DYNAMIC COMPRESSION AND PHASE TRANSFORMATIONS OF BADDELEYITE IN SYNCHROTON-BASED DIAMOND ANVIL CELL EXPERIMENTS.

F. Langenhorst[1], E. Adelhardt[1], U. Mansfeld[1], and H.P. Liermann[2], [1]Institute of Geoscience, Friedrich Schiller University Jena, Germany (Falko.Langenhorst@uni-jena.de), [2]Deutsches Elektronen-Synchrotron (DESY), FS-PE, P02, Notkestraße 85, 22607 Hamburg, Germany.

Introduction: Recently, fast membrane-driven diamond anvil experiments (mDAC) were tested for their suitability to simulate the effects of shock compression in rock-forming minerals [1-3]. A major advantage of such experiments is the ability to obtain time-resolved *in-situ* X-ray diffraction data of rapidly compressed crystalline solids and thus to detect their structural changes directly during the compression and decompression paths. Despite the absence of shock waves in DAC experiments and their distinctly slower compression rates compared to shock experiments, the mDAC technique yielded known shock effects such as the amorphization of quartz and feldspars [1,3] and [001] dislocation glide in olivine [2].

In this study, we performed dynamic mDAC experiments on baddeleyite (monoclinic form of ZrO_2), a reliable geochronometer and accessory mineral in planetary basaltic rocks. The ZrO_2 phase diagram displays several phase transformations along the temperature and pressure axes. Phase transitions along the pressure axis involve the formation of various orthorhombic structures at 6 GPa (ort-I), 22 (ort-II), and 40 GPa (ort-III) [4,5]. Further orthorhombic polymorphs and cotunnite-structured ZrO_2 are predicted to occur at even higher pressures [6]. Here, we focus on the nature and kinetics of these pressure-induced phase transformations, which are much less constrained than the temperature-induced martensitic and displacive transitions.

Methods: Polycrstalline, 1 µm sized ZrO_2 powder served as starting material in two mDAC experiments run up to 37 GPa and 64 GPa. The ZrO_2 powder was loaded into a Re gasket and then dynamically compressed under non-hydrostatic conditions at a rate of 1 GPa/s. X-ray diffraction patterns were acquired at beamline P02.2 of PETRAIII, DESY Hamburg at 25.6 keV using a Perkin-Elmer XRD 1621 fast flat panel detector. Powdered gold was used as an internal pressure standard. Recovered samples were investigated by transmission electron microscopy (TEM) to detect persistent structural modifications in baddeleyite.

Results and Discussion: In line with previous slow compression experiments [4,5], our synchroton X-ray diffraction experiments reveal the immediate transformation of monoclinic baddeleyite to the orthorhombic phases I, II, and III at 6, 22, and 40 GPa, respectively. On unloading these phases even persist without reversion to the baddeleyite structure. The lattice constants *a*, *b*, and *c* of the orthorhombic phases abruptly change at the transformation pressures (Fig. 1), while the overall cell volume almost linearly increases with increasing pressure.

TEM inspection of recovered samples confirms the persistence of orthorhombic phases at ambient pressure and shows the conversion of the material into nanocrystalline aggregates. Individual nanocrystals are strongly strained but otherwise defect-free. These observations indicate the displacive nature of the pressure-induced phase transformations. The persistence of the orthorhombic phases at ambient pressure is attributed to internal strain in nanocrystalline aggregates.

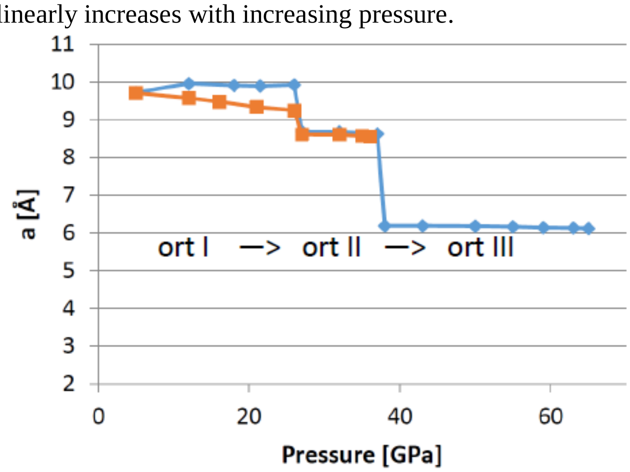

Fig. 1: Lattice constant a of the orthorhombic phases I, II, and III in two mDAC experiments as a function of pressure.

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Additional Information: This work was funded by the DFG via the Gottfried-Wilhelm Leibniz price (LA830/14-1) and the research unit FOR 2285 (LA830/20-1). V. Mohrholz is thanked for their help in preparing DAC experiments.