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Characterization of Metal Doped Polymer Capsules using Confocal Micro X-ray Fluorescence Spectroscopy and X-ray Computed Tomography

Nikolaus L. Cordes[1], George J. Havrilla[2], Kimberly A. Obrey[1], Brian M. Patterson[1]

[1]. Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM USA
[2]. Los Alamos National Laboratory, Chemistry Division, Los Alamos, NM USA

Defect Induced Mix Experiment (DIME) spherical capsules, utilized as National Ignition Facility (NIF) targets, are composed of a 42 µm-thick polymer shell which has been doped with a 2 µm-thick inner layer of 1.5 at.% germanium and separate 2 µm-thick outer layer of 1.5 at.% gallium. The metal-doped layers are separated by a 3 µm-thick polymer layer. The LANL DIME campaign requires that the characterization of these capsules must provide better accuracy than the fabrication tolerances. X-ray characterization techniques, such as confocal micro X-ray fluorescence (MXRF) spectroscopy and micro- and nano-scale computed tomography (CT) allow for the nondestructive elemental and spatial characterization, respectively, of such samples.

Confocal MXRF was performed using a custom-built confocal MXRF instrument [1-3] with a spatial resolution of 30 x 30 x 50 µm. Line scans of DIME capsules were performed in the y direction at 3 µm intervals over a 200 µm distance. The capsule was then rotated one degree and the y-direction line scan repeated; this procedure was repeated over two full sample rotations (720°) [4]. This procedure was used to keep instrument artifacts to a minimum. The counts of the y-direction line scan were then summed and averaged as a function of sample rotation (see Fig. 2). Micro-CT was performed using a Micro XCT instrument (Xradia, Pleasanton, CA); the voxel sizes of reconstructed micro-CT slices are 600 nm, however the dopant layers were not completely distinct. Nano-CT was performed using an Ultra XRM (Xradia, Pleasanton, CA); the voxel sizes of reconstructed nano-CT slices are 65 nm, which was more than enough resolution to resolve the layers and directly measure the dopant layer thickness.

Figure 1 shows an X-ray fluorescence spectrum using confocal MXRF of a DIME capsule, acquired at a 1.3 mm distance from the capsule outer edge. The K_α emission lines of both Ga and Ge are detected. The averaged summation of Ga K_α and Ge K_α emission counts, as a function of sample rotation, (Fig. 2), indicates small variations of Ga and Ge concentrations within the DIME capsule. Preliminary statistical analyses of confocal MXRF data (not shown) acquired from four separate DIME capsules indicate no significant sample-to-sample variations in Ga and Ge concentrations. Reconstructed micro- scale and nano- scale CT slice images (Fig. 3) resolve the metal doped and polymer regions, allowing for the nondestructive quantification of the metal doped and polymer region thicknesses. Preliminary analysis of nano-CT data reveal a 44 µm-thick polymer shell, and Ga- and Ge-doped region thicknesses of 1.5 and 2.4 µm. The combination of these three characterization techniques allows for the elemental and spatial resolution of DIME capsules required by the LANL DIME campaign.

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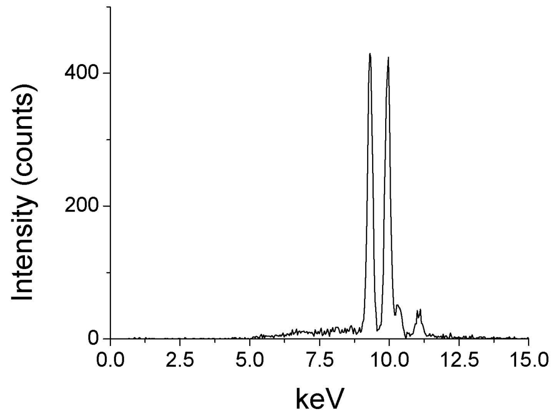

Figure 1. Confocal MXRF spectrum of a DIME capsule acquired at 1.3 mm from the capsule outer edge. Both Ga (9.30 keV) and Ge (9.97 keV) K_α emission lines are detected.

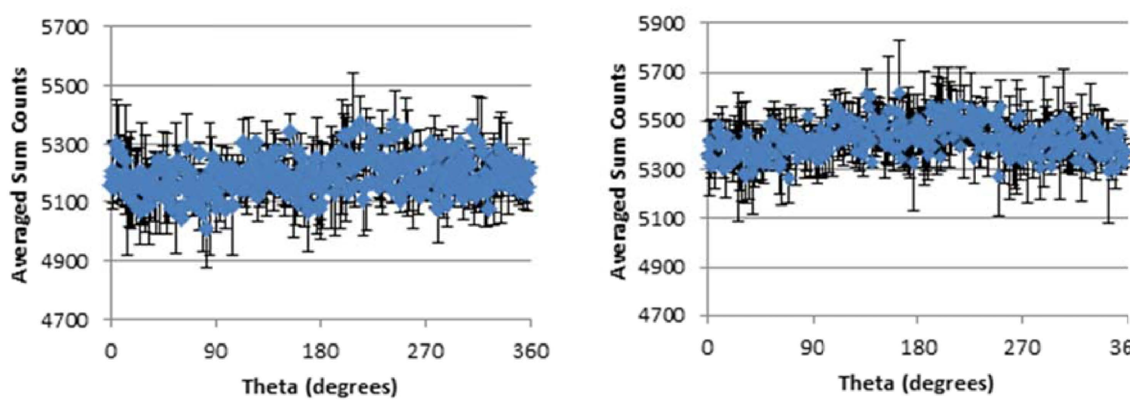

Figure 2. Averaged sum Ga K_α counts (left) and averaged Ge K_α counts of a DIME capsule as a function of sample rotation. Error bars are standard deviations of the averages.

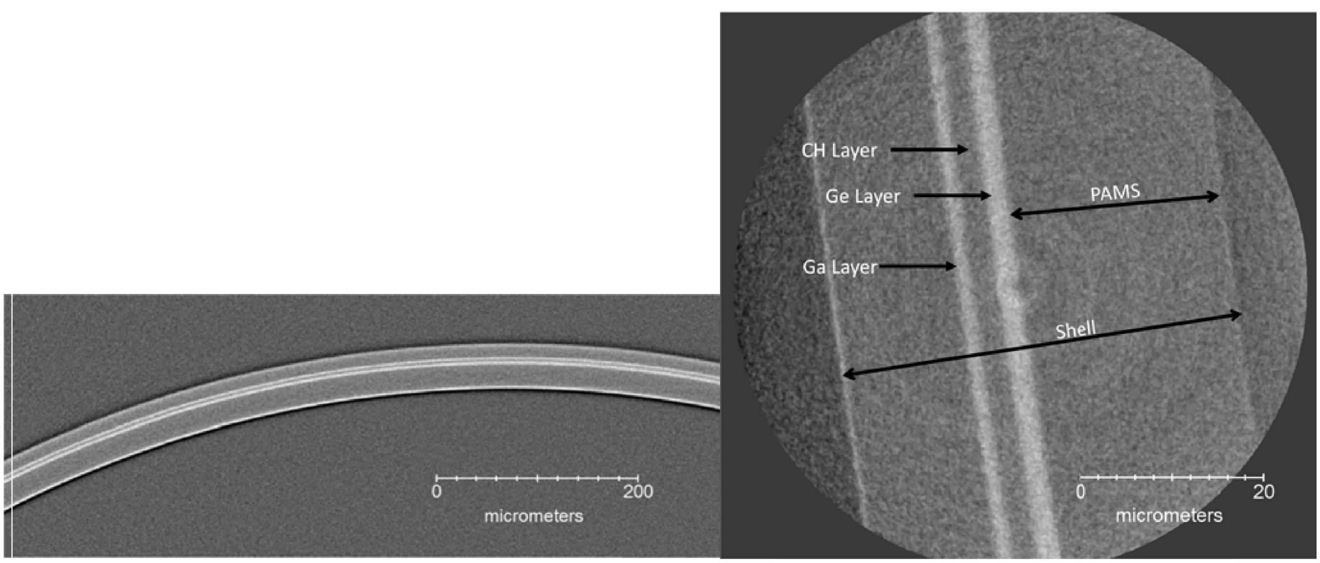

Figure 3. Micro-CT (right, 600 nm voxel size) and nano-CT (left, 65 nm voxel size) reconstructed slice images of a DIME capsule.

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