

1 **Revision 1**

2 **Electrical Cell Assembly**

3 **for Reproducible Conductivity Experiments in the Multi-Anvil**

4  
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10  
11 **Abstract**

12 Electrical conductivity experiments under pressure and temperature conditions relevant to  
13 planetary interiors are a powerful tool to probe the transport properties of Earth and planetary  
14 materials as well as to interpret field-based electrical data. In order to promote repeatability  
15 and reproducibility of electrical experiments among multi-anvil facilities that use this technique,  
16 we designed and developed an electrical conductivity cell for multi-anvil experiments based on  
17 the 14/8 assembly that was developed to allow access to high temperatures. Here we present  
18 the details of design and parts developed for this cell that is available via the Consortium for  
19 Material Properties Research in Earth Sciences (COMPRES). The electrical cell has been tested  
20 up to 10 GPa and about 2,000°C on different materials (silicates and metals, both in the solid  
21 and liquid state).

## 23 INTRODUCTION

24 Laboratory measurements of the electrical properties of Earth's and planetary materials  
25 benefitted directly from the knowledge gained in material science and, in particular,  
26 electrochemical studies on aqueous solutions (e.g., Noyes et al., 1907; Fogo et al., 1954; Quist  
27 and Marshall, 1968) and on solids (e.g., Bauerle, 1969) that have been conducted since the  
28 beginning of the 20<sup>th</sup> century. Among the different setups developed, a common challenge  
29 consisted of avoiding polarization of the electrodes and in the sample, which can be  
30 accomplished by applying a small alternating current through the cell. Because semi-conductor  
31 materials present a frequency dependent electrical behavior, efforts were also made to  
32 conduct electrical measurements over a scan in frequency (e.g., Koops, 1951). The abundance  
33 of semi-conductor silicates in the crust and mantle of the Earth (and other terrestrial bodies)  
34 (e.g., Knight, 1984; Sato et al., 1986) motivated geoscience studies to develop setups allowing  
35 measurements of the dispersion in frequency of rock samples under ever-increasing pressure  
36 and temperature. Among the different methods to visualize the electrical response of a  
37 semiconductor, the graphic representation of the sample's complex impedance (e.g., Roberts  
38 and Tyburczy, 1991; Huebner and Dillenburg, 1995) has become the most commonly used and  
39 experimental petrology facilities show a widespread usage of the impedance spectroscopy  
40 technique. In contrast, electrical studies of conductors (such as metallic core analogues) under  
41 pressure and temperature do not require measurements to be conducted over a range in  
42 frequency (e.g., Secco and Schloessin, 1989; Deng et al., 2013; Silber et al., 2017); conductors  
43 present a very low capacitance and the frequency where induction equals zero is temperature-  
44 independent (Constable, 2015).

45 Different technical challenges arise from *in situ* and real-time electrical measurements under  
46 pressure and temperature, such as minimizing noise from the furnace on the electrical data and  
47 avoiding the breaking of the electrodes during compression. Among the different pressure  
48 device used in experimental geosciences, the multi-anvil apparatus is a relevant tool to conduct  
49 electrical investigations. First, by covering a broader pressure and temperature range than  
50 piston-cylinders and internally or externally-heated pressure vessels, it reproduces the pressure  
51 conditions of the Earth's crust and upper mantle as well as the entire interior of several small  
52 terrestrial bodies (such as Mars, Mercury, Ganymede, the Moon). Furthermore, recent  
53 development of high pressure techniques using a Kawai-type multi-anvil press suggests that the  
54 maximum attainable pressure can reach up to 60 and 120 GPa using WC anvil and sintered  
55 diamond anvil, respectively. Second, multi-anvil investigations can be conducted on larger  
56 sample volumes than in very high-pressure devices such as diamond-anvil cells (DAC) and  
57 provide better constraints on temperature than DAC experiments.

58 The success of high-pressure and high-temperature electrical experiments to address  
59 geoscience questions depends on the quality and reproducibility of the measurements, as well  
60 as precise inter-laboratory comparisons. COMPRES, the Consortium for Material Properties  
61 Research in Earth Sciences, supported this study that consists of designing a 14/8 electrical  
62 assembly for the multi-anvil apparatus that can be used at pressure (up to about 10 GPa) and  
63 temperature (up to about 2,000°C) conditions relevant to terrestrial bodies, including the Earth.  
64 Here we present the development and testing of this electrical cell, which is adapted from the  
65 COMPRES cell assemblies (Leinenweber et al., 2012), and document the materials used as well  
66 as the calibration. This cell is available through COMPRES and interested experimentalists

67 should be able to reproduce and adapt the cell with the designs and descriptions provided in  
68 this paper.

69

## 70 **DESIGN OF THE ELECTRICAL CELL ASSEMBLY**

### 71 **Assembly parts and sources**

72 The electrical cell is adapted from the 14/8 COMPRES high-temperature equatorial assembly  
73 and uses the same suppliers (Leinenweber et al., 2012). These suppliers have shown to be able  
74 to provide all the required parts in sufficient quantities to support a large usage in numerous  
75 multi-anvil facilities. A straight-heater rhenium assembly was developed to allow access to  
76 higher temperatures (where graphite breaks down) in the range of 8-14 GPa. The octahedron  
77 from the Bayreuth 14/8 assembly (with a 5.6 mm hole) was used, a straight full-length zirconia  
78 sleeve fits inside the octahedron, and a Re heater with a one-layer 125-micron thick foil is  
79 employed inside the zirconia sleeve. MgO rods and tubes with 3.5 mm outer diameter are used  
80 inside the furnace, and a maximum sample volume of 3 mm outer diameter and 3 mm length  
81 can be achieved in this assembly.

82 For electrical conductivity measurements, the sample length is reduced and all parts are made  
83 from porous MgO, with no alumina included. The sources and dimensions of materials are  
84 listed in **Table 1** and a sketch of the electrical assembly is presented in **Figure 1**. Two types of  
85 electrical experiments can be performed with the electrical cell assembly: 2-electrode (**Figure**  
86 **1a**) or 4-electrode (**Figure 1b**) experiments. Three W-Re wires are present in the 2-electrode  
87 cell, with one wire serving as both a thermocouple wire and an electrode. The 4-electrode cell  
88 contains four W-Re wires, serving either as four electrodes or as two thermocouples. As

89 discussed below, in the case of 2-electrode experiments, the electrical contribution of the  
90 electrodes involved in the bulk measurements resistance typically corresponds to a few ohms  
91 and needs to be removed from the bulk resistance in order to obtain the sample (Pommier et  
92 al., 2010). If this resistance value is negligible compared to the much higher electrical resistance  
93 (lower electrical conductivity) of silicate minerals (e.g., Tyburczy and Fislser, 1995), it can  
94 represent a significant amount of the bulk electrical response and even hide the sample's  
95 response in case the resistance of the sample is smaller than that of the electrodes, which  
96 happens for samples containing silicate and carbonate liquids and metals. Getting rid of most of  
97 the electrode contribution can be achieved with the 4-electrode setup, where the configuration  
98 prevents the applied current from being delivered in the loop of measurement formed by the  
99 electrodes. With this setup, four thermocouple wires are connected two-by-two on the metal  
100 disk, each of them corresponding to either a voltage probe or a current probe (**Figure 1b**). In  
101 this configuration, the measured bulk resistance only needs to be corrected for the metal disks,  
102 as the wires are not part of the electrical measurement like they are with the 2-electrode setup.  
103 For both 2-electrode and 4-electrode experiments, the sample is in direct contact with the two  
104 metal disks and the MgO middle sleeve that contains the sample. The purpose of the metal  
105 disks is two-fold: 1) to prevent direct contact between the sample and thermocouple wire, and  
106 2) by covering the sample's extremities, to promote the distribution of current through the  
107 entire volume of the sample (e.g., Kennedy, 1960). Note that in semiconductors, current  
108 distribution also depends on the sample length:electrode diameter ratio and is optimized when  
109 the ratio is close to 1 (Kennedy, 1960).

110 The chemical interactions between the sample and these parts are discussed below. No capsule  
111 contains the sample and the absence of capsule is motivated by the requirement of having the  
112 electrode disks in direct contact with the sample and the W-Re leads. We observe that the  
113 effect of pressure helps contain a partially molten sample until high melt fractions (Pommier et  
114 al., 2015a, 2015b). However, depending on the sample's texture and melt chemistry, the  
115 mobility of the liquid phase at high temperature may be high enough to escape along the inner  
116 wall of the MgO sleeve and eventually leave the cell (e.g., Yoshino et al., 2003; Zhang and  
117 Pommier, 2017). One way to circumvent this issue is to minimize the melt fraction and the time  
118 spent at temperature above the melting point of the phase that melts first. At high  
119 temperatures (> melting point), there is a balance to find between reaching electrical  
120 equilibrium (which does not guarantee chemical equilibrium) and minimizing melt escape.

121 Crushable MgO sleeves and MgO 4-bore beads are preferred to alumina parts due to the low  
122 hardness of crushable MgO compared to fully dense alumina. Tests on partially molten samples  
123 using a cell containing 4-bore alumina showed that sample deformation is higher than with 4-  
124 bore MgO. This implies that the geometric factor of the sample, corresponding to the electrode  
125 area/sample length, is affected during the experiment with alumina. Since the electrical  
126 conductivity  $\sigma$  of the sample directly depends on the geometric factor G with the relationship

$$127 \quad \sigma = 1/(RxG) \quad (1)$$

128 with R being the electrical resistance, a higher uncertainty on the electrical measurements  
129 occurs when the 4-bore beads that host the metallic wires are made of alumina. All MgO parts  
130 used in this assembly are fired at 1400°C to fully remove any carbon contaminants, and also to

131 increase the crystallinity and reduce the possibility of surface hydration of the MgO during  
132 exposure to air.

133

#### 134 **Other cell components**

135 Similarly to the 14/8 COMPRES assemblies (Leinenweber et al., 2012), the electrical cell  
136 assembly is used with Toshiba Tungaloy carbide cubes for the 14/8 size, and can also be used  
137 with Rockland Research carbide cubes. The plastic pads at the interface between the tungsten-  
138 carbide cubes and the anvils are made of fiberglass-impregnated phenolic resin (called "G-10").  
139 Twelve pyrophyllite gaskets (6 short and 6 long) have the same dimensions and source as the  
140 ones used in Leinenweber et al. (2012) for the 14/8 assemblies. Depending on the type of  
141 electrical experiment (2 or 4 W-Re wires, **Figure 1**), 3 or 4 grooves need to be made on the side  
142 of gaskets, using either a thin diamond file or a hand drill with appropriate drill bit. Four pieces  
143 of 3-ply paper are used on the four tungsten-carbide cubes as backing of the pyrophyllite  
144 gaskets. Their dimensions and source are similar to the other 14/8 COMPRES assemblies. We  
145 recommend to wrap the other four tungsten-carbide cubes with 5 mil Teflon tape in order to  
146 minimize risks of short-circuit between the electrode wires and the tungsten-carbide cubes.

147

#### 148 **Development of the electrical cell: modifications from the standard 14/8 assembly**

149 The presence of 3 or 4 W-Re wires (for 2- and 4-electrode experiments, respectively) required  
150 the development of a 4-notch octahedron. The design of the pressure medium is illustrated in  
151 **Figure 2a**. As in the COMPRES 14/8 equatorial assemblies, the octahedron is an injection-  
152 molded ceramic essentially made of MgO and alumina that turn into spinel and periclase after

153 firing at high temperature (1535°C) (Leinenweber et al., 2012). In the case of 2-electrode  
154 experiments, one of the four notches is left unused, as this configuration involves only 3 wires  
155 (**Figure 1a**). We recommend to simply fill this notch with the electrically resistant cement that is  
156 applied on both ends of the cell assembly.

157 The laser-cut rhenium furnace is also re-designed to contain four grooves for this assembly.  
158 The four notches are aligned with the four grooves in the octahedron. The geometry of the  
159 rhenium foil is presented in **Figure 2b**. One improvement of the furnace from the COMPRES  
160 14/8 equatorial assemblies is the increase in the groove width (1.2 mm), improving the isolation  
161 between the furnace and the W-Re wires, hence minimizing the risk for short-circuit. Again, one  
162 notch in the rhenium foil will be left unused when preparing a 2-electrode cell assembly.

163

164

## 165 **TESTING AND CALIBRATION OF THE ELECTRICAL CELL**

166 This cell has been tested up to 2000°C (Pommier et al., 2015a) and 10 GPa (Pal et al., in prep.).

167 It should work up to 14 GPa, like the standard 14/8 cell it is derived from.

### 168 **Effect of the furnace on electrical measurements**

#### 169 *Furnace chemistry and electrical measurements*

170 Two types of furnaces were tested, rhenium and graphite. In order to evaluate the effect of  
171 furnace chemistry, and in particular, the redox environment it may contribute to impose on the  
172 sample's response, electrical measurements were conducted on the same sample  
173 (polycrystalline San Carlos olivine) at similar pressure and temperature conditions. Olivine  
174 conductivity being sensitive to oxygen fugacity (e.g., Schock et al., 1989), it is a relevant



175 material to use to probe the effect of the furnace type on the electrical measurements. As  
176 illustrated in **Figure 3**, sample conductivity is not affected by furnace type (that is graphite  
177 versus rhenium) at the timescale of electrical experiments (duration of about two hours each),  
178 though these two furnaces would produce relatively different redox conditions, i.e., reducing  
179 and oxidizing environment, respectively. This observed insignificant effect of heater chemistry  
180 on oxygen fugacity is also due to the fact that the experiment is not buffered (for instance, at  
181 the Re-ReO<sub>2</sub> oxygen buffer) since the components of the cell and the sample do not bring both  
182 sides of the redox buffer equation.

183

#### 184 *Noise from the furnace on electrical measurements*

185 The electrical cell does not contain a shielding such as a Faraday cage. A Faraday cage is needed  
186 when electromagnetic radiation reaches the sample while electrical measurements are being  
187 performed, and the presence of a thermal insulator (in our case, the MgO sleeves) will not stop  
188 the radiation. This radiation strongly depends on the level of electromagnetic noise in the lab  
189 and can be minimized or erased with shielding. Having the multi-anvil press shielded represents  
190 an efficient Faraday cage, and the cell was tested both at ASU and UCSD-SIO, where the presses  
191 are shielded to the ground with no major source of radiation around. In case the cell is used in a  
192 press that is not shielded or is located in an environment with nearby sources of  
193 electromagnetic noise, interferences might affect the electrical measurements and one solution  
194 is to wrap the sample with a metal foil (e.g., Xu et al., 1998; Kavner and Walker, 2006). In this  
195 case, it is critical to ensure that the shielding foil and the electrodes are not in contact, to avoid  
196 a short-circuit.

197 Interferences between furnace and electrical measurements would be directly detected with  
198 the impedance technique, as any coupling between the sample and the furnace would affect  
199 the shape of the impedance response and increase noise on the collected data over at least  
200 part of the frequency range. As illustrated in **Figure 4**, the electrical cell provides high-quality  
201 spectra over wide frequency ranges.

202

### 203 **Interactions between the sample and cell parts**

204 Experiments on silicates and on metals suggest that at the timescale of electrical experiments  
205 (typically a few hours), the MgO middle sleeve does not significantly react with the sample  
206 (Pommier et al., 2015a,b; Zhang and Pommier, 2017). Experiments on Fe-bearing materials  
207 (polycrystalline San Carlos olivine and iron alloys) at temperature up to 1800°C present the  
208 development of a thin (<100 microns) ferropericlase layer at the sample-MgO sleeve interface  
209 (**Figure 5**). The contribution of this thin (Mg,Fe)O layer to the bulk electrical response can be  
210 estimated using the conductivity of ferropericlase. Since the sample and ferropericlase  
211 arrangement corresponds to a parallel model, the bulk electrical conductivity (effective  
212 resistance) of the circuit can be written as follow (e.g., Glover et al., 2000)

$$213 \quad \frac{1}{R_{bulk}} = \frac{X_{per}}{R_{per}} + \frac{1-X_{per}}{R_{sample}} \quad (2)$$

214 where  $R_{bulk}$  is the bulk electrical resistance (ohm),  $X_{per}$  the volume fraction of periclase,  $R_{per}$  the  
215 resistance of periclase (ohm) from Yoshino et al. (2011) with a  $X_{Fe}$  of 0.2-0.3, and  $R_{sample}$  the  
216 resistance of the sample (ohm). The electrical conductivity is calculated using Eq. 1. Application  
217 of Eqs. 1 and 2 to the experiment on San Carlos olivine (sample shown in **Figure 5b**),  
218 considering a volume fraction of periclase of about 0.2 and the estimated sample conductivity

219 over the T range 700-1864°C (we do not expect periclase formation to occur at lower  
220 temperature), suggests that the sample conductivity represents >99.6 % of the measured bulk  
221 conductivity, highlighting the negligible contribution of periclase on the bulk electrical  
222 response.

223 The electrical cell has been tested with two types of electrodes, molybdenum and iron. These  
224 materials have been selected mostly because of their high melting point (1538°C and 2623°C at  
225 1 bar for Fe and Mo, respectively) that allows collecting electrical data over a wide temperature  
226 range. Another important criterion is the minimization of chemical interactions with the sample  
227 bracketed by the metal disks. Experiments on silicates up to 6 GPa suggested that chemical  
228 contamination from the electrodes is insignificant up to very high temperature when a high  
229 melt fraction is present in the sample (> about 1800°C for partially molten olivine samples and  
230 MORB+olivine samples; Pommier et al., 2015a, 2015b). Electrical experiments up to 6 GPa on  
231 olivine-iron alloys showed that Mo contamination of liquid metallic alloys is significant if the  
232 sample is kept for a few minutes above the melting temperature of the iron alloy (Yoshino et  
233 al., 2004; Zhang and Pommier, 2017). The samples being in direct contact with molybdenum or  
234 iron, it is possible that the metal disks get lightly oxidized during the experiments when the  
235 sample contains oxygen. However, it is unlikely to be a significant oxidation due to the limited  
236 time that the sample spends at high temperature. In any case, it is necessary to analyze the  
237 chemistry of retrieved samples to estimate the level of contamination from the electrodes  
238 using EDS SEM or electron microprobe analyses.

239 Electrode deformation (as observed in **Figure 5b**) can occur and may be favored at high  
240 pressure and high temperature. It may also occur during decompression, after quenching the

241 experiment. If necessary, electrode disks can be doubled on each side to minimize deformation  
242 (a blank experiment will be required to estimate the bulk electrodes contribution). Electrode  
243 deformation should be accounted for as part of the calculation of uncertainty on conductivity,  
244 as it affects the sample thickness that is part of the geometric factor  $G$  (Equation 1). The change  
245 in sample length with  $P$  and  $T$  introduces a systematic bias because the final length of the  
246 sample is usually a minimum length for the entire experiment. Based on experience with *in-situ*  
247 radiography, the final length is reached once the sample reaches the temperature condition  
248 where it is soft enough to lose all its porosity (usually around 800 °C for silicates). The sample  
249 length then varies primarily with thermal expansion, elastic response to pressure changes,  
250 phase transformations, and ductile flow. Elastic considerations mean that the sample length is  
251 less at high pressure, while ductile flow means the sample length could have been greater.  
252 Because we cannot estimate these effects well without having an *in-situ* capability, we consider  
253 the length after the experiment to be the preferred representation of sample length during the  
254 measurement.

255

#### 256 **Contribution of the electrodes to the measured electrical resistance**

257 In the case of 2-electrode experiments, it is critical to evaluate the electrode contribution to the  
258 measured (bulk) resistance in order to have an accurate estimate of the sample's electrical  
259 resistance (e.g., Pommier et al., 2010). The setup we used to test and calibrate the electrical cell  
260 considers electrodes made of four components: two metal disks (each being 0.2 mm thick and 2  
261 mm in diameter), two W-Re thermocouple wires protected by a Teflon sleeve (each being  
262 about 10 cm long and 178 micron in diameter, which is thin enough for the anvil gap (at least 1

263 mm) up to the highest pressure), two W-Re metallic wires that connect the wires inside the  
264 press to the outside (each being about 30 cm long and 0.5 mm in diameter), and four 50 cm-  
265 long BNC cables that are plugged to the Solartron impedance spectrometer. Minimizing the  
266 length of cables and wires between the spectrometer and the press minimizes the electrode  
267 resistance. We performed a blank electrical experiment at 3 GPa and over a wide temperature  
268 range (up to 1450°C) at UCSD-SIO. No sample was placed in the cell, i.e. the metal disks were in  
269 direct contact, and measurements were taken during heating and cooling cycles. As highlighted  
270 in **Figure 6**, the results suggest that 1) the electrode resistance is about 7.5 Ohm, 2) this value is  
271 not temperature-dependent, and 3) this value does not change significantly at the timescale of  
272 the electrical experiments (total duration of about 2 hours). A similar value was obtained with  
273 the two-electrode setup at ASU for a blank experiment conducted at 4 GPa and temperature up  
274 to 1900°C and using the same electrical cell (Pommier et al., 2015a).

275 In 4-electrode experiments, only the Mo or Fe disks are added to the bulk measurements.  
276 Based on the geometry of the disks and on the conductivity of the metal, their contribution to  
277 the electrical measurement can be easily estimated but it can also be measured as part of a  
278 blank experiment. From 700 to 1500°C, the calculated resistance of Mo disks represents 4.3-  
279  $8.3 \times 10^{-5}$  ohm and the one of Fe disks  $2-2.7 \times 10^{-4}$  ohm. These resistance values need to be  
280 deduced from the bulk resistance, as sample and disks form a series circuit.

281

## 282 **Thermal gradient**

283 Electrical conductivity of Earth's and planetary materials being very sensitive to temperature, it  
284 is important to minimize temperature variations across the sample. The thermal gradient in the

285 electrical cell was estimated in different ways. Because 4-electrode experiments contain two  
286 thermocouples (**Figure 1b**), it provides a temperature reading on both sides of the sample  
287 during the electrical experiment. If this technique does not measure directly the thermal  
288 gradient across the sample, it allows checking the temperature at the extremities of the sample  
289 and verify whether or not the sample is centered in the hotspot zone of the assembly. The  
290 absence of a thermal gradient across the sample can be evaluated after the electrical  
291 experiment by checking the chemical homogeneity of the phases present in the retrieved  
292 samples. Samples from experiments using this electrical cell did not present any chemical  
293 heterogeneity in the samples due to a significant thermal gradient across the sample (Pommier  
294 et al., 2015a,b; Zhang and Pommier, 2017).

295 We also computed the thermal gradient in the electrical cell using the CellAssembly numerical  
296 model for temperature distributions in cell assemblies developed by Hernlund et al. (2006). An  
297 example is presented in **Figure 7**. The sample considered for the calculations corresponds to a  
298 1mm-long forsterite olivine disk and the electrodes are made of molybdenum. At 1200°C, the  
299 computed isotherms predict a small thermal gradient across the sample of 10°C or less, with  
300 the highest variations occurring laterally. The estimated current at this temperature is 187 A,  
301 which is in very good agreement with experimental values.

302

303

#### 304 **IMPLICATIONS FOR THE EXPERIMENTAL COMMUNITY**

305 The main motivation for developing this electrical cell for the 14/8 multi-anvil assembly is to  
306 make it available to the experimental community, in order to enhance cross-laboratory

307 comparisons and thus, improve the quality of the electrical database for geoscience  
308 applications. The cell can be used to conduct both 2-electrode and 4-electrode electrical  
309 experiments, and has been tested with different types of materials (semi-conductors and  
310 conductors). The description of the cell parts used, their source and dimensions have been  
311 presented in order to make this technology available and facilitate the reproducibility of  
312 electrical experiments. We hope that the use of this electrical cell will promote technical  
313 discussions among the experimental community that will lead to improvements of the cell and  
314 new designs for future investigations. This electrical cell is available via COMPRES as a new cell  
315 assembly.

316

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323 the freely available thermal modeling program CellAssembly (available at  
324 <http://multianvil.asu.edu/Kurt's%20Thermal%20modelling/ThermalModeling.htm>).

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416 **FIGURE CAPTIONS**

417 **Figure 1:** Electrical cells developed for 2- and 4-electrode experiments using 14/8 COMPRES  
418 assemblies (A) and B), respectively). In both cases, W-Re wires are used as both  
419 thermocouples and electrodes.

420 **Figure 2:** Electrical cell design: Modifications from traditional 14/8 COMPRES assemblies to  
421 accommodate the electrode wires. A) 4-notch octahedron (pressure medium). B) 4-groove  
422 rhenium foil (furnace).

423 **Figure 3:** Effect of furnace chemistry on electrical measurements. Example of deformed  
424 polycrystalline San Carlos olivine at about 3 GPa (Pommier et al., under rev.) using graphite  
425 (black circles) or rhenium (empty circles) furnaces. No significant difference is observed on  
426 the electrical response, suggesting that the potential changes in the redox environment due  
427 to the furnace chemistry are negligible at the timescale of the experiments (about 2 hrs).  
428 The experiment performed with the rhenium furnace is more noisy because of issues due to  
429 the power supply that were unrelated to the cell assembly.

430 **Figure 4:** Examples of impedance spectra collected using the electrical cell (2-electrode  
431 measurements). X and Y axes correspond to the real and imaginary part of the complex  
432 impedance, respectively. Intersection of the sample's response with the real axis provides  
433 the value of the bulk electrical resistance. A) Hydrated olivine at 600°C and 4 GPa, from  
434 Pommier et al. (2015a). The semi-circle arc is typical of semiconductors. The presence of a  
435 second loop may indicate adsorption relaxation mechanisms (e.g., Lee and Pyun, 2009 and  
436 Kriaa et al., 2009) or chemical reactions (Wang et al., 2013) and its understanding requires  
437 further work. B) Two layer sample (one layer of San Carlos olivine, one layer of iron alloy,

438 with the volume ratio of olivine:metal being 0.7:1) at 400 and 975°C and 6 GPa (Zhang and  
439 Pommier, 2017). Here, the electrical response is dominated by the metal part of the sample.  
440 Spectra corrected from electrodes' contribution. C) Natural lawsonite at 500°C and 10 GPa  
441 (Pal et al., in prep.).

442 **Figure 5:** SEM images of retrieved samples after electrical experiments. A) EDS map on Fe-S  
443 alloy sample quenched at 4.4 GPa and 1470°C, showing the distribution of Mg, O, and Fe.  
444 The thin layer (<80 micron, in orange) at the interface between the metal sample and the  
445 MgO sleeve corresponds to ferropericlase. B) Back-scattered electron image of hydrous  
446 polycrystalline San Carlos olivine sample from Pommier et al. (2015a). Small electrode  
447 deformation is observed and should be accounted for as part of uncertainty calculations.  
448 Ferropericlase forms a layer (<100 micron thickness) at the interface between sample and  
449 MgO sleeve. The thin layer has a negligible contribution on the electrical measurements  
450 (see text for details).

451 **Figure 6:** Electrode contribution to the bulk electrical measurements using the 2-electrode  
452 configuration. The blank experiment was conducted at UCSD-SIO at about 3 GPa and at  
453 temperature up to about 1450C during heating (blue and green circles) and cooling (red  
454 circles) cycles. The constant value of about 7.5 ohms is similar to the one obtained at ASU  
455 using a comparable setup at 4 GPa (Pommier et al., 2015a).

456 **Figure 7:** Thermal profile in the electrical cell using the numerical model by Hernlund et al.  
457 (2006). Simulations are considering a forsterite sample at a temperature of 1200°C.  
458 Isotherms are indicated as black lines with the temperature interval between two isotherms  
459 being 100 °C. The temperature at the center of the assemblies is 1203°C; the temperature

460 at the contact between TC wire(s) and Mo electrode is 1200 °C and the temperature at the  
461 sample/MgO sleeve is 1211°C. The computed current is 187 A, which is in very good  
462 agreement with experiments at this temperature.

**Table 1. Components of the 14/8 electrical cell assembly (TC = thermocouple).**

Material	Part	Source	Quantity for one experiment	Density <sup>a</sup> (g/mL)	Porosity <sup>a</sup> (%)
MgO-spinel (MG-ASU)	Octahedron	Ceramco	1	2.97	16.70
Zirconia	Inner sleeve	Mino Yogyo OZ-8C-HD	1	3.80	28.80
Rhenium, 63 micron foil	Furnace	Rhenium Alloys	1	21.02	0.00
MgO	4-bore (TC)	Saint-Gobain	2	2.41	32.60
MgO	Sample sleeve	Saint-Gobain	1	2.41	32.60
MgO	TC sleeve	Saint-Gobain	2	2.41	32.60
Mo	Electrode (disk or square)	Alfa	2	10.22	0.00
Fe	Electrode (disk or square)	Goodfellow	2	7.87	0.00
W-Re	TC wire (C-type)	Concept Alloys	3 or 4 <sup>b</sup>	-	-
Mullite	TC insulation sleeve	Coors	6 or 8 <sup>b</sup>	2.10	>30
Ceramic adhesive	Part # 919	Cotronics	-	-	-
Paper, 3-ply	Sheet around WC cubes	Anchor paper	4	-	-
Pyrophyllite	Gaskets around WC cubes	Bar-Io	12 <sup>c</sup>	2.69	4.30

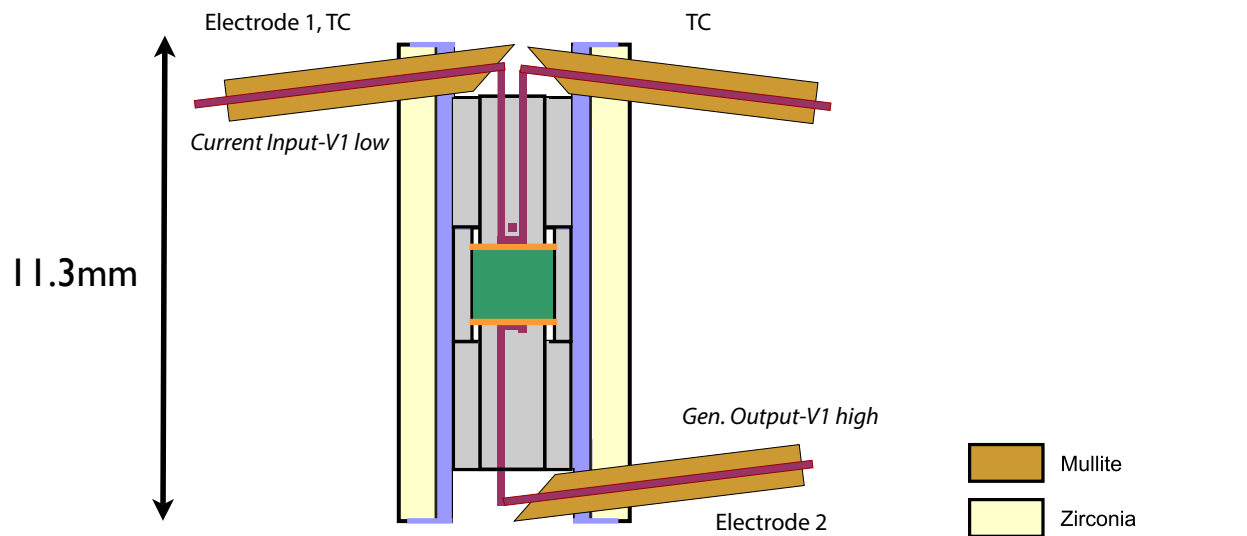
<sup>a</sup> From Leinenweber et al. (2012)

<sup>b</sup> The number depends on the type of electrical measurements (2 or 4-electrode experiments)

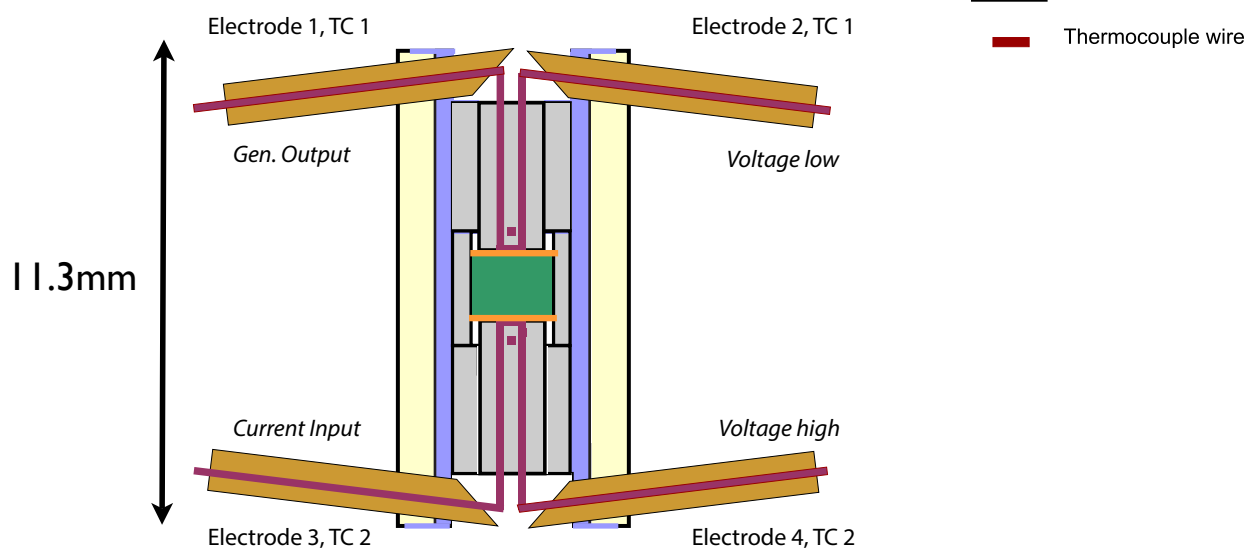
<sup>c</sup> Two sizes ("long" and "short"). See Leinenweber et al. (2012) for details.

**Figure 1**

**A) 2-electrode cell**



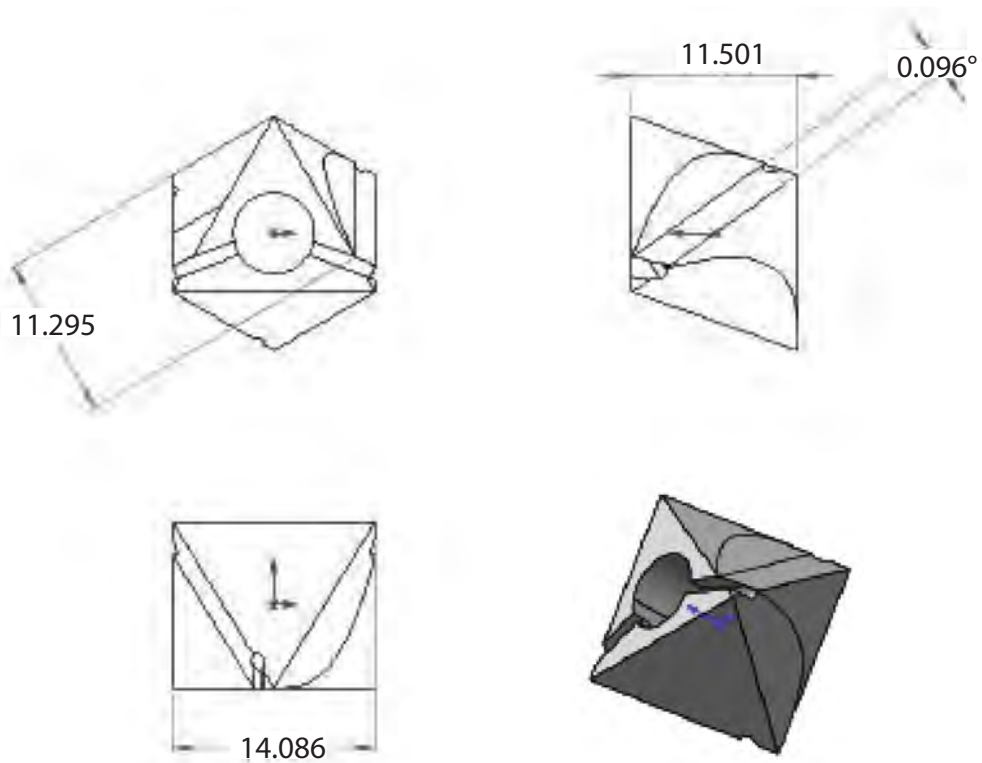
**B) 4-electrode cell**





**Figure 2**

A)



B)

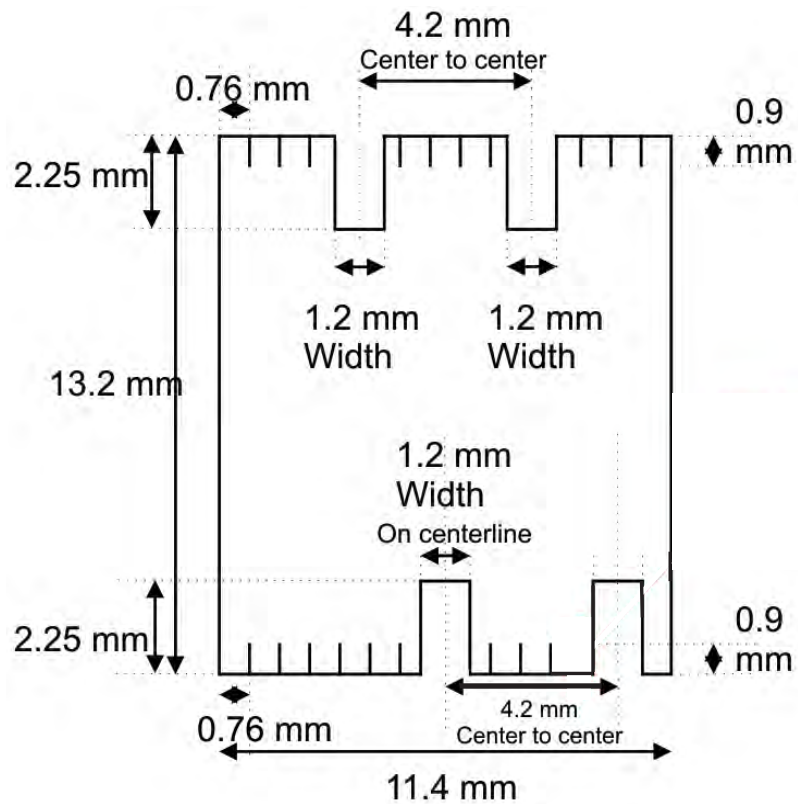


Figure 3

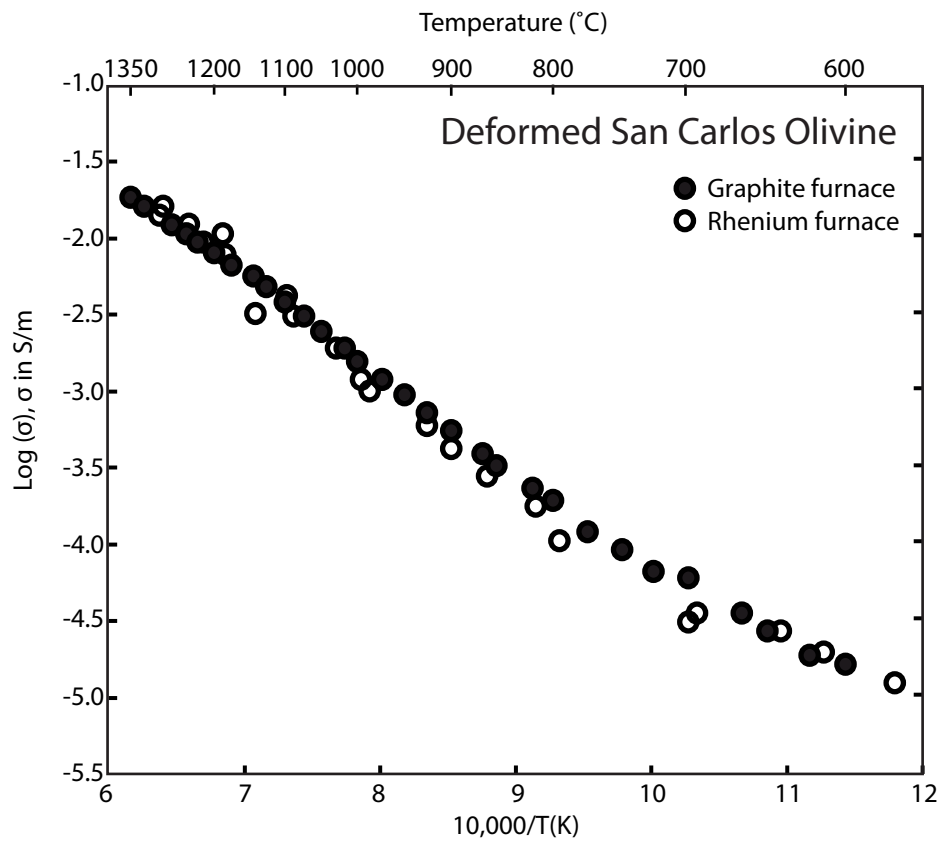


Figure 4

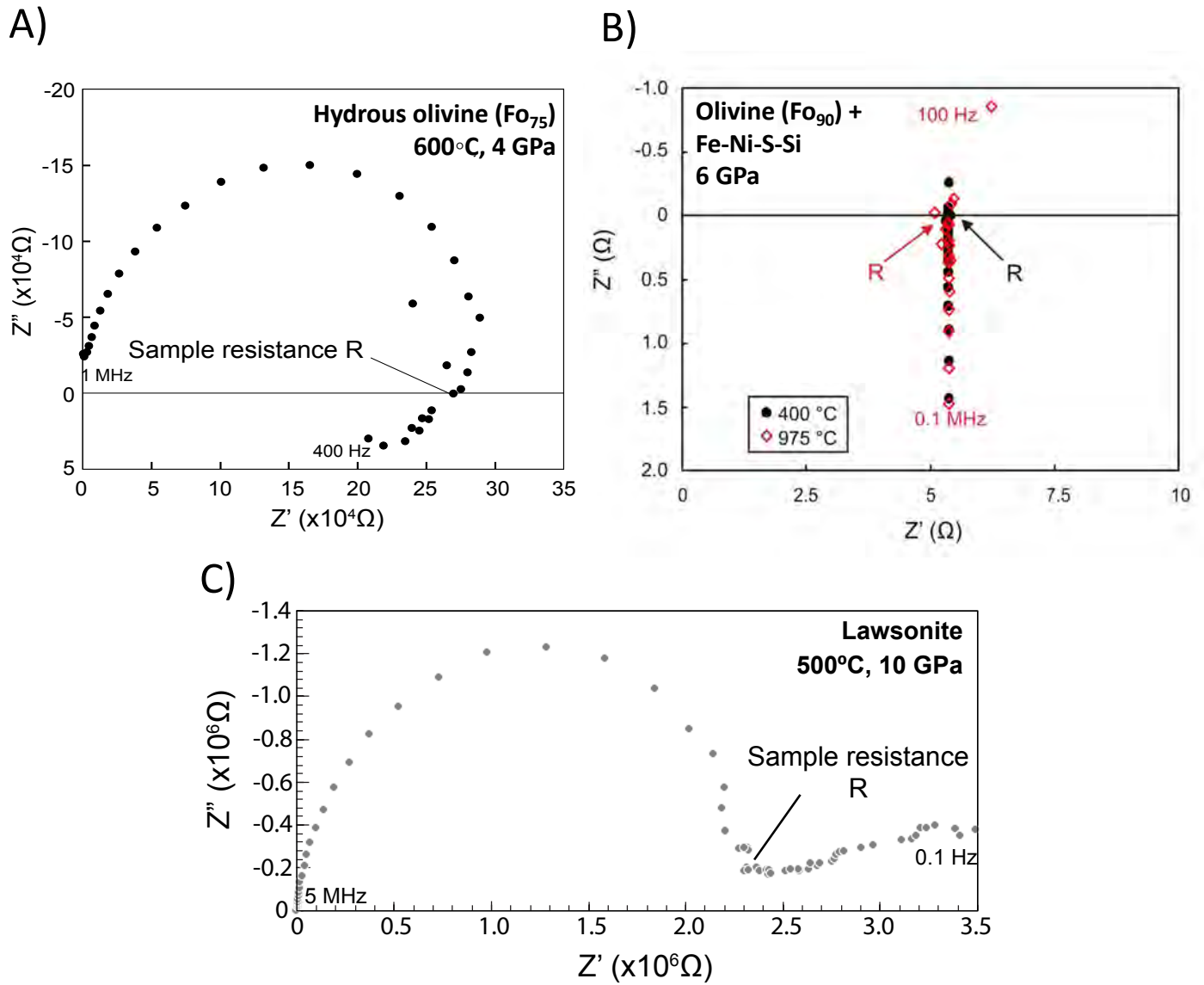


Figure 5

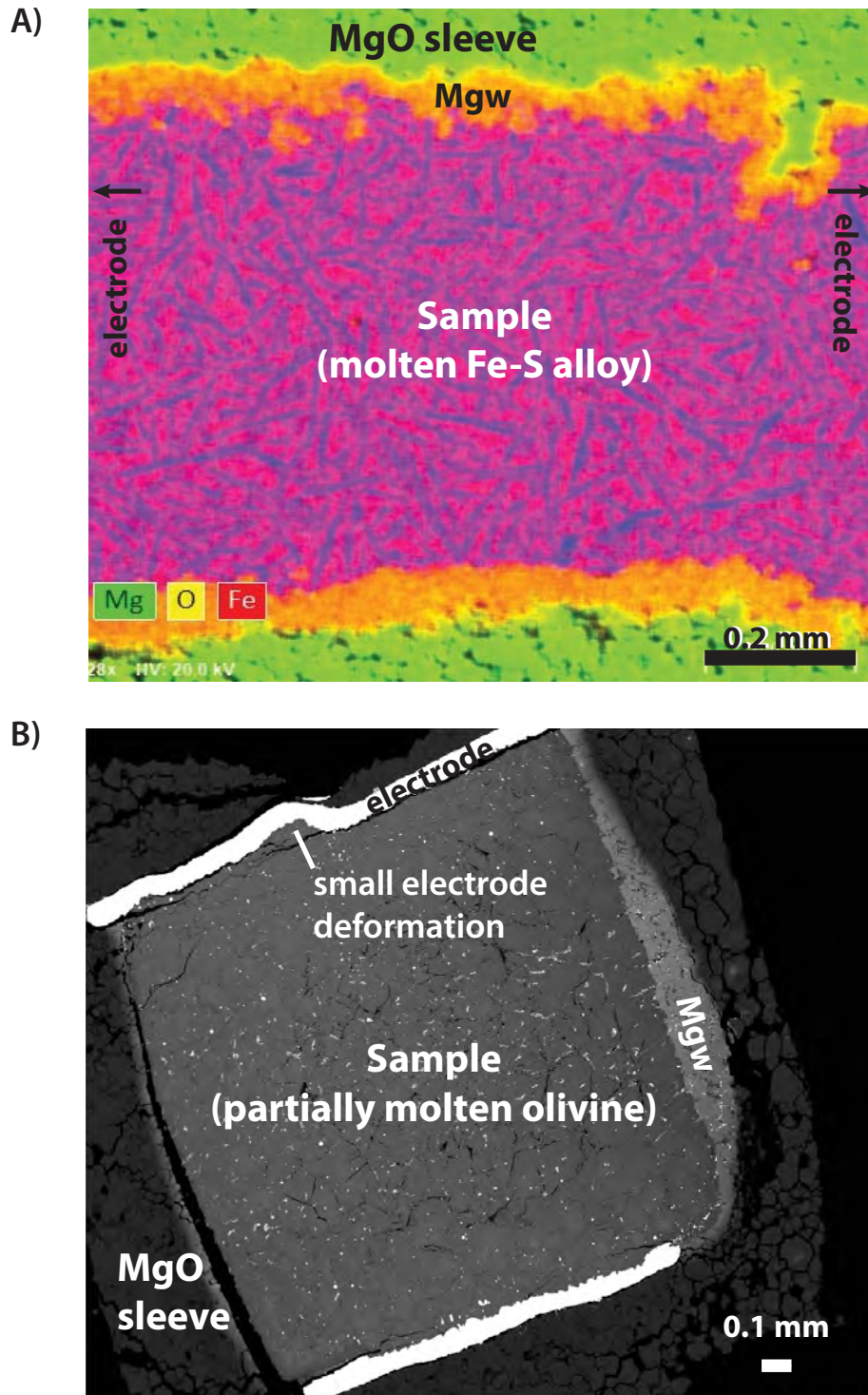


Figure 6

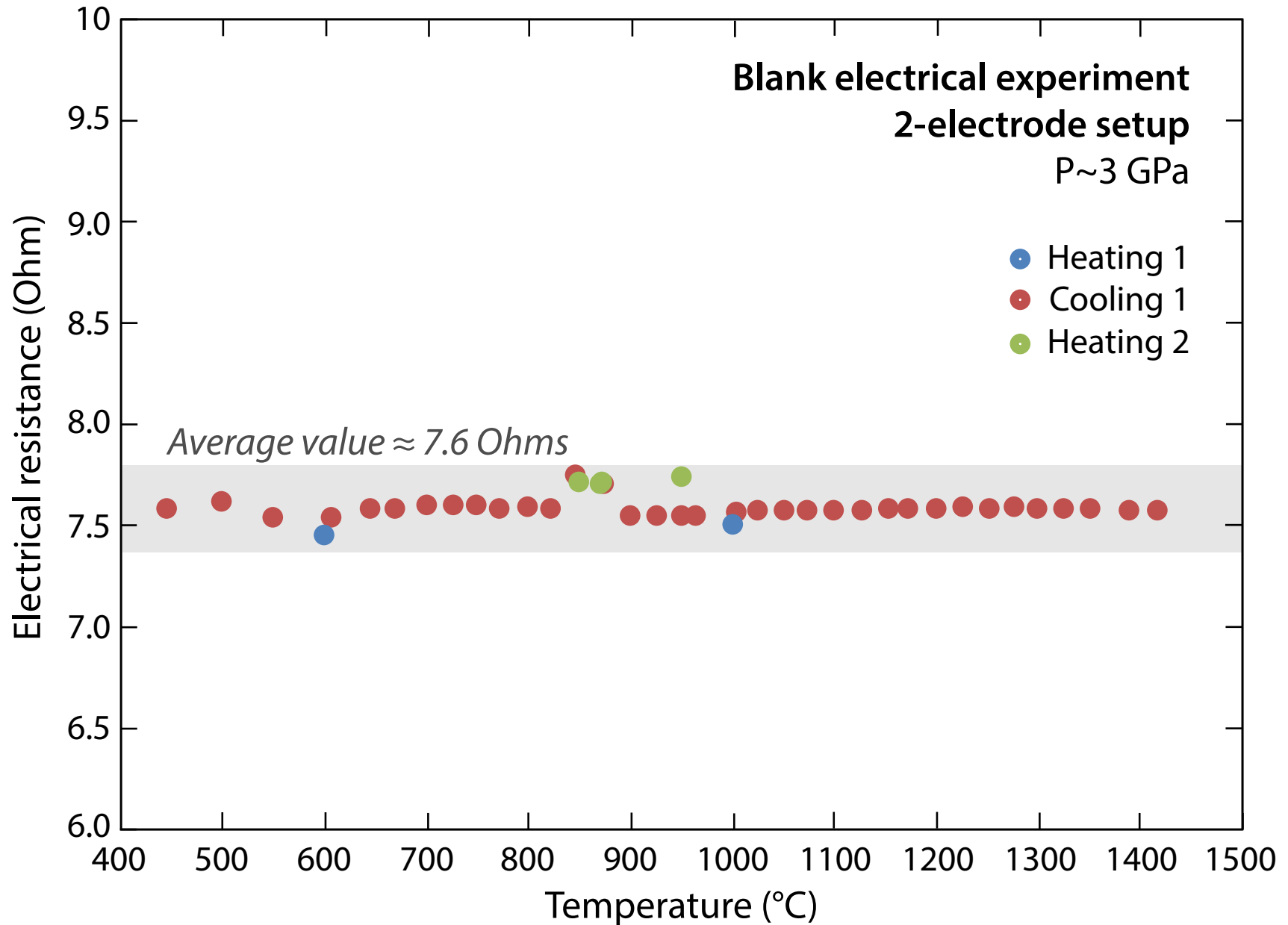


Figure 7

