## First Results of Integrating a Compression/Tension Load Cell with Nano-scale X-ray Transmission Microscopy

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Understanding mechanical failure, crack propagation, and compressive behavior at the submicrometer scale is essential for tailoring material properties for structural performance. Typically, tension or compression loading is needed to understand these processes. Here we demonstrate the coupling of a custom compression/tension load cell with a nano-scale X-ray transmission microscope.

The load cell, capable of 9 N of force, is custom designed to fit into an Xradia (now Carl Zeiss X-ray Microscopy Inc., Pleasanton, CA) UltraXRM-L200 nano-scale X-ray transmission microscope. Driven by a piezoelectric motor, the load cell has a total displacement of 500  $\mu$ m and can be operated in either uniaxial tension or uniaxial compression configurations. In the latter configuration, the bottom platen is driven upwards into a stationary top platen. Figure 1 (left) presents a photograph of the custom load cell. A magnified view (Fig. 1, right) shows the cell in uniaxial compression mode, before alignment of the bottom platen with the stationary top platen. A variety of samples will be described, such as the compression of silicone polymer foam ligaments (Fig. 2A-D) and hollow glass microspheres (Fig. 3A-B). The microspheres were acquired from  $3M^{TM}$  (S22 Glass Bubble Series) and vary in diameter, ranging from ~20  $\mu$ m to ~65  $\mu$ m, with a median diameter of 35  $\mu$ m [1]. The representative crush strength of the microsphere is quoted as 2758 kPa. In addition, we will present results of tensile strain of Al-Cu eutectics (data not shown).

Figure 2A-D displays nano-scale X-ray transmission radiographs of the compression of a single silicone polymer ligament, imaged in Zernike phase contrast mode. Edges are visible inside the ligament, corresponding to the diatomaceous earth filler used in the silicone polymer synthesis. The ligament can be seen bending in Fig. 2B and 2C, leading to densification in Fig. 2D. Figure 3A-B highlights the uniaxial compression of a hollow glass microsphere, used in the synthesis of syntactic (i.e., glass microsphere-templated) polymer foams. Several microspheres of varying diameters can be seen on the bottom platen (Fig. 3A); however, the large sphere (diameter = 22.7µm) is aligned so that it is the only microsphere being compressed. Figure 3A shows this microsphere in contact with the top platen, which is fabricated from diamond, thus allowing a sufficient number of X-ray photons to transmit through the platen to the detector. Figure 3B shows the compressed microsphere at 19% strain, as the bottom platen is being driven upwards. The software which drives the load cell is capable of acquiring and recording force data. By measuring the diameter of the sphere from the radiograph and by averaging the acquired force data, it is possible to construct a stress-strain curve of the uniaxial compression (Fig. 3C). From the manufacturer, the representative crush strength of the microspheres is 2758 kPa; here, we find the crush strength of one microsphere to be  $1338 \pm 127$  kPa at a strain of 44%.

From these first results, we show the feasibility of coupling a custom load cell with nano-scale X-ray transmission microscopy. Radiographs acquired through this coupling reveal the damage of materials due to compressive or tensile strains, with sub-micrometer resolution.

## References

[1] 3M<sup>TM</sup> Microspheres Selection Guide. http://multimedia.3m.com/mws/media/130063O/3mtm-glass-bubbles-selection-guide.pdf?fn=MicroSelectGuide\_Celum\_9841447.p (02/12/2015).

Figure 1. Optical picture of the custom load cell (Left) and a magnified view of the load cell in uniaxial compression mode before fine alignment.



Figure 2. Nano-scale X-ray transmission radiographs, acquired in Zernike phase contrast mode, of a polymer foam ligament (A-D) under uniaxial compression. The displacement of the compression cell was  $0 \mu m$  (A),  $10 \mu m$  (B),  $20 \mu m$  (C), and  $30 \mu m$  (D).



Figure 3. Nano-scale X-ray transmission radiographs, acquired in Zernike phase contrast mode, of a glass microsphere before (A) and during (B) uniaxial compression. C) The stress-strain curve of a glass microsphere, obtained while undergoing uniaxial compression in the custom load cell. Data points are the average stress and error bars correspond to the standard deviation of the measured stress.