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1 An improved setup for radial diffraction experiments at high
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3 diamond anvil cell.

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13

14 Abstract

15 We present an improved setup for the experimental study of deformation of solids at
16 simultaneous high pressures and temperatures by radial X-ray diffraction. The technique
17 employs a graphite resistive heated Mao Bell type diamond anvil cell (DAC) for radial X-ray
18 diffraction in combination with a water-cooled vacuum chamber. The new chamber has been
19 developed by the sample environment group at PETRA III and implemented at the Extreme
20 Conditions Beamline (ECB) P02.2 at PETRA III, DESY (Hamburg, Germany). We discuss
21 applications of the new setup to study deformation of a variety of materials, including
22 ferropericlase, calcium perovskite, bridgmanite, and tantalum carbide at high-
23 pressure/temperature.

24

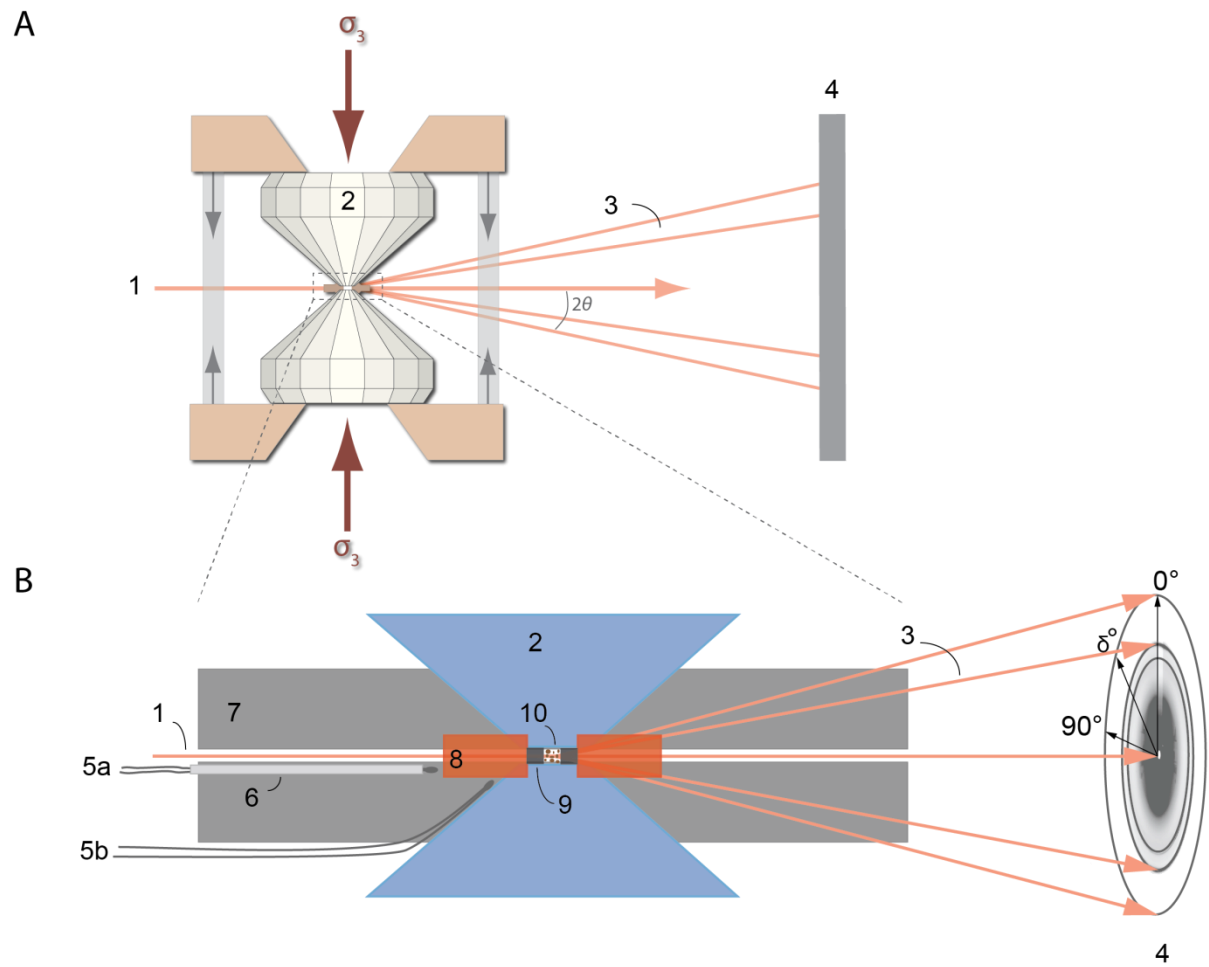
25 Introduction

26 Understanding the physical and rheological properties of materials at simultaneous high
27 pressures and temperatures is of key importance in Earth science as well as materials sciences.

28 The rheological properties of Earth's mantle materials, for example, govern large-scale mantle
29 convection¹⁻⁴. In addition, Crystallographic Preferred Orientation (CPO) caused by the
30 alignment of mantle minerals during deformation, leads to seismic anisotropy, providing a
31 means to link seismic observations to mantle flow⁵⁻¹³. Several techniques have been
32 developed in the past to study the rheology of materials under high pressure and temperature,
33 but achievable pressures are mostly limited to those typical of the crust and upper mantle^{3,14-}
34 ¹⁸.

35 Deformation experiments at deep lower mantle pressures are almost exclusively performed in
36 diamond-anvil cells in combination with synchrotron-based in-situ X-ray diffraction in a
37 radial geometry (Fig. 1A, B). By employing radial diffraction geometry, lattice strains and
38 deviatoric stress as well as evolution of CPO can be derived from analysis of in situ
39 diffraction images. Radial X-ray diffraction in DACs has been widely employed in Earth as
40 well as materials sciences, but high-pressure experiments have been mostly limited to room
41 temperature¹⁹⁻²⁵. Early high-temperature employed a laser-heated DAC, but data analysis and
42 interpretation is challenging due to temperature gradients in the sample^{26,27}. Liermann *et al.*,
43 (2009)²⁸ developed a resistive-heated DAC, which was tested up to 36 GPa and 1100 K while
44 performing in-situ radial X-ray diffraction. The use of a resistive-heating setup reduces
45 temperature gradients and provides more homogeneous heating of the entire sample chamber.

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46

47 Figure 1: (A) angle-dispersive high-pressure radial X-ray diffraction in a DAC (modified after

48 ²⁸); σ_3 : stress along compression direction; 2θ : diffraction angle; 1: incoming X-ray Beam; 2:

49 Diamond anvil; 3: Diffracted beam; 4: Area detector. (B) Magnification of the diamond-anvil

50 culets showing the position for the two thermocouples (5a: between both graphite heaters; 5b:

51 on the diamond anvil next to the culet). 6: Ceramic sleeve; 7: Flexible graphite sheet; 8:

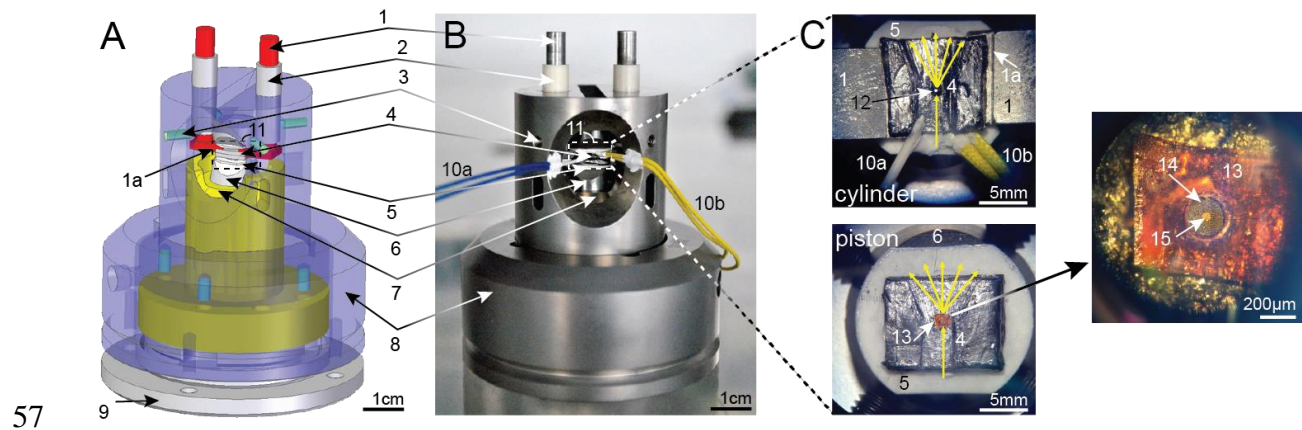
52 Kapton; 9: Boron gasket; 10: sample.

53

54 Here, we present a modified resistive-heated-radial-X-ray-diffraction-diamond-anvil-cell

55 (RH-rXRD-DAC) (Fig. 2A, B) and report on its performance during in-situ radial X-ray

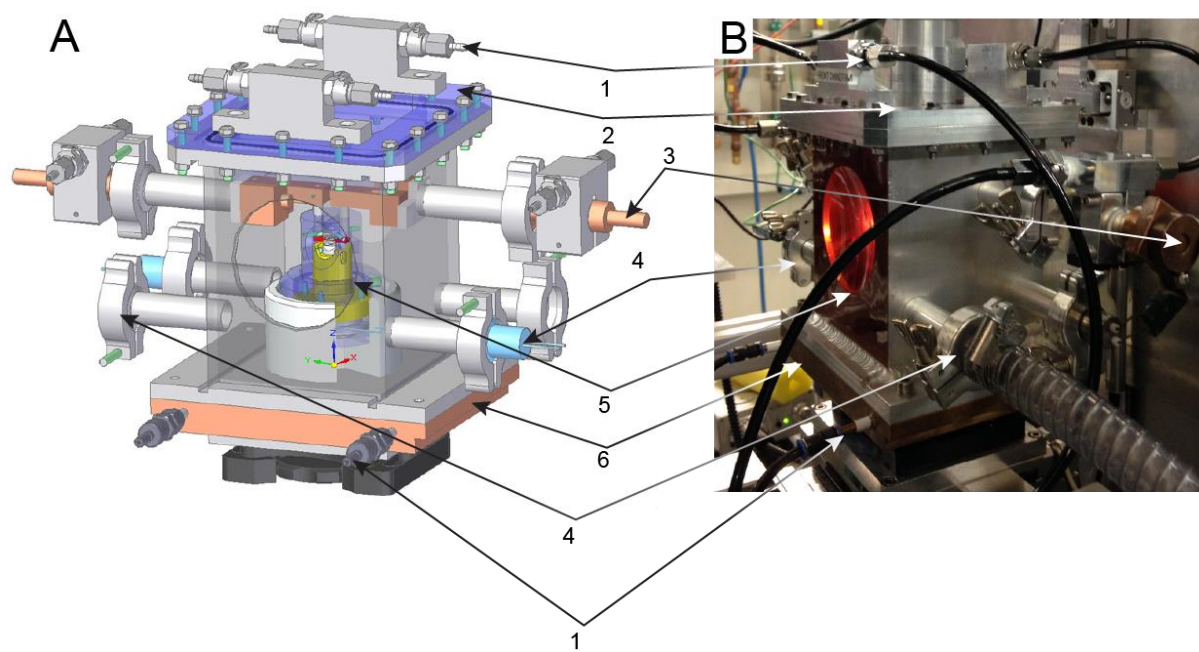
56 diffraction experiments at simultaneous high pressure and high temperature on several



57
58 Figure 2: Experimental setup of a resistive-heated DAC (modified after¹⁰). (A) 3D CAD
59 model and (B) photograph of the resistive-heated DAC used for the experiments. 1:
60 Molybdenum rods (electrical contacts); 1a: Strip with step at the end of molybdenum rod; 2:
61 Ceramic sleeves; 3: Screws; 4: Graphite sheet; 5: Ceramic plate; 6: Tungsten carbide seat; 7:
62 Piston; 8: Cylinder; 9: Membrane cup (missing in B); 10a: Thermocouple with ceramic
63 sleeve; 10b: Thermocouple; 11: Graphite-heater. (C) Close-up of 11. Cylinder: 12: Culet of a
64 diamond; 4: Flexible graphite sheet with carved space for X-ray beam. Inset shows the lower
65 diamond pressed into the graphite heater and illustrates the positions of the thermocouples.
66 Piston: 13: Kapton, which supports a cubic boron nitride gasket or amorphous boron epoxy
67 gasket (14) with powder sample in the sample chamber (15).

68
69 polycrystalline samples. The main improvement as compared to the setup described by
70 Liermann et al. (2009) is the development and implementation of a water-cooled vacuum
71 chamber (Fig. 3A, B) that also enables cooling of the piston of the Mao Bell type DAC. This
72 modification decreases heating and thermal expansion of the piston of the Mao Bell DAC but
73 allows the cylinder to heat up, thus reducing friction between the piston and the cylinder
74 during compression at high temperatures. The use of a vacuum chamber prevents the
75 oxidation of the cell, the Molybdenum rods and the diamonds at very high temperatures. We

76 discuss applications of the improved setup for studying the deformation behavior of major
77 materials expected in Earth's lower mantle as well as tantalum carbide.



78
79 Figure 3: (A) 3D CAD model of the vacuum chamber. (B) Photograph of the vacuum
80 chamber while performing a high-temperature experiment (~ 1400 K). 1: Water cooling
81 inlet/outlet; 2: Lid with screws; 3: Power supply connector; 4: Vacuum pump connection; 5:
82 Kapton window; 6: Copper cooling plate.

83

84 Experimental Method

85 In radial X-ray diffraction experiments, the incoming X-ray beam is oriented perpendicular to
86 the compression direction, i.e. the axis of the diamond anvils (Fig. 1A, C: 1). This setup
87 provides the possibility to study the lattice strains, resulting from the effect of differential
88 stress, together with the CPO of powder samples¹⁹. A pressure-transmitting medium is not
89 used. This enhances the development of differential stress and texture. In order for X-rays to
90 reach the sample chamber in the radial diffraction geometry, X-ray transparent gaskets are
91 required. Here, we used either X-ray transparent amorphous boron epoxy + kapton²⁹ or cubic
92 boron nitride (cBN) epoxy²⁷ (10:1 Epotech 353ND) + kapton gaskets (Fig. 1: 8, 9; Fig. 2C:

93 13, 14) to reach high pressures³⁰. The culet sizes of the employed diamonds were 200 μm or
94 300 μm (Fig. 2C: 12). The setup of the graphite heater is similar on the piston and cylinder
95 side of the DAC (Fig. 2C). Diamonds are glued on tungsten carbide seats that are truncated at
96 the side to increase the opening angle for diffracted X-rays (Fig. 2: 5). The seats are insulated
97 from the graphite by a ceramic ring (Fig. 2: 4) fixed to the seat with OMEGABOND 500
98 liquid. The gaps between the ceramic plates and the diamonds are filled with ceramic glue
99 (Resbond 989). The heads of the molybdenum rods end in horizontal strips with a small step
100 at the end. A piece of graphite foil connects the molybdenum rods (Fig. 2A: 1a; Fig. 2B: 11;
101 Fig. 2C: 1a) and serves as heating element surrounding the diamond-anvils. A space is carved
102 in the graphite foil to prevent diffraction of the graphite contaminating the diffraction image.
103 Two thermocouples (R-type) are attached to the cylinder side. One thermocouple is placed
104 close to the tip of the diamond on the upstream side of the DAC and to the side of the path of
105 the incident X-ray beam (Fig 2C: 10a). The second thermocouple is positioned on the graphite
106 sheet, likewise upstream and to the side of the incident X-ray beam (Fig. 2C: 10b). When the
107 cell is closed, the second thermocouple rests between the graphite sheets of the piston and the
108 cylinder.
109 During the experiment, the temperature of the sample can be increased/decreased by varying
110 an analog I/O signal from 0-10 V using the beamline control system. This proportionally
111 adjusts the power of the DC power supply from 0-1800 W (0–8 V and 0–220 A)³¹. Pressure
112 is changed remotely using a gas membrane device that is operated by the membrane pressure
113 controller APD200 from Sanchez Technology^{27,28,32}.
114 For high-pressure and high-temperature experiments, the RH-rXRD-DAC is placed in a
115 newly-designed water-cooled vacuum chamber that serves to both cool the DAC and to
116 prevent oxidation of the DAC, the molybdenum electrodes, the diamond anvils and the
117 graphite heater (Fig. 3A, B). The piston of the DAC is indirectly cooled through a steel pin

118 that is connected to the base of the vacuum chamber, which is water-cooled. The differential
119 cooling between the piston and the cylinder reduces the friction between the two parts of the
120 DAC and enables a smoother pressure increase as compared to the previous experimental
121 setup^{27,28}. During the experiment the vacuum in the chamber can be as good as 5×10^{-3} mbar.
122 Note that, due to connections between the pump and the cell chamber, vacuum levels around
123 the diamond anvil cells may not be as efficient. Nevertheless, the achieved vacuum is
124 sufficient to perform experiments with minimal oxidation of the heating elements.

125

126 Results and discussion

127 The new setup has been tested during different experimental campaigns at the ECB P02.2 at
128 PETRA III, DESY (Hamburg, Germany). Diffraction images were collected with a XRD
129 1621 flat panel detector from Perkin Elmer. In the following, we will describe some selected
130 experiments in order to illustrate the capability of the new setup for Earth and materials
131 science research. We report on the deformation of polycrystalline samples of ferropericlase
132 ($(\text{Mg}_{0.8}\text{Fe}_{0.2})\text{O}$), the in-situ synthesis and deformation of cubic Ca-Pv (CaSiO_3), experiments
133 performed on a two-phase mixture of bridgmanite (MgSiO_3) and ferropericlase, synthesized
134 from a mixture of enstatite glass + ferropericlase in the resistive-heated DAC, as well as the
135 high-temperature compression of tantalum carbide ($\text{TaC}_{0.99}$), an ultra-high temperature
136 ceramic material.

137

138 *1. In-situ deformation of ferropericlase*

139 Ferropericlase is the second most abundant mineral in Earth's lower mantle. It may play a key
140 role in mantle dynamics since it is rheologically weaker than bridgmanite, the dominant lower
141 mantle phase. Furthermore, it shows a pronounced elastic anisotropy, making it one of the
142 candidates to explain seismic shear wave polarization anisotropy in the lower mantle³³.

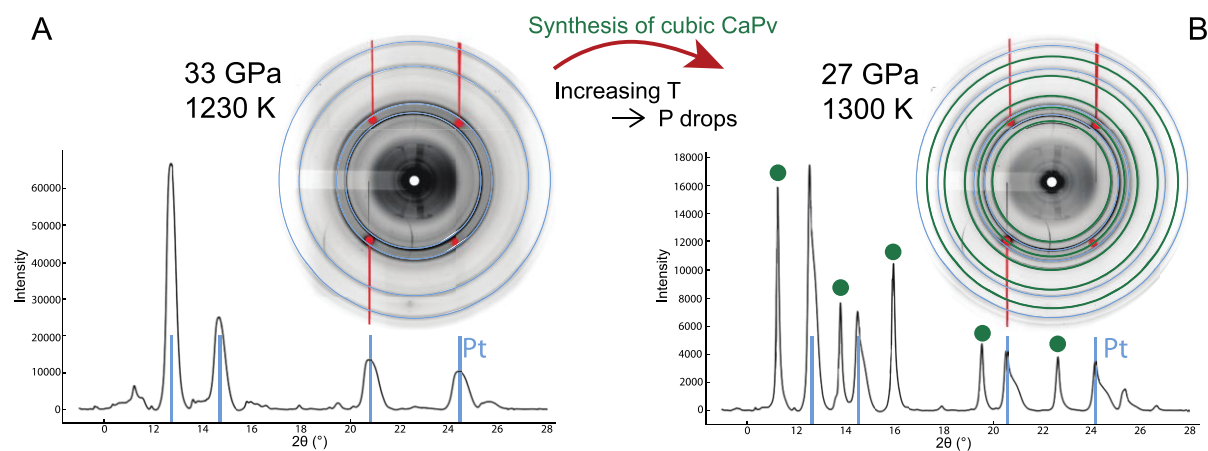
143 However, previous deformation studies at pressures of the lower mantle were limited to room
144 temperature due to experimental complexity^{19,21–23,34,35}. In Immoor *et al.* (2018), we used the
145 described setup to measure the deformation of ferropericlase to 62 GPa at 1400 K as well as
146 to higher pressures but lower temperature. In our 1400 K run, the pressure of the sample was
147 increased up to 40 GPa at room temperature and afterwards heated up to 1400 K. During
148 heating, the pressure of the sample dropped to below 20 GPa. We increased the pressure again
149 and reached 62 GPa when diamond failure stopped the experiment. During compression, we
150 collected high quality diffraction images of ferropericlase. Based on these results, we were
151 able to monitor the evolution of CPO in ferropericlase and confirm a change of slip system
152 activity at high temperature as predicted by computations¹⁰.

153

154 2. *In-situ synthesis and deformation of cubic CaSiO₃*

155 CaSiO₃ perovskite is expected to be an important mineral in Earth's transition zone and lower
156 mantle, where it is the third most abundant phase for a pyrolytic mantle composition³⁶. In a
157 deeply subducted oceanic slab, CaSiO₃ perovskite may account for up to 25 Vol.% of the
158 transformed basaltic crust³⁷ and will affect the bulk rheological properties of the lithospheric
159 slab. According to a recent computational study³⁸, the shear wave anisotropy of CaSiO₃
160 perovskite is about 15-30% at conditions of the lower mantle. A strong CPO of CaSiO₃
161 perovskite may, therefore, contribute to seismic anisotropy observations, in particular in the
162 shallow lower mantle or lowermost transition zone, where the elastic anisotropy is strongest.
163 A previous CaSiO₃ perovskite study has been limited to 49 GPa at ambient conditions²³. At
164 these conditions, however, CaSiO₃ perovskite forms a pseudo cubic structure and the exact
165 nature of the distortion is still under debate^{39–42}. Whereas at temperatures typical for the lower
166 mantle, the structure is cubic (*Pm3m*)^{40–43} and may show a different rheological behavior.
167 CaSiO₃ perovskite can be experimentally synthesized from CaSiO₃ wollastonite at pressures

168 of about 20 GPa and temperature of about 1300 K⁴⁴, but is not quenchable to ambient
169 conditions. This implies that studies of the physical properties of CaSiO₃ perovskite need to
170 be performed in-situ and in the same pressure device where it has been synthesized.
171 Using the improved RH-rXRD-DAC, we were able to synthesize CaSiO₃ perovskite and
172 performed several successful deformation experiments reaching temperatures of up to 1500 K
173 at pressures of 45 GPa (Immoor *et al.* in prep.). The starting material was amorphous CaSiO₃
174 mixed with platinum powder as pressure standard. Figure 4 shows two diffraction images
175 collected during a compression experiment that reached a final pressure of 40 GPa at 1300 K.
176 Cubic CaSiO₃ perovskite was synthesized after increasing the temperature to 1300 K at which
177 point the pressure dropped from 33 GPa to 27 GPa as a result of the phase transition (Fig.
178 4B). The collected diffraction patterns show smooth diffraction rings, indicating a relatively
179 small and homogeneous grain size of the synthesized cubic CaSiO₃ perovskite. The large
180 pressure drop at the transition also caused strain heterogeneity in Pt finely mixed with the
181 sample (see Pt peaks asymmetry in Fig. 4B).



182
183 Figure 4: Synthesis of cubic calcium perovskite. The pressure was calculated from the
184 stronger Pt peak. (A) X-ray diffraction image at 33 GPa and 1230 K shows rings of Pt (blue),
185 which are used to estimate the pressure during the experiment and diamond single-crystal
186 diffraction spots (red). The corresponding integrated diffraction pattern is shown below with

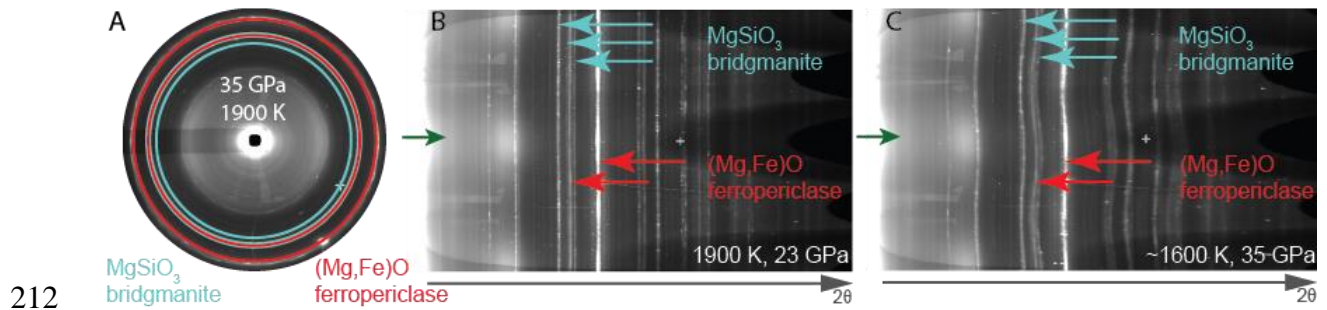
187 blue line indicating Pt peaks. (B) X-ray diffraction image at 27 GPa and 1300 K with Debye
188 rings of Pt (blue), Debye rings of calcium perovskite (green) and diamond diffraction spots
189 (red). At the bottom integrated diffraction pattern with blue lines indicating Pt peaks and
190 green dots indicating calcium perovskite peaks.

191 In both panel (A) and (B) the diffraction lines of diamond are masked in the integration and
192 absent in the diffraction patterns.

193

194 *3. Synthesis of Bridgmanite and Ferropericlase*

195 The deformation behavior of multiphase rock assemblies might substantially differ from the
196 behavior of single-phase assemblies, particularly if the phases show large differences in
197 rheological properties^{35,45–48}. The lower mantle can be modeled as a two-phase mixture of
198 bridgmanite and ferropericlase, two phases that show large differences in plastic strength and
199 viscosity^{3,5,22}. Because of this large contrast in rheological strength, it is difficult to predict
200 mantle properties, including viscosity and seismic anisotropy, from single-phase
201 measurements. There have been few deformation experiments on analogues^{49,50}, as well as on
202 a true two-phase lower mantle mixture at pressures and temperature of the very top of the
203 lower mantle using a rational Drickamer apparatus³. Here, we used the improved RH-rXRD-
204 DAC to synthesize a bridgmanite and ferropericlase assembly (Fig. 5A, B) from an enstatite
205 glass powder mixed with ferropericlase, and applied deviatoric stress to the two-phase
206 mixture at high temperatures (Fig. 5C). In one successful run, we first increased the pressure
207 at 1600 K. Afterwards the pressure in the sample decreased while increasing the temperature
208 continuously to 1900 K. A peak splitting of ferropericlase was observed, likely as a result of
209 pressure gradients in the sample chamber, followed by the appearance of the typical
210 diffraction ring triplet of the new phase bridgmanite. Bridgmanite grew while the pressure
211 continued to decrease when the thermocouples stopped working. During a subsequent



212
 213 Figure 5: Synthesis of bridgmanite (blue) + ferropericlase (red). Green arrows indicate the
 214 compression direction in the unrolled radial X-ray diffraction image. (A) shows the
 215 diffraction rings of bridgmanite and ferropericlase at 35 GPa and 1900 K. (B) shows the
 216 unrolled image with the straight unrolled diffraction rings of bridgmanite and ferropericlase at
 217 23 GPa and 1900 K. (C) The unrolled diffraction image shows the curved unrolled diffraction
 218 rings of bridgmanite and ferropericlase after pressure increase up to 35 GPa at ~ 1600 K.
 219 Ferropericlase was used as pressure calibrant.

220
 221 decrease of voltage and therefore presumably of temperature (based on power-temperature
 222 relation; see Fig. 6), the pressure in the sample chamber increased again to 35 GPa leading to
 223 deformation of the sample (Fig. 5C).

225 4. Compression of tantalum carbide ($TaC_{0.99}$)

226 Carbides are characterized by high mechanical and thermal stability and play an important
 227 role in industrial applications, where they are used, for example, as coatings for abrasive
 228 tools. Many experimental and computational studies have been conducted on tantalum carbide
 229 (see references in⁵¹). However, no experiments have been performed to study the behavior of
 230 this phase under simultaneous high pressure, high temperature and deviatoric stress. We
 231 performed two successful experimental deformation runs on tantalum carbide ($TaC_{0.99}$) and
 232 constrained the pressure-volume-temperature equation of state⁵¹. The starting material was

233 TaC_{0.99} powder and the pressure was determined by a thin piece of Au foil (less than 5 μm)
234 using the EOS published in Fei et al. (2007). In the first run, we started heating when the
235 pressure reached 2 GPa, we increased the temperature to 673 K, and measured X-ray
236 diffraction up to a final pressure of 33 GPa along the 673 K isotherm. In the second run, we
237 increased temperature at a pressure of ~ 2 GPa up to 1073 K, and we collected X-ray
238 diffraction images up to a final pressure of ~ 38 GPa along an isothermal path. The data
239 collected under non-hydrostatic conditions were used to constrain the quasi-hydrostatic high
240 temperature EOS by extracting the hydrostatic unit-cell parameter from the x-ray diffraction
241 data⁵³. In addition these data can be used to determine the strength and activity of the slip
242 systems of TaC_{0.99} at simultaneous high-pressure and -temperature (Speziale *et al.* in prep.).

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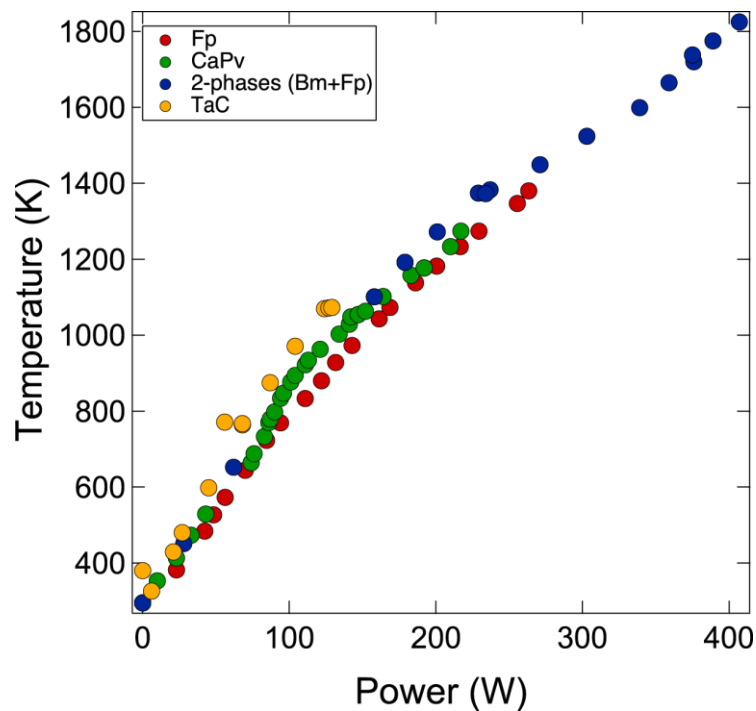
244 *5. Challenges and solutions*

245 In several experimental runs the DAC jammed during high-temperature experiments leading
246 to discrete pressure jumps as opposed to smooth increases of pressure. Because of their
247 brittleness the ceramic gaskets were not able to buffer these pressure jumps resulting in failure
248 of the diamond anvils. The reason for our difficulties to smoothly increase pressure at very
249 high temperatures could be the expansion of both the piston and the cylinder of the Mao Bell
250 DAC leading to increased friction between both parts. However, at moderately high
251 temperatures, up to 1400 K, the differential cooling was effective such that a smooth pressure
252 increase is generally possible. For higher temperature experiments the indirect piston cooling
253 is still insufficient, and needs to be improved in order to reduce the thermal expansion of the
254 piston and thus the friction.

255 Using the water-cooled vacuum chamber, temperatures up to 1900 K have been reached in the
256 RH-rXRD-DAC, but no pressure increase was possible. Generally, both thermocouples

257 recorded stable temperatures during the experiments, with a reproducible dependence of
258 temperature on power (Fig. 6).

259 In a few runs, the difference in temperature reading between the two thermocouples was very
260 large (the maximum difference observed was 400 K). In these cases higher temperature values
261 were recorded by the thermocouple situated between the graphite sheets. Large differences in
262 temperature reading usually occurred when one of the thermocouples, i.e. the one at the tip of
263 the diamond was placed too far from the culet of the diamond.



264

265 Figure 6: Power – Temperature curves of different experiments. The temperature is read using
266 two thermocouples, one placed between the two graphite heaters and one placed on the
267 diamond-anvil close to the sample. Symbols: Fp is ferropericlase; CaPv is calcium perovskite;
268 Bm is bridgmanite; TaC is tantalum carbide.

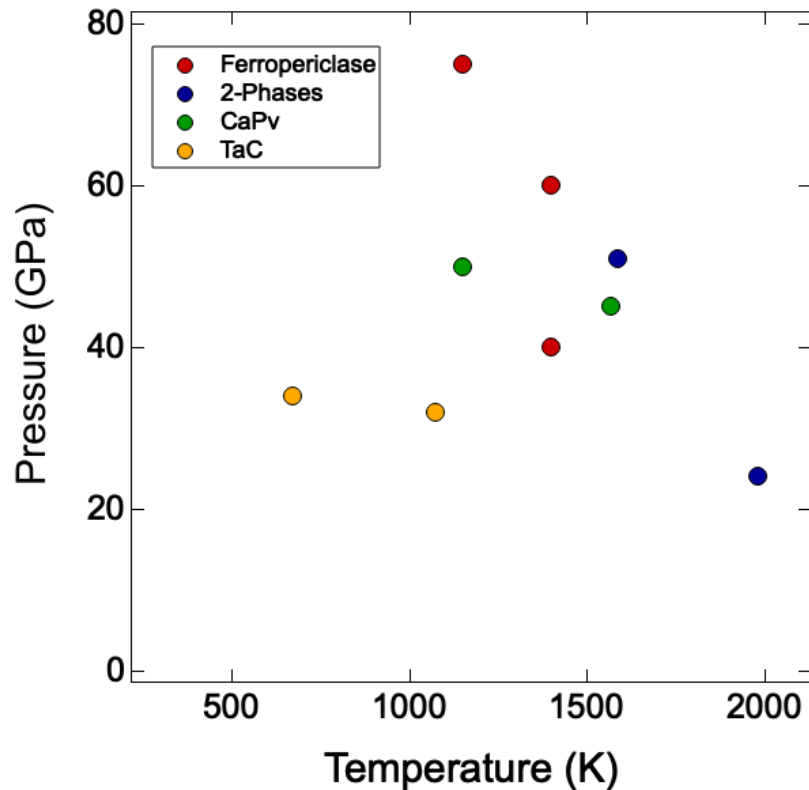
269

270 Conclusion

271 We have presented an improved experimental setup for radial X-ray diffraction measurements

272 based on a graphite-heated Mao Bell type diamond-anvil cell contained in a water-cooled

273 vacuum chamber. The setup is available for users at the Extreme Conditions Beamline P02.2
274 at DESY, Hamburg and allows for reaching temperatures of up to 1900 K at high pressures
275 (Fig. 7).



276
277 Figure 7: Maximum pressure-temperature- conditions reached in experiments on
278 ferropericalse (red, calculated pressure), 2-phases (enstatite (bridgmanite) + ferropericalse)
279 (blue, estimated pressure), calcium perovskite CaPv (green, 45 GPa estimated pressure; 50
280 GPa calculated pressure) and tantalum carbide TaC (yellow, calculated pressure) in different
281 experimental runs.

282 Temperature and pressure in the diamond-anvil cell are controlled remotely during the
283 experiment. Several successful experiments studies were performed by using the improved
284 setup on a variety of Earth materials (ferropericalse, calcium perovskite, a two-phase
285 bridgmanite-ferropericalse mixture) and tantalum carbide in order to show the capabilities of
286 the resistive-heated-radial-X-ray-diffraction-diamond-anvil-cell. A major priority is currently

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287 the search for a better gasket material which combines mechanical strength and high-
288 temperature stability.

289

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303

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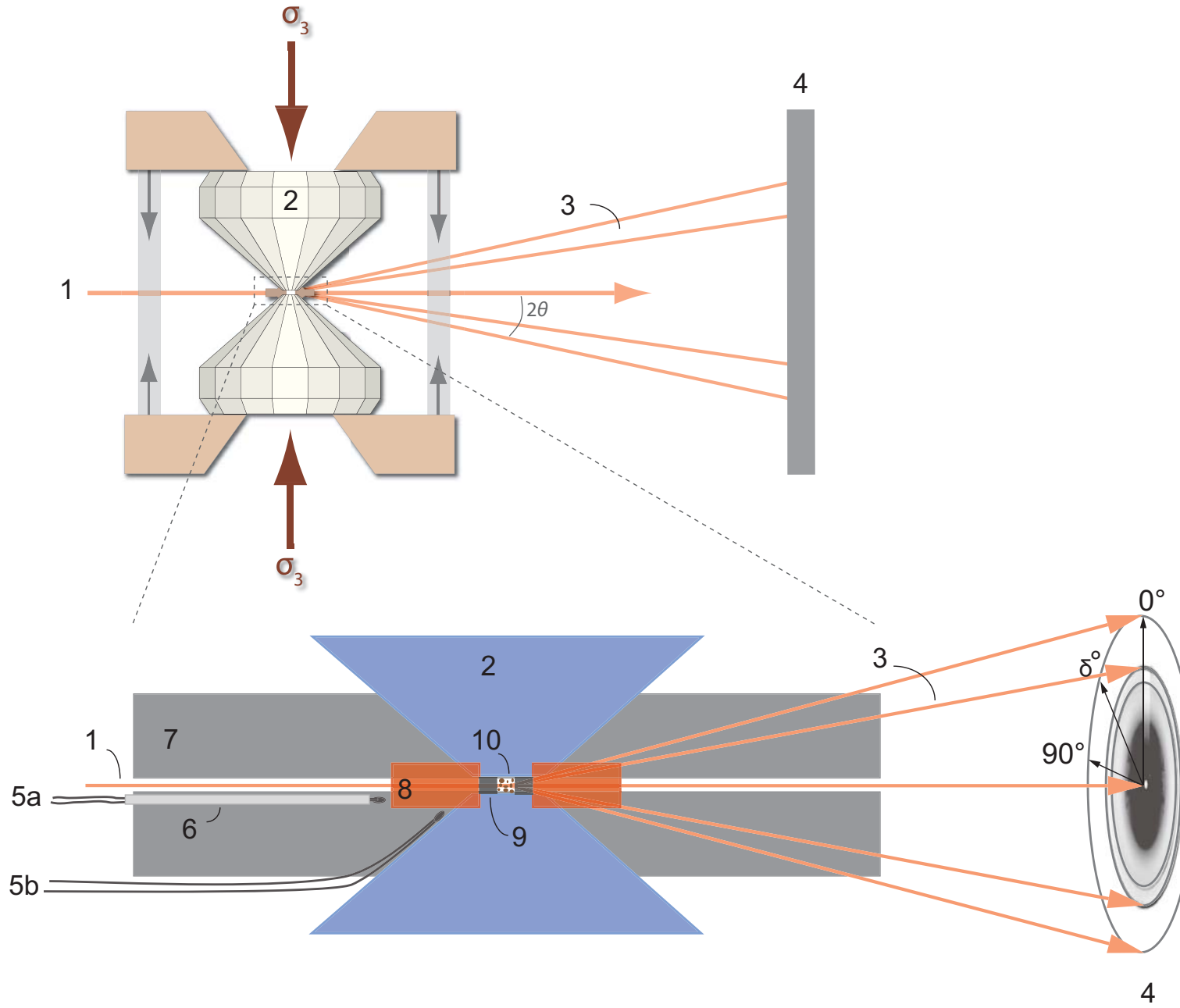
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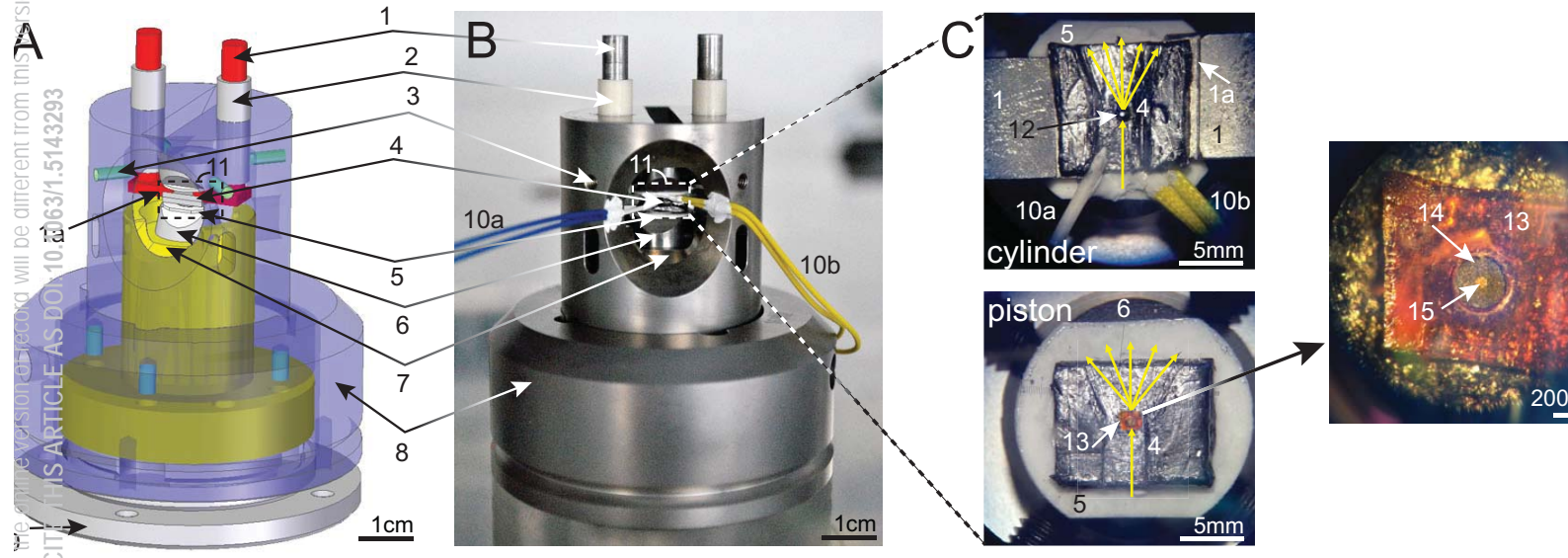
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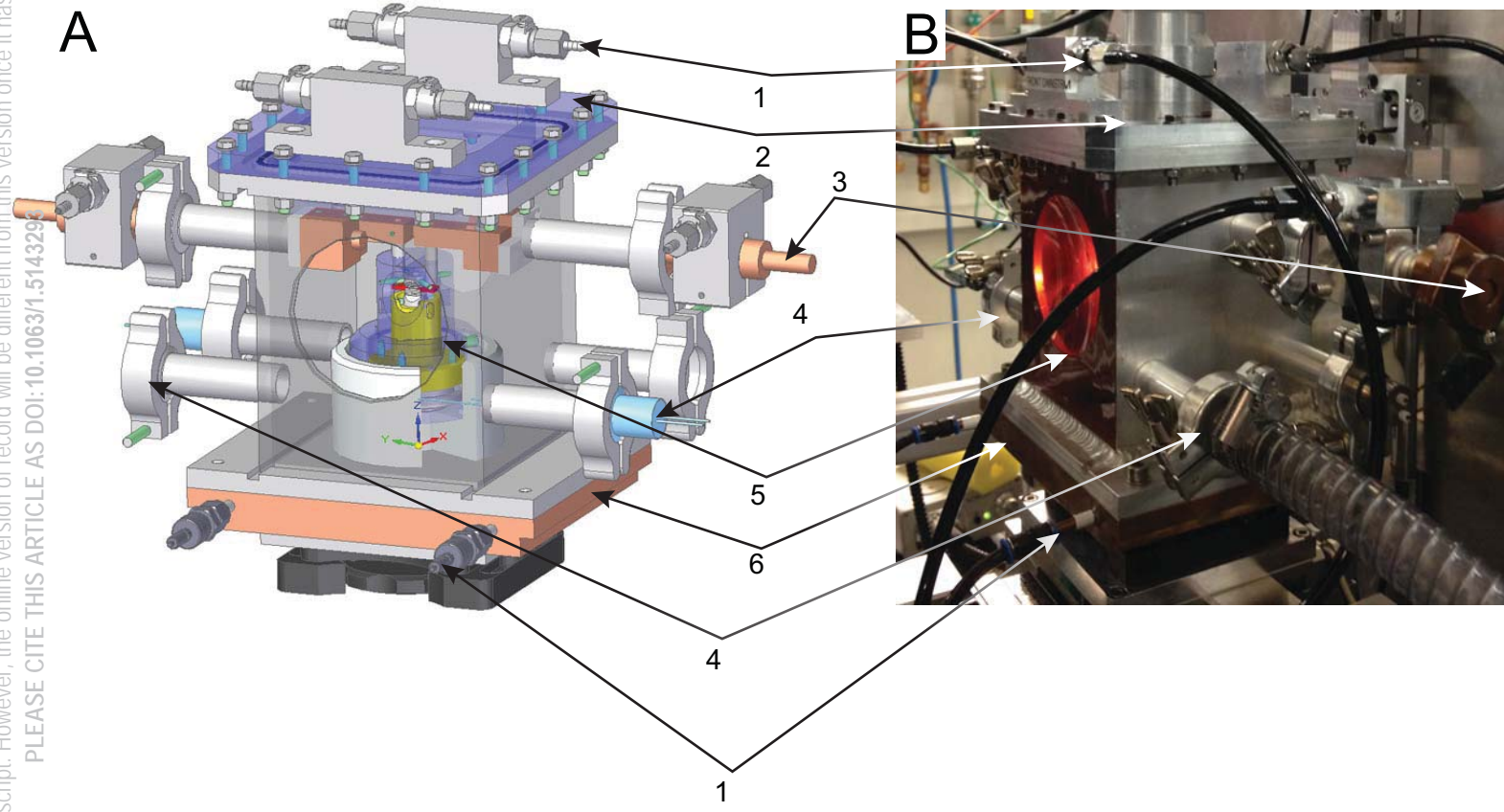
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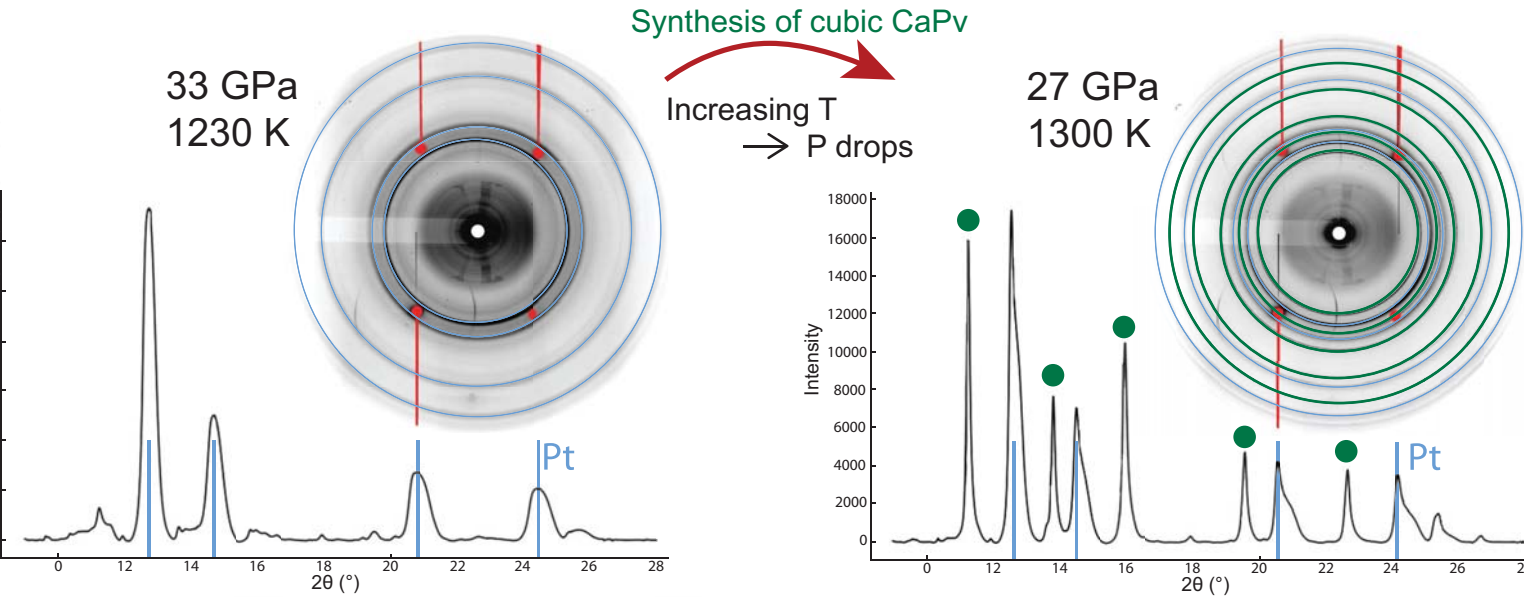
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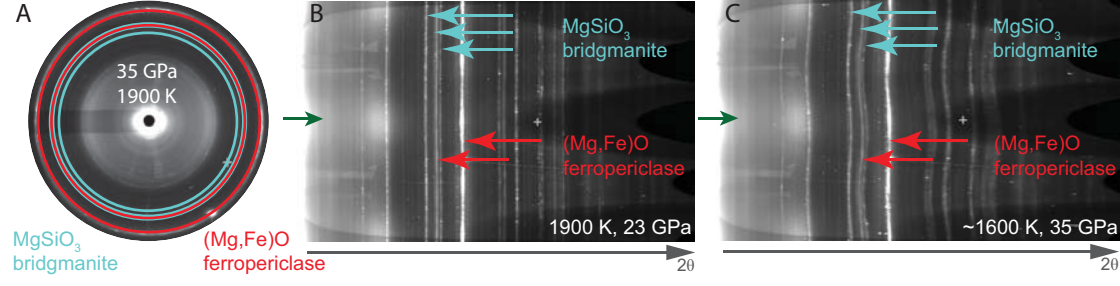
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