IN-SITU HIGH TEMPERATURE SPATIALLY RESOLVED X-RAY DIFFRACTION OF TiB_2 UP TO ~3250 ˚C

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In-situ high temperature X-ray diffraction experiments were performed TiB_2 (reported melting point of ~3230 ˚C) up to ~ 3250 ˚C at Argonne National Laboratories, Advanced Photon Source, beam-line 6-ID-D. TiB_2 powders were fabricated into spherical beads via gel casting methods and were densified in a high temperature graphite furnace at 2300 ˚C. These spheres were then levitated in a conical nozzle levitator (CNL) using a forming gas (3%H_2-Ar) to prevent oxidation, while being heated with a 400 W CO_2 laser. The CO_2 laser (10.6 µm) and pyrometer (0.9 µm, with an emissivity correction of 0.35) were aligned to the tip of the TiB_2 bead. The X-ray beam was focused to a width of 0.5 mm and height of 0.2 mm and was used to scan the bead from the tip down until the beam came into contact with the nozzle. A multi-wavelength spectrometer (0.5 µm to 1 µm) is being integrated into a CNL system at UC Davis that will be used in the future to assist with *in-situ* high temperature emissivity corrections. The high-temperature, high resolution, spatially resolved X-ray diffraction data was used to calculate the anisotropic thermal expansion of TiB_2 from room temperature up to ~3250 ˚C along with temperature gradients within the levitating TiB_2 bead. Melting was not observed. These *in-situ* high temperature measurements will be critical in developing ultra-high temperature material systems for applications in hypersonic vehicles, nuclear fission/fusion reactors, and spacecraft.

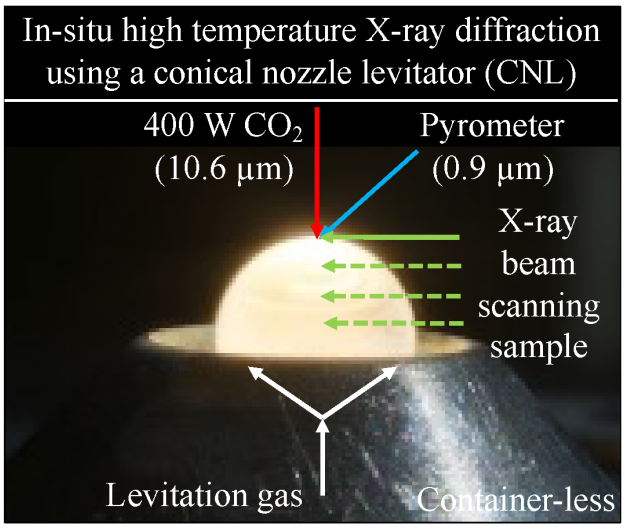

Figure: In-situ high temperature X-ray diffraction of a levitated ~3 mm ceramic bead. The CO2 laser and pyrometer are aligned at the tip of the bead, while the X-ray beam scans the bead from the tip (incident with the CO2 laser and pyrometer) with 0.2 mm resolution, down until the beam encounters the nozzle.