Development of In-situ X-ray imaging and μ-CT facility under load conditions at imaging beamline Indus-2

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ABSTRACT

Synchrotron based X-ray micro tomography is a versatile technique to study material microstructure, density, composition etc Especially under variable load conditions, such studies are extremely important to evaluate material behavior in real life applications. We have developed an in-situ X-ray micro tomography facility at imaging beamline Indus-2 to study material microstructure under compression and tensile load conditions. This allows visualization as well as image based quantitative analysis of microstructural changes under external loads and obtains its correlation with mechanical, and transport properties. In this report, we have discussed the details of this experimental facility, challenges, advantages and a case study of Aluminium (Al) foam sample under compression load.

Keywords: In-situ loading, X-ray tomography, synchrotron imaging, material microstructure

Introduction

Microstructure, along with physical properties of constituent material phases, plays an important role in defining the macroscopic properties of the material. Deformation mechanism, strain accumulation and localization, cracks initiation and failure under load during operation, are extremely important properties of materials for deciding their utility in real-life applications[1,2]. A strong and defining correlation of microstructure with mechanical and transport properties of materials is established through empirical numerical relations or finite element modeling[3-5]. Advanced materials such as polymers, ceramics, composites, bio-materials etc. are being designed and developed with specific microstructure, density and composition to achieve desired mechanical and transport properties[6-8]. 3D evaluation of microstructure is conventionally done using electron microscopy with successive serial sectioning of the sample but modification of structure during sample preparation leads to some ambiguity in the structure property relation models[9,10]. In-situ micro-tomography , which provides 3D microstructure and density map with high resolution and contrast at different load conditions ,is one of the most suitable techniques for this purpose[11]. Among various mechanical loads, compression and tensile stress is most commonly encountered in practical conditions [12-14]. Therefore we have developed a facility for carrying out synchrotron based high resolution and high contrast X-ray micro-CT experiments under in-situ compression or tensile load conditions. In this report, we discuss details of the experimental facility developed at Imaging beamline, Indus-2 synchrotron source ,RRCAT Indore, and a case study of Aluminium (Al) foam under compression load.

Materials and Methods

For In-situ X-ray micro-CT experiment under compression mode, samples of standard size need to be prepared either in cubic/cuboidal or cylindrical form. The height of the sample need to be 15 mm, which can be compressed upto 5 mm. In tensile mode, sample need to be prepared in the form of dog-bone shape for which active length between jaws should be 10 mm before initiating the tensile loading which is stretched upto maximum 20 mm. In-situ experiments require acquisition of several micro-CT scans at different loading condition under the identical beam and detector conditions. A faster rate of data acquisition (typically few minutes per scan) at Synchrotron beam is particularly important to acquire

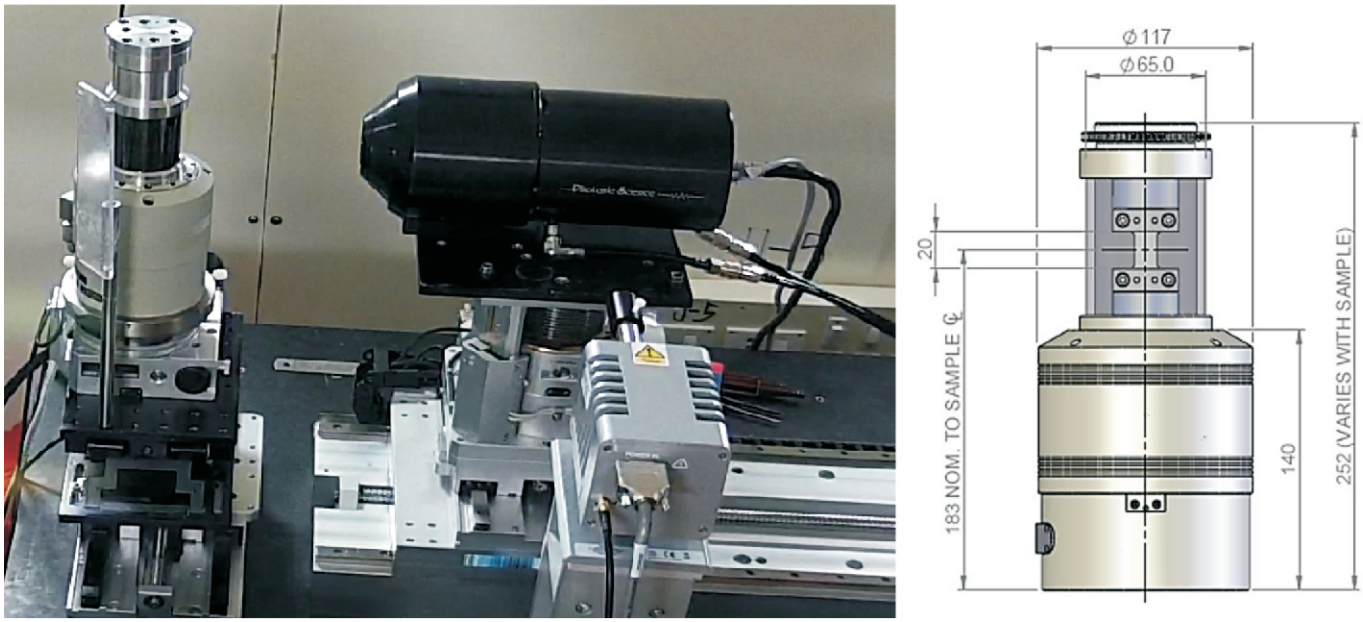

Fig. 1: The In-situ X-ray µCT facility at imaging beamline, BL-4 of Indus-2 and design of In-situ compression and tensile stage

several such scans at different loading conditions, which in turn, is helpful in analysis of stress-strain behavior of materials, and defining structure-property relation. The facility for In-situ X-ray micro-CT developed at imaging beamline consists of synchrotron beamline operated in monochromatic and white beam modes, a sample rotation stage, a high resolution, high frame rate X-ray imaging camera, manipulation system for sample and detector to allow their alignment by orthogonal translation and rotation, and an In-situ loading device (Fig. 1)[15]. The system is equipped with 500 N & 3000 N load cell with 1% accuracy. The smallest step of load applied is 1/1000 fraction of maximum range of load cell being used. The samples can be compressed or pulled with speeds in the range 0.1 mm/minute to 1 mm/minute.

Data acquisition protocol and automation

Data acquisition for In-situ micro-CT experiments includes several tasks such as optimization of experimental parameters, sample alignment with beam and camera, applying suitable load or extension to the sample, collection of micro-CT scan data at different load condition, re-initialization of set-up after each micro-CT scan and collection of reference, background images. The first tomography scan of the sample is taken initially in the unloaded condition and then sample is stepwise loaded to a predefined stress (loads) or strains (extensions). At each loaded position, micro-CT scan is taken while keeping the sample at constant load/extension. Entire process is carried out remotely from the control room by establishing remote communication of in-situ rig controller from control room PC without any human intervention once it is initiated by user.

Post processing and analysis

Each micro-CT scan data is normalized , background corrected and then tomography reconstruction is carried out.The quantitative parameters from micro-CT projections and reconstructed slice images under various load conditions are derived. Variation of measured parameters such as porosity, pore size, shape, etc. can be studied with load. These microstructural properties are related to stress-strain behavior and other physical properties of materials through empirical relations or finite element modeling. The study is also useful for visualization of strain field mapping, strain localization at pores, voids, interfaces, cracks and inclusions geometry, surface under load condition, and measures their capacity to cause fracture in the materials. Other potential studies are 3D fatigue crack initiation and propagation under cyclic load and its dependence on cracks, microstructure, pores, particles, surface, defects, dislocation etc.

Case Study: Al foam under compression load

We have carried out In-situ X-ray micro-CT study under compression mode on several materials such as polyurethane foam, metal foam (Al) and Polymer fiber aerogels materials. As an example study, aluminum foam sample under compression is presented here. An Al foam sample of dimension 6.4 mm x 6.2 mm x 14.4 mm was first extracted from the large block of sample. The initial load measured at this position was offset to zero and In-situ

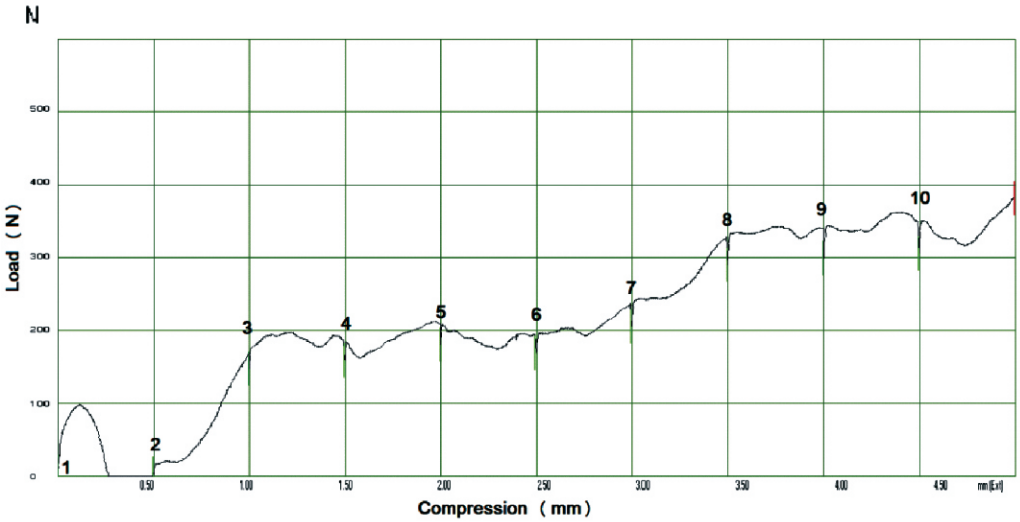

Fig. 2: Load vs. compression curve for In-situ compression of Al foam sample

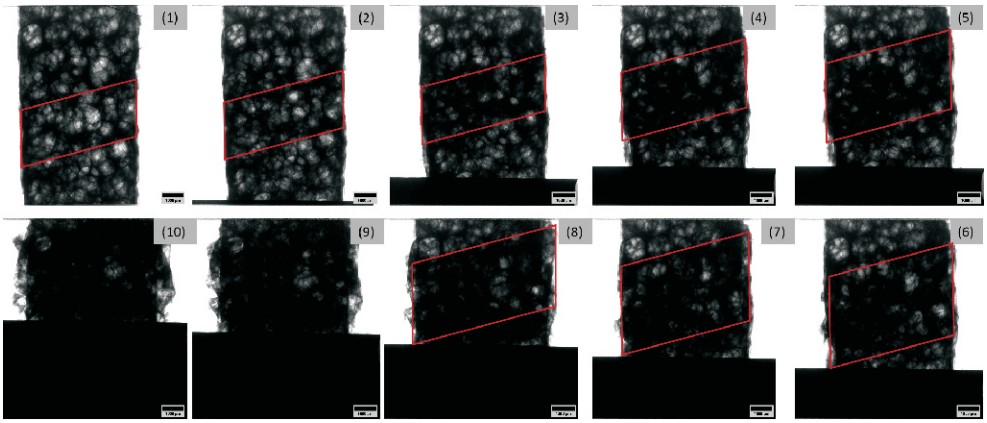

Fig. 3: X-ray projection images of Al foam at different compression loads. Sample densification with load and buckling is highlighted in red quadrangle

micro-CT scan was completed under this unloaded conditions. Further to this, automated data acquisition code was run to carryout complete in-situ loading micro-CT scan in total 10 steps with extensometer increments of 0.5 mm at each step until the sample was crushed at approximately 400 N loads. The force compression curve shows (Fig. 2) an initial rise and then fall in the force during first compression of 0.5 mm. In the second compression step of 0.5 mm, elastic behavior with linear rise in load with compression is observed. From 3rd to 6th compression, no further rise in the force is seen with increasing compression which is first densification region. Again, at 7th compression, some rise in the force is seen followed by further densification of the sample in 8th to 10th compression. This behavior of force-compression curve need to be explained by microstructural changes in the samples.

Fig. 3 shows the first projection images of the sample at different compression conditions. These images clearly show that there is a region in the sample (highlighted in the red quadrangle), which is more specifically compressed under load. As the load increases, the densification of that particular region also increases whereas other regions of the samples are more or less unaffected until the compression reaches to 6th position. This particular phenomenon in mechanical engineering is known as strain localization and strain band formation in the sample[16]. All the compression applied to the sample is localized and transferred to the strain bend until a specific level of compression. Further compression leads to the densification of other regions too which is reflected in the projection images of 6th to 10th steps of compression. Observation of these projection images under compression load partially explains the behavior of Al foam sample in force-compression curve.

In order to get more insight to the microstructural changes in Aluminum foam under load, the vertical micro-CT slices of the sample under different load conditions are shown in Fig. 4. The reconstructed slice images clearly show the cellular microstructure of the foam which is

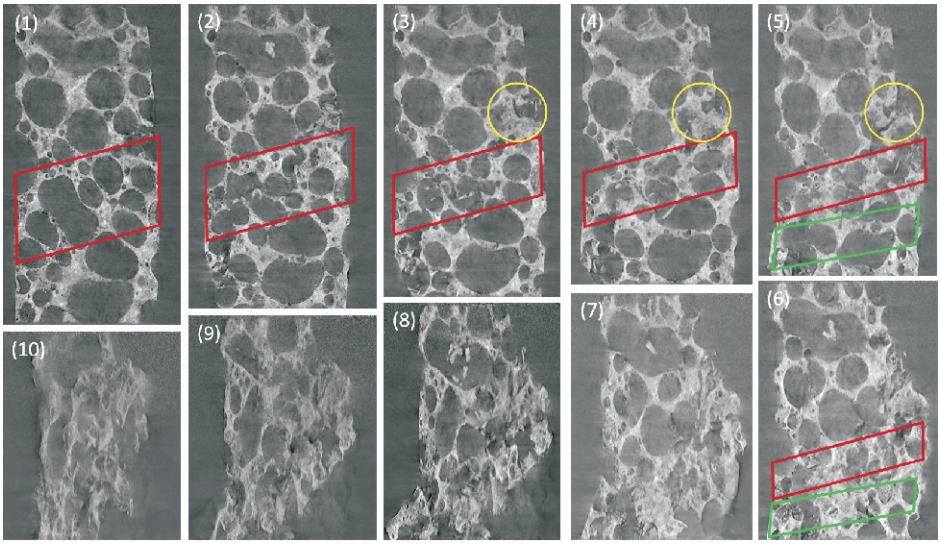

Fig. 4: Tomographic slice images at identical depth in the sample show microstructural changes in the Al foam. Cell collapse band in the middle is highlighted in red quadrangle

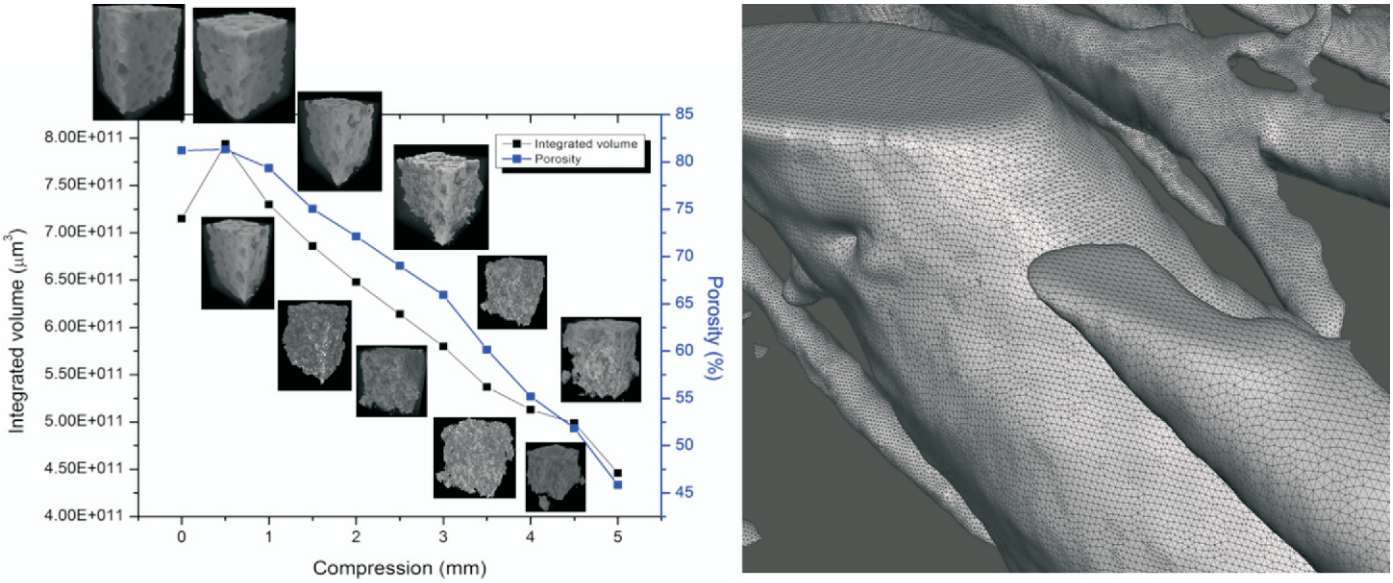

Fig. 5: Quantitative variation of porosity, integrated volume and 3D volume images of sample with compression. 3D volume mesh data generated from the micro-CT data

composed of interconnected large open cells of various sizes separated by Aluminum struts of varying thicknesses. The struts also enclose some closed pores of small volumes at random locations. When load is applied, it affects the shape, size of cellular structure and struts. It is seen in the foam of cell volume reduction, cell wall breaking etc. leading to densification of the sample as observed in the projection images in Fig. 3. The initial dip observed in the load compression curve is due to sudden breaking of certain struts and collapse of some cells in the region highlighted in the red quadrangle in Fig. 4. This collapse of the cells is due to brittle nature of aluminum and strain localization at the thin walled struts in that region. Further load leads to reduction in the cell volume in the form of elastic deformation at the 2nd compression step followed by plastic deformation which is seen in form of densification observed in load compression curve and projection images from steps 3rd to 6th compression. Further compression leads to elastic deformation and cell collapse in the second quadrangle region highlighted in the green, which is seen at 7th to 10th compression.

Discussion

The microstructural changes in the cellular structure and struts of Aluminum foam sample under in-situ compression load are observed in the form of cell volume reduction, cell collapse, and strut breaking etc. which clearly explain its compression behavior. Quantification of porosity and integrated volume of the sample at different load conditions and their corresponding 3D images are shown in Fig. 5. This plot identifies that both, the volume as well as porosity of the sample reduces as the load

increases which is seen in the force-compression curve of the sample. Further to this, 3D mesh data of Al foam sample in unloaded condition is also generated as shown in Fig. 5, which can further be used for finite element modeling of the sample to establish structure-property relations.

Conclusion

In-situ micro-CT facility developed at imaging beamline is useful in studying microstructural variation in the materials under compression and tensile load condition and establishing correlation of these changes with its deformation behavior. A case study of Al foam sample under compression load is presented to show the potential of the facility. In this experiment, the compression behavior of Al foam is quite clearly explained through underlying microstructural changes in the cellular shape, size, volume, porosity and strut breaking. Strain localization, stain band formation and sample buckling are some of the effect causing the peculiar force compression curve of the sample recorded during in-situ micro-CT experiment. Further development of this In-situ X-ray micro-CT facility is planned under other mechanical loads such as impact, shear and torsion loading as well as under thermal load condition at elevated and cooled temperatures.

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