In-Situ Phase Equilibria in the TiO_2-HfO_2-WO_3 System up to 2000 ˚C

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The TiO_2-HfO_2-WO_3-temperature phase diagram was investigated up to 3000 ℃ in air with in-situ synchrotron powder diffraction. The high melting temperatures of these multi-component Hf-Ti-W-oxides and their applications as refractory structural materials and in high temperature electronics and sensors motivated this study. The high temperatures required for this study were reached with a quadrupole lamp furnace (QLF) with powder samples mounted in sapphire capillaries in air to achieve temperatures from 25 ℃ to 1800 ℃. Additionally, a 400 W CO_2 laser, with a conical nozzle levitator (CNL), heated solid spherical samples while they were suspended in a stream of air at temperatures from 900 ℃ to 3000 ℃. Powder X-ray diffraction (XRD) data from beamlines 6 ID-D (with CNL system) and 33 BM-C (with QLF) at the Advanced Photon Source at Argonne National Laboratory at 100 ℃ intervals allowed phase-fraction determination by Rietveld refinement and temperature-dependent molar volume measurements. Equilibrium phases were determined by checking the reversibility of phase transformations in XRD experiments. Temperature-dependent thermal expansions of all phases present were measured. This included the isotropic *negative* thermal expansion of HfW_2O_8, which was measured for the first time at its equilibrium temperature range of about 1100 ℃ to 1300 ℃. Phase diagram development is vital in the ceramics industry, as all engineers refer to them during the material selection process to ensure thermodynamic stability of the materials under the required conditions.