, 300 μm thick, occupies the middle horizontal cell section and is thin enough to allow x-rays to illuminate the sample (60% transmission at 20 keV). The window is part of the loading train and finite element analysis indicated that ~2 kN is about the maximum load the 300 um thick window can operate before buckling. The entire cell is required to rotate 180° for recording a tomographic data set. The cell can be evacuated or operated under selected gas pressure. The example shown in Figure 1c is of a 3D woven structure of C fibers embedded in a SiC matrix. The sample has been heated to 1750^{o}C in vacuum and loaded with a tension of 130N. The tomography image reveals a crack in a transverse tow that had propagated across the specimen width parallel to the tow axis but deflected and arrested when it impinged on the longitudinal fiber tows.

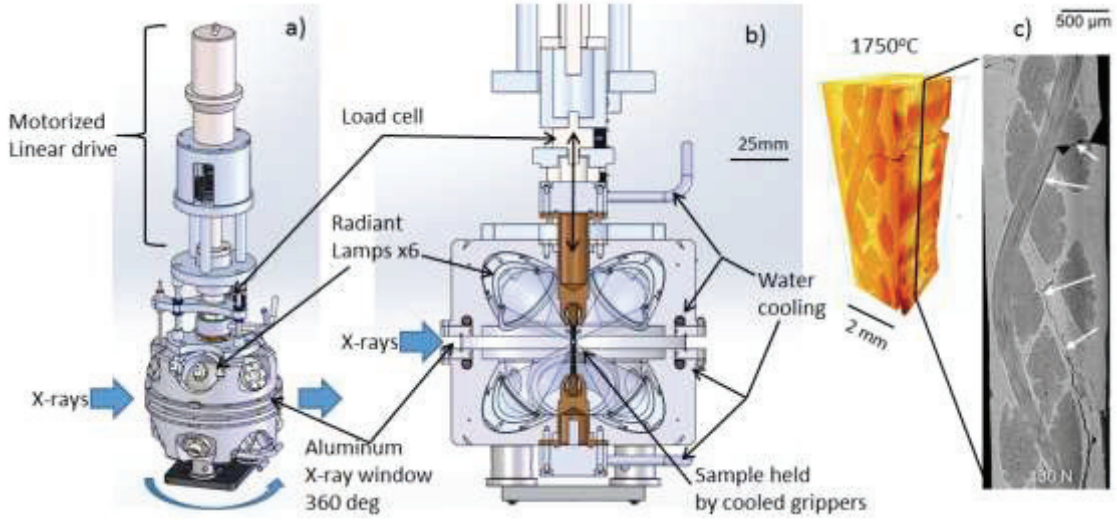

FIGURE 1. a) 3D model of the high temperature cell used for mechanical loading of samples. b) Section through load cell showing centered sample held by cooled grippers. c) Woven CMC under 130 N axial tension at 1750 C showing internal crack pattern as indicated by arrows in vertical tomographic section (right image).

Infiltration and Pyrolysis during CMC Fabrication

A second class of CMCs comprise SiC fibers in SiC matrices. Targeted applications include critical components in combustors and turbines of aero engines which are more efficient when operated at higher temperature [5]. Among several candidate processing methods, one involves infiltration of organometallic polymer precursors (e.g. allylhydridopolycarbosilane) into fiber preforms via resin transfer molding, followed by thermal decomposition (i.e.

pyrolysis) at moderately high temperatures to form SiC. [6]. To address fundamental issues in the evolution of matrix microstructure during processing, infiltration experiments were performed on unidirectional SiC fiber preforms packed within thin-walled quartz tubes (outside diameter 1.8 mm, wall thickness 0.15 mm). Following polymer infiltration and subsequent curing at 120°C for 2 hours in an external oven, the sample was mounted on the lower gripper in the hot cell and underwent pyrolysis over a 16 hour period as the temperature was raised to 1200°C in an argon atmosphere. During his time, 3D tomographic data sets were recorded every 15 min., allowing observation of shrinkage and crack formation of the SiC polymer during pyrolysis.

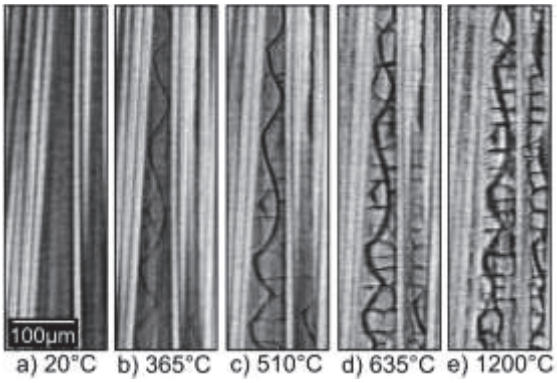

FIGURE 2. Sequential section images showing CMC matrix shrinkage crack formation from 20-1200°C.

Figure 2 shows the formation and evolution of matrix cracks, driven by mass loss (23%) during pyrolysis and material property changes upon conversion from polymer to ceramic. One of the key characteristics of the cracks (presently under investigation) is the degree of contiguity: contiguity being essential if subsequent infiltration cycles are to be successful without void formation which, if formed act as mechanical defect centers.

High Temperature Cell for Ablative Heat Shields

Spacecraft and planetary probes use designed ablative materials as heat shields when entering atmospheres at high speed. A detailed understanding of the response and decomposition of the material under high-enthalpy is required to optimize the heat shield design to ensure that the spacecraft survives re-entry without an unduly heavy shield that would reduce the available weight budget for the payload. To that end, an extensive modelling effort is underway [7] where key aspects of the material response model can be confirmed by 3D imaging at various stages of the decomposition of heat shield material. One particular heat shield material developed by NASA is PICA (Phenolic Impregnated Carbon Ablator) and consists of a network of carbon fibers with phenolic filler. As a first stage to understanding the oxidative decomposition, the carbon fiber network alone was heated to 900 K in 100 torr air. The sample was contained in a thin walled quartz tube (internal diameter = 3mm) through which air was flowed (rate = 1 mg/sec) yielding laminar flow of 2.7 m/sec ahead of the sample. Three intervals of carbon fiber oxidation were achieved as follows:- air flow was switched on for 10 minutes followed by helium to freeze the reaction and a tomography scan recorded. Figure 3 shows significant loss of material over a 30 min reaction time. The data is being used to directly measure the change in tortuosity and porosity values and validate oxidative reaction models currently under development.

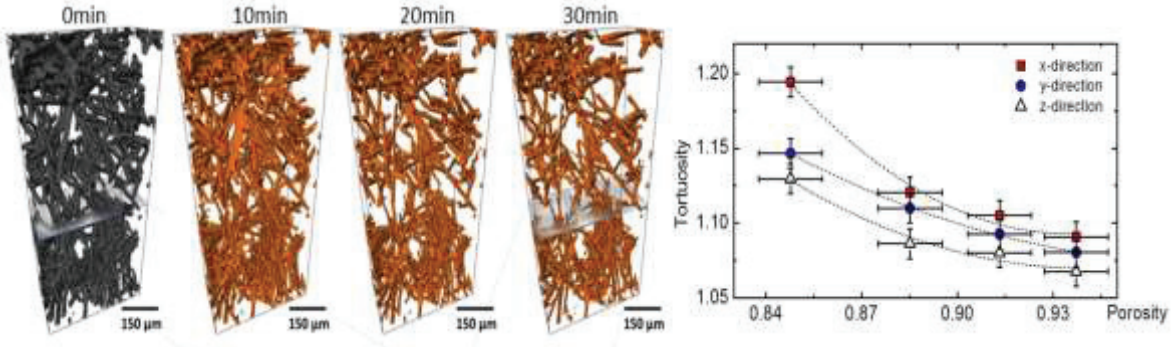

FIGURE 3. Left- time sequence of the oxidation of bare PICA carbon network in 100 torr air at 900K. Right – Tortuosity versus Porosity plot as measured from the 3D data.

Three-Point Bending of Nuclear Reactor Core Graphite at 1000°C

A graphite reactor is a nuclear reactor that uses graphite as a neutron moderator, thus allowing un-enriched uranium to be used as nuclear fuel. In many of these reactor designs, the graphite also functions as structural

components; therefore the deformation and fracture of this material is of great importance to the reliability and the integrity evaluation of the core. The requirement is to measure the graphite's mechanical properties at temperature, typically ~1000°C - this being the outlet temperatures from a Gen IV reactor. The three-point bending (3PB) test is one of the ASTM recommended mechanical property measurements and can be carried out in the hot cell. Flexural strength and fracture toughness can be measured directly and this technique avoids the specimen gripping problem associated with the uniaxial tension setup. Figure 4a shows the 3PB assembly as an additional fixture to the cooled top and bottom grippers of the hot cell. The specimen contact bend points are by solid ceramic cylinders producing a bending span of 10 mm. The ceramics are captured in stainless steel supports that are in turn held in the cooled bronze grippers. The rectangular beam specimen is trapped between the three ceramic rollers in the horizontal plane and receives the radiant heat from the six lamps directed at it. Temperature gradients from the specimen to the cooled grippers are managed as the region of interest is at the center of the specimen which is thermally isolated by the three ceramic contacts. The central part of the specimen has its temperature monitored by a thermocouple held in place with ceramic glue. A stable temperature of 1000°C was achieved in an argon atmosphere. A typical specimen size was of dimensions 15x4x4mm. As 180 degree of specimen rotation was required during a tomography scan, imaging through the long axis of the specimen (15 mm) is necessary. However, for the low absorbing graphite material and the filtered white light used this was not a problem. An exposure of 50 msec and a total scan time of approximately 8 mins was adopted at each load step; incremental steps of loading were applied to allow the measurement of R-curve as the cracks extend through the complex porous microstructure.

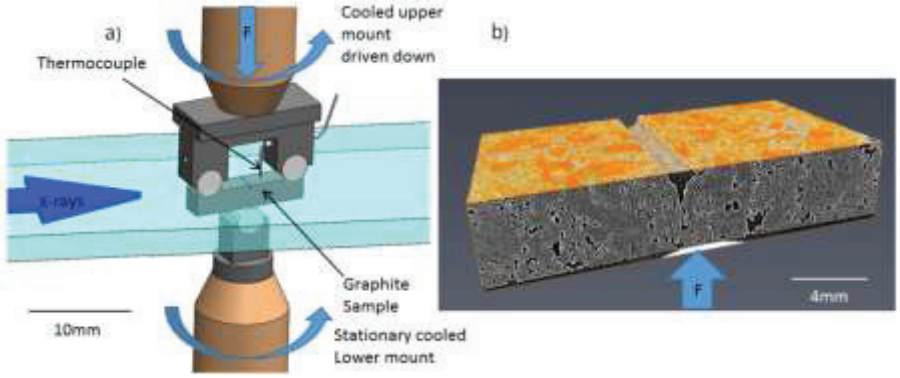

FIGURE 4. a) Schematic layout of the three-point bender inside the hot cell. b) 3D view of a notched Gilsocarbon nuclear graphite specimen fractured under load at 1000°C

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REFERENCES

1. B.Cox, Q.Yang, Science , **314** no. 5802 pp. 1102-1107 (2006)
2. A. A. MacDowell ; D. Y. Parkinson ; A. Haboub ; E. Schaible ; J. R. Nasiatka ; C. A. Yee ; J. R. Jameson ; J. B. Ajo-Franklin ; C. R. Brodersen ; A. J. McElrone, "X-ray micro-tomography at the Advanced Light Source", Proc. SPIE 8506, Developments in X-Ray Tomography VIII, 850618 (2012);
3. H.Bale, A. Haboub, A.A. MacDowell, J.R. Nasiatka, D.Y. Parkinson, B.N. Cox, D.B. Marshall, and R.O. Ritchie, Nature Materials **12**, 40-46 (2013).
4. A.Haboub, H.Bale, J.R.Nasiatka, B.N.Cox, D.B.Marshall, R.O.Ritchie, and A.A.MacDowell, Rev. Sci. Instrum. **85**(8), 083702 (2014).
5. K. Wood, "Ceramic-matrix composites heat up", High-Performance Composites, November 2013
6. R. Naslain. Composites Science and Technology, **64**,155–170 (2004).
7. J.Lachaud, I.Cozmuta, N.N.Mansour , *J. Spacecraft Rockets* **47**(6):910-921 (2010)