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Report:

Geobarometers are essential in Earth sciences for determining the last equilibration depth of mantle derived rocks, and are important for determining the Earth's heat flux and the depth at which metasomatic processes operate such as those that lead to diamond growth. Some barometers are routinely used at pressures up to 6 GPa, where pressure cannot be calculated directly due tu friction losses, but must be calibrated against phase transitions. A more rigourous approach, however would be to calibrate barometer equilbria directly using x-ray diffraction analyses of calibrant materials.

We compared the pressures calculated form the x-ray-determined unit-cell volumes of well-known pressure standards with the pressures obtained by the geobarometer equilibria and sought corrections, if necessary. The resulting directly comparable calibrations will give confidence in the application of different geobarometers to the same suite of rocks and will yield mutually consistent depths of last equilibration. Moreover, accurate barometers are of economic interest because they are an important tool to trace diamond-bearing rocks.

Experiments were performed at beamline ID06 using a 2000-tonne modified DIA press in 6/8 mode. The assembly is showin Figure 1. Samples were loaded into AuPd multi-chamber capsules. Mixtures of MgO + h-BN and Au+ NaCl were chosen as x-ray standards. These measurements required the collection of angle-dispersive x-ray diffraction (ADX) data, on a linear pixilated GOS X-scan series detector from Detection Technology, through the horizontal anvil gap. For this we used a monochromatic beam of wavelength, $\lambda = 0.2254$ Å (55 keV), which was selected from the emission of a U18 undulator by a Cinel Si(111) double-crystal monochromator.





run (ES313-1), using a cross calibration of Re and h-BN lattice volumes For runs ES313-2 and ES313-3, pressure and temperature were calculated from the EoS of the reference materials and thermocouple

temperature, following established procedures ². For the final run (ES313-5), we used the third-order Birch-Murnaghan P-V-T EoS for NaCl³. A representative diffraction pattern is shown in Figure 2. Experiments of this study were combined with similar experiments conducted at Advanced Photon Source, Argonne, beamline 13-ID-D in order to ensure simulation pressure and temperature conditions from shallower towards greater mantle depth.

400 - ES313-5, 9.4(2) GPa, 1400°C 300 - Gan - Gan

The deviation between experimental and calculated pressure is

shown in Figure 3, highlighting the discrepancies between the *Figure 2 ADX pattern of run ES313-5*.

Collerson barometer⁴ and experimental pressure. By applying a simple linear correction, the barometer reporduced the experimental data within the uncertainties. On the basis of our results, it is apparent that a direct calibration of geobarometers was long overdue. While the precision of the barometers remains the same, the accuracy and thus the external reproducibility, has been improved. Systematic errors between barometer P, T corrections have been minimized by our multi-chamber runs, conducted under the sample conditions, to offer a consistent set, where, the comparability between pressures obtained with different barometer is given. Moreover, it is now possible to obtain a comprehensive depth of last equilibration of eclogite and peridotite suites from the same. With the corrections presented in this study we can rule out inconsistencies that were inevitable due to different pressure calibrations between different laboratories. In future studies we will verify the apparent offsets observed in this study for pressures below 3 GPa and above 11 GPa. Obtaining this information will greatly increase the confidence of how geobarometers can be applied to natural rocks over a larger range of depths. The data obtained during this ESRF-session (ES-313) and the APS one (APS-No. 42985) are combined and written up as a manuscript for publication, which will be submitted soon.

The unfocussed incident beam size was 0.5 mm

x 0.5 mm. The detector ran continuously, collecting a new image at 10 Hz rate and wrote every 3.2 s. The 3.2 s detector images were integrated after correction according to the detector's location on the calibrated x-ray detector plane, using v17 of Fit2D ¹, which was at approximately 2050 mm from the sample.

As the thermocouple failed on compression, we calculated pressure and temperature of the first



Figure 3 (left) Calculated vs. experimental pressure for the Collerson et al. (2010) majorite barometer. (right) ΔP vs. experimental pressure. The broken line represents the best fit to the data $\Delta P(C10)_{corr}[GPa] = 0.52(4) * P(C10) - 3.9(3)$, $R^2 = 0.96$.

References

- 1 Hammersley, A. FIT2D: a multi-purpose data reduction, analysis and visualization program. *Journal of Applied Crystallography* **49** (2016).
- 2 Crichton, W. A. & Mezouar, M. Noninvasive pressure and temperature estimation in large-volume apparatus by equation-of-state cross-calibration. *High Temperatures. High Pressures* **34**, 235-242 (2002).
- 3 Matsui, M. Temperature–pressure–volume equation of state of the B1 phase of sodium chloride. *Phys. Earth Planet. Inter.* **174**, 93-97 (2009).
- 4 Collerson, K. D. *et al.* Majoritic garnet: A new approach to pressure estimation of shock events in meteorites and the encapsulation of sub-lithospheric inclusions in diamond. *Geochimica et Cosmochimica Acta* **74**, 5939-5957 (2010).