Video Article

High-pressure, High-temperature Deformation Experiment Using the New Generation Griggs-type Apparatus

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Abstract

In order to address geological processes at great depths, rock deformation should ideally be tested at high pressure (> 0.5 GPa) and high temperature (> 300 °C). However, because of the low stress resolution of current solid-pressure-medium apparatuses, high-resolution measurements are today restricted to low-pressure deformation experiments in the gas-pressure-medium apparatus. A new generation of solid-medium piston-cylinder ("Griggs-type") apparatus is here described. Able to perform high-pressure deformation experiments up to 5 GPa and designed to adapt an internal load cell, such a new apparatus offers the potential to establish a technological basis for high-pressure rheology. This paper provides video-based detailed documentation of the procedure (using the "conventional" solid-salt assembly) to perform high-pressure, high-temperature experiments with the newly designed Griggs-type apparatus. A representative result of a Carrara marble sample deformed at 700 °C, 1.5 GPa and 10^{-5} s^{-1} with the new press is also given. The related stress-time curve illustrates all steps of a Griggs-type experiment, from increasing pressure and temperature to sample quenching when deformation is stopped. Together with future developments, the critical steps and limitations of the Griggs apparatus are then discussed.

Video Link

The video component of this article can be found at https://www.jove.com/video/56841/

Introduction

Rock deformation is one of the most important geological processes. It strongly contributes to human-time-scale phenomena, like earthquakes or landslides, but also to the large-scale mass movements of the solid outer shell in telluric planets, including plate tectonics on Earth[1]. For instance, depending on the rheology of the shell-like lithosphere, which defines the strength of both the crust and sub-solidus mantle ($\lesssim$ 1200 °C), the scheme of plate tectonics and related features may vary significantly[2,3,4,5]. On the one hand, the presence of a strong uppermost mantle and/or lower crust is required to sustain mountain belts or stabilize subduction zones[6]. But on the other hand, numerical models have also shown that plate boundaries cannot develop from mantle convection if the lithosphere is too strong, giving rise to a rigid lid behavior as observed on Venus[7]. Thus, the strength of the lithosphere as dictated by rock rheology has a direct control on the plate-like behavior of active planets.

For more than half a century, the rock rheology has been investigated at high temperatures (> 300 °C), giving rise to state-of-the-art techniques that mainly differ in the pressure range they can achieve. This includes the gas-medium Paterson-type apparatus[8] at relatively low pressures (<0.5 GPa), the solid-medium Griggs-type apparatus[9,10,11] at intermediate to high pressures (0.5-5 GPa), and the deformation-Dia apparatus[12,13] (DDia: up to ~20 GPa) or diamond anvil cell at very high pressures[14] (up to more than 100 GPa). Thus, the pressures and temperatures encountered in the deep Earth can nowadays be achieved experimentally. However, rock deformation also relies on differential stress that needs to be measured with high accuracy and precision, so that constitutive relationships can be formulated. Thanks to its gas-confining medium, the Paterson apparatus is today the only technique able to perform stress measurements with an adequate accuracy (± 1 MPa) to extrapolate the data over 6 orders of magnitude in strain rate, but it can only explore deformation processes at low pressures. Conversely, solid-medium apparatuses can deform rocks at high pressures, but with a lower accuracy of the stress measurements. While stress accuracy has been estimated at ± 30 MPa for the Griggs-type apparatus[15,16], the synchrotron-based DDia produces mechanical laws with an error of more than ± 100 MPa[17]. In the Griggs-type apparatus, stress could also be overestimated by up to 36% with respect to stress measurements in the Paterson one[15]. Performing accurate and precise stress measurements at high pressures - and high temperatures - therefore remains a major challenge in Earth Sciences.

Excluding deep subduction slabs where pressures may exceed 5 GPa, the Griggs-type apparatus is presently the more appropriate technique to study deformation processes over the pressure (< 4 GPa) and temperature ($\lesssim$ 1200 °C) ranges in a large part of the lithosphere. On this basis, significant endeavors have been undertaken in the 1990's to improve stress measurements, particularly to reduce friction effects by using eutectic salt mixtures as a confining medium around the sample[11,18]. Such a molten salt assembly gave rise to a better accuracy of the stress

measurement, reducing the error from ± 30 to ± 10 MPa[15,19], but additional disadvantages have been encountered when applying this type of assembly. These have a much lower success rate, great difficulties to perform non-coaxial (shear) experiments, and a more complicated sample assembly. Moreover, the accuracy of stress measurements remains ten times lower than that of the low-pressure Paterson-type apparatus. These issues limit the quantification of rheological processes using the Griggs-type apparatus, which today is more commonly applied to explore the deformation processes and their related microstructures. A new approach will be therefore required to perform rheological quantification at high lithospheric pressures.

This paper gives detailed documentation of the "conventional" procedure to perform high-pressure deformation experiments using a newly designed solid-medium Griggs-type apparatus. In the framework of new "Griggs" laboratories implemented at the *ISTO* (Orléans, France) and *ENS* (Paris, France), the main purpose is to properly illustrate each step of the protocol in details, so that scientists from all fields can decide whether the apparatus is appropriate or not to their aims of study. The critical steps and limitations of this state-of-the-art technique are also discussed, together with new approaches and possible future developments.

The new Griggs-type apparatus

Based on the piston-cylinder technology, the Griggs-type apparatus has been formerly designed by David T. Griggs in the 1960's[9], and then modified by Harry W. Green in the 1980's[11] (mainly to achieve higher pressures during deformation experiments). In both cases, the Griggs apparatus is characterized by a metal frame that includes: 1) three horizontal platens mounted on vertical columns, 2) a main hydraulic cylinder (confining pressure ram) suspended to the middle platen and 3) a deformation gear box and piston/actuator fixed on top of the upper platen (**Figure 1**). The "confining" ram and deformation actuator are each connected to independent pistons that transmit forces to the sample assembly within a pressure vessel. With such a vessel, deformation can be achieved at confining pressures of up to 2 or 5 GPa, depending on the apparatus and diameter of the sample assembly.

Thanks to a resistance furnace, the sample temperature is increased by Joule effect (up to ≈1300 °C[20]), while the pressure vessel is water cooled on top and bottom. In Green's design, the Griggs apparatus also includes an end-load system that homogenizes the pre-stress in the pressure vessel (**Figure 1**). This permits to achieve deformation experiments at higher pressures (max. 5 GPa), particularly using a small bore in the pressure vessel. For further details about the Griggs press, the readers are referred to the excellent description of the modified Griggs apparatus design by Rybacky *et al.*[19].

Arising from a close collaboration between the *Institut des Sciences de la Terre d'Orléans* (ISTO, France) and *École Normale Supérieure de Paris* (ENS Paris, France), the new generation Griggs-type apparatus is directly based on the design from H. W. Green[11], but some improvements have been made to comply with European standards for safety of high-pressure experiments. In this new press, the confining and deformation actuators are driven by servo-controlled hydraulic syringe pumps, giving the possibility to perform either constant load or constant displacement experiments at high pressures (up to 5 GPa). The confining (isostatic) pressure, force, and displacement are respectively monitored using oil pressure sensors, a load cell (max. 200 kN) and displacement transducers (**Figure 1**). The pressure vessel is made of an inner tungsten-carbide (WC) core inserted into a 1° conical steel ring and pre-stressed using the strip winding technique[21]. For transmitting forces, the pressure vessel and sample assembly lie between WC-removable pistons that include a deformation piston (σ_1), confining piston (σ_3), end-load piston and base plate (**Figure 1**). Together with regular cooling on top and bottom of the pressure vessel, water flows through the steel vessel around the tungsten-carbide core within 6 mm diameter holes for better cooling (**Figure 1**). The hydraulic cylinder for the confining pressure is also cooled by silicon oil flow. In addition, the deformation apparatus in Orléans employs larger sample size up to 8 mm diameter, so that 1) microstructures can be better developed, and 2) the Griggs press and Paterson press share a common sample dimension for future comparisons. This requires an increased diameter of the WC bore in the pressure vessel (27 mm, instead of 1 inch, i.e., 25.4 mm), reducing the maximum attainable pressure to 3 GPa.

The present paper describes the procedure to perform an experiment with the new Griggs-type apparatus, which includes the description of all pieces that compose the conventional solid-salt sample assembly using alumina pistons (**Figure 2A** and **2B**), as well as the successive steps to produce them and introduce them into the pressure vessel. This description follows in large parts the routine developed over many years by Prof. Jan Tullis and co-workers at Brown University (R.I., USA). The resulting sample assembly is fully appropriate to perform either co-axial (pure shear) or non-coaxial (general shear) deformation experiments over the whole range of pressures and temperatures of the Griggs-type apparatus. While a pure shear experiment typically requires a cored drill sample of a certain length (commonly ≈2 times the sample diameter), a general shear deformation is commonly applied to a zone cut at 45° to the piston axis (**Figure 2B**). The sample material can either be a slice of a core sample or fine-grained powder of a chosen grain size. All pieces are wrapped into a metal foil and jacketed within a platinum tube welded (or folded flat) at both sides. The temperature is commonly monitored using either S-type ($Pt_{90\%}Rd_{10\%}$ alloy) or K-type (Ni alloy) thermocouple, but only the preparation of an S-type thermocouple using a mullite 2-hole sheathing tube is here described (**Figure 2C**).

Protocol

1. Prepare the sample assembly

1. Grind at least 60 g of NaCl powder (99.9% purity) in a ceramic mortar.
   NOTE: The NaCl powder should look like caster sugar for baking. While preparing other pieces of the assembly, store the salt powder in an oven at 110 °C to prevent salt from pumping humidity.
2. Cold press salt pieces (lower and upper outer, and inner salt pieces; **Figure 2B**) using the specific tools adapted to the size of the sample assembly (**Figure 3**).
   1. To produce the lower outer salt piece, coat the pressing tools with soap (with fingers). This includes all surfaces of the piston components (tools number #2, #5 and #6 of **Figure 3A**) and borehole surface of the vessel components (tool components #3 and #4 of **Figure 3A**) .
   2. Put 17.5 g of ground NaCl powder into a beaker. Add ≈0.1 mL of distilled water and make sure that salt and water are well mixed.
   3. Assemble the pressing tool components #3, #4, #5 and #6 and put them below the piston of a 40-ton hydraulic press.

4. Fill the wet salt powder into the borehole of the components #3 and #4, and put the piston components #1 and #2 of **Figure 3A** on top of the salt powder.
5. Press the powder at 14 tons for 30 s, and then unload the salt piece.
6. Take out the lower vessel component #4 of **Figure 3A**, put the component #3 on two metal pieces leaving an empty space below the bore hole, replace component #1 by component #8 and use the hydraulic press again to extract the salt piece from below (**Figure 3A**).
7. To produce the upper salt piece, repeat the steps from §1.2.1 to §1.2.6 but using component #7 instead of component #5 of **Figure 3A** and filling 16.5 g of ground NaCl powder into the borehole of the vessel (components #3 and #4).
8. Use medium-grit (400) sandpaper to adjust the length of the lower and upper salt pieces to the graphite furnace (*i.e.*, graphite tube protected by two fired pyrophyllite sleeves). While the lower salt piece should be ≈24 mm long, the upper one should be ≈22.5 mm long (or ≈19 mm and ≈18 mm for a regular 1-inch borehole pressure vessel, respectively).
9. To produce the inner salt pieces around alumina pistons, repeat the steps from §1.2.1 to §1.2.4 but using tool components from #1 to #4 of **Figure 3B** and pressing 8 g of NaCl powder plus ≈0.05 mL of distilled water at 6 tons for 30 s. Using the piston component #7 of **Figure 3B**, repeat the steps of §1.2.6 to extract the inner salt piece from below. The whole piece should be ≈40 mm long, but it will be cut and adjusted to the graphite furnace later in the protocol.
10. Repeat the steps of §1.2.9 to produce the inner salt piece around the jacketed sample, but using tool components #5 (instead of #2) and #6 (instead of #4) of **Figure 3B**.

3. Make S-type thermocouple by cutting two metallic wires (Ø 0.3 mm) of around 350 mm long, one made of pure platinum ($Pt_{100\%}$) and a second one made of platinum/rhodium ($Pt_{90\%}Rh_{10\%}$)
   1. Use a PUK 5 welding microscope (or equivalent) at a power of 15% and a welding time of 7 ms to weld one tip of each wire together. Flatten the weld-bead using a flat micro-plier and remove around ¾ of the upper part of the welded tip using a diagonal micro-cutter.
   2. Use a low-speed diamond saw with water bath to cut two sections of mullite sheathing (1.6-mm-diameter mullite round double bore tubing), one of around 10 mm long and a second one of around 80 mm long.
   3. With the low-speed saw, cut one tip of each mullite piece at 45° of the long axis, making sure that the inner holes are aligned with the short axis of the resulting elliptical section (**Figure 2C**). Adjust the dimensions of the mullite sections to 6.8 mm for the short one and 76 mm (or 56 mm for a regular 1-inch borehole pressure vessel) for the long section of the thermocouple (**Figure 2C**).
   4. Cut a small groove of the thickness of the diamond saw blade and of around 1 mm deep on the flat tip of the short mullite tube. The groove should be parallel to the alignment of the inner holes.
   5. Thread carefully each wire of the thermocouple in their respective hole of the mullite. To adjust the two mullite sections at 90° from each other, bend the wires of a few degrees, thread them into the long section, bend the wires a bit more, thread them again, and so on until the two 45° surfaces face each other as close as possible.
   6. Use ceramic glue to fill the tip of the short section and to solidly fix the two sections at the 90° elbow of the thermocouple sheathing.

4. Use a milling machine, a stainless steel drill bit of 1.8 mm Ø and the tool shown in **Figure 4** to drill a hole of 2 mm diameter all through the length of the lower salt piece.
5. At the top of the lower salt piece, use a scalpel with triangular blade and sharp point to carve a small channel (around 1 mm deep and 2 mm large) from the thermocouple hole to the bore.
   NOTE: Make sure that the short section of the thermocouple fully fit in there as close as possible to the top surface of the salt piece (see **Figure 2B**).
6. Make shear alumina forcing blocks (only for a general shear experiment) by using the low-speed diamond saw to cut an 8-mm-diameter alumina piston of around 13 mm long.
   1. Use a lathe with a diamond tool (or equivalent) to make the tip surfaces parallel to each other (to ≈ ± 0.002 mm) and to reduce the length of the alumina piston at 12 ± 0.1 mm.
   2. 1.6.2. Use the low-speed diamond saw with water bath to cut the piston into two parts at 45° of the piston axis. To prevent from any sliding between the sample and alumina piston, grind(gently) the 45°-surface of each piston using medium-grit (800) sandpaper.

7. Calculate the dimensions of the top and bottom alumina pistons based on the size of the jacketed sample and dimensions of the sample assembly.
   NOTE: For a coaxial experiment, the jacketed sample size only includes the length of the core and twice the thicknesses of platinum (or gold) jacket (0.15 mm thick). For a general shear experiment, the sample is replaced by the two shear forcing blocks and the sample slice, which is commonly ≈1 mm thick (*i.e.*, around 1.4 mm measured along the piston axis). Here, the jacketed sample is ≈13.5 mm, so the top piston is ≈19.5 mm and the bottom one is ≈16.6 mm long.
8. Use the low-speed saw to cut two alumina pistons of ≈20 and ≈17 mm long, and repeat the steps of §1.6.1 to adjust their length at the right dimensions (here 19.5 and 16.6 mm) and to parallelize them (to ≈ ± 0.002 mm).
9. To jacket the sample, use a round-shaped hollow punch (Ø 10 mm) to extract two discs of 10 mm diameter (for an 8-mm diameter sample) from a platinum foil of 0.15 mm thick. Make two platinum cups (**Figure 5A**) by bending a 1 mm rim of each disc into a cup-shape using the tool components #1, #2 and #3 of **Figure 5A**.
   1. Use a tubing cutter to cut a platinum tube of the length of the "full" sample (*i.e.*, the core sample only for a pure shear experiment or the sample + shear forcing blocks for a general shear experiment) plus ≈3 mm (1-1.5 mm sticking out from each end of the "full" sample). Use a Benchtop Muffle furnace to anneal the Pt tube for at least 30 min at 900 °C.
   2. Fit one cup into the platinum tube, use a file tool to grind the end of the tube and cup flat, and weld the platinum cup and tube together using the tool shown in **Figure 5B** and the PUK 5 welding microscope (power: 18%; welding time: 10 s).
   3. Wrap (by hand) the "full" sample into a nickel foil of 0.025 mm thick and fit them into the platinum tube. Close the tube with the second platinum cup and grind them (with the file tool). Weld the cup and tube together using the tool components of **Figure 5B**.
      NOTE: For a 45° sample, do not forget to put a mark (using a permanent pencil) on the platinum jacket to remember the position of the sample after welding, so that the thermocouple will be well seated on the sample side (along strike).
   4. Slightly bend the tips of the platinum tube using a pair of flat needle nose micro-pliers, so that each alumina piston (top and bottom ones) can fit as far as possible into the platinum tube. Using the same pair of flat pliers, press the tube onto the alumina pistons all around to maintain a small total diameter.

10. Using the low-speed diamond saw (without water bath), cut two tubes of inner salt pieces for the pistons (8 mm inner diameter) and one tube for the jacket (8.8 mm inner diameter). Adjust their length using medium-grit (800) sandpaper.
    NOTE: While the inner salt piece around the sample should fully cover the platinum jacket, the lower and upper inner salt pieces respectively cover the bottom and top alumina pistons all over the length of the graphite furnace. For instance, with a "full" sample of 10 mm length, the lower and upper inner salt pieces are respectively of ≈14.40 mm and ≈15.20 mm long.
11. Put together by hand and in the following order: the lower outer salt piece, bottom copper disc and graphite furnace (**Figure 2B**). Use a pencil to mark a dot at the expected position of the thermocouple on the outer pyrophyllite sleeve of the furnace.
    1. Take the outer salt piece out and insert by hand the inner salt pieces (around pistons and jacket) within the graphite furnace.
    2. While maintaining by hand the graphite furnace, inner salt pieces and bottom copper disc together, use the milling machine to drill a hole of ≈2 mm diameter (stainless steel drill bit of 1.8 mm Ø) where the position of the thermocouple has been estimated (dot mark). The drill should go through half of the furnace and inner salt piece sections (without the sample inserted) .

12. Prepare the lead piece by putting 50 g of lead into a ceramic recipient, and leave the recipient into a Benchtop Muffle furnace at 400 °C for around 30 min.
    CAUTION: Use nitrile gloves to manipulate the lead.
    1. When the lead has entirely melted, pour it quickly onto the tool component #2 while seated onto components #3 and #4 of **Figure 6**.
    2. Right after the step of §1.12.1, use the 40-ton hydraulic press to press the lead at 4 tons for 30 s using tool component #1 of **Figure 6**.
    3. Take out the lead piece by repeating the steps of §1.2.6, but using the tool components of **Figure 6B**.
    4. Use the low-speed diamond saw (without water bath) to produce the NaCl insert (**Figure 2B**) by cutting a section of 2 mm thick of an inner salt piece (around-piston inner diameter). Fit the NaCl insert into the lead piece, and using any type of scalpel, push some lead between the NaCl insert and lead piece to maintain them together. Use medium-grit (400) sandpaper to adjust the NaCl insert to the lead piece.

2. Charge the sample assembly

1. Put together by hand all pieces that compose the sample assembly, except for the top copper disc, lead peace and packing rings. Wrap with Teflon (either tape or grease PTFE) the outer salt pieces, lead piece and base pyrophyllite piece (**Figure 2B**).
    1. Place the base plate at the base of an arbor press, mount the pressure vessel on the piston of the arbor press, and use a 27-mm-diameter steel cylinder to align the base plate with the pressure vessel.
    2. Leave the vessel suspended as high as possible above the base plate, and while carrying the sample assembly, carefully fit the thermocouple into the thermocouple hole of the base plate. Put the sample assembly at the center of the base plate.
    3. Once in place, add a foil of Mylar in-between the base plate and pressure vessel around the assembly.
       NOTE: Make sure that its surface fully covers the top surface of the basal piston around the sample assembly.
    4. Use the arbor press to carefully lower the pressure vessel onto the base plate and fit the sample assembly into the borehole of the pressure vessel.
       NOTE: Make sure that the mullite sheath does not break at this step. If it does break, the steps from §1.3 to §1.3.6 should be repeated.
    5. Use adapted clamps (see **Figure 7**) to fix the pressure vessel and base plate together tightly, and add the top copper disc, lead piece and σ_3 packing ring (using the σ_3 WC piston) on top of the sample assembly.

2. Carry (by hand or using a cart) the pressure vessel upside down and put it on a workbench.
    1. Slide plastic tubes (1.5 mm outer Ø; 1 mm inner Ø) over each wire of the thermocouple to insulate them from any metal piece, and fix each wire to an S-type universal flat pin thermocouple connector.
    2. Bend and fit the wires into the basal groove of the base plate, and put a piece of a regular paper sheet between the two wires in order to avoid any contact between each other, particularly at the tip of the thermocouple sheath.

3. Turn the pressure vessel into an upright position and place the end-load piston, σ_3 WC piston, and σ_1 WC piston (including σ_1 packing ring) on top of the sample assembly.
4. Place the base plate, pressure vessel, and pistons onto the bottom platen of the Griggs apparatus, and plug the thermocouple connector to the temperature regulation system.

3. Perform the deformation experiment

1. Launch the software *Falcon* (or equivalent) to monitor the hydraulic pumps (a scheme of the display is shown in **Figure 8**)
2. Lower the deformation piston by opening the electro-valves EV2 and EV6 (mouse left-click on the screen display) and valve V4 (manually on the control panel). Close the other valves (to close an electro-valve, right-click on the screen display).
    1. On the software, left-click on "run" by the deformation pump and choose the option "Constant Flow Rate". Set the flow rate to 150 mL/min, left-click on "Inject", and then click on "Start".
    2. When the deformation piston is around 3 to 4 mm above the σ_1 piston, left-click on "stop" to stop the pump and move by hand the pressure vessel to align the σ_1 piston with the deformation actuator of the Griggs-type apparatus.
    3. Launch the software *CatmanEasy-AP,* Left-click on "open a project" and choose the project "Griggs_exp".
    4. Left-click on "start" in the top left corner, and select the panel "Force, Differential stress/Temperature" to have a look at the "Force" graph.
    5. Repeat the step of §3.2.1 to start the deformation pump again, but at a flow rate of 20 mL/min. When the deformation actuator is touching the σ_1 piston - the force should be sharply increasing - left-click on "stop" on *Falcon*.

3. Lower the confining and end-load actuators by closing EV6 and V4, and then opening EV3, V5 and V6.
    1. On *CatmanEasy*, left-click on the panel "Pressure/Stress/LVDT" to have a look at the "confining ram pressure" graph.

2. Repeat the step of §3.2.1 with the deformation pump at a flow rate of 150 mL/min. When the confining and end-load actuators are touching the σ_3 piston and end-load piston, respectively - the confining ram pressure should be sharply increasing -, left-click on "stop" to stop the deformation pump.
3. Stop *CatmanEasy* by left-clicking on "stop" in the top left corner.

4. Use the 8-mm-diameter plastic tubes with double-self-sealing coupler to plug the vessel and pistons to the cooling system.
NOTE: As shown on **Figure 8**, make sure that the cooling water flows from bottom to top around the pistons and through the vessel, and then through the flow meter.
    1. Open V7 and V8, switch on the cooling system of the pressure vessel (blue path in **Figure 8**) and check at the flow meter (the water flow should be around 3 L/min).
    2. Switch on the cooling system of the confining/end-load ram (Yellow path in **Figure 8**).

5. Refill the confining pump by closing EV2, EV3 and V4, and by opening EV4.
    1. Using confining air pressure, turn on the pressure relief valve on top of the oil tank (**Figure 8**) to increase the pressure at around 0.4 MPa.
    2. On *Falcon*, left-click on "run" for the confining pump, then select "Constant Flow Rate". Set the flow rate to 20 mL/min. left-click left on "Fill", and then on "Start".
    3. When the pump automatically stops, close EV4, open EV1 and repeat the step of §3.5.2 to refill the deformation pump at a flow rate of 150 mL/min.
    4. When the confining pump is full, open EV4 and turn off the pressure relief valve to release the air pressure in the oil tank.
    5. Close EV1 and EV4, and open EV2, EV5, EV6, V4.

6. On *CatmanEasy_AP*, select the panel "Measuring channels (*Voies de mesure*)", select the digital channels of the two displacement transducers (LVDT) and set them to zero (left-click on zero at the top window). Left-click on the panel "Measuring jobs (*Jobs de mesure*)", then on "Job parameters (*paramètres du job*)", and enter the experiment name in the "job name" box. Start again *CatmanEasy* (left-click on start).
7. On *Falcon*, start pumping by left-clicking on "run" for the confining pressure pump, and then by selecting "Constant Flow Rate". Set the flow rate to 1 mL/min, left-click on "Inject", and then on "start".
    1. When the confining pressure is around 10 MPa, stop the confining pressure pump and start the deformation pump by repeating the step of §3.2.1 at a flow rate of 3 mL/min. Stop the deformation pump when the force is sharply increasing on *CatmanEasy*.
    NOTE: While the σ_3 piston is advancing, the σ_1 piston will be first driven by the σ_3 piston at the very beginning, but it will stop at one point.
    2. Repeat the step of §3.7.1 every increment of 10 MPa of confining pressure until the pressure has reached 50 MPa, so that the σ_1 piston keeps in touch with the lead piece. When the confining pressure is around 50 MPa, stop the pump (left-click on "stop").
    3. Unscrew the top part of the clamps fixing the base plate to the pressure vessel (**Figure 7**) and slide a foil of Teflon between each clamp and the pressure vessel.

8. Start heating by switching on the furnace (green button on the temperature control panel), and use the arrows of the temperature controller to set the electrical output between 6 and 7%.
NOTE: The temperature should slowly increase.
    1. Play with the arrows of the temperature controller to set the temperature at around 30 °C, and then switch to the automatic ("auto") mode by pushing once on "man".
    2. Push once on the button "prog", select the desired heating program (preset using the software *Eurothermitools)*, and push once again on "prog" to start the program. The temperature should increase at a rate of around 0.3 °C/s.
    3. When the temperature reaches 200 °C, push twice on "prog" to hold the program.

9. Continue pumping by starting (and stopping) alternatingly both pumps (repeat the steps of §3.7 and §3.2.1) and using flow rates of 2 mL/min for the confining pump and 3 mL/min for the deformation pump.
NOTE: Both pistons should react each other because of the lead flux; while one piston is advancing, the second one is moving back.
CAUTION: Make sure that σ_1 remains between 2 and 3 mm behind σ_3, but not more than 3 mm to avoid striping the σ_1 packing ring. If the σ_1 packing ring strips from the σ_1 piston, a critical lead leak will occur and the experiment should be repeated from the beginning, including the preparation of the sample assembly.
    1. During pumping, when the confining pump is empty, close V4 and EV5, open EV4, and repeat the steps of §3.5.1 and §3.5.2 to refill the pump.
    2. When the pump is full, close EV4 and start the confining pump at a flow rate of 3 mL/min. Stop the pump when the pump pressure equals the pressure value of the confining ram as indicated on *CatmasEasy* ("confining ram pressure" graph).
    3. Release the pressure in the oil Tank and open EV5 and V4.

10. Continue pumping and heating alternatingly until the target pressure and temperature are reached. When the target temperature is achieved, push twice on "prog" to hold the heating program.
NOTE: During pumping and heating, the chosen values to define the pressure and temperature plateaus may change depending on the melting curve of NaCl and purpose of the experiment (*e.g.*, taking into account the pressure-temperature stability of phases in the sample). In any case, the plateaus are chosen so that NaCl does not melt (see Li and Li[22] for the melting curve of NaCl).
11. To start deforming, left-click on "run" of the confining pump, select "Constant Pressure", set the pump pressure to the pressure value indicated on the "confining ram pressure" graph (on *CatmanEasy*), and left-click on "start" to regulate at the target pressure.
    1. Repeat the step of §3.2.1 to start the deformation pump at a flow rate that corresponds to the desired displacement rate (for instance, a flow rate of 4.71 mL/min equal to a displacement rate of 10^{-2} mm/s).
    2. When the sample strain has reached the desired value, stop both the deformation and confining pumps and push twice on "prog" of the temperature controller to start quenching, *i.e.*, to quickly decrease the temperature to 200 °C at a rate of ≈300 °C/min.

3. While the temperature is decreasing, start both the confining pressure and deformation pumps by left-clicking on "run" and selecting "Constant Flow Rate" for the two pumps. Set the flow rate to 0.5 mL/min for the confining pump and 0.1 mL/min for the deformation pump, left-click on "Fill", and then on "Start" for each pump.
4. When the temperature has reached 200 °C, push twice on the button "prog" of the temperature controller to hold the heating program.
5. Use the "+" and "-" of the increment windows on *Falcon* to adjust the flow rate of both pumps, so that 1) the pressure decreases at a rate of ≈5 MPa/min and 2) the deformation ram pressure remains ≈50 MPa above the confining ram pressure.
6. During decompression, when the confining pump is full, stop the deformation pump, close EV5, open EV4, and repeat the step of §3.7 at a flow rate of 20 mL/min. Stop the pump when ≈5% of the oil volume are remaining in the pump.
    1. Close EV4 and repeat the step of §3.7 to start the pump at a flow rate of 3 mL/min. Stop the pump when the pump pressure equals the pressure value of the confining pressure ram as indicated on *CatmanEasy* ("confining pressure ram" graph).
    2. Open EV5, start both the confining and deformation pumps again to decrease the pressure ("Fill" option) using flow rates of 0.5 and 0.1 mL/min, respectively, and repeat the step of §3.11.5.
7. When the confining pressure has reached ≈100 MPa, push twice on the button "prog" of the temperature controller to decrease the temperature to 30 °C. Push twice again on "prog" to stop the program.
8. When the pressure is around 0.1 MPa in both pumps, stop the pumps and switch off the furnace (red button on the temperature control panel) and cooling systems.

4. Remove the sample

1. Re-attach the base plate to the pressure vessel using the adapted clamps (**Figure 7**).
2. Close EV5, EV6, V4, V5, V6, V7 and V8, open V1, V2 and V3, and unplug the thermocouple and tubes of the cooling system for the pressure vessel.
3. Use the hand pump to lift up the confining and end-load actuators as much as possible.
4. Repeat the step of §3.2.1 to start the deformation pump at a flow rate of 150 mL/min and lift the deformation actuator up a few millimeters more than the confining actuator.
   CAUTION: The deformation actuator should not retreat of more than 10 mm with respect to the confining actuator to avoid stripping the internal O-rings.
5. Take out (by hand or using a cart) the vessel and pistons (σ_1, σ_3, end-load and base plate) from the Griggs-type apparatus.
6. Remove the σ_1, σ_3 and end-load pistons and put the vessel upside down on the workbench. Unscrew the S-type thermocouple connector, remove the isolating plastic tubes, unscrew the clamps and take the base plate and Mylar foil away.
7. Turn the vessel upright, put a piece of lead on top of the σ_3 packing ring and use the 40-ton hydraulic press to press out the sample assembly from below.
8. Dismantle carefully the sample assembly using pliers and curve cutting edge scalpel.
   NOTE: While dismantling the sample assembly, check for the exact position of the thermocouple tip and any trace of possible jacket leak during the experiment. This may be important for the interpretation of the mechanical data (temperature offset, contamination, etc.). Only the lead piece (through melting), thermocouple wires and WC plug may be used again for the next experiment.

Representative Results

Figure 9 shows an example of a stress-time curve resulting from the new generation Griggs-type apparatus during pure shear (co-axial) deformation of Carrara marble (8-mm-long core sample) at a strain rate of 10^{-5} s^{-1}, a temperature of 700 °C and a confining pressure of 1.5 GPa. During such an experiment, both the pressure and temperature are first increased alternatingly, mainly to prevent the NaCl from melting. The molten NaCl is very corrosive for the sample and it may irreversibly damage the thermocouple. All along the successive steps of increasing pressure and temperature - here referred as the "pumping stage" (**Figure 9**) – the lead piece has the function of preventing the sample from becoming deformed by transferring stresses from σ_1 to σ_3 and conversely, maintaining a more or less isostatic stress state in the sample assembly.

When the target pressure and temperature are achieved, a period of "hot-pressing" may be applied. Although optional, this step – of commonly 24 h duration – may be required to sinter the sample powder before deformation, if applicable. The σ_1 piston/actuator is then advanced to deform the sample, giving rise to the so-called "deformation stage". This latter is first characterized by a steep-to-gentle increase of differential stress (σ_1 - σ_3), which is due to friction induced by 1) the packing rings and 2) the increasing surface of contact between the σ_1 piston and lead piece while σ_1 is moving through the lead. This "run-in" section should have a sufficient duration in order to determine the hit point (contact between the σ_1 piston and top alumina piston) accurately by curve fitting (**Figure 9**). For this purpose, a significant thickness of lead (≥ 2 mm) between the σ_1 piston and alumina piston is required before starting advancing σ_1. When approaching the top alumina piston, lead is extruded faster as a thin sample thickness, causing strain hardening in the lead and promoting a progressive stress increase until the σ_1 piston is pushing on the sample column. The stress curve then increases steeply up to yield stress conditions, which theoretically defines the transition from elastic to plastic behaviors (**Figure 9**). As required to define the differential stress, the hit point of the experiment is subsequently deduced from the intersection between the extensions of the "elastic" curve and "run-in" curve (**Figure 9**).

When deformation is finally stopped, the temperature is decreased very quickly (≈ 300 °C/min) to preserve microstructures. A substantial drop of pressure necessarily occurs during the "sample quenching", but following this drop, both the σ_1 and σ_3 pistons are moved back slowly by reducing the oil pressure in the hydraulic rams (≈ 5 MPa/min). This is required to limit the formation of unloading cracks, although some cracking is inevitable. After experiment, the stress-time curve is subsequently corrected to produce a stress-strain curve of the deformed sample up from the hit point (see inset in **Figure 9**). These corrections include 1) the stiffness/extension of the apparatus and 2) the friction induced by the packing rings and lead piece[15,19]. **Figure 10** also shows two examples of the post-experiment sample assembly, one containing the core sample of Carrara marble (**Figure 10A** and **10B**) and a second one of an olivine powder sintered, and then deformed in general shear at 900 °C and 1.2 GPa using the former Griggs-type apparatus[23] (**Figure 10C** and **10D**).

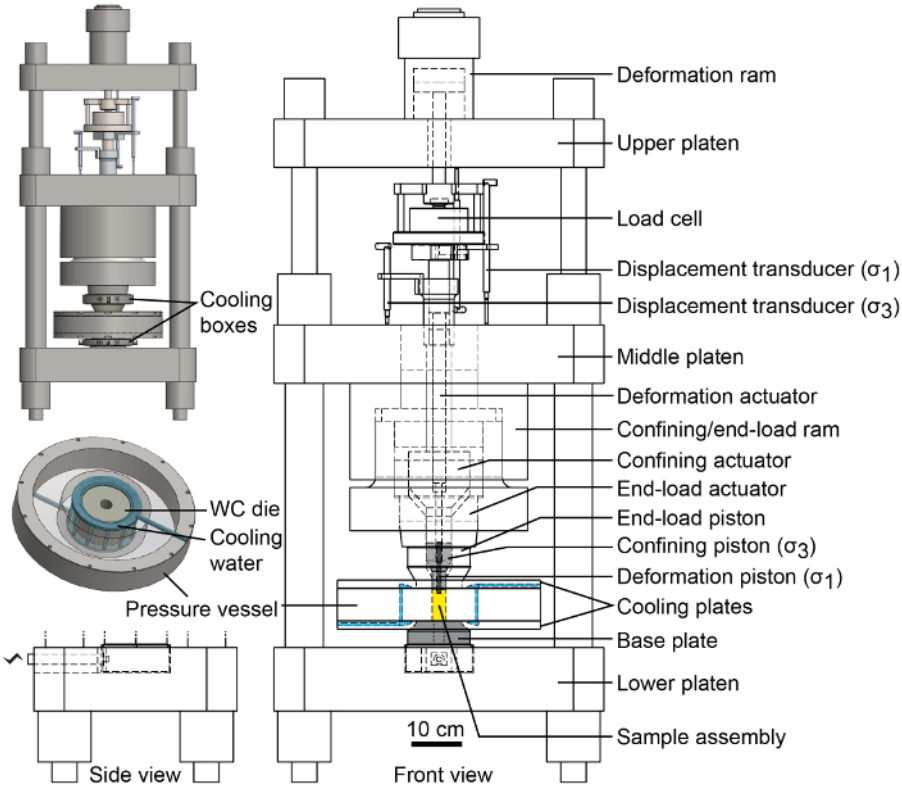

Figure 1: The new generation Griggs-type apparatus. Schematic drawings of the Griggs-type apparatus now available at the *Institut des Sciences de la Terre d'Orléans* (ISTO, France) and *École Normale Supérieure de Paris* (ENS Paris, France). While the sample assembly lies within the pressure vessel, high confining pressure and differential stress are applied by independent syringe pumps through hydraulic rams and pistons/actuators. The temperature is increased using a low-voltage/high-amperage electrical current injected from below the assembly (see side view) through a resistive graphite furnace. To preserve the tungsten carbide (WC) die, the pressure vessel is also cooled by water flow from bottom to top through the cooling plates/boxes and the vessel itself. Please click here to view a larger version of this figure.

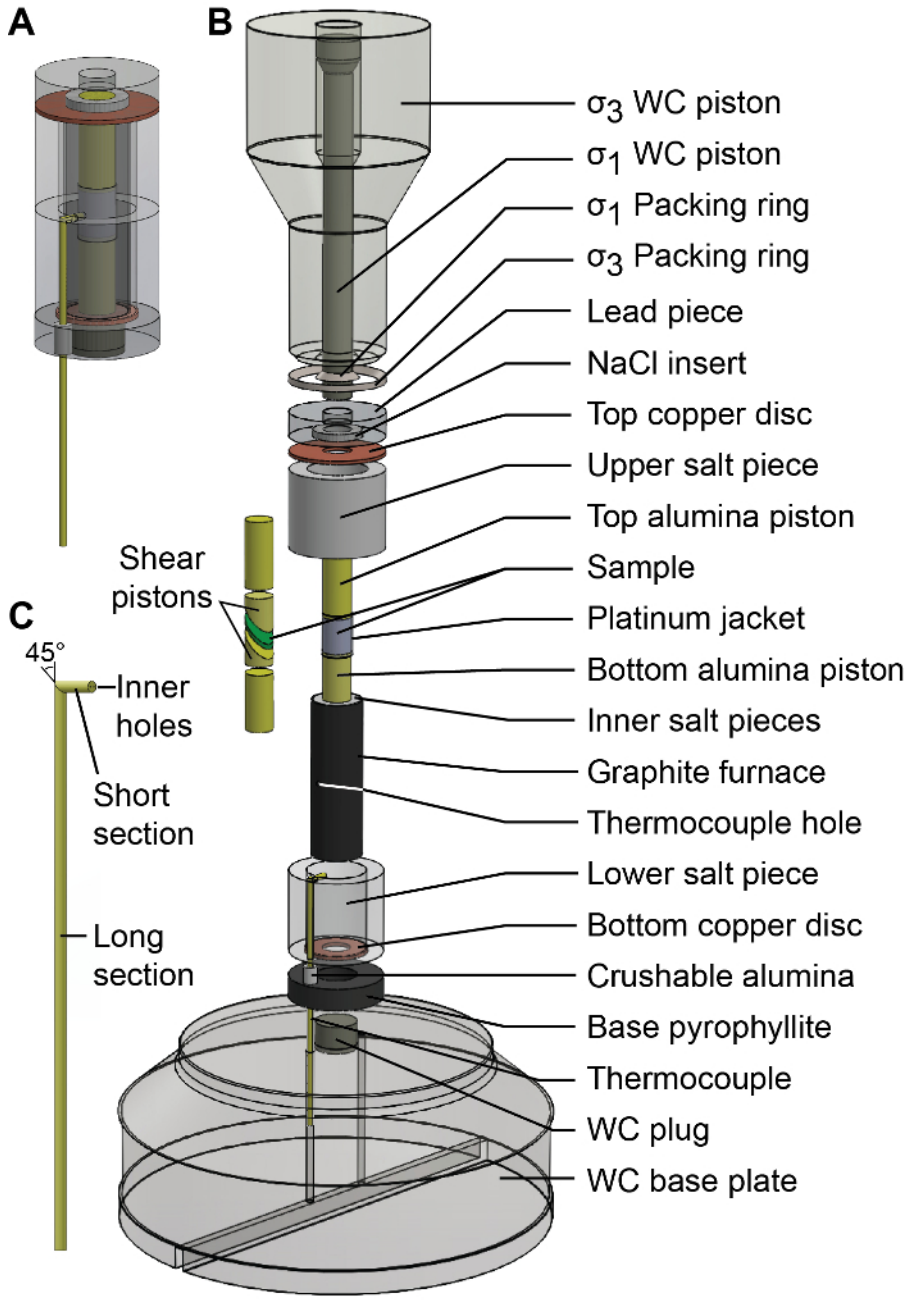

Figure 2: Sample assembly. Detailed view of the pieces that compose the sample assembly. The σ_1 piston, σ_3 piston and base plate are also shown – part of them in transparency – to locate the position of each piece with respect to the Griggs-type apparatus. **A)** Sample assembly of a coaxial experiment. **B)** Exploded view of the sample assembly, either for a "coaxial" (white) or "general shear" sample (green). The lead piece and lower salt piece are shown in transparency. **C)** 3D view of a mullite 2-hole sheathing S-type thermocouple used to monitor the temperature during an experiment. WC = tungsten carbide. Please click here to view a larger version of this figure.

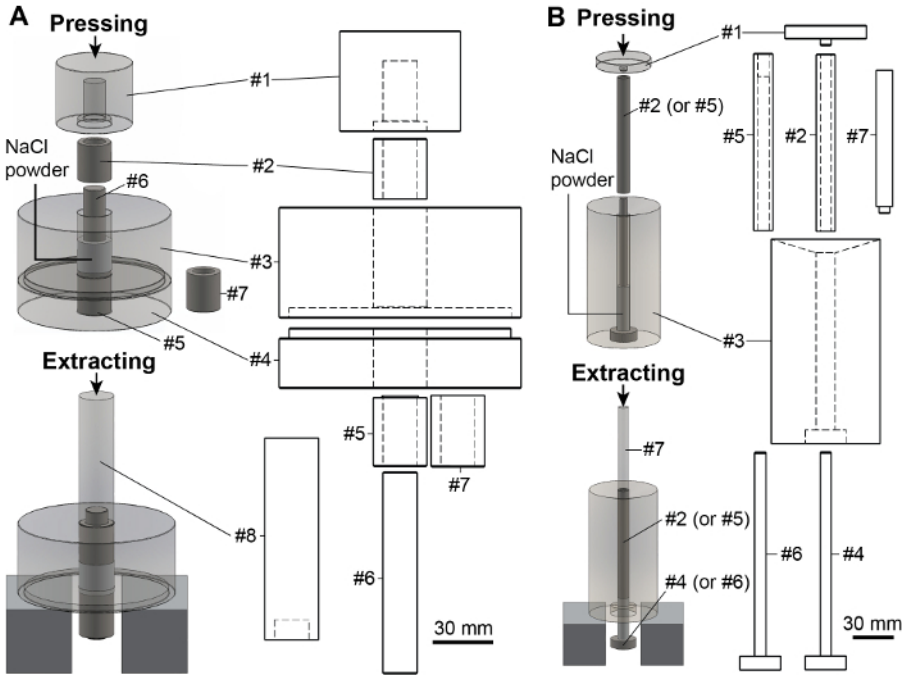

Figure 3: Tools required to cold press the outer and inner salt pieces from NaCl powder. A) 3D views during pressing (14 tons for 30 s) and extraction of the outer salt pieces (left), and scaled drawings of the related tool components (right). **B)** 3D views during pressing (6 tons for 30 s) and extraction of the inner salt piece (left), and scaled drawings of the related tool components (right). Some parts are shown in transparency. Please click here to view a larger version of this figure.

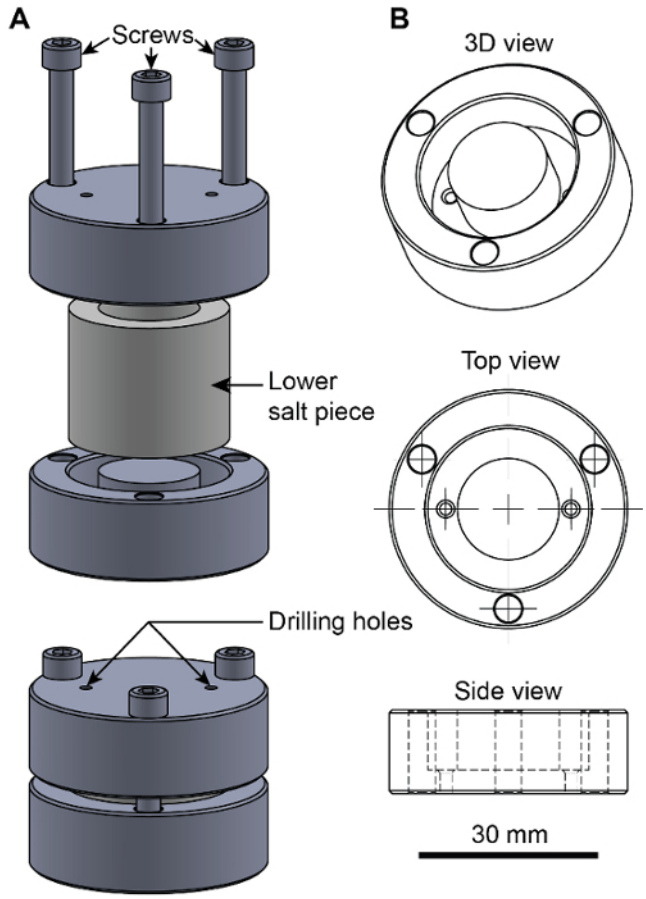

Figure 4: Tool required to drill the lower outer salt piece. A) 3D views before (top) and during (bottom) drilling. **B)** Scaled drawings (3D, top and side views) of the tool (only one part is shown). Please click here to view a larger version of this figure.

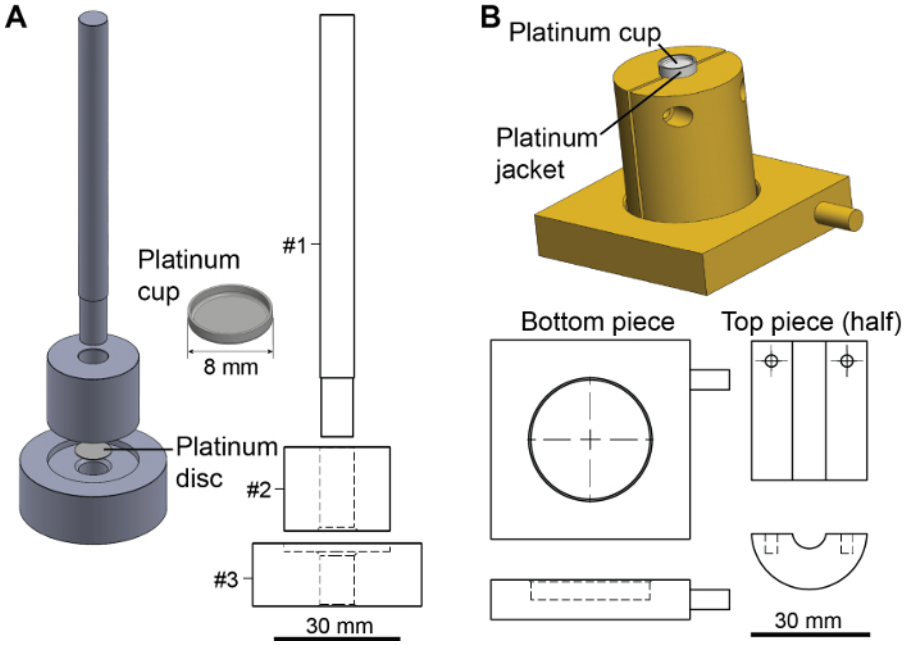

Figure 5: Tools required to produce the platinum jacket. A) 3D view (left) and scaled drawings (right) of the tool needed to produce the platinum cups. By pushing on the 10-mm-diameter platinum disc, its outer part is bent up over 1 mm thickness into a cup-shape, so that it can fit into and be welded together with the 8-mm-diameter platinum jacket. **B)** 3D view (top) and scaled drawings (bottom) of the tool needed to weld a platinum cup to the platinum jacket (only half of the top piece is shown). Please click here to view a larger version of this figure.

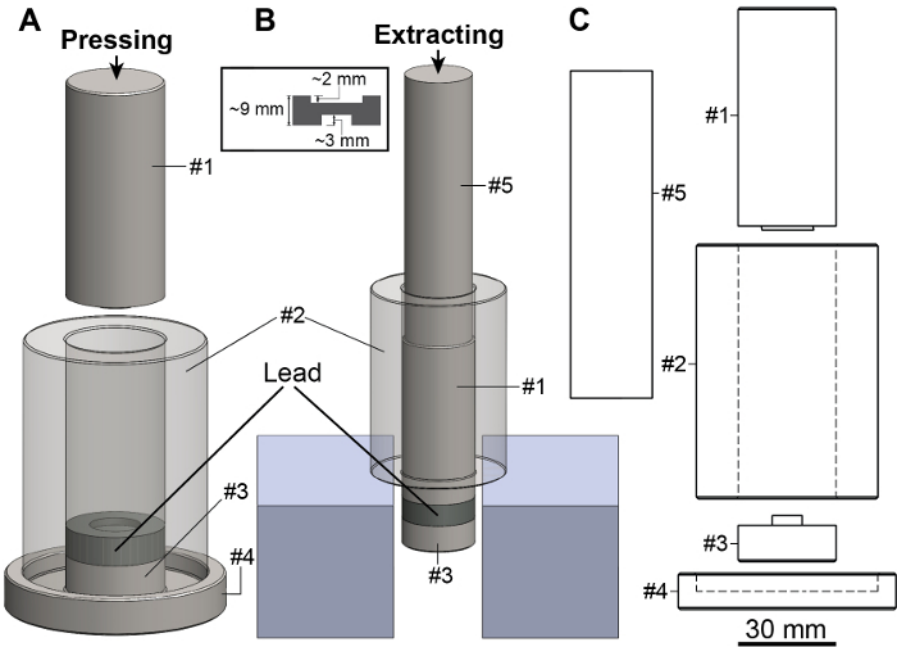

Figure 6: Tool required to produce the lead piece. A) 3D view during pressing (4 tons for 30 s) of the molten lead (50 g). The component #2 is shown in transparency. **B)** 3D view during extraction of the lead piece (the dimensions are shown in the top left inset). **C)** Scaled drawings of the tool components. Please click here to view a larger version of this figure.

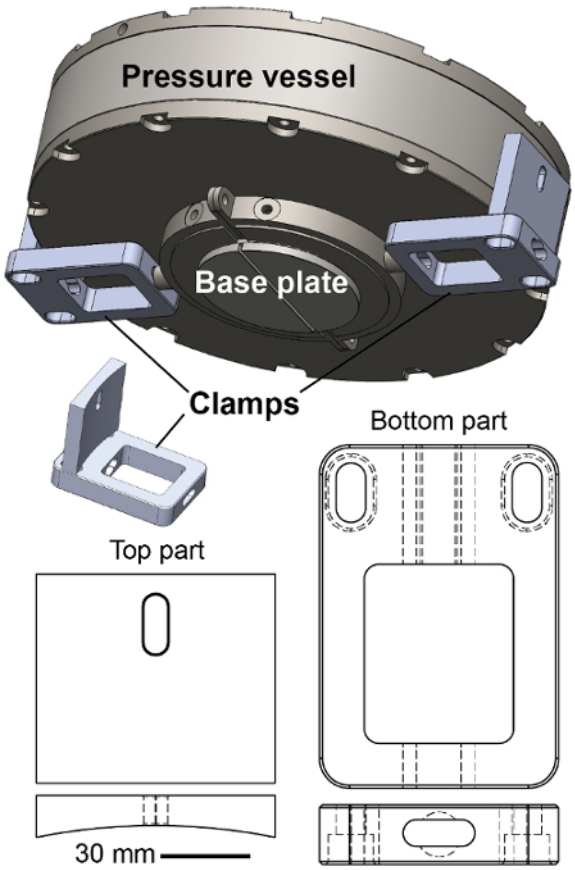

Figure 7: Clamps to fix the basal piston to the pressure vessel. 3D view of the pressure vessel, basal piston and clamps (top), and scaled drawings of the top and bottom parts of one clamp, including a 3D view (bottom). Please click here to view a larger version of this figure.

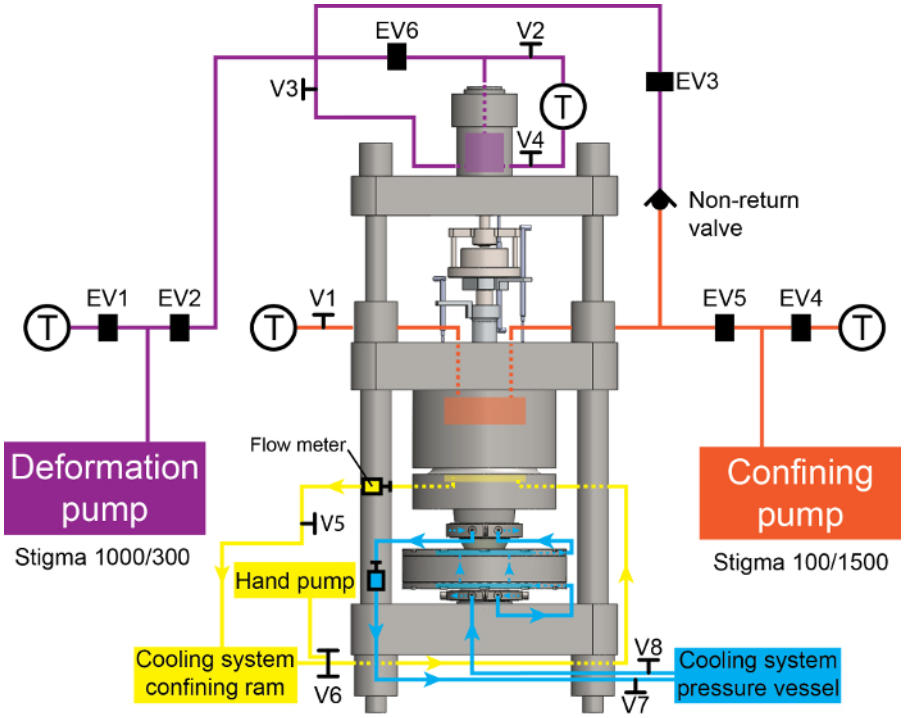

Figure 8: Hydraulics of the pumps and cooling systems. Scheme of the hydraulics – including valves (V), electro-valves (EV) and oil tank (T) – of the deformation pump (purple), confining pump (orange), cooling system of the pressure vessel (light blue) and cooling system of the confining/end-load ram (yellow). Please click here to view a larger version of this figure.

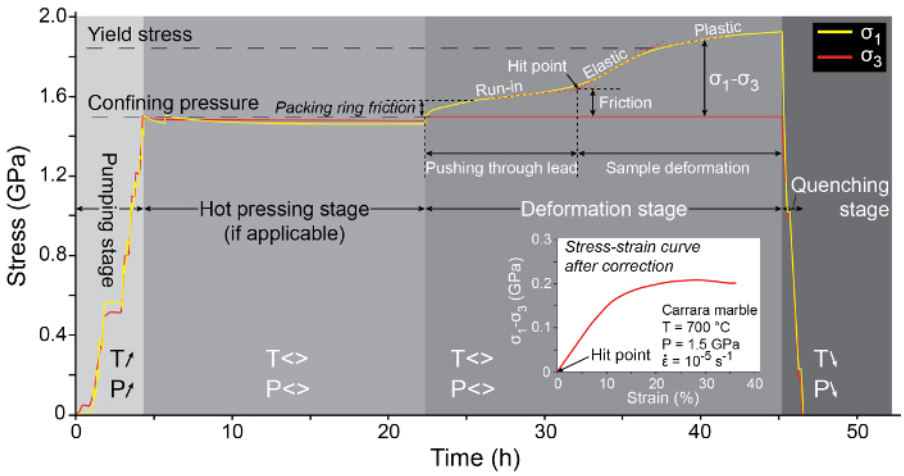

Figure 9: Representative result. Example of a stress-time curve of a deformation experiment using the new generation Griggs-type apparatus. This experiment has been performed coaxially on a core sample (8 mm long) of Carrara marble at 700 °C, 1.5 GPa and a strain rate of 10^{-5} s^{-1}. This result illustrates the successive steps of a Griggs-type experiment, which includes 1) a "pumping stage" to increase pressure and temperature, 2) a "hot-pressing stage" to sinter the sample, if applicable, 3) a "deformation stage" to deform the sample, and 4) a "quenching stage" to decrease pressure and temperature. During deformation, the σ_1 piston first advances through the lead ("run-in" step), and then pushes on the alumina piston to properly deform the sample (up from the hit point), giving rise to elastic-then-plastic behavior (see text). After correction of the stress-time curve from friction and stiffness/extension of the apparatus, a stress-strain curve is produced up from the hit point (inset). σ_1 = stress applied by the σ_1 piston; σ_3 = stress applied by the σ_3 piston; P = confining (isostatic) pressure; T = temperature. σ_1-σ_3 = differential stress. Please click here to view a larger version of this figure.

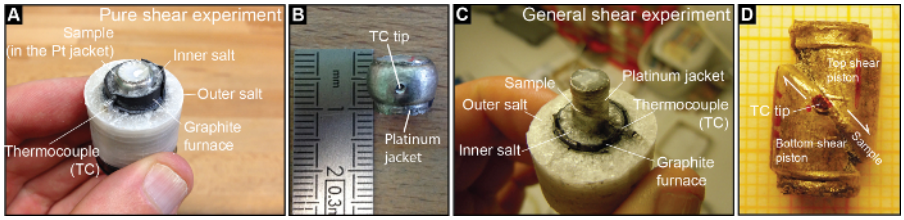

Figure 10: Sample extraction. A) Lower part of the sample assembly extracted after the experiment described in **Figure 9**. **B)** Sample of Carrara marble (still wrapped into its platinum jacket) after pure shear deformation at 700 °C and 1.5 GPa in the new Griggs-type apparatus. **C)** Lower part of a sample assembly containing a sample of olivine powder sintered, and then deformed in general shear at 900 °C and 1.2 GPa with the former Griggs-type apparatus[23]. **D)** Olivine sample and alumina shear pistons (still wrapped into the platinum jacket) after extraction from the sample assembly. TC = Thermocouple. Please click here to view a larger version of this figure.

Discussion

Initially, the Griggs-type apparatus was designed to perform deformation experiments as slowly as possible to approach geological strain rates closer than other techniques, *i.e.*, over weeks, months or even years[9]. Thus, Griggs-type experiments may run as long as the electrical supply and water cooling are functioning, particularly overnight when no operator is required. As mentioned before, the Griggs press can also explore most of the range of pressure and temperature encountered in the lithosphere. However, this technique presently is subjected to some limitations that may reduce the accuracy of the stress determination.

The success of a Griggs-type experiment relies on several critical points that mainly include the quality of the thermocouple sheath, the shape of packing rings, and the alignment of shear pistons (only for general shear experiments). Indeed, the thermocouple wires should be well insulated from each other and from the confining medium (NaCl). Otherwise, the temperature recording may be either modified through touching of the two wires outside of the sample chamber, leading to a dramatic increase of temperature (this could break the pressure vessel), or the thermocouple may break and the experiment fails. The top surface of each packing ring (σ_1 and σ_3) should be flat and large enough (around half a millimeter). This is required to avoid any lead leak during pressure increase. For general shear experiments, the top and bottom shear pistons should be perfectly aligned, so that no asymmetrical deformation occurs during experiment. If not, the sample may come into contact with the confining medium through a jacket leak, giving rise to possible contamination and sample failure. In addition, such a jacket leak will likely occur in a general shear experiment if the deformation piston is not stopped early enough. The capability of the platinum jacket in being deformed without any breaking may significantly vary from one experiment to another. Nevertheless, although shear deformation has been already achieved at more than gamma = 7 on samples of 2 mm thickness (an example is given in Heilbronner and Tullis[24]), a gamma = 5 is routinely applied with a good success rate and significantly higher shear strains can be achieved by reducing the sample thickness.

Nowadays, the Griggs press is subject to friction effects that reduce the accuracy of stress measurements, particularly when the "hit point" is defined by curve fitting. Most of the friction occurs while the deformation piston is advancing through the σ_1 packing ring, lead piece and confining medium (NaCl). This can be seen from the stress-time curve during the "run-in" step of the deformation stage (**Figure 9**), but also during loading after the hit point. While elastic behavior is not dependent on the sample stiffness, the slope of the loading curve increases with the sample strength in the Griggs-type apparatus. This is mainly due to non-elastic sample strain while the σ_1 piston pushes through the lead. Indeed, the slope of the load curve before yield stress conditions does not represent pure elastic loading of the sample, but a combination of different components that include friction and some sample deformation/compaction. Unfortunately, this type of behavior is hardly reproducible as it depends on the sample strength, which is low at high temperature, and the error induced by friction that strongly varies from 3 to 9%[18]. Some other weaker materials like Indium, Bismuth or Tin have been used instead of lead[19], but they always give rise to some leak at pressures higher than 1 GPa. In addition, whereas km-scale objects and very slow strain rates (10^{-15}-10^{-12} s^{-1}) need to be considered for geological purposes, the Griggs-type apparatus - like any other deformation apparatus - is limited in terms of sample size (max. 8 mm diameter for the Griggs press) and strain rate (min. 10^{-8} s^{-1}). These geological conditions require indeed unrealistic forces and impractical duration of experiment to be applied. Nevertheless, this inevitable gap between deformation experiments and geological circumstances may be partly substituted by numerical models, provided that lab-based mechanical laws are fully valid through extrapolations. This definitely requires developing high-pressure apparatuses with better accuracy, at least as good as the one of the gas-pressure-medium-type apparatus (*i.e.*, ± 1 MPa).

At present, only the gas-medium apparatuses are accurate enough to perform rheological experiments, and most of the available mechanical laws come from the Paterson apparatus at confining pressure of 0.3 GPa. The high accuracy on stress measurements mainly relies on the presence of an internal load cell that undergoes the confining pressure, in contrast to an external one that only suffers room pressure, and its combination with a gas pressure vessel, which permitted to apply a specific design that cannot be transferred as-is into a solid-medium press. Today, the solid-medium apparatus only uses an external load cell – some of them do not even have any load cell – to measure the differential stress, giving rise to a poor resolution and substantial overestimation because of friction.

In the Griggs-type apparatus, the using of a molten salt assembly may significantly reduce the friction around the sample (by a factor of 3). But as mentioned before, it also gives rise to further issues and the accuracy of stress measurement remains 10 times lower than that in the Paterson apparatus. Another approach would consist in implementing an internal load cell, or something similar, to get rid of the friction effects in the Griggs press. Considering the size and capacities of "regular" load cells, such as found in the industry, it seems unrealistic to include some of them within the sample chamber of the pressure vessel. They could not sustain the confining pressure and a high-capacity load cell (max. 200 kN), such as required for high-pressure experiments in the Griggs-type apparatus, and they would be too large to be included in the sample chamber. However, one possibility would imply using the basal piston of the sample column as an internal load cell[25], provided that its deformation can be precisely measured (Andreas K. Kronenberg, personal communications). This requires a room below the base plate to adapt a specific load cell, which has been anticipated in the new Griggs-type apparatus (**Figure 1**). But today, such an internal load cell in solid-medium deformation apparatus remains to be implemented.

Disclosures

The authors have nothing to disclose.

Acknowledgements

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