In Situ 3D Synchrotron Imaging of Failure Processes in Engineering Materials

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Abstract. The micrometer range resolution of Synchrotron Radiation Tomography has developed an important technique for characterizing the three dimensional (3D) microstructure of materials. This technique was used for imaging Aluminum-alumina composites, porous aluminum and AA 6061 alloy under different loading conditions. The experimental set-up was installed at the Biomedical Imaging and Therapy (BMIT)'s 05B1-1 beamline at Canadian Light Source (CLS). The experimental stage has been designed for conducting the in-situ experiments. The unique feature of this experimental stage is that the sample can be rotated torsion free for taking the images for Computed Tomography imaging during any loading conditions. The developed experimental system and technique allows deciphering the internal structures of composites, porous materials, multiphase alloys and to observe the failure processes such as crack initiation, crack propagation, void formation, void coalescence and the fracture process during loading of materials.

1. Introduction
To optimize materials properties and performance, it is important to relate mechanical or physical properties to microstructure of the materials. In manufacturing processes, these relationships can be used to optimize the properties of materials for various applications and service conditions. In 2D analysis of crack propagation the crack pass cannot be easily imaged since in some cases the same crack disappears from view and re-emerged in other place. 3D imaging of the structure will allow understanding better the true path of crack propagation. 3D imaging would be an important tool for investigation of the porous structure of materials and their failure mechanisms. Metal matrix composites (MMC's) have various industrial applications because of theirs high specific strength. 3D imaging can be used to investigate the structural changes during loading and failure processes of the
composites. An experimental sample stage has been designed and fabricated for performing various
types of dynamic experiments. Aluminum-alumina composite manufactured by ARB process [1, 2],
Porous aluminum without loading and AA 6061 under different loading conditions were imaged by
using Computed Tomography (CT) at the Biomedical Imaging and Therapy (BMIT)’s 05B1-1 beamline at Canadian Light Source (CLS).

2. Experiments
Computed tomography using synchrotron radiation at CLS is used to reconstruct 3D images of the
model samples. The energy range at BMIT-BM at CLS is 8 – 40 KeV that energy is suitable for light
weight materials like Aluminum, however is not suitable for the material having high atomic weight.
For high Z materials we need higher energy. The imaging resolution of our present study is 4 µm.
Since our present experiments are focused on understanding failure mechanism of aluminum and
aluminum composites, beam energy of about 30 KeV is sufficient for obtaining measurable contrasts.
3D images are reconstructed using softwares e.g. ImageJ, Syrmeq and Amira.

2.1. Experimental Sample Stage
Experimental stage was designed and fabricated for in-situ dynamic loading experiments at BMIT
beamline in CLS. Sample size range is 1/2” (before elongation) to 3.5” (after elongation). The load up
to 5kN can be applied. Sample is strained from both sides with
same strain rate. Thrust bearing at the sample mountings and
gear transmission system facilitate torsion free rotation of
the sample for taking the image
projections by rotating the
samples for Computed
Tomography (CT) during
loading conditions. Rotational
step resolution is 0.003˚ per step.
For our experiments we used
angular rotation of 0.12˚ per
projection step. Rotational
Speed is up to 68 rpm. The
tensile and rotational power
transmissions are shown in
Figure 1 and Figure 2.

Figure 1. Tensile load power transmission
1. Stepper motor, 2. Worm gear box 3. Mountings 4. Top sliding plate 5. Bottom sliding plate 6. Gear transmission shaft 7. Transmission gears 8. Thrust bearings in sample mountings 9. Thrust bearings in gear transmission 10. Supporting ball bearings 11. Sliding shaft around the transmission shaft 12. Sample

**Figure 2.** Rotational power transmission

2.2. *Model Samples*
Aluminum-alumina composite is made by oxidizing and rolling after folding of aluminum sheet (ARB process). Sintered aluminum is made by sintering the aluminum powder at 550 °C for an hour after cold pressing it at 150 MPa. These two samples were imaged without any loading condition. The sample tested under tension is made by AA 6061 alloy (Figure 3). The central area of the specimen, marked in fig.3 is imaged.

**Figure 3.** Tensile model sample

3. *Results and Discussion*
This imaging technique allows us to see the distribution of alumina inside the aluminum matrix (Figure 4a) and also to observe formation of voids near the aluminum-alumina interfaces which can be considered failure in service condition. This 3D imaging technique could be helpful to improve manufacturing process (ARB) and to manufacture desired structure of the composites. In case of sintered porous aluminum (Figure 4b) we found that there is a gradient of porosity in the direction from top to bottom. Obtaining such information should be helpful for a design of the manufacturing process of such type of porous structures.

**Figure 4a.** ARBed Aluminum-alumina Composite  
**Figure 4b.** Sintered Porous Aluminum

The in-situ 3D imaging is done in AA 6061 model sample at different strain levels. Figure 5a is showing the 3D image of the selected region (see Figure 3) of the sample after 4% elongation. Figure
5b and 5c are showing the magnified views of the crack propagation inside the sample. It is visible how the cracks are propagating and changing the direction of propagation in 3D structure. Figure 5d (top part) figure 5e (bottom part) is showing the SEM Fractography after fracture of the sample which are conforming the similar crack propagation.

Figure 5a. 3D view after 4% elongation
Figure 5b. Magnified view of crack propagation
Figure 5c. Magnified view of crack propagation

Figure 5d. Fractography of top part of the fractured sample
Figure 5e. Fractography of bottom part of the fractured sample

4. Conclusion
High-resolution tomography (micro CT and nano CT) is used for inspecting metals, composites, porous materials and ceramics. Material phase compositions and distribution as well as voids and cracks are visualized in three-dimensions at microscopic resolution. The method can therefore, be used to analyze the failure processes of technologically important materials. It is evident that void formation as well as alumina in aluminum matrix can be visualized in 3D using SR-CT. The proposed imaging technique can help in deciphering the failure mechanisms of important engineering materials

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