In-Situ High Temperature Phase Equilibria and Thermal Expansion Studies Up to 3000 °C
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Bio: Scott McCormack grew up in the small fishing village of Eden on the Far South Coast of Australia. He completed a Bachelor of Engineering with First Class Honors (H1), majoring in Materials Engineering at the University of Wollongong, NSW, Australia in 2013. He then completed his Ph.D. in Materials Science and Engineering from the University of Illinois at Urbana-Champaign, IL, USA in 2019. He is now an Assistant Professor of Materials Science and Engineering at the University of California, Davis. His research focuses on the interplay of crystal symmetry and energetics of materials in extreme environments for applications in space exploration. More information can be found at: https://mccormacklab.engineering.ucdavis.edu

Abstract

In-situ X-ray diffraction in conjunction with laser-heated, levitated samples and a Quadrupole Lamp Furnace (QLF) have been used for studying material systems, in air, at high temperatures (up to 3000 °C). These in-situ devices have allowed for the elucidation of new crystal structures, anisotropic coefficients of thermal expansion (CTE’s), phase transformations/reactions (symmetry relations) and complex phase equilibria. Here, I will focus on two key experiments conducted within the HfO$_2$-Ta$_2$O$_5$-TiO$_2$ ternary system: (i) The elucidation of the HfO$_2$-Ta$_2$O$_5$ binary in-situ up to 3000 °C and (ii) Directions of zero thermal expansion in the orthorhombic HfTiO$_4$ oxide.

Phase Equilibria in the HfO$_2$-Ta$_2$O$_5$ phase Diagram up to 3000 °C

Ceramic equilibrium phase diagrams have proven to be difficult to produce for materials above 1500 °C. I demonstrate that in-situ X-ray powder diffraction on laser-heated, levitated samples can be used to build phase diagrams. In these experiments, solid spherical samples were suspended and rotated by a gas stream through a conical nozzle levitator, heated by a 400 W CO$_2$ laser at beamline 6-ID-D of the Advanced Photon Source at Argonne National Laboratory. X-ray diffraction patterns suitable for Rietveld refinement were collected at 100 °C temperature intervals and were used to identify phase fields. The liquidus temperatures were determined from levitation and laser-heated recoalescence experiments. The Hf$_6$Ta$_2$O$_{17}^{Orthorhombic}$ crystal structure was solved using the charge flipping method and the Hf$_6$Ta$_2$O$_{17}^{Orthorhombic}$ ⇌ HfO$_2^{Tetragonal}$ + L peritectic reaction at 2250 °C was analyzed using symmetry decomposition.
**Directions of Zero Thermal Expansion in Orthorhombic HfTiO$_4$**

Oxide materials tend to have anisotropic crystal structures, which results in material properties being direction dependent. Typically, oxide materials have positive CTE’s. Yet, in specific directions, negative thermal expansion has been observed over certain temperature ranges. Oxide materials, which exhibit both positive and negative thermal expansion will also have a series of directions which exhibit zero thermal expansion. In-situ X-ray diffraction in conjunction with a QLF has been performed at the National Synchrotron Light Source II (NSLSII) powder diffraction beamline (XPD-28-ID) to track these directions of zero thermal expansion in orthorhombic HfTiO$_4$. These results have important implications for next generation low thermal expansion materials.

Refs:


