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Influence of intensive parameters and assemblies on friction evolution during piston-cylinder experiments

Pierre Condamine, Simon Tournier, Bernard Charlier, Etienne Médard, Antoine Triantafyllou, Célia Dalou, Laurent Tissandier, Delphine Lequin, Camille Cartier, Evelyn Fűri, et al.

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1 Influence of intensive parameters and assemblies on friction
2 evolution during piston-cylinder experiments

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Authors

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8

9

Affiliations

10 ¹*Université de Lorraine, CNRS, CRPG, F-54000 Nancy, France*

11 ²*Department of Geology, University of Liège, 4000 Sart Tilman, Belgium*

12 ³*Université Clermont Auvergne, CNRS, IRD, OPGC, Laboratoire Magmas et Volcans, F-63000
13 Clermont-Ferrand, France*

14 ⁴*Géosciences Montpellier, CNRS, Université de Montpellier, F-34095 Montpellier, France*

15 ⁵*Geology laboratory of Lyon - Earth, Planets and Environment (LGL-TPE), Université Lyon 1, ENS
16 de Lyon, CNRS, UMR 5276, Villeurbanne, France*

17

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Correspondence (*)

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pierre.condamine@univ-lorraine.fr

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Abstract

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Piston-cylinder assemblies exhibit inhomogeneous pressure distributions and biases compared to the theoretical pressure applied to the hydraulic press because of the thermal and mechanical properties of the assembly components. Whereas these effects can partially be corrected by conventional calibration, systematic quantification of friction values remain very sparse and results vary greatly among previous studies. We performed an experimental study to investigate the behavior

27 of the most common cell assemblies, i.e., talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$), NaCl, and BaCO_3 , during piston-
28 cylinder experiments to estimate the effects of pressure, temperature, run duration, assembly size, and
29 assembly materials on friction values. Our study demonstrates that friction decreases with time and
30 also partially depends on temperature but does not depend on pressure. We determined that friction
31 decreases from 24 to 17% as temperature increases from 900 to 1300 °C when using talc cells,
32 indicating a friction decrease of ~2% per 100 °C increase for 24-h experiments. In contrast, friction
33 becomes independent of time above 1300 °C. Moreover, at a fixed temperature of 900 °C, friction
34 decreases from 29% in 6-h runs to 21% in 48-h runs, corresponding to a decrease of friction of 0.2%
35 per hour. Similar results obtained with NaCl cell assemblies suggest that friction is constant within
36 error, from 8% in 9-h runs to 5% in 24-h runs. At 900 °C, possible steady-state friction values are only
37 reached after at least 48 h, indicating that friction should be considered a variable for shorter
38 experiments. We establish that assembly materials (and their associated thermomechanical properties)
39 influence the friction correction more than the dimensions of the assembly parts. Finally, we show that
40 the use of polytetrafluoroethylene film instead of conventional Pb foil does not modify friction, but
41 significantly reduces the force required for sample extraction, thus increasing the lifetime of the
42 carbide core, which in turn enhances experimental reproducibility.

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Keywords

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Experimental petrology | Piston-cylinder | Friction | Assembly | Calibration

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1. Introduction

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The piston-cylinder apparatus (Boyd and England 1960) is well established in experimental
petrology and mineralogy for the synthesis of high-pressure and high-temperature geomaterials.
Typical setups can attain pressures of 0.5–4 GPa and temperatures of 600–2000 °C, and special setups
extend these ranges down to 0.3 GPa (e.g., Mirwald et al. 1975; Moore et al. 2008) and up to 2500 °C
(Cottrell and Walker 2006). Piston-cylinders are thus particularly well suited to investigate material
properties at crustal to upper mantle conditions on Earth and even at core conditions on smaller

54 planetary bodies. Furthermore, many thermodynamic models concerning the properties of the mantle
55 and their evolution through geological time rely on experimental databases. It is thus critical to
56 provide accurate experimental data with minimal uncertainties.

57 During high-pressure and high-temperature (*HP-HT*) piston-cylinder experiments, multiple
58 factors can lead to important biases on the pressure applied to the sample. Laboratories worldwide
59 employ various pressurization and heating procedures to reach the *P-T* conditions of interest, such as
60 the commonly used hot piston-in and piston-out techniques, leading to noticeably different applied
61 pressures (Johannes et al. 1971; Shimizu and Kushiro 1984; McDade et al. 2002). It has also been
62 suggested that a fraction of the hydraulic pressure is not transmitted to the sample due to
63 heterogeneous pressure distributions and/or frictional strain between the carbide core and the cell
64 assembly (Tamayama and Eyring 1967; Edmond and Paterson 1971). These pressure losses can be
65 characterized by the friction value *F* (in %), defined as the difference between the applied hydraulic
66 pressure P_{app} and the effective pressure on the sample P_{eff} :

$$67 \quad F = \left(\frac{P_{app}}{P_{eff}} - 1 \right) \times 100 \quad (1)$$

68 The materials used in piston-cylinder cell assemblies vary widely depending on the purpose of
69 the experiments (see Dunn 1993 for a review). For instance, different spacer and sleeve materials can
70 be used around the sample, each possessing unique thermal and mechanical properties (Bell et al.
71 1971; Longhi 2005). Alumina is sometimes substituted for MgO spacers to complete graphite furnaces
72 (Johannes et al. 1971; McDade et al. 2002); however, the different rheological properties of alumina
73 and MgO (elasticity, ductility, hardness) can lead to over- and under-pressured zones around the
74 sample. Furthermore, assemblies made with high-strength materials require greater pressure
75 corrections than those made with low-strength materials (Johannes, 1978). It is therefore important to
76 understand and quantify the role of material properties such as hardness, density, isothermal
77 compressibility, and thermal expansion during *HP-HT* experiments.

78 Several studies have sought to (i) understand and model thermal uncertainties and
79 reproducibility during piston-cylinder experiments (e.g., Watson et al. 2002; Médard et al. 2008) and
80 (ii) estimate the friction correction required depending on cell materials, including the commonly used

81 talc, NaCl, and BaCO₃ pressure media (e.g., Fram and Longhi 1992; Bose and Ganguly 1995; McDade
82 et al. 2002; Longhi 2005). However, the reproducibility of pressure and associated corrections (i.e.,
83 friction) during *HP-HT* experiments and which parameters control their evolution remain unclear. For
84 example, Fram and Longhi (1992) claimed that the friction correction is highly dependent on pressure
85 for BaCO₃ cells, whereas McDade et al. (2002) reported that friction is independent of temperature
86 and pressure at 1000–1600 °C and 1.5–3 GPa. Therefore, the main objective of this study is to
87 experimentally quantify the friction induced by the use of talc, NaCl, and BaCO₃ cells during piston-
88 cylinder experiments. Whereas the use of talc cells is known to produce important pressure losses on
89 the sample compared to the applied hydraulic pressure, friction values are only sparsely reported and
90 vary widely (Johannes et al. 1971; McDade et al. 2002). Our second aim, then, is to understand and
91 quantify the roles of experimental parameters (pressure, temperature, and experimental duration) and
92 assembly materials and size on friction and its evolution during short- and long-duration experiments.

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2. Methods

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2.1. Experimental strategy

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Experiments were performed on three different reactions. First, we chose the quartz-coesite transition for its shallow *P-T* slope (Bose and Ganguly 1995, and references therein), which limits biases due to thermal gradients. This simple reaction precludes problems observed in other commonly used transitions, such as synthesis of the starting material in the fayalite + quartz = ferrosilite transition (see Bohlen et al. 1980). In addition, the kinetics of this transition are remarkably fast (Lathe et al. 2005), allowing experiments to be performed over both short (2.5 h) and long durations (48 h), in turn allowing us to investigate the evolution of friction over time. We calibrated the quartz-coesite transition over a wide range of typical piston-cylinder temperatures, i.e., at 900 and 1300 °C, to understand the role of temperature on frictional issues in the three cell types (Figure 1). Second, we investigated the albite (NaAlSi₃O₈) = jadeite (NaAlSi₂O₆) + quartz (SiO₂) reaction at 800 and 1000 °C using BaCO₃ cells (Holland 1980). Third, we investigated the anorthite (CaAl₂Si₂O₈) + gehlenite (Ca₂Al₂SiO₇) + corundum (Al₂O₃) = kushiroite (3CaAl₂SiO₆) reaction (Hays 1966a, b) at a constant

108 pressure of ~1.3 GPa using talc cells to understand the role of pressure and assembly size (1/2" vs.
109 3/4") on friction at 1300 °C.

110 Because low-temperature and short-duration experiments exacerbate friction (Bose and
111 Ganguly 1995), we performed additional experiments on the roles of assembly size and component
112 materials at 900 °C during 6 h. These experiments included replacing the MgO sleeve around the
113 capsule with an alumina sleeve, using a polytetrafluoroethylene (PTFE) film wrapping the cell, and
114 using higher density MgO spacers (2.8 vs. 2.0 g/cm³) in the graphite furnace (Table 1). A last series of
115 experiments was performed on a new batch of assemblies (thicker talc cell, thinner Pyrex glass) at
116 more conventional piston-cylinder conditions (1300 °C, 24 h) to understand the potential role of
117 assembly part sizes on the generated friction (batch #2; Table 1, Figure 1d).

118

119 **2.2. Starting materials**

120 For the quartz-coesite calibration experiments, we used analytical grade amorphous SiO₂ (99.9
121 wt% purity) and added 6 wt% H₂O to each capsule with a micro syringe to enhance the reaction
122 kinetics (Lathe et al. 2005). To study the kushiroite = anorthite + gehlenite + corundum reaction, we
123 prepared a mixture based on the stoichiometry of the kushiroite, using analytical grade SiO₂, Al₂O₃,
124 and CaCO₃ powders, ground in ethanol for 1 h, and then decarbonated in a furnace at 1200 °C during
125 24 h. The procedure was repeated to ensure complete decarbonation. Oxide powders were stored in an
126 oven at 110 °C prior to capsule assembly. For the albite = jadeite + quartz reaction, the starting
127 material consisted of a mixture of pure powdered crystals, validated by X-ray diffraction
128 measurements on a Bruker D8-Advance diffractometer with CuK α radiations (ULiege). Proportions
129 were set during mixture preparation, with 70 wt.% quartz, 20 wt.% jadeite and 10 wt.% albite. The
130 powder's mixture was stored in an oven at 120°C prior to capsule assembly.

131

132 **2.3. Experimental techniques**

133 We performed 38 experiments on identical Voggenreiter Mavo LPC 250-300/50 end-loaded
134 12.7-mm (1/2") and 19.0-mm (3/4") piston-cylinders at three different institutes: the Centre de

135 Recherches Pétrographiques et Géochimiques (CRPG, Nancy, France) for talc cell assemblies, the
136 Laboratoire Magmas et Volcans (LMV, Clermont-Ferrand, France) for NaCl cell assemblies, and the
137 University of Liège (ULiège; Belgium) for BaCO₃ cell assemblies. The talc-based piston-cylinder
138 assembly (Ceramic Substrates and Components Ltd.) comprised a talc cell wrapped in an outer PTFE
139 film, a Pyrex cylinder, a graphite furnace, and inner MgO spacers (Figure 1a). The current was
140 conducted to the graphite heater through a stainless-steel plug, which was electrically isolated with a
141 pyrophyllite sleeve. The use of PTFE film instead of conventional Pb foil for the outer sleeve allows
142 covering the base plug, which tends to drastically decrease the force required to extract the assembly at
143 the end of an experiment from ~35 to ~5 kN, which in turn better preserves the carbide core from
144 fracturing. PTFE is also a more environment- and health-friendly alternative compared to Pb and/or
145 Mo-based lubricants. The NaCl cell assembly was similar to the talc cell assembly (Figure 1b). At
146 ULiège, the assembly is composed of a BaCO₃ cell wrapped in Pb foil, a graphite furnace, and MgO
147 spacers (Figure 1c). Additional details on assembly materials and dimensions are provided in
148 Supplementary Materials 1. Calibrated W₇₄Re₂₆-W₉₅Re₅ thermocouples protected by a 4-bore alumina
149 sleeve were used to control temperature to within 1 °C of the setpoint. Temperature gradients
150 throughout the capsule are estimated to be about 20 °C (Sorbadere et al. 2013). The hot spot is located
151 in the center of the capsule, implying a thermal gradient of about 10 °C/mm. To preclude perforation
152 of the capsule, an Al₂O₃/MgO cap 0.6 mm thick was inserted between the capsule and the
153 thermocouple. During experiments, the pressure was first increased to about 0.7 GPa (1/2" assemblies)
154 or 0.3 GPa (3/4" assemblies) at room temperature. In all experiments, the temperature was then
155 increased to 650 °C at 50 °C/min, held for 15 min to reach the target pressure, and then heated to the
156 target temperature.

157 Several series of experiments were conducted to understand the role of assembly parts on
158 friction in talc cells (Table 1). In experiments V32 and V42, we replaced the MgO sleeve with an
159 alumina sleeve, a method used to preserve the experimental charge because alumina sleeves are harder
160 and denser than MgO sleeves. We also investigated the role of the density of MgO spacers by
161 replacing the conventional spacers (2.0 g/cm³) with higher density spacers (2.8 g/cm³) in experiments
162 V109 and V117. In experiments V110 and V116, we tested a last batch of assemblies (batch #2) with

163 thicker talc cells (1.35 mm instead of 0.75 mm) and thinner Pyrex glass cylinders (1 mm instead of 1.6
164 mm; see details in Supplementary Materials 1). Finally, in experiments V39 and V107, Pb foil was
165 used instead of the PTFE film.

166 For quartz-coesite transition experiments, Au₈₀Pd₂₀ capsules (4 mm high) were used to limit
167 water loss (Gaetani and Grove 1998; Kägi et al. 2005). For the kushiroite reaction, Pt capsules (4 mm
168 high) were employed. For the albite = jadeite + quartz reaction, graphite containers (3.75 mm high)
169 were used. At the end of each experiment, the capsule was cut in half longitudinally using a wire saw,
170 mounted in epoxy, and polished to 0.25 µm on nylon pads with diamond pastes.

171

172 **2.4. Analytical techniques**

173 Experimental run products of the kushiroite reaction were analyzed with a JEOL JSM-6510
174 scanning electron microscope (SEM) at CRPG using an accelerating voltage of 15 kV, a beam current
175 of 10 nA, and a spot size of ~ 1 µm. Back-scattered electron images of experimental run products of
176 the albite = jadeite + quartz reaction were acquired on the QEMSCAN FEI Quanta 650F at RWTH
177 Aachen (Germany).

178 Raman spectroscopy was used to determine the nature of silica polymorphs in all experiments.
179 Raman spectra were acquired using a LabRAM HR spectrometer (Horiba Jobin Yvon) at
180 GeoRessources (Nancy, France). The spectrometer is equipped with a 600 gr/mm grating and an edge
181 filter. The confocal hole aperture is 500 µm and the slit aperture is 100 µm. The excitation beam
182 (wavelength, 514.53 nm; power, 200 mW) was produced by a Stabilite 2017 Ar⁺ laser (Spectra
183 Physics, Newport Corporation) and focused on the sample using a 50× objective. The acquisition time
184 (2 s) and the number of accumulated spectra (20) were chosen to optimize the signal-to-noise ratio. All
185 spectra were recorded over Raman shifts of 150–1400 cm⁻¹. Quartz and coesite were identified by
186 their respective peaks at 470 and 520 cm⁻¹.

187

188

3. Experimental textures and products

189 Experimental conditions and results of quartz-coesite, kushiroite, and albite reactions are
190 reported in Table 1, and calculated frictions are reported in Table 2. Pictures of typical run products
191 are provided in Supplementary Material 2. In quartz-coesite experiments, coesite was always observed
192 with variable amounts of quartz. Coesite mainly occurs as straight veins in the euhedral quartz matrix,
193 with no preferential orientation. In kushiroite reaction experiments, grain growth was observed in all
194 runs, with anhedral grains ranging from 20 to 50 μm . Kushiroite mainly appears near corundum and
195 anorthite, replacing gehlenite. In runs in which kushiroite was observed (V106 and V113), anorthite,
196 gehlenite and corundum were still present after 24 h.

197 Non-negligible amounts of the low-pressure phases in the three investigated reactions (i.e.,
198 quartz, anorthite + gehlenite + corundum, and albite) persisted after the transition reaction occurred.
199 This result is consistent with Hays (1966a, b), who observed low-pressure phases persisting above the
200 transition to kushiroite, indicating a continuous reaction over a pressure interval of a few kilobars. In
201 contrast to our experiments with a transitional pressure interval of ~ 0.1 GPa, Bose and Ganguly (1995)
202 found only either quartz or coesite in their experiments with a pressure interval of ~ 0.05 GPa. We
203 could not estimate modal abundances from our experimental textures due to significant sample losses
204 during cold decompression.

205

206

4. Roles of intensive parameters on friction

207 In the following sections, experiments from Bose and Ganguly (1995) were chosen as the
208 reference pressure (P_{eff}) for the quartz-coesite system. They indeed showed that after 30 h, the friction
209 is null in their assemblies, based on crossed calibrations using high pressure vessel experiments. In the
210 quartz-coesite system, our experiments at both 900 and 1300 $^{\circ}\text{C}$ allow us to investigate the role of
211 temperature on friction (Figure 2). In 24-h experiments in talc cells, the quartz-coesite transition
212 occurred between applied pressures of 3.68 and 3.77 GPa at 900 $^{\circ}\text{C}$ and between 3.77 and 3.87 GPa at
213 1300 $^{\circ}\text{C}$. In contrast, Bose and Ganguly (1995) observed the transition at 3.00 and 3.28 GPa at 900
214 and 1300 $^{\circ}\text{C}$, respectively, in CsCl cells. Taking their values as the effective pressure on the sample,

215 our results indicate friction values (F) for talc cells of 24.1% at 900 °C and 16.5% at 1300 °C (Table
216 1). A linear fit to these values (Figure 2) shows that friction decreases by about 2% when temperature
217 increases by 100 °C in 24-h experiments. In NaCl cells, the quartz-coesite transition occurred between
218 3.11 and 3.21 GPa at 900 °C ($F = 5.4 \pm 3.0\%$) and between 3.41 and 3.46 GPa at 1300 °C ($F = 4.8 \pm$
219 3.0%), indicating no friction evolution within error. In BaCO₃ cells, the albite = jadeite + quartz
220 reaction occurred between 2.35 and 2.4 GPa at 800 °C and between 2.8 and 2.9 GPa at 1000 °C.
221 Compared to 2.16 and 2.69 GPa at 800 and 1000 °C obtained in Holland (1980), $F = 10.2 \pm 4.3$ and
222 $6.1 \pm 3.4\%$, respectively, indicating no friction evolution in this range of temperature.

223 Our experiments on the kushiroite = anorthite + gehlenite + corundum reaction were
224 conducted at 1300 °C to understand the role of pressure on the friction value in talc cells. In 1/2"
225 assemblies, this transition occurred between applied pressures of 1.45 and 1.54 GPa in 24-h
226 experiments. According to the equation of Hays (1966a), the reference pressure for this transition is
227 1.3 GPa, resulting in a friction value of $15.1 \pm 4.3\%$.

228 This value is identical, within errors, to the friction value obtained from the quartz-coesite
229 transition at the same temperature ($16.6 \pm 3.2\%$), implying that the friction value is independent of
230 confining pressure at identical temperatures and experimental durations. This result is partially
231 consistent with McDade et al. (2002), who argued that the friction correction is independent of
232 pressure and temperature at 1000–1600 °C and 1.5–3.2 GPa in BaCO₃ cells. However, our results
233 show that friction is temperature dependent in talc cells, decreasing by 2% over a temperature increase
234 of 100 °C between 900 and 1300 °C. This difference can be explained by the slower stress
235 accommodation of talc compared to NaCl and BaCO₃ cells due to its higher strength. It is also possible
236 that friction becomes more strongly temperature dependent at low temperature (<1000 °C). We
237 speculate that stress accommodation is fast enough above 1000 °C to be undetectable under these
238 experimental conditions.

239

240

5. Friction evolution

241 We performed experiments on the quartz-coesite reaction at 900 °C and over various run
242 durations between 2.7 and 48 h to investigate the evolution of friction during a single run (Figure 3).

243 In talc cells, the transition occurred at progressively lower applied pressures with increasing
244 duration (Table 1); friction thus evolved from $33.8 \pm 3.4\%$ after 2.7 h to $30.7 \pm 3.3\%$ after 4.5 h, 27.3
245 $\pm 3.3\%$ after 6 h, $24.1 \pm 3.2\%$ after 24 h, and to $20.9 \pm 3.2\%$ after 48 h (Figure 3). Whereas a linear fit
246 to the data for longer durations (≥ 6 h) indicates that friction decreases by $\sim 2\%$ every 10 h, a power-law
247 fit better describes the full dataset. This may explain the ubiquitous presence of quartz in all
248 experiments conducted in talc cells via the early crystallization of quartz from the amorphous SiO_2
249 starting material at low effective pressures and its subsequent metastability. In contrast, in talc cell
250 assemblies at 1300°C , friction did not evolve between 6- and 24-h duration experiments, indicating
251 that a frictional steady state was achieved within less than 6 h.

252 In NaCl cells at 900°C , the apparent pressure of the quartz-coesite transition decreased from
253 between 3.21 and 3.30 GPa after 9 h ($F = 8.4 \pm 3.1\%$) to between 3.11 and 3.21 GPa after 24 h ($F =$
254 $5.4 \pm 3.0\%$), indicating no friction evolution within error, contrary to what is reported in talc cells over
255 longer duration experiments. The friction decrease in talc cell is however consistent with the data of
256 Bose and Ganguly (1995) in CsCl cell assemblies (both $1/2''$ and $3/4''$), which show that friction
257 decreased from 6.6% after 2 h to negligible after 35 h, or by $\sim 2\%$ every 10 h (Figure 3).

258

259 **6. Thermomechanical properties of assembly components**

260 **Alumina sleeve**

261 Experiments V32 and V42 (Table 1) were performed at 900°C and applied pressures of 3.87
262 and 3.97 GPa, respectively, with an alumina sleeve around the capsule instead of a conventional MgO
263 sleeve. In both experiments, only quartz was observed, and the pressure limitation of the carbide core
264 precludes experiments at pressures above ~ 4 GPa. Thus, the minimum friction observed using alumina
265 sleeves is $>32.3\%$ (Figure 4). However, the main advantage of alumina sleeves is that the sample
266 capsule conserves its cylindrical shape, preventing excessive deformation during quenching and cold
267 depressurization, a net advantage when working with single crystals. McDade et al. (2002) obtained a
268 friction value of 3.6% in NaCl cells by using an alumina sleeve and Alsimag (Al_2O_3) plugs to fill the
269 tapered graphite furnace. Given the high density and hardness of Alsimag, these plugs are better

270 pressure transmitters than the crushable MgO used in our assembly setup. The combination of an
271 alumina sleeve with MgO spacers thus greatly decreases pressure transmission and should only be
272 used to limit fracturing of the experimental product in cases requiring particular care for the capsule.

273

274 **Density of MgO spacers**

275 Experiments V109 and V117 made with talc-Pyrex assemblages were conducted 900 °C and
276 applied pressures of 3.68 and 3.77 GPa, respectively, with higher density MgO spacers (2.8 vs. the
277 usual 2.0 g/cm³). Both coesite and quartz were observed in V117, positioning the apparent quartz-
278 coesite transition at 3.72 GPa. The friction obtained using these MgO spacers ($24.1 \pm 3.2\%$; Figure 4)
279 is identical within error to that obtained using the standard assembly under the same conditions ($27.3 \pm$
280 3.3%). This result demonstrates that the densities of assembly components does not significantly affect
281 the effective sample pressure. This however further highlights that the Alsimag spacers used by
282 McDade et al. (2002) are a good pressure medium, as they achieved a friction value of only 3.6%.

283

284 **Assembly size (1/2" vs. 3/4")**

285 Kushirolite reaction experiments were performed in both 1/2" and 3/4" assemblies at 1300 °C
286 for 24 h (Table 1). The calculated friction values are identical within errors, i.e., $15.1 \pm 4.3\%$ and 15.9
287 $\pm 4.4\%$ for the 1/2" and 3/4" assemblies, respectively (Figure 3), suggesting that the size of assembly
288 parts has a negligible effect on friction compared to assembly materials (and their associated
289 thermomechanical properties).

290 Experiments V110 and V116 were performed using assemblies with thicker talc cells and
291 thinner Pyrex sleeves ('batch #2', see Supplementary Materials 1 for size details). The friction
292 determined for this assembly ($16.6 \pm 3.2\%$) is identical to that for the standard assembly ($16.6 \pm 3.2\%$)
293 at 1300 °C for 24 h (Figure 4), further demonstrating that assembly materials have a stronger influence
294 on friction than their sizes.

295

296 **Pb foil vs PTFE foil**

297 Experiments V107 and V39 were performed at 900 °C and applied pressures of 3.77 and 3.87
298 GPa, respectively, using talc cells and standard Pb foil instead of the PTFE film used elsewhere in this
299 study (excluding ULiège experiments, Table 1). Coesite was only observed in experiment V39,
300 implying that friction ($27.3 \pm 3.3\%$) was identical within errors to that using a PTFE-wrapped
301 assembly ($27.3 \pm 3.3\%$; Figure 4). As previously mentioned, the key advantage of using PTFE film
302 instead of Pb foil is the preservation of the carbide core of the pressure plates. The steel base plug,
303 surrounded by pyrophyllite, is frequently stuck in the carbide core when removing the assembly,
304 especially after experiments run at pressures above 2.0 GPa. The use of Pb foil precludes covering the
305 base plug to avoid melting the Pb at the contact point with the thermocouple plate, which can
306 electrically short-circuit the experiment. Therefore, the use of PTFE film is recommended to limit the
307 development of fractures within the carbide core, which will both extend the life of the carbide core
308 and enhance pressure reproducibility over that lifetime.

309

310 **7. Implications for pressure reproducibility during piston-cylinder experiments**

311 In this section, we provide general recommendations to improve pressure and temperature
312 reproducibility during piston-cylinder experiments. These recommendations can be adapted,
313 depending on the capsule material and the scientific purpose, e.g., for oxygen-fugacity buffering or
314 volatile-bearing experiments. These recommendations are depicted in P - T - t space in the form of
315 preferential practices in Figure 5.

316 At temperatures above 1600 °C, the NaCl cell is no longer usable because of the melting point
317 of NaCl. Consequently, only talc or BaCO₃ cells should be used for very-high temperature
318 experiments. Talc cells require a very-high frictional correction factor, regardless of P - T - t conditions
319 (Table 2) and should thus be avoided in very high pressure experiments (>3 GPa) because the carbide
320 core could suffer from excessive applied pressure. To generate pressures above 3 GPa, we thus
321 recommend using NaCl cells below 1600 °C because they exhibit the lowest friction corrections
322 (Figures 2 and 3) or BaCO₃ cells at higher temperatures. For experimental durations of a few to ~48 h

323 and pressures below 3 GPa, talc cells are as competent as NaCl and BaCO₃ cells because they all show
324 similar frictional evolution with time. However, for short experiments (≤ 6 h), we recommend using
325 NaCl or BaCO₃ cells as the decrease in friction value with time is comparable and because the friction
326 in talc cells is much greater (following a power law) in shorter duration experiments (Figure 3). As
327 described in section 5, this could lead to unexpectedly low effective pressures, promoting the
328 crystallization and metastability of low-pressure phases. For experiments longer than 48 h (and below
329 1600 °C), we recommend using NaCl cells: whereas friction becomes negligible in CsCl cells after
330 ~30 h (Bose and Ganguly 1995), the behavior of talc cells in experiments longer than 48 h remains
331 unclear. Therefore, for temperatures above 1600 °C and durations longer than 48 h, we recommend
332 using BaCO₃ cells because of their lower frictional correction compared to talc cells.

333

334

8. Conclusions

335 We determined the effects of pressure, temperature, time and assembly parts (materials and
336 size) on the frictional correction factors to be applied to piston-cylinder experiments using talc cell
337 assemblies. Whereas pressure calibrations in most laboratories assume a single value for friction
338 correction for a given assembly, our study demonstrate that friction is time- and temperature-
339 dependent, and particularly elevated friction can be produced for short experiments at low
340 temperature, especially for talc-cell assemblies. Whereas the size of assembly parts has a negligible
341 effect on friction, different sleeve and spacer materials (here, MgO vs. alumina) can significantly
342 change the friction factors. Friction decreases by about 2% per 100 °C increase between 900 and 1300
343 °C at a given duration. Experimental duration also has a strong effect on the friction value at low
344 temperatures, with friction decreasing by about 0.2% per hour at 900 °C; this effect is not observed at
345 1300 °C. In contrast to CsCl cells, in which friction becomes negligible after around 30–35 h, the
346 friction in talc cells, although decreasing with increasing run duration, is never negligible, especially at
347 low temperatures. Therefore, special care must be taken for low-temperature experiments (roughly
348 <1000 °C) using talc cells, especially at short durations, because the slow stress accommodation of the
349 assembly results in slow frictional evolution during the first several hours. Other key parameters not
350 studied here may also critically affect friction and its evolution. In particular, the quality of the carbide

351 core and the development of fractures are expected to influence the friction generated on the assembly.
352 A more systematic calibration procedure including the status of the core is thus necessary to provide a
353 complete model of friction and its evolution during an experiment. This is fundamental for phase
354 relationship experiments or the quest for low-degree melts from mantle lithologies, for which accurate
355 and reproducible *P-T* conditions are essential. Furthermore, a dynamic model for friction correction
356 should be developed to ensure pressure reproducibility among *HP-HT* petrological studies.

357

358

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426

Figure captions

427 **Figure 1.** Schematic diagrams of the four different piston-cylinder cell assemblies used in this
428 study. a) talc cell assembly used at CRPG (Nancy), b) NaCl cell assembly used at LMV (Clermont-
429 Ferrand), c) BaCO₃ assembly used at ULG (Liège), d) batch #2 of the talc cell assembly used at
430 CRPG, using a thicker cell and thinner Pyrex sleeve compared to *a*. The talc cell is 0.75 mm thick in *a*
431 against 1.35 mm in *d*. Contrary to other assemblies, no Pyrex glass is present in the BaCO₃ cell
432 assembly and a Pb film is used instead of PTFE to wrap the cell. See Supplementary Materials 1 for
433 additional details.

434

435 **Figure 2.** Friction evolution as a function of temperature for 24 h experiments in 1/2" cell
436 assemblies. The durations of experiments from McDade et al. (2002) were 22 h. Linear fits describe
437 the change in friction as a function of temperature (°C): $F = -0.019T + 40.991$ for talc cells; $F = -$
438 $0.006T + 10.370$ for NaCl cells; $F = -0.020T + 26.463$ for BaCO₃ cells.

439

440 **Figure 3.** Evolution of the friction value as a function of experimental duration. B&G1995
441 data correspond to the study of Bose and Ganguly (1995), performed at 900 °C with CsCl cells. At 900
442 °C, the friction value decreases of about 2 % every 10 h for experimental durations > 6 h. At 1300 °C,
443 no friction decrease is observed between 6 and 24 h. Lower pressure (kushiroite experiments) and
444 sizing of assemblies (1/2" and 3/4") do not seem to have a critical influence on the friction value and
445 its evolution. The slope of the friction decrease observed in NaCl cell assemblies is comparable to the
446 slope observed in Bose and Ganguly (1995).

447

448 **Figure 4.** Variations in friction with changes in assembly size and components, relative to
449 experiments performed using the assembly shown in Figure 1a (talc cell, MgO sleeve, PTFE film, 2.0
450 g/cm³ MgO spacers) at 900 °C for 6 h and 1300 °C for 24 h (1/2" assembly). 'Alumina sleeve'
451 indicates that the regular MgO sleeve around the capsule was replaced by an alumina sleeve. 'Pb foil'
452 indicates that the cell was wrapped in standard Pb foil instead of the PTFE film used throughout this
453 study. 'MgO spacers' indicates that higher density MgO spacers (2.8 g/cm³) were used within the

454 graphite furnace (including the sleeve around the capsule). ‘Assembly batch #2’ indicates the
455 assembly shown in Figure 1d, using a thicker talc cell and thinner Pyrex sleeve than those in the
456 regular assembly (Figure 1a). See Supplementary Materials 1 for details. Note that the friction
457 reported for ‘Alumina sleeve’ is a minimum value because coesite was not observed in this series of
458 experiments due to the pressure limitations of the press and carbide core.

459

460 **Figure 5.** Recommended cell types for piston-cylinder experiments as a function of pressure,
461 temperature, and experimental duration. Note that the duration axis is not linear. These general
462 recommendations limit the uncertainty on pressure and its evolution during *HP-HT* experiments,
463 except for avoiding NaCl cells in experiments at very high temperature (>1600 °C) because the NaCl
464 cell no longer acts as an electrical insulator. See discussion in section 7 for further details. Cell types
465 are recommended as ‘best > intermediate > worst’ according to the color scale at bottom right.

466

467 **Table 1**

Experimental conditions and results						
Assembly	T (°C)	Duration (h)	Run	P _{app} (GPa)	Product	
Talc cell experiments (CRPG laboratory)						
PTFE - Talc - Pyrex - C - MgO	900	2.7	V130	3.97	qz	
			V16	4.06	qz + co	
		4	V10	3.87	qz	
		5	V19	3.97	qz + co	
		6	V108	3.77	qz	
			V55	3.87	qz + co	
		15	V41	3.77	qz	
			V38	3.87	qz + co	
		24	V30	3.68	qz	
			V24	3.77	qz + co	
		48	V33	3.58	qz	
			V56	3.68	qz + co	
			6	V37	3.77	qz
				V18	3.87	qz + co
			24	V22	3.77	qz
		PTFE - Talc - Pyrex - C - Al ₂ O ₃	900	6	V57	3.87
V114	1.45				an + gh + crn	
V113	1.54				kush + an + gh + crn	
V32	3.87				qz	
Pb - Talc - Pyrex - C - MgO	900	6	V42	3.97	qz	
			V107	3.77	qz	
PTFE - Talc - Pyrex - C - MgO (2800 kg/m ³)	900	6	V39	3.87	qz + co	
			V109	3.68	qz	
PTFE - Talc - Pyrex - C - MgO (batch #2)	1300	24	V117	3.77	qz + co	
			V110	3.77	qz	
PTFE - Talc - Pyrex - C - MgO (3/4")	1300	24	V116	3.87	co	
			V112	1.46	an + gh + crn	
			V106	1.55	kush + an + gh + crn	
NaCl cell experiments (LMV laboratory)						
PTFE - NaCl - Pyrex - C - MgO	900	9	PC/2014/11	3.21	qz	
			PC/2014/14	3.30	qz + co	
		24	Cal-LMV-03	3.11	qz	
			Cal-LMV-01	3.21	qz + co	
		1300	24	Cal-LMV-02	3.41	qz
				Cal-LMV-04	3.46	qz + co
BaCO₃ cell experiments (ULG laboratory)						
Pb - BaCO ₃ - C - MgO	800	24	A070	2.35	ab	
			A058	2.40	jd + qz	
	1000	24	A073	2.80	ab	
			A060	2.90	jd + qz	

468

469 **Table 2**

Friction values										
Size (")	T (°C)	Duration (h)	P _{app}	ΔP _{app}	P _{eff}	ΔP _{eff}	F (%)	ΔF	Comment	
Talc cell (CRPG laboratory)										
Friction as a function of temperature										
1/2	900		3.72	0.10	3.00	0.05	24.1	3.2		
	1000 ^a		3.75	0.10	3.07	0.06	22.0	3.2		
	1100 ^a	24	3.77	0.10	3.14	0.06	20.1	3.2		
	1200 ^a		3.79	0.10	3.21	0.06	18.3	3.2		
	1300		3.82	0.10	3.28	0.07	16.6	3.2		
Friction as a function of duration										
1/2	900	2.7	4.01	0.10	3.00	0.05	33.8	3.4		
		4.5	3.92	0.10	3.00	0.05	30.7	3.3		
		6	3.82	0.10	3.00	0.05	27.3	3.3		
		15	3.82	0.10	3.00	0.05	27.3	3.3		
		24	3.72	0.10	3.00	0.05	24.1	3.2		
		48	3.63	0.10	3.00	0.05	20.9	3.2		
		6	3.82	0.10	3.28	0.07	16.6	3.2		
1300	24	3.82	0.10	3.28	0.07	16.6	3.2			
Friction as a function of pressure and assembly size										
1/2	1300	24	1.50	0.10	1.30	0.03	15.1	4.3		
3/4			1.51	0.06	1.30	0.03	15.9	4.4		
Friction as a function of assembly parts										
1/2	900	6	3.82	0.10	3.00	0.05	27.3	3.3	Standard calibration (900 °C, 6 h)	
			> 3.97		3.00	0.05	> 32.2		Alumina sleeve	
			3.82	0.10	3.00	0.05	27.3	3.3	Pb foil	
				3.72	0.10	3.00	0.05	24.1	3.2	MgO spacers density
	1300	24	3.82	0.10	3.28	0.07	16.6	3.2	Standard calibration (1300, 24 h)	
			3.82	0.10	3.28	0.07	16.6	3.2	Assembly batch #2	
NaCl cell (LMV laboratory)										
1/2	900	9	3.25	0.10	3.00	0.05	8.4	3.1		
1/2		24	3.16	0.10	3.00	0.05	5.4	3.0		
1/2	1300	24	3.38	0.10	3.28	0.07	4.8	3.0		
BaCO₃ cell (ULG laboratory)										
1/2	800	24	2.38	0.10	2.16	0.05	10.2	4.3		
1/2	1000		2.85	0.10	2.69	0.05	6.1	3.4		

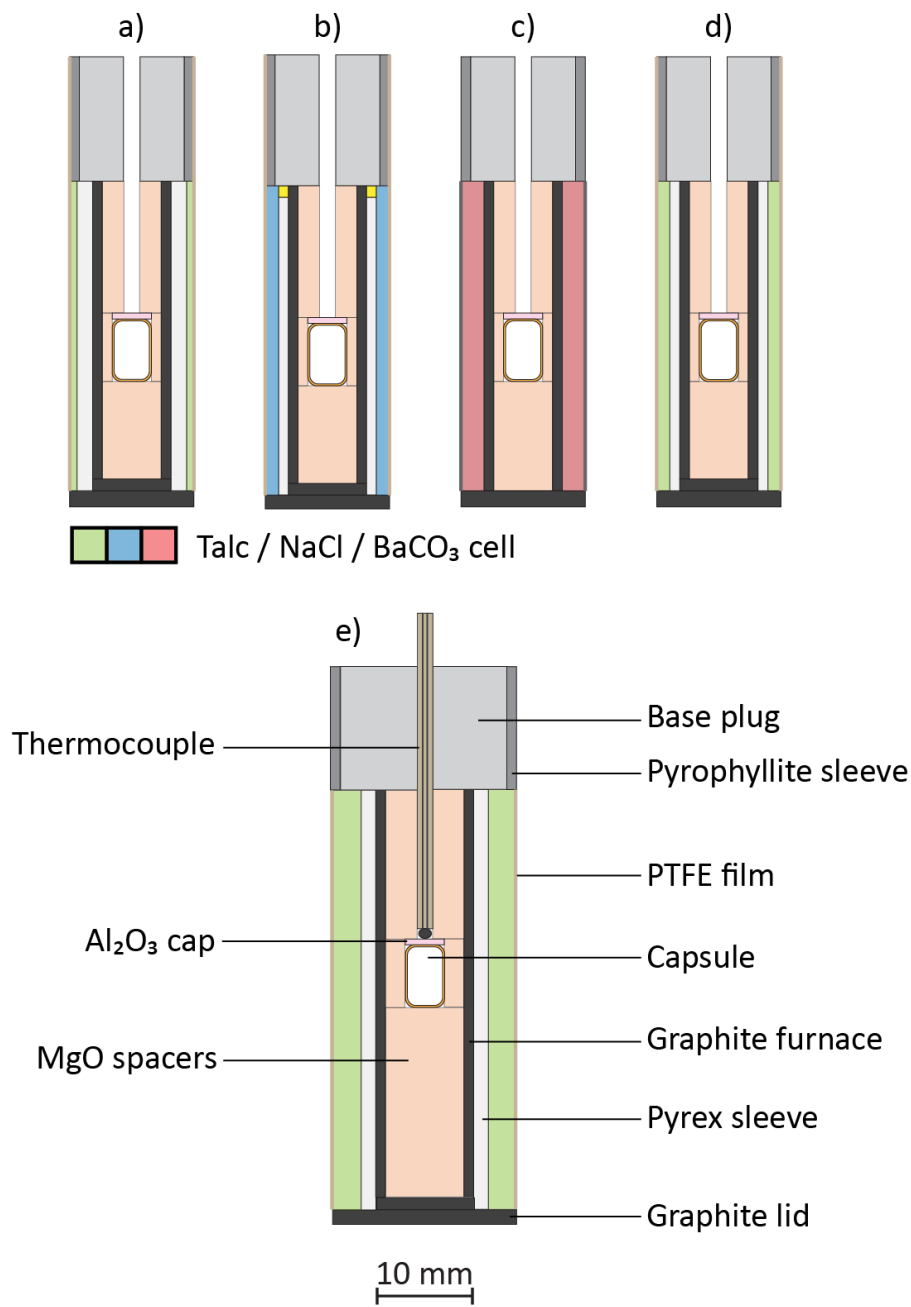
P_{app} and P_{eff} represent the pressure (GPa) applied by the press and the pressure on the sample, respectively. F represents the friction in %. ΔP_{app}, ΔP_{eff} and ΔF are 2σ standard deviations of applied pressure, sample pressure and friction, respectively.

^aValues from the linear fit in Figure 2.

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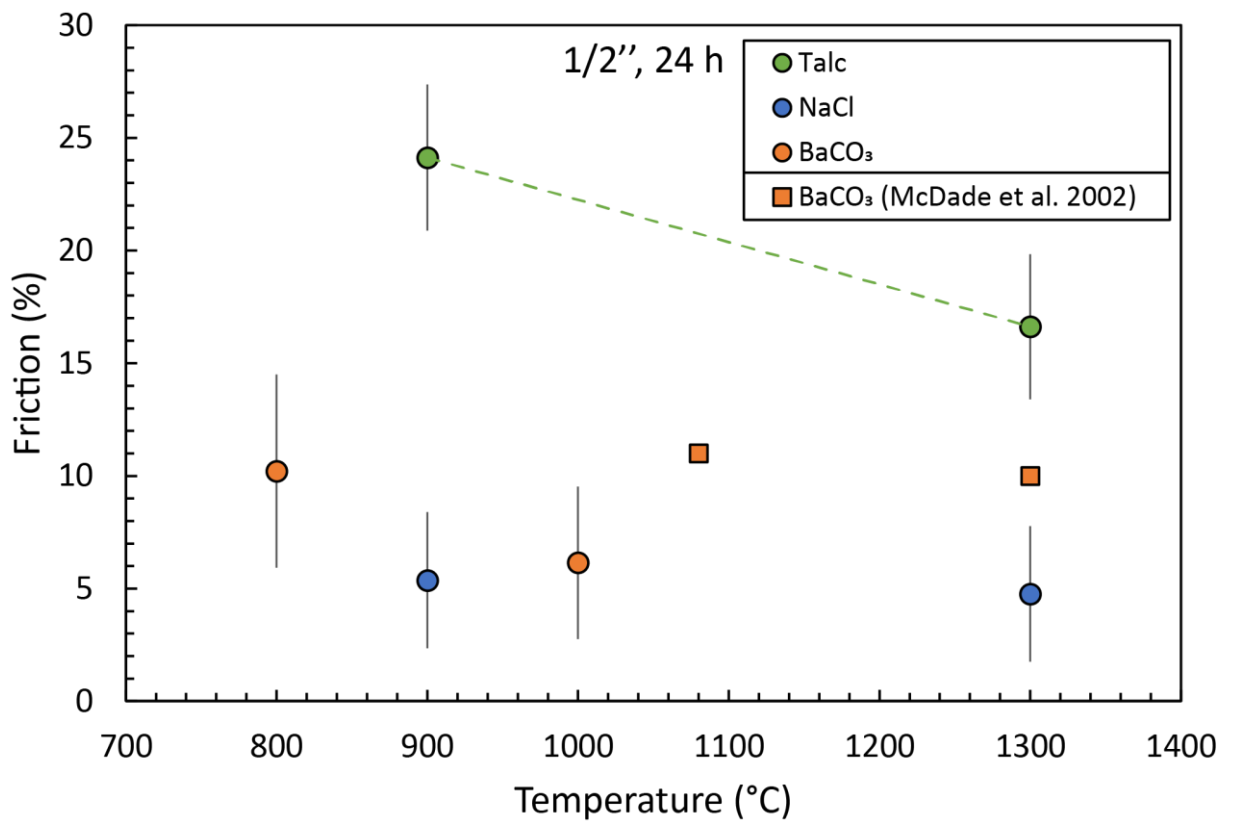
472 **Figure 1**



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474

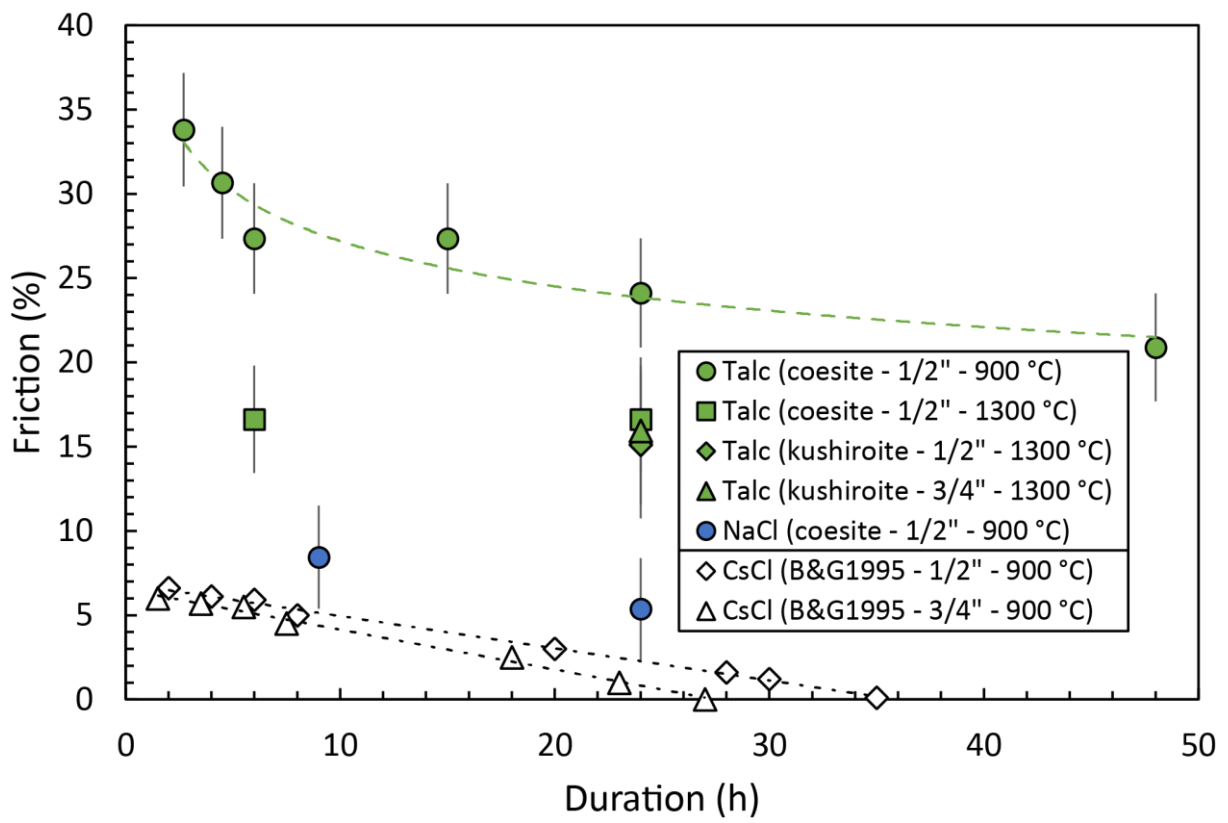
475 **Figure 2**



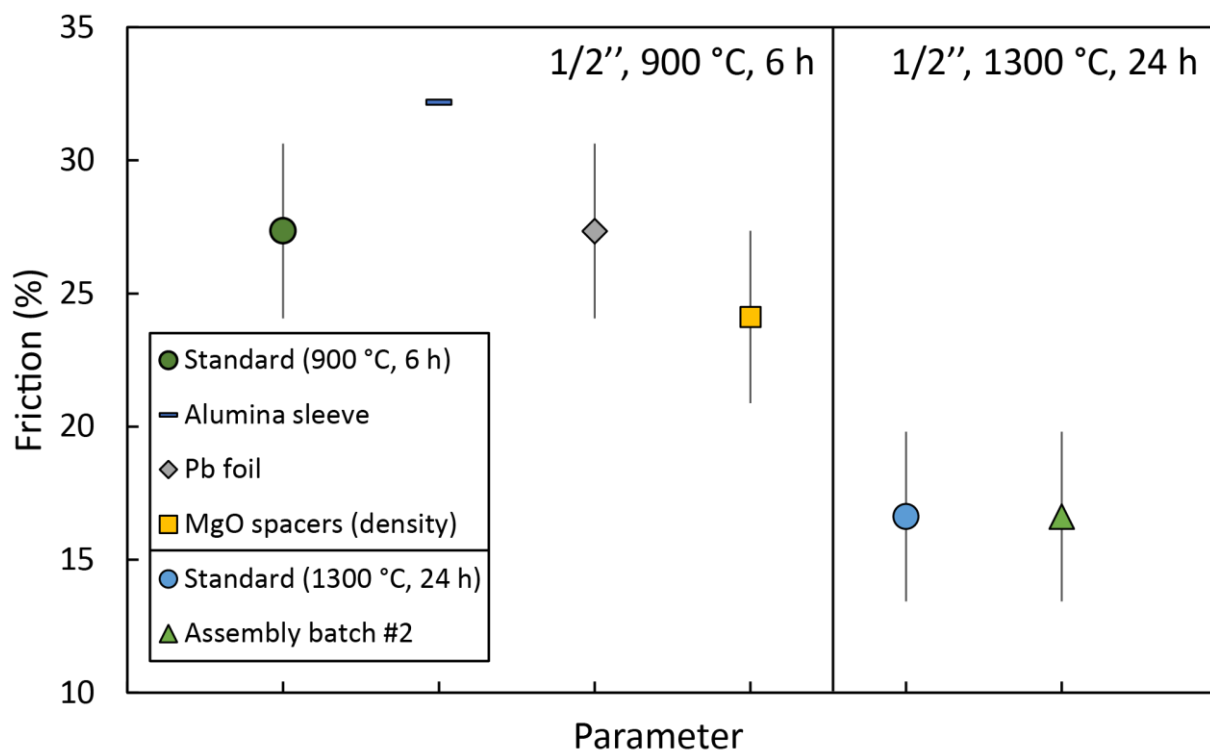
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478 **Figure 3**



481 **Figure 4**

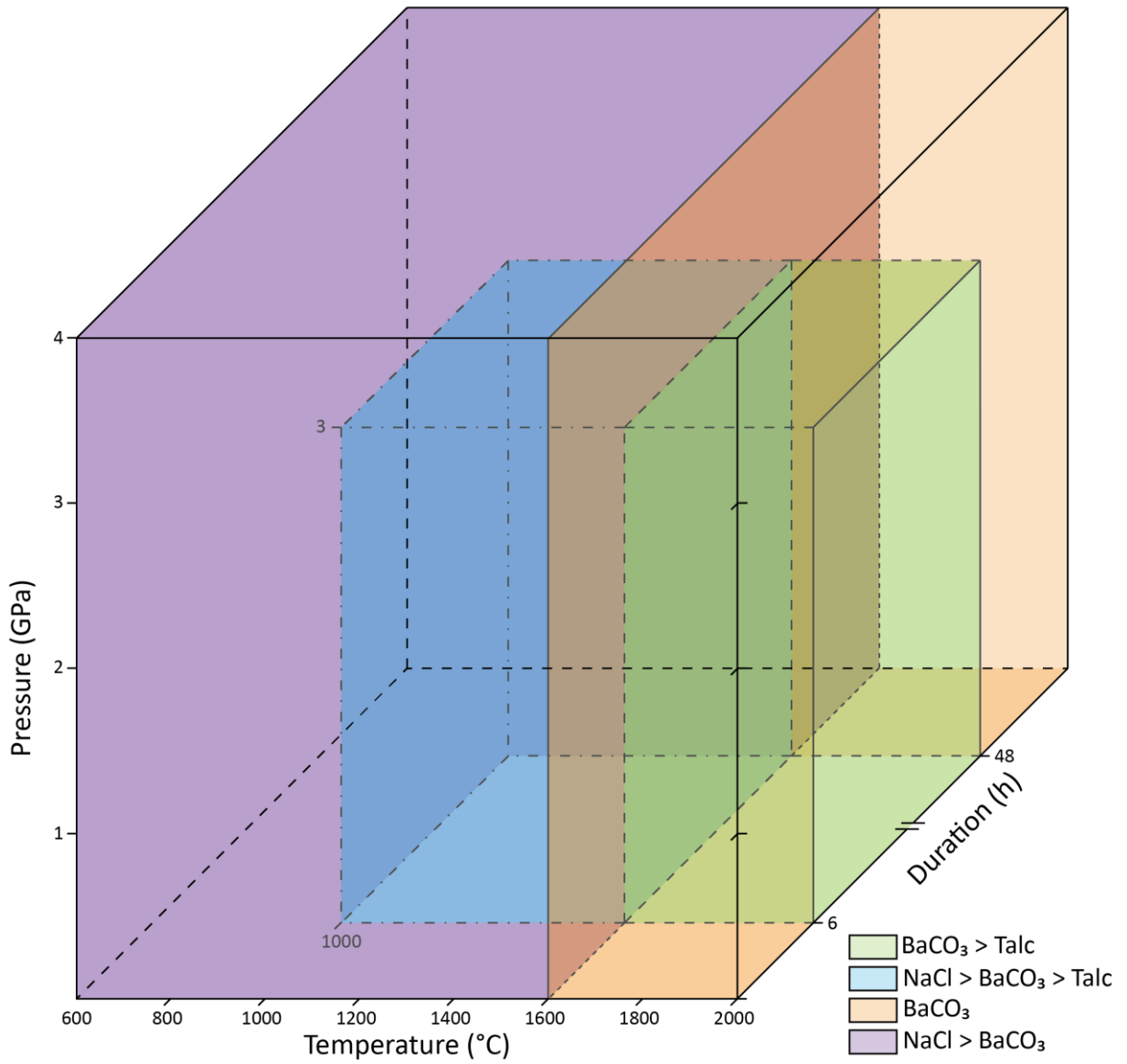


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483

484 **Figure 5**

Fig. 5



485

486

487

Supplementary Material

488 **Influence of intensive parameters and assemblies on friction evolution during piston-cylinder**
489 **experiments**

490 P. Condamine, S. Tournier, B. Charlier, E. Médard, A. Triantafyllou, C. Dalou, L. Tissandier, D.
491 Lequin, C. Cartier, E. Fűri, P. Burnard, S. Demouchy, Y. Marrocchi

492

493

List of Supplementary Figures

494 **Supplementary Material 1:** Assembly sizes.

495 **Supplementary Material 2:** Pictures of typical run products from the three reactions investigated. a)
496 Reflected light microscope image of the reaction quartz (Qz) - coesite (Co) in V18 (3.87 GPa, 1300
497 °C, 24 h). b) Back-scattered electron image of the reaction anorthite (An) + gehlenite (Gh) +
498 corundum (Crn) = kushiroite (Ku) in V113 (1.54 GPa, 1300 °C, 24 h). c) Back-scattered electron
499 image of the reaction jadeite (Jd) + quartz (Qz) = albite (Ab) in A070 (2.35 GPa, 800 °C, 24 h).

500 **Supplementary Material 1**

		CRPG (a)	CRPG (b)	LMV (c)	ULG (d)	CRPG (e)
Piston cylinder diameter	inch	1/2"	1/2"	1/2"	1/2"	3/4"
	mm	12.7	12.7	12.7	12.7	19.1
Pressure vessel height		52.00	52.00	52.00	52.00	63.00
Cell	type	Talc	Talc	NaCl	BaCO ₃	Talc
	h.	31.70	31.70	31.70	31.70	43.00
	o.d.	12.70	12.70	12.70	12.70	18.80
	i.d.	11.20	10.00	10.00	8.00	13.00
Glass	type	Borosilicate	Borosilicate	Borosilicate	/	Borosilicate
	h.	31.70	31.70	30.50	/	43.00
	o.d.	11.20	10.00	10.00	/	13.00
	i.d.	8.00	8.00	8.00	/	10.00
Furnace	h.	30.50	30.50	30.50	31.80	41.70
	o.d.	8.00	8.00	8.00	8.00	10.00
	i.d.	6.00	6.00	6.00	6.00	8.00
Furnace lid	h.	1.20	1.20	1.20	/	1.30
	d.	8.00	8.00	8.00	/	10.00
Graphite lid	h.	1.65	1.65	1.40	1.65	1.50
	d.	12.70	12.70	12.70	12.70	18.90
Base plug	h.	12.75	13.75	13.40	12.75	12.60
	o.d.	10.70	10.70	10.70	10.70	17.00
Pyrophyllite sleeve	h.	12.75	13.75	13.40	12.75	12.60
	i.d.	10.70	10.70	10.70	10.70	17.00
	o.d.	12.70	12.70	12.70	12.70	18.90
Total	h.	46.10	47.10	46.50	46.10	57.10
Density of MgO spacers (kg/m ³)		2000	2000	2300	2800	2000

All dimensions in mm

h.: height

d. : diameter

o.d.: outer diameter

i.d.: inner diameter

Letters in parenthesis correspond to the labels on Figure 1.

