



# An internally consistent pressure calibration of geobarometers applicable to the Earth's upper mantle using *in situ* XRD

Christopher Beyer<sup>a,b,\*</sup>, Anja Rosenthal<sup>c,d</sup>, Robert Myhill<sup>e</sup>, Wilson A. Crichton<sup>f</sup>,  
Tony Yu<sup>g</sup>, Yanbin Wang<sup>g</sup>, Daniel J. Frost<sup>a</sup>

<sup>a</sup> Bayerisches Geoinstitut, Universität Bayreuth, 95440 Bayreuth, Germany

<sup>b</sup> Institut für Geologie, Mineralogie und Geophysik, Ruhr-Universität Bochum, 44780 Bochum, Germany

<sup>c</sup> Institute of Petrology and Structural Geology, Charles University in Prague, Albertov 6, 12843 Prague, Czech Republic

<sup>d</sup> Laboratoire Magmas et Volcans, Université Blaise Pascal – CNRS – IRD, OPGC, Campus Universitaire des Cézeaux, 6 Avenue Blaise Pascal, 63178 Aubière Cedex, France

<sup>e</sup> School of Earth Sciences, University of Bristol, Bristol BS8 1RJ, United Kingdom

<sup>f</sup> ESRF – The European Synchrotron, 71 avenue des Martyrs, 38000 Grenoble, France

<sup>g</sup> Center of Advanced Radiation Sources, The University of Chicago, 5460 S. Ellis Avenue, Chicago, IL, USA

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## Abstract

We have performed an experimental cross calibration of a suite of mineral equilibria within mantle rock bulk compositions that are commonly used in geobarometry to determine the equilibration depths of upper mantle assemblages. Multiple barometers were compared simultaneously in experimental runs, where the pressure was determined using *in-situ* measurements of the unit cell volumes of MgO, NaCl, Re and h-BN between 3.6 and 10.4 GPa, and 1250 and 1500 °C. The experiments were performed in a large volume press (LVPs) in combination with synchrotron X-ray diffraction. Noble metal capsules drilled with multiple sample chambers were loaded with a range of bulk compositions representative of peridotite, eclogite and pyroxenite lithologies. By this approach, we simultaneously calibrated the geobarometers applicable to different mantle lithologies under identical and well determined pressure and temperature conditions. We identified discrepancies between the calculated and experimental pressures for which we propose simple linear or constant correction factors to some of the previously published barometric equations. As a result, we establish internally-consistent cross-calibrations for a number of garnet-orthopyroxene, garnet-clinopyroxene, Ca-Tschermaks-in-clinopyroxene and majorite geobarometers.

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## 1. INTRODUCTION

Geobarometers are an essential tool for understanding geochemical and geodynamic processes of the Earth's inte-

rior. Their uses are manifold, ranging from the modelling of P-T paths of high-pressure rocks in mountain belts (e.g. Dale and Holland, 2003), the reconstruction of the chemical and thermal state of the lithosphere (Shirey et al., 2001; McKenzie et al., 2005; Smart et al., 2009) to the economic exploration of diamond-bearing rocks and the composition of the transition zone based on inclusions in diamonds (Walter et al., 2011). Geobarometers are routinely used to

\* Corresponding author at: Institut für Geologie, Mineralogie und Geophysik, Ruhr-Universität Bochum, 44780 Bochum, Germany.

determine equilibration depths of mantle xenoliths (Brey et al., 1990; Simakov, 2008; Ashchepkov et al., 2010; Beyer et al., 2015) and have also been used to infer inclusion formation depths in diamonds (Collerson et al., 2010; Wijbrans et al., 2016; Beyer and Frost, 2017). The majority of barometers used on mantle rocks have been calibrated using experiments where sample pressures have been previously calibrated using known high pressure mineral transformations or equilibria. Although the procedures and the transformations employed to calibrate high pressure apparatuses are well established and use transformations such as quartz – coesite (e.g. Bohlen and Boettcher, 1982), and fayalite – ringwoodite (see review in Keppler and Frost, 2005; Holloway and Wood, 2012), these *ex-situ* calibrations have uncertainties propagated from the method used to initially determine the transformation pressure and related to the reproducibility in pressure determination during the barometer calibration. This could potentially result in differences between apparent pressures determined by different barometer equilibria for rocks that came in fact from the same depth. Uncertainties in pressure determinations for calibration reactions arise from the fact that once frictional corrections for high pressure assemblies become necessary, absolute pressure determinations quite often rely on assumptions concerning equation of state measurements employed and the effect of thermal pressure. The reasons for poor pressure reproducibility are multifarious, and include for example, differences in the mode of piston operation in the piston-cylinder apparatus (Bose and Ganguly, 1995), differences in friction corrections of pressure cells (Bohlen and Boettcher, 1982), drift in the oil pressure vs. sample pressure in the multi-anvil apparatus over time and with changing temperature, differences in transformation volume changes and in the assembly design. Furthermore, there is the additional, although probably small uncertainty arising from the pressure effect on the thermocouple emf (Getting and Kennedy, 1970; Rubie, 1999). Some identified inter-laboratory discrepancies, for example, are lacking a plausible explanation, such as the 0.15 GPa difference between the quartz-coesite transition in “friction-free” NaCl cells of Bohlen and Boettcher (1982) and Bose and Ganguly (1995).

Here, we take a more rigorous approach and compare pressures calculated from the equation of states of widely used X-ray diffraction standard materials (Morard et al., 2007; Sokolova et al., 2016) directly with pressures obtained from a series of barometer equilibria, which are applicable to xenoliths and inclusions in diamonds from Earth’s upper mantle (Brey et al., 1990, 2008; Simakov and Taylor, 2000; Simakov, 2008; Collerson et al., 2010; Beyer et al., 2015; Wijbrans et al., 2016; Beyer and Frost, 2017). Multi-chamber capsules, filled with various typical mantle bulk compositions, were employed in order to test a number of geobarometers in the same experiment under equivalent conditions. The multi-chamber capsule method has been successfully applied in several other studies where identical pressure and temperature conditions were essential (Liebske and Frost, 2012; Myhill et al., 2017). We compare the pressures calculated from the X-ray-determined unit-cell volumes of the pressure standards with the pressures

estimated from the geobarometer equilibria and propose corrections, where necessary. The resulting directly comparable calibrations should give confidence in the application of different geobarometers to the same suite of rocks and should yield mutually consistent depths of last equilibration.

## 2. METHODS

### 2.1. Starting materials

A series of bulk compositions were selected to cover the most common mantle lithologies with mafic and ultramafic compositions (Table 1). NMORB4 (Beyer and Frost, 2017) and Mix-1G (Hirschmann et al., 2003) are synthetic glasses with normal hydrous mid-ocean ridge basalt (MORB) and pyroxenitic compositions respectively. Details on the fabrication of the glasses are given in Beyer and Frost (2017). KLB-1 is a fused glass that represents a synthetic spinel lherzolite (Takahashi, 1986), whereas KLB-1n is a simplified synthetic harzburgite glass (Walter, 1998), being more refractory (higher Mg#) and containing no Na<sub>2</sub>O relative to KLB-1 (Table 1). Both glasses were synthesized at 1600 °C in a 1-atm furnace for 90 min. The peridotitic glasses contain μm-sized phenocrystals that crystallize unavoidably during quenching. We also conducted experiments using a synthetic average altered MORB composition, GA20, which is similar to GA2 (Spandler et al., 2008) but with Cr<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub> removed. P2 is a fertile lherzolite oxide mix guided by the composition HZ-1, representative of a MORB source (Green et al., 2010). Oxide powders of GA20 and P2 were prepared using established procedures (Green et al., 2010; Rosenthal et al., 2014). Ir was added as IrO<sub>2</sub> to both GA20 and P2 to act as a redox sensor, however, due to the small size of the resulting alloy and due to mixing with Re we ultimately did not employ the sensor. Approximately 1 μl of distilled water was also added to the dry peridotitic compositions to promote equilibration.

### 2.2. Experimental methods

Experiments were performed at beamline ID06 of the European Synchrotron Radiation Facility, Grenoble, France using a 2000-tonne modified DIA press in 6/8 configuration and at the 13-ID-D beamline of GSECARS at the Advanced Photon Source, Argonne, Illinois, USA using a 1000-tonne press and a T-25 double-stage multi-anvil module (Wang et al., 2009). Experiments were conducted between pressures of 3.6 to 10.4 GPa at temperatures from 1200 to 1500 °C. An octahedron with 14 mm truncated edge length, made from semi-sintered Cr<sub>2</sub>O<sub>3</sub>-doped MgO, was compressed using 8 tungsten carbide anvils of 25 mm edge length and with 8 mm edge length corner truncations. The assembly was drilled for the insertion of a Re-foil furnace. The Re-foil (250 μm thickness, 99.995% purity) was modified with two opposing slits (0.5 × 2.7 mm) for unimpeded X-ray transmission. The furnace was enclosed within a cylindrical sleeve of MgO or h-BN at the center of the assembly and a ZrO<sub>2</sub> sleeve above and below the beam path

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