

A new Kumazawa-type rock deformation apparatus for improved stress measurements at confining pressures up to 2 GPa

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Solid-medium deformation apparatuses of the Griggs type have been widely used for experimental studies of rock and mineral rheology at high pressure and temperature, but precise determination of differential stress has long been limited by friction within solid confining media. In the 1960s, M. Kumazawa independently developed a double piston–cylinder type deformation apparatus, which enables independent and simultaneous measurements of axial and confining forces using two pairs of load cells. However, the potential of this design for accurate stress measurements has not been fully explored. Here, we present a newly developed Kumazawa-type deformation apparatus designed for high-pressure (up to 2 GPa confining pressure) and high-temperature experiments. The apparatus allows simultaneous, real-time monitoring of axial stress and confining pressure, making it possible to quantitatively evaluate frictional stresses acting within solid sample assemblies. We review the design principles of the original Kumazawa apparatus, describe the methodology for stress measurement and correction, and report results from preparatory calibration tests and deformation experiments at room temperature using a newly developed system. The results demonstrate that frictional stress variations during pressurization, heating, and axial deformation can be resolved in real time, providing a basis for improving the reliability of differential stress measurements in solid-medium deformation experiments.

I. INTRODUCTION

Laboratory studies on plastic flow of rocks and minerals under controlled confining pressures (P_c) and temperatures (T) have been conducted using triaxial deformation apparatuses and torsion apparatuses, which utilize liquids, gases, and solid materials as confining media.¹ Gas-medium apparatuses equipped with an internal load cell provide the highest accuracy in stress measurements,^{2–4} but their use is generally restricted to $P_c \leq 0.3$ GPa, corresponding to a depth of ~ 10 km in the Earth's upper crust.

To achieve plastic deformation of quartz in the laboratory, David T. Griggs⁵ developed a double-piston-cylinder-type solid-medium apparatus, which enabled deformation of cylindrical samples with a diameter of 3.2 mm at higher P_c . Its revised model, equipped with a two-post frame and designed for larger sample diameters (6.4 mm), referred to as the “modified” Griggs apparatus, has become the world standard for solid-medium deformation experiments.^{1, 6–8} Weak solid confining media such as pyrophyllite, talc, and NaCl enabled deformation experiments at $P_c < 2$ GPa under upper mantle conditions. By adopting the Boyd–England-type loading system,⁹ the pressure range of the Griggs-type apparatus was further extended to ~ 5 GPa.^{10–14} However, the considerable frictional stress generated in solid materials within the pressure vessel makes precise determination of differential stress difficult. For this reason, some previous studies employed dislocation-density piezometers to quantify differential stress instead of using direct mechanical data obtained from the external load cell of the Griggs apparatus.¹⁵

To reduce friction between the sample and the confining medium, eutectic mixtures of salts have also been used imme-

diately around the sample. A capsule of a eutectic salt mixture such as NaCl/KCl, referred to as the “molten salt cell,” forms a liquid cell at high temperature.^{16, 17} Flow-law parameters for dislocation creep of wet quartz determined using molten salt cell assemblies^{18, 19} have been considered among the most reliable data sets at high P_c .²⁰ However, calibration tests using high-purity metals demonstrated that even when the molten salt cell is used in the Griggs apparatus, the measured stress values are always $\sim 25\%$ greater than those obtained using gas-medium apparatuses.²¹ These results have been duplicated in other laboratories.²²

For deformation experiments at ultra-high pressures exceeding 5 GPa, synchrotron X-ray radiation techniques have been introduced for stress and strain measurements using rotational Drickamer apparatuses (RDA)^{23, 24} and multi-anvil apparatuses, such as the deformation-DIA (D-DIA)²⁵, Kawai-type (6/8-type) presses, and their variations.^{26, 27} Owing to these techniques, deformation experiments at pressures exceeding 15 GPa and high temperatures under conditions corresponding to the mantle transition zone and lower mantle have been realized.²⁸ In these experiments, pressure and deviatoric stress are calibrated based on X-ray diffraction patterns of crystals within the sample itself and are therefore not affected by friction in the solid confining medium. However, application of this technique to long-term experiments is limited by cost and available beamtime. In addition, when low-symmetry minerals such as olivine are used, stress values derived from different diffraction peaks can deviate significantly owing to plastic anisotropy within the polycrystalline sample, making the physical meaning of the calculated stress ambiguous.^{26, 29}

The synchrotron X-ray technique is not applicable to conventional piston-cylinder apparatuses because the thick walls of the pressure vessel prevent penetration of X-ray beams. Compared with multi-anvil apparatuses, however, piston-cylinder deformation apparatuses offer several advantages,

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including larger sample sizes, independent control of axial¹⁴¹
and confining piston movements, and the ability to jacket¹⁴²
samples with metal tubes. Although metallic jackets may¹⁴³
also be used in multi-anvil deformation apparatuses (e.g., D-¹⁴⁴
DIA), their use is often limited in synchrotron-based studies¹⁴⁵
due to X-ray absorption and diffraction interference. These¹⁴⁶
features are particularly important for studies of microstruc-¹⁴⁷
tures and lattice preferred orientation (LPO) in polycrystalline¹⁴⁸
aggregates,^{15,30,31} deformation mechanisms in the brittle-¹⁴⁹
ductile transition zone,³² and the influence of fluids and chem-¹⁵⁰
ical environments on crustal deformation.^{33,34} Probing the¹⁵¹
actual stress state within the pressure vessel therefore re-¹⁵²
mains a major technical challenge in high-pressure and high-¹⁵³
temperature deformation experiments on Earth materials.^{12,35}

In this paper, we describe a sophisticated stress measure-¹⁵⁵
ment method that was first implemented in a solid-medium¹⁵⁶
piston-cylinder apparatus developed by Mineo Kumazawa in¹⁵⁷
1963–1965.³⁶ This apparatus was originally designed to ex-¹⁵⁸
plore mantle anisotropy resulting from lattice preferred ori-¹⁵⁹
entation of minerals,^{37–41} although its maximum designed P_c ¹⁶⁰
was limited to 1 GPa, corresponding to continental Moho¹⁶¹
depths. Deformation and recrystallization microstructures of¹⁶²
wet quartz were also investigated using this apparatus.^{42,43}¹⁶³
However, Kumazawa's original concept for stress measure-¹⁶⁴
ments was not fully utilized in these early studies and was not¹⁶⁵
further pursued until its potential for precise determination of¹⁶⁶
differential stress was later demonstrated.^{44,45} We have devel-¹⁶⁷
oped a new Kumazawa-type apparatus capable of achieving
confining pressures up to 2 GPa and tested the performance of¹⁶⁸
its stress measurement system. In the following sections, we
briefly review the design of the original Kumazawa apparatus¹⁶⁹
and its stress measurement method, then describe the details
of the new Kumazawa-type apparatus, the procedures for ini-¹⁷⁰
tial calibration tests, and preliminary results on internal stress
states within the solid assemblies.¹⁷¹

172 II. ORIGINAL KUMAZAWA APPARATUS

173 A. Overview of design

The original Kumazawa apparatus (MK65S) was con-¹⁷⁸
structed at Nagoya University in 1965³⁶ (Figure 1a,b). Al-¹⁷⁹
though it was developed independently, its basic concept was¹⁸⁰
similar to that of the modified Griggs apparatus: a pressure¹⁸¹
vessel equipped with two concentric pistons, where the outer¹⁸²
piston generates confining pressure and the inner piston ap-¹⁸³
plies axial deformation to the sample. A major difference is¹⁸⁴
that, in the Kumazawa apparatus, both pistons are driven from¹⁸⁵
the lower side, whereas in the Griggs apparatus they are driven¹⁸⁶
from the upper side. The axial movement of the inner piston is¹⁸⁷
controlled by a motor-gear train coupled with a ball screw and¹⁸⁸
thrust bearing, allowing a wide range of displacement rates¹⁸⁹
from 2×10^{-2} to 2×10^{-7} mm s⁻¹, corresponding to strain¹⁹⁰
rates of 10^{-3} – 10^{-8} s⁻¹ for a sample length of 20 mm. A dis-¹⁹¹
tinctive feature of Kumazawa's design is the vertical symme-¹⁹²
try of the upper part of the apparatus, where two pairs of load¹⁹³
cells are installed: the inner load cells measure axial stress¹⁹⁴

(σ), whereas the outer load cells monitor confining pressure
(Figure 1c,d).

The pressure vessel consists of two concentric steel cylin-
ders, with vertical water-cooling channels machined into the
outer ring (Figure 1a,c). The apparatus was originally de-
signed to accommodate relatively large cylindrical samples,
typically 10 mm in diameter and less than 25 mm in length, in
order to investigate lattice preferred orientation development
in polycrystalline aggregates. This sample size is compar-
able to that used in early gas-medium apparatuses⁴⁶ and sig-
nificantly larger than that of early Griggs-type solid-medium
apparatuses.⁵ It is also similar to those used in recent gen-
erations of Griggs-type apparatuses.¹² Samples are internally
heated using a graphite furnace, and thermal insulation is
achieved by a thick solid confining medium with an outer
diameter of approximately 40 mm (Figure 1e,f). The maxi-
mum designed confining pressure and axial stress were 1 GPa
and 2 GPa, respectively. To generate high pressures in such
large assemblies, a 200 ton four-post press and a hydraulic
pumping system capable of oil pressures up to 100 MPa were
employed. The electric power required to heat the sample
to 1000°C was approximately 3.5 kW, supplied through a
200 V AC to 20 V AC transformer with a rated capacity of
4 kVA. In practice, deformation experiments were conducted
at confining pressures up to 0.8 GPa³⁷ and temperatures as
high as 1265°C.^{40,41}

175 B. Principle of stress measurements

176 1. Loading system

In deformation experiments using the Griggs apparatus, the
axial force acting on the inner piston is measured by an exter-
nal load cell placed above the pressure vessel (Figure 2a). To
minimize the effects of friction during initial pressurization,
the sample, ceramic pistons, and confining medium are loaded
together through a Pb spacer, while the outer and inner pis-
tons are advanced either alternately or simultaneously. Before
deformation, the inner piston is finally advanced to establish
contact with the sample. Confining pressure estimated from
the oil pressure deviates from the axial stress measured by
the load cell due to friction of packing rings and resistance of
the pressure medium.^{12,14,35} The differential stress is therefore
evaluated relative to the hit point, assuming a near-hydrostatic
stress state, where the axial stress approximates the confining
pressure. In contrast, in the Kumazawa apparatus (Figure 2b),
the internal pistons are engaged from the beginning of pres-
surization, and both axial stress (σ) and confining pressure
are increased simultaneously and independently. Two pairs
of load cells are used to monitor axial and confining forces
separately.

Before an experiment, the pressure vessel is positioned be-
tween deformable spacers that support its weight. During
pressurization, the distance between the upper and lower con-
fining pistons decreases owing to compaction of the solid
confining medium. With increasing P_c , the frictional force
between the pressure vessel and the sample assembly ex-

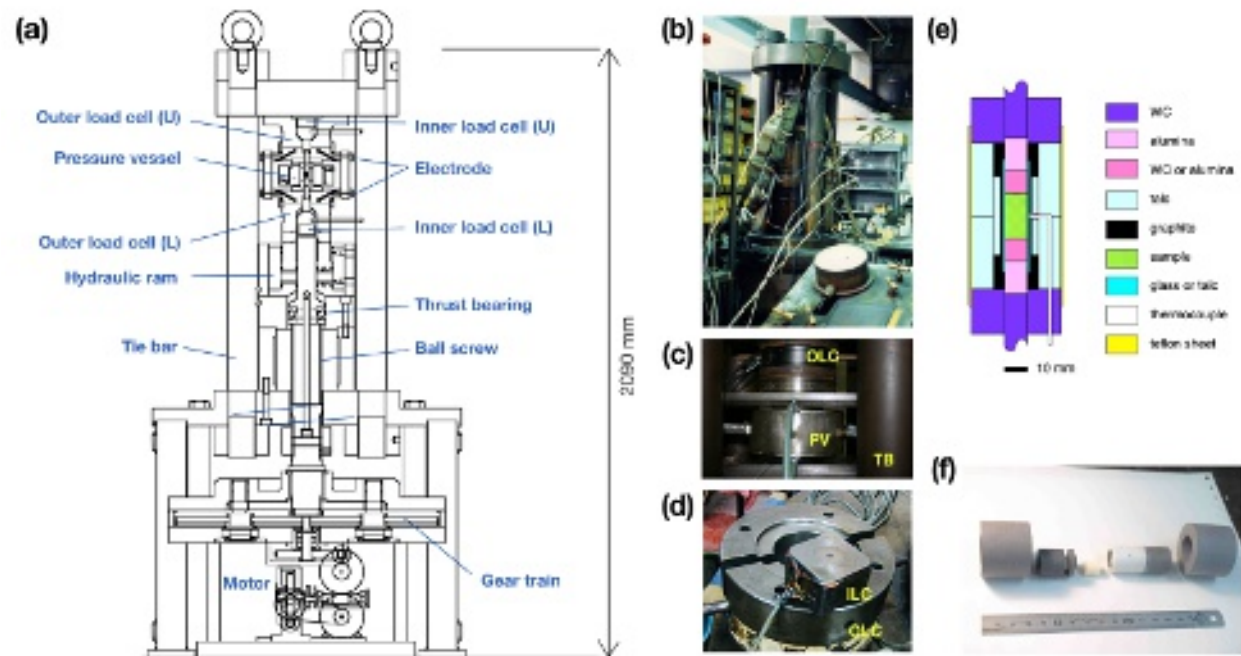


FIG. 1. The original Kumazawa apparatus: (a) a copy of the original blueprint drawn in 1965³⁶; (b) the 200 ton press and hydraulic pumping system at Nagoya University; (c) pressure vessel placed between electrode plates and the upper outer load cell (OLC); (d) the lower inner load cell (ILC) extracted from the central hole of the lower OLC; (e) a representative sample assembly^{37,38}; (f) assembly parts using pyrophyllite and talc for the outer and inner sleeves, respectively.

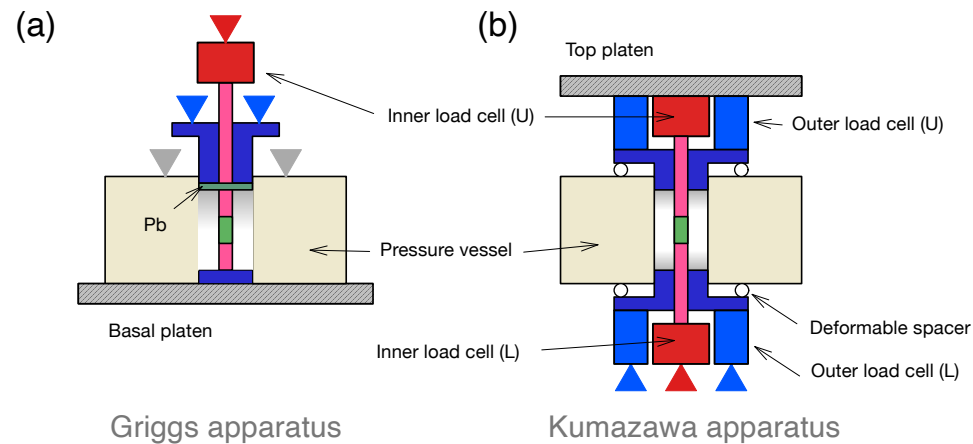


FIG. 2. Schematics of loading and stress measurement systems in (a) the Griggs apparatus and (b) the Kumazawa apparatus. Red and blue triangles indicate loading directions of the inner and outer pistons, respectively. Grey triangles show Boyd-England-type end loading on the pressure vessel for higher- P_c (>3 GPa) experiments adopted in some Griggs-type apparatuses.

ceeds the gravitational force, causing the vessel to be lifted upward. Because no external forces act on the pressure vessel other than its own weight, the confining medium is compressed symmetrically from the upper and lower sides, thereby minimizing pressure gradients within the confining

medium. A similar technique, known as floating-die compaction, is widely used in powder pressing to produce homogeneous products. In this configuration, relative motion between the sample and the confining medium is minimized, and the influence of friction at the inner wall of the pressure

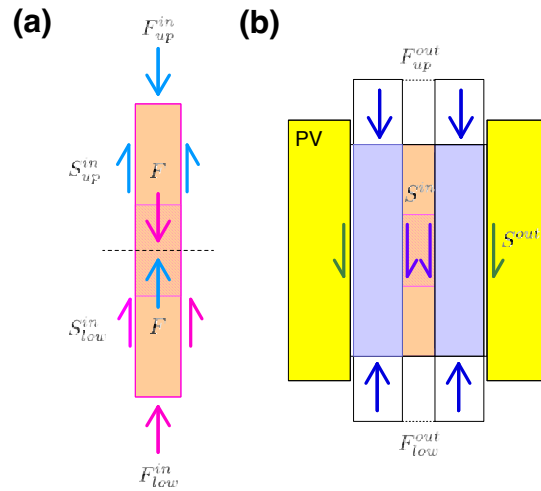


FIG. 3. Force balance in the sample assembly with respect to (a) the sample column and (b) the confining medium. See text for details. PV: pressure vessel.

vessel on P_c measurements is reduced.

2. Axial stress

The axial stress is calculated from the axial force (F) acting on the horizontal cross-sectional area (A^{in}) of the cylindrical sample:

$$\sigma = \frac{F}{A^{in}}. \quad (1)$$

However, the axial forces measured at the upper and lower sides of the assembly, denoted as F_{up}^{in} and F_{low}^{in} , respectively, are generally different from the axial force at the center of the sample owing to shear forces acting within the confining medium (Figure 3a). Here, the central part of the assembly consisting of the sample and ceramic pistons is referred to as the “sample column”. The force balance for the upper and lower halves of the sample column is expressed as

$$\begin{cases} F_{up}^{in} = F + S_{up}^{in} \\ F_{low}^{in} = F - S_{low}^{in} \end{cases} \quad (2)$$

where S_{up}^{in} and S_{low}^{in} are the shear forces acting on the upper and lower halves of the sample column, respectively. The total shear force required to drag the sample column upward is given by $S^{in} = S_{up}^{in} + S_{low}^{in}$.

The axial stresses acting on the top and basal surfaces of the sample column are written as $\sigma_{up} = F_{up}^{in}/A^{in}$ and $\sigma_{low} = F_{low}^{in}/A^{in}$, respectively. The sample assembly of the Kumazawa apparatus is designed to be vertically symmetric (Figure 1c). Therefore, as a first approximation, we assume

$$S_{up}^{in} = S_{low}^{in} = \frac{S^{in}}{2}. \quad (3)$$

Under this assumption, the effects of shear forces can be eliminated by averaging F_{up}^{in} and F_{low}^{in} . From Eqs. (1)–(3), we obtain

$$\sigma = \frac{\sigma_{up} + \sigma_{low}}{2}. \quad (4)$$

From Eq. (2), the shear stress τ acting on the sample column can also be evaluated as

$$\tau = \frac{S^{in}}{A_s^{in}} = \frac{F_{up}^{in} - F_{low}^{in}}{A_s^{in}}. \quad (5)$$

Here, A_s^{in} denotes the side area of the cylindrical surface of the sample column.

3. Confining pressure

The pressures acting on the upper and lower sides of the confining medium, denoted as P_{up} and P_{low} , respectively, are expressed as

$$\begin{cases} P_{up} = F_{up}^{out}/A^{out} \\ P_{low} = F_{low}^{out}/A^{out} \end{cases}, \quad (6)$$

where F_{up}^{out} and F_{low}^{out} are the forces measured by the outer load cells placed at the upper and lower sides of the assembly, respectively (Figure 3b). Owing to frictional stresses acting on both the outer and inner surfaces of the confining medium, pressure gradients develop within the solid confining medium, and consequently P_{up} and P_{low} deviate from the confining pressure at the center of the assembly. In addition, because of the finite shear strength of the solid confining medium, the vertical stress is generally not equal to the radial confining stress acting on the side surface of the cylindrical sample.⁴⁷

We therefore assume that the mean value

$$P_{mean} = \frac{P_{up} + P_{low}}{2} \quad (7)$$

is proportional to the confining pressure P_c :

$$P_c = \gamma P_{mean}. \quad (8)$$

The proportionality coefficient γ is determined by pressure calibration tests.

III. NEW KUMAZAWA-TYPE APPARATUS

A. Overview of configuration

The new deformation apparatus (named CHERRY), built by Sumitomo Heavy Industries, Ltd., is installed at Kyoto University (Figure 4). This apparatus follows the original Kumazawa design but employs a smaller sample assembly to generate higher pressures, and the overall size of the machine was reduced for easier handling. The upper four posts sustain a total load of 100 tons (~ 1000 kN), whereas the lower two

posts support an axial load of 20 tons (~ 200 kN). The total height of the press is approximately 1.3 m, and its total weight is about 600 kg (Figure 4a). Because the pressure vessel of the Kumazawa-type apparatus is not end-loaded (Figure 2), the theoretical limit of pressure resistance is approximately 3 GPa. Focusing on brittle-to-ductile (plastic) deformation in the middle to lower crust and in subducting slabs down to depths of about 60 km, we designed the pressure vessel for a maximum confining pressure of 2 GPa (Figure 4b).

In the new apparatus, the confining force is generated by a center-hole hydraulic cylinder (Riken Seiki Co., Ltd.) operated at oil pressures up to 100 MPa. The hydraulic ram is actuated by a hand pump equipped with an analog pressure gauge and an adjustment syringe. The inner piston is driven by a servomotor connected to a worm jack through a gear reducer. The vertical displacement of the worm jack is calculated from the gear ratio and the rotation angle of the servomotor (Mitsubishi Electric Co.) and is displayed on a touch panel of the control box (Figure 4d). The resolution of vertical motion, defined as the minimum step per servomotor pulse, is $0.015 \mu\text{m}/\text{pulse}$. The movements of the axial pistons at the position indicators are independently monitored using laser displacement meters (OPTEX FA Co., Ltd., CD33) with a repeatability of $2 \mu\text{m}$ (Figure 5a). The axial displacement velocity can be continuously varied from 2×10^{-1} to $1 \times 10^{-6} \text{ mm s}^{-1}$, yielding strain rates on the order of 10^{-2} – 10^{-7} s^{-1} .

The pressure vessel consists of three concentric rings with cooling-water jackets at the top and base (Figure 4b; Figure 5a). The tapered cylinder core, made of high-speed tool steel (JIS SKH51), withstands an internal pressure of 2 GPa with a safety factor greater than 1.9 at room temperature. A sample assembly with an outer diameter of 24 mm is inserted into the center hole of the cylinder core; this assembly size is comparable to the standard 25.4 mm (1 inch) diameter used in conventional Griggs apparatuses. The inner and outer forces are transmitted to the sample assembly through σ pistons and center-hole P_c anvils made of tungsten carbide (WC), respectively, which are designed for cylindrical samples with diameters of either 8 mm or 6 mm (Figure 4c). The compressive strength of WC is approximately 5.7 GPa. Using 8 mm inner pistons, the outer force reaches ~ 800 kN at the designed maximum confining pressure of 2 GPa, and the inner-force capacity of 200 kN corresponds to a maximum axial stress of 4 GPa. Using 6 mm inner pistons, the outer force reaches ~ 850 kN at $P_c = 2$ GPa, while the corresponding inner-force capacity is ~ 150 kN, giving a maximum axial stress of approximately 5 GPa.

Figure 5b shows examples of the sample assemblies, which consist of ceramic inner pistons sandwiching the sample, inner and outer sleeve confining media, a graphite heater, a thermocouple, and ceramic tubes. Alumina or zirconia is commonly used for the inner pistons. A Teflon sheet with a thickness of 0.1 mm is placed along the inner wall of the pressure vessel to reduce friction and to electrically insulate the vessel. No packing ring is used at the upper and lower rims of the combining medium.

B. Heating and cooling systems

Power is supplied to the graphite heater by a single-phase 200 V/50 A power source through a 200 V AC to 10 V AC transformer with a capacity of 6 kVA, which can deliver currents of up to 600 A. The press frame and load cells are electrically insulated from the heater circuit by mica sheets (Figure 5a). The specific electrical resistivity of the graphite heater, manufactured by TOYO TANSO Co., Ltd., is $11.0 \mu\Omega\text{m}$ at room temperature. Using a graphite sleeve with a thickness of 1 mm, the power required to heat the sample to 1000°C was approximately 2.5 kVA. The measured temperature–power relationship indicates that maximum temperatures exceeding 1500°C are attainable with the same assembly. At present, deformation experiments are conducted at temperatures up to 1100°C using a K-type (alumel–chromel) thermocouple sheathed with MgO in a metal jacket with a diameter of 1 mm, which minimize significant drift and breakage at temperatures above 800°C , associated with the so-called the green rot phenomenon. The thermocouple is connected to a proportional–integral–derivative (PID) temperature controller (Chino Corporation) via an extension lead cable. For experiments at higher temperatures or requiring higher accuracy, an R-type (Pt/Pt–13%Rh) or a S-type (Pt/Pt–10%Rh) thermocouple can be used with the same control system.

During high-temperature experiments, the pressure vessel as well as the upper and lower electrode plates are cooled by circulating water. Cooling water supplied by a 1400 W recirculating chiller (AS ONE Corporation) is first introduced into the pressure vessel, then passes through the upper and lower electrode plates in sequence, and finally returns to the chiller. Because the inner wall of the pressure vessel experiences the highest circumferential tensile stress, effective cooling of the cylinder core is essential to prevent rupture. To enhance cooling efficiency and maintain the mechanical strength of the core, the second ring made of die steel (SKD) is penetrated by twelve vertical channels through which cooling water circulates sequentially (Figure 5b). This cooling concept is inherited from the original Kumazawa apparatus³⁶ (Figure 1a), and similar internal cooling systems are adopted in several recent high-pressure deformation apparatuses.^{12,48} Thermosensitive papers recorded maximum temperatures of approximately 110°C on the upper surface of the cylinder core and 70°C on the outer surface of the third ring, confirming the effectiveness of the cooling system. The quench time required to cool the sample from 1000°C to 500°C was about 7 s, and further cooling to below 100°C was achieved within 1 min.

C. Data acquisition system

Axial and confining forces are measured using inner and outer load cells installed below and above the pressure vessel, respectively (Figure 5a). Because there is insufficient space to accommodate commercially available load cells with capacities of 200 kN and 1000 kN, structural steel components of the apparatus, each instrumented with four strain gauges, are used

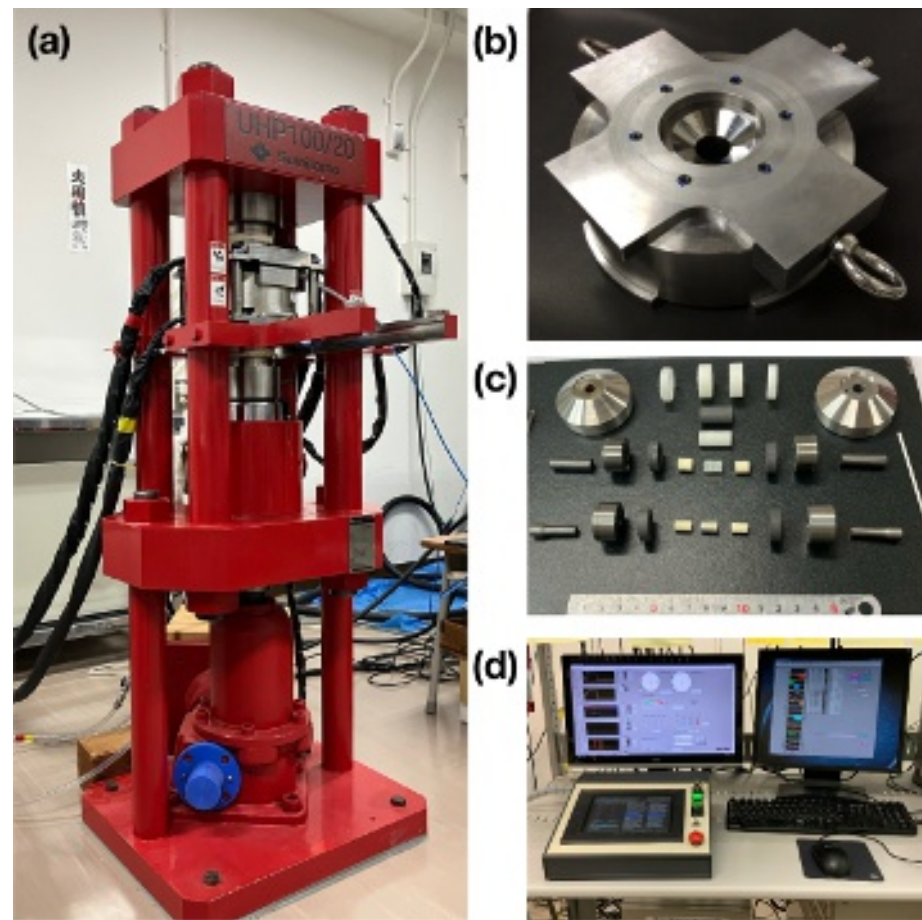


FIG. 4. (a) New Kumazawa-type apparatus (CHERRY) installed at Kyoto University (Sumitomo Heavy Industries, Ltd., 100-ton press). (b) Pressure vessel; the outer ring has a diameter of 180 mm. (c) Pistons, anvils, and solid components of the sample assemblies; the diameters of the sample and inner pistons are either 8 mm or 6 mm. (d) Servomotor control box and PC-based monitoring system.

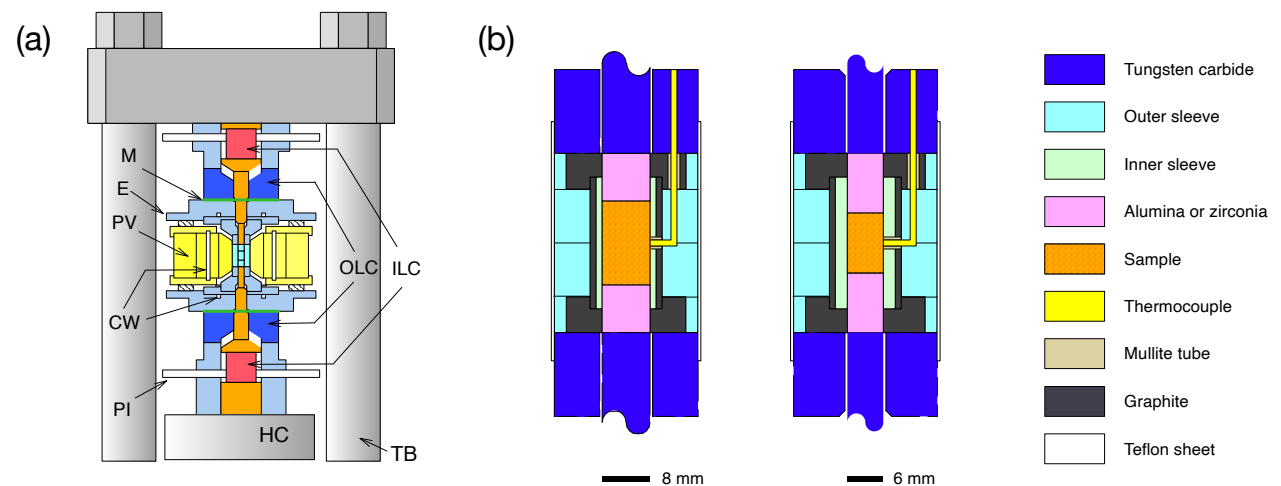


FIG. 5. (a) Schematic cross section of the upper part of the new Kumazawa-type apparatus. PV, pressure vessel; ILC, inner load cell; OLC, outer load cell; CW, cooling-water passage; E, electrode; M, mica sheet; PI, position indicator for the axial piston; TB, tie bar; HC, hydraulic cylinder. (b) Examples of sample assemblies using cylindrical samples with diameters of 8 mm (left) and 6 mm (right); see also Figure 4c.

as load cells. These custom-made load cells were calibrated against commercially available load cells with capacities of 200 kN and 1000 kN.

Analog signals from the load cells, thermocouples, and laser displacement meters are collected and digitized using a 16-channel data logger (PCD300A and PCD320A, Kyowa Electronic Instrument Co., Ltd.) and transferred to a personal computer. The available sampling rates range from 1 Hz to 5 kHz, although typical recording is performed at 2–5 Hz. Data processing, real-time visualization on a CRT monitor, and recording on the PC are managed using a LabVIEW-based system (National Instruments Corporation). The raw load-cell outputs are transformed into F_{low}^{in} , F_{up}^{in} , F_{low}^{out} , and F_{up}^{out} using nonlinear calibration relations. The axial stress (Eq. 4), confining pressure (Eq. 8), and shear force acting on the sample column (Eq. 5) are calculated and displayed on the monitor in real time (Figure 4d).

IV. CALIBRATION TESTS

A. Machine stiffness

Uniaxial compression tests of a tungsten carbide rod with a diameter of 8 mm and a length of 28 mm were conducted to evaluate the elastic distortion of the apparatus (Figure 6). The stress (or force)–displacement curve shows a non-linear increase at low stresses, and the slope of the curve represents the machine stiffness (G). Because the inner piston assembly consists of multiple WC and steel components (Figure 5a), the initial slope reflects the stiffness associated with asperity contacts between individual parts. At axial stresses exceeding 1 GPa, these solid–solid contacts are nearly fully closed, and G attains a constant value of 95.7 kN mm^{-1} .

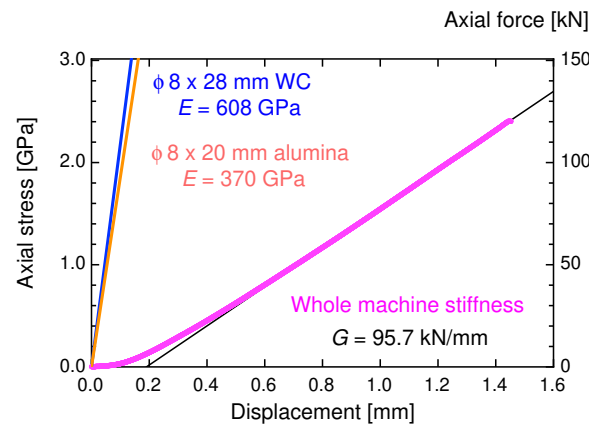


FIG. 6. A uniaxial compression test using a tungsten carbide rod with a diameter (ϕ) of 8 mm. Axial stress is plotted against the displacement of the inner piston calculated from the rotation angle of the servomotor. The slope of the fitted line (black) represents the overall machine stiffness G at axial stresses greater than 1.0 GPa. Elastic deformation of the WC rod (blue) and alumina pistons (orange) calculated from Young's modulus E are also shown.

B. Calibration of confining pressure

The calibration constant γ in Eq. (8) was determined at room temperature using the phase transition points of ammonium fluoride (NH_4F). A simple all-talc assembly containing surface-ground specimens of Sivec marble with a diameter of 8 mm and a height of 10 mm was used for a series of calibration tests (Figure 7a). Sivec marble, produced in North Macedonia, is white in color and is homogeneous and isotropic, consisting of nearly 100% calcite with typical grain sizes of 0.2–0.5 mm (Figure 7b). Figure 8a shows representative mechanical data obtained during a calibration test. While the hydraulic ram was driven upward using a hand pump, the movement of the inner piston was manually controlled so that the axial stress σ increased simultaneously with P_c . Although both the outer and inner pistons were continuously advanced, the increase in P_c was temporarily halted at the I–II transition (0.36 GPa) and the II–III transition (1.2 GPa) of NH_4F . In this figure, the calibrated upper and lower pressures, γP_{up} and γP_{low} , are replotted as P_{up} and P_{low} , respectively.

C. Constant strain-rate tests

During the pressure calibration test, the axial stress σ_{up} increased faster than σ_{low} because the sample and alumina pistons were dragged upward by the confining medium (Figure 8a). After pressurization to 1.5 GPa, both the outer and inner pistons were held stationary for 10 min. Subsequently, the inner piston was moved upward to deform the sample at a constant strain rate of $2.0 \times 10^{-4} \text{ s}^{-1}$. During this stage, σ_{low} caught up with σ_{up} and then exceeded it. The values of P_{up} and P_{low} changed slightly in opposite directions, whereas the confining pressure P_c remained nearly constant.

Figure 8b shows a similar example of a constant strain-rate test. In this case, the sample was pressurized to the first phase transition point of NH_4F (360 MPa) and then axially deformed at a slower strain rate of $6.7 \times 10^{-6} \text{ s}^{-1}$. These mechanical behaviors can be understood in terms of the shear force associated with the upward movement of the inner piston.

The force balance with respect to the confining medium is expressed as

$$F_{low}^{out} = F_{up}^{out} + S^{in} + S^{out}, \quad (9)$$

where S^{out} is the shear force acting on the outer surface of the sample assembly. At the beginning of the experiment, the weight of the pressure vessel ($\sim 15 \text{ kg}$) is supported by deformable spacers (Figure 2b). With increasing confining pressure, however, the frictional force acting on the outer surface of the assembly exceeds the gravitational force, and the pressure vessel is lifted upward. For example, even when a Teflon sheet with a friction coefficient of 0.02 is used, the frictional resistance exceeds 6 kN at a confining pressure of 100 MPa, which is much larger than the gravitational force of the pressure vessel ($\sim 0.15 \text{ kN}$). In this state, S^{out} is balanced by the gravitational force acting on the pressure vessel.

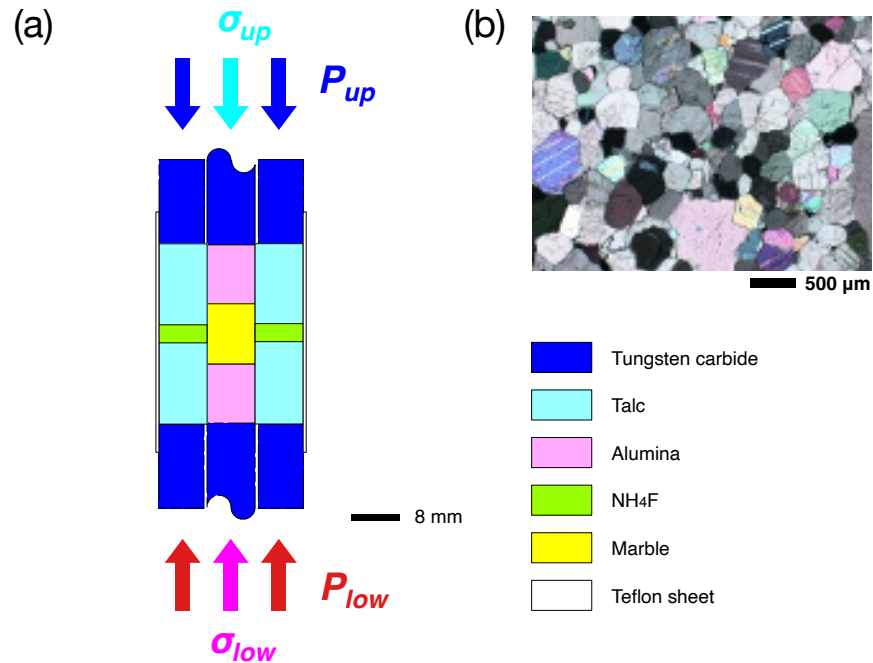


FIG. 7. (a) The all-talc assembly used for pressure calibration. NH_4F powder was inserted between the upper and lower talc pieces. Arrows indicate the upper and lower axial stresses (σ_{up} and σ_{low}) and confining pressures (P_{up} and P_{low}), respectively. (b) A microphotograph of a thin section of starting Sivec marble taken under crossed polarized light.

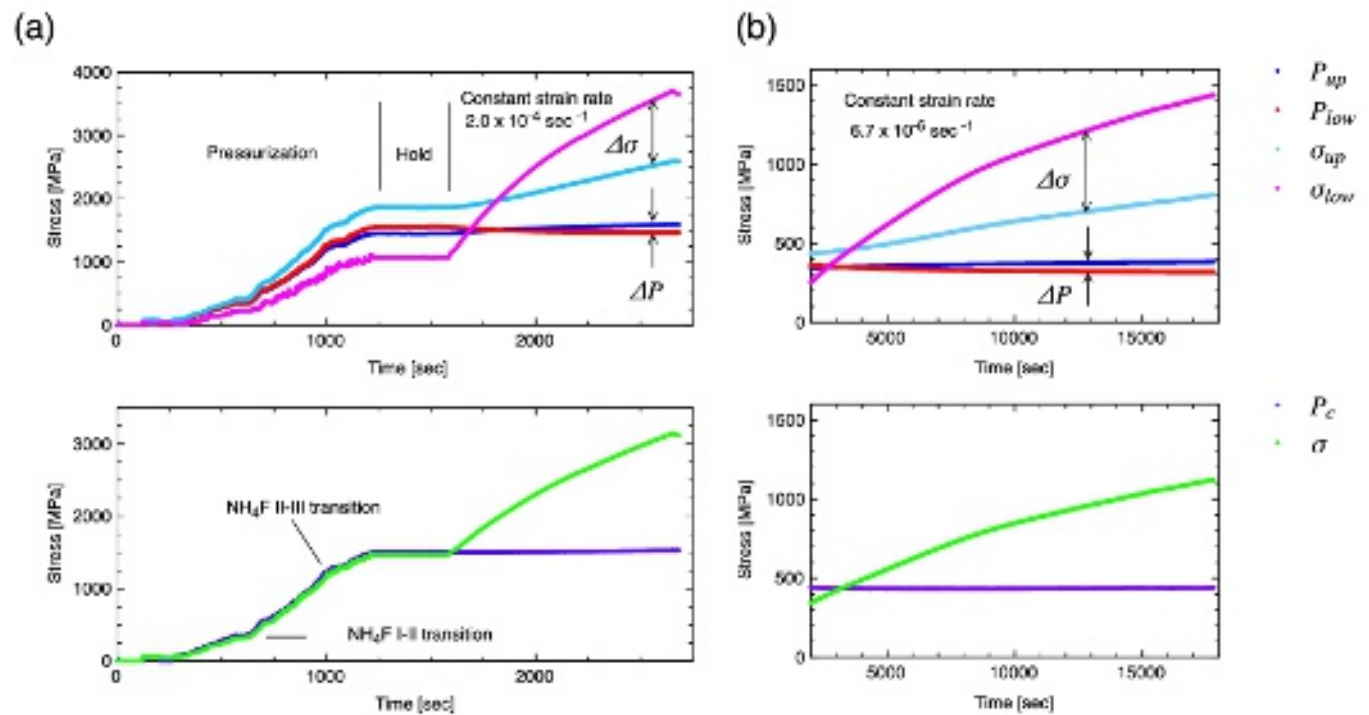


FIG. 8. Mechanical data acquired during (a) a pressure calibration test and constant strain-rate tests of Sivec marble at a confining pressure of 1.5 GPa and a strain rate of $2.0 \times 10^{-4} \text{ s}^{-1}$ (CIV07), and (b) a confining pressure of 0.36 GPa and a strain rate of $6.7 \times 10^{-6} \text{ s}^{-1}$ (CIV03).

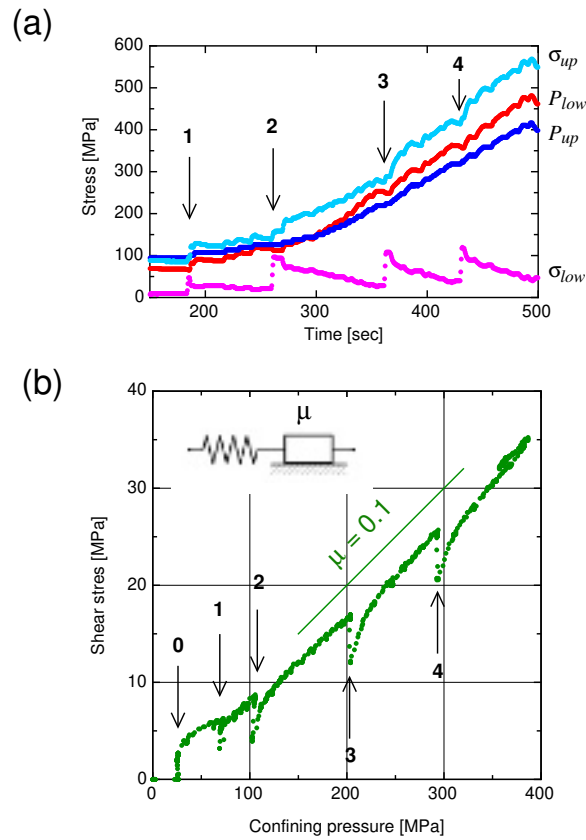


FIG. 9. Pressure stepping test (CIV04) conducted at room temperature. (a) Variations in apparent stresses during stepwise pressurization. Arrows indicate the timing of upward movements of the inner piston. (b) Changes in shear stress acting on the side surface of the sample column, calculated using Eq. (5). The inner piston was moved upward at the times indicated by the numbered arrows.

Neglecting the weight of the pressure vessel, another expression for Eq. (5) is obtained:

$$\tau = -\frac{F_{up}^{out} - F_{low}^{out}}{A_s^{out}}, \quad (10)$$

where A_s^{out} is the side area of the solid assembly. It follows that

$$-\frac{\Delta P}{\Delta \sigma} = \frac{A_s^{out}}{A_s^{in}} \cdot \frac{A^{in}}{A^{out}}, \quad (11)$$

where $\Delta P = P_{up} - P_{low}$ and $\Delta \sigma = \sigma_{up} - \sigma_{low}$. In the present case, $-\Delta P / \Delta \sigma = 0.38$. The observed changes in the outer pressures on the upper and lower sides (Figure 8d) are therefore reasonably explained by the effect of internal friction. In contrast, the net change in confining pressure during axial deformation was negligible (Figure 8d).

D. Pressure stepping test

To investigate the frictional behavior of the solid confining medium, a pressure stepping test was conducted at room temperature using an all-talc assembly (Figure 8a). In this test, the outer and inner pistons were moved alternately while the other piston was held stationary. The force data recorded by each load cell are shown in Figure 9a.

When the hydraulic ram was actuated by the hand pump and the confining pressure P_c increased, the inner pistons were kept at fixed positions. During this stage, the apparent axial stress σ_{up} increased automatically, whereas σ_{low} decreased. In contrast, when the inner pistons were moved upward at a constant velocity by the servomotor while the outer pistons were held fixed, σ_{low} increased and σ_{up} decreased. These contrasting behaviors of P_c and σ can be explained by changes in the dragging force acting on the sample column (Figure 3). Figure 9b shows the shear stress τ , calculated from Eq. (5), plotted as a function of P_c .

During pressurization by the outer pistons, τ approached an envelope with a slope of approximately 0.1. This slope is consistent with the friction coefficient (μ) of talc reported in the literature (0.1–0.2).⁴⁹ When the inner pistons were moved upward, τ deviated from the line with a slope of 0.1. The transient increases and decreases in τ associated with piston movements represent the elastic component of shear deformation in the talc confining medium.

E. Heating test

Internal friction during heating was examined using an assembly similar to that shown in Figure 5b, in which talc was used for both the inner and outer sleeves. For comparison with the room-temperature tests (Figure 9), the Sivec marble sample was not jacketed, except for copper discs placed at the top and bottom of the sample. After hydrostatic pressurization, the sample was heated stepwise to 900°C over a period of 90 minutes. Although the outer and inner pistons were held at fixed positions during heating, both the confining pressure P_c and axial stress σ increased from approximately 1.0 GPa to 1.2 GPa in response to the temperature increase (Figure 10a), due to thermal expansion. At the same time, the friction coefficient μ decreased from about 0.1 to 0.05 (Figure 10b).

F. Room-temperature experiment with a liquid cell

To clarify the effects of the solid confining medium on the mechanical data, a talc-graphite assembly enclosing high-viscosity silicone oil with a dynamic viscosity of 1 million cSt was tested at room temperature (Figure 11a). The sample was covered with a heat-shrink tube and separated from the lower alumina piston by the silicone oil. The sample was therefore expected to be hydrostatically compressed before contact with the inner piston. However, the inner stress observed was approximately 30 MPa lower than the confining pressure P_c determined from the outer load-cell measurements (Figure 11b).

After the hit point was confirmed, the inner piston was moved upward at a constant rate. When the upper and lower axial stresses increased by about 350 MPa, clear yielding was observed. Notably, even when using a “liquid cell”, $\Delta \sigma$ did

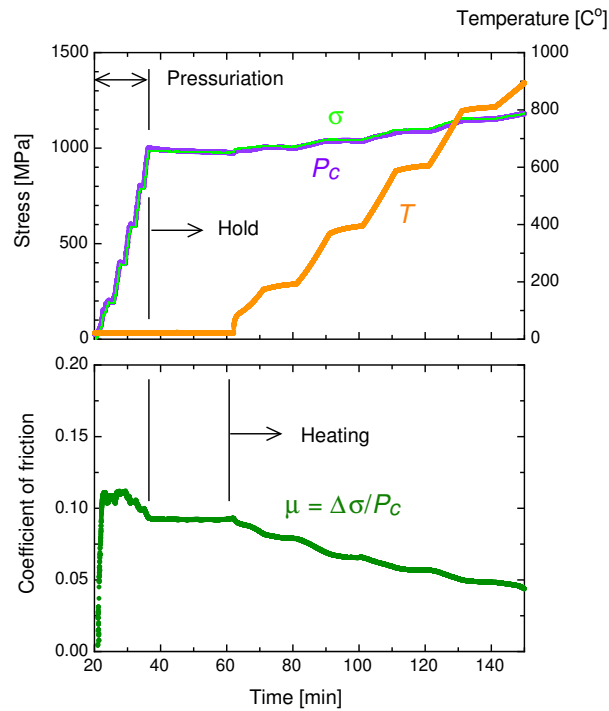


FIG. 10. Changes in axial stress (σ), confining pressure (P_c), and friction coefficient (μ) acting on the surface of the sample column during pressurization and heating of Sivec marble. Talc was used for both the inner and outer sleeves (CIV16).

not diminish, but instead remained at an approximately constant value of ~ 100 MPa after yielding. This value likely reflects internal friction within the solid parts of the assembly, particularly at the interfaces between the alumina pistons and graphite.

V. STRESS-STRAIN CURVES

A. Data processing after deformation experiments

In the standard procedure of high-temperature deformation experiments using the Kumazawa-type apparatus, P_c and σ are continuously and simultaneously increased and adjusted to the experimental conditions (Figure 8a, 10), or σ is set slightly smaller than P_c (Figure 8b, 11c), and then the inner piston is moved upward at a constant displacement rate. To construct stress-strain curves, we assume that axial deformation starts when σ exceeds P_c . The differential stress, σ_{diff} , is calculated from the real-time values of σ and P_c as

$$\sigma_{\text{diff}} = \sigma - P_c \quad (12)$$

The axial shortening strain (ϵ) is defined as the axial displacement divided by the initial length (L_0) of the cylindrical sample. Here, the displacement is calculated from the strain rate and the duration of deformation (Δt). The apparent displacement, given by $\Delta x = \dot{\epsilon} \Delta t$, includes elastic deformation of

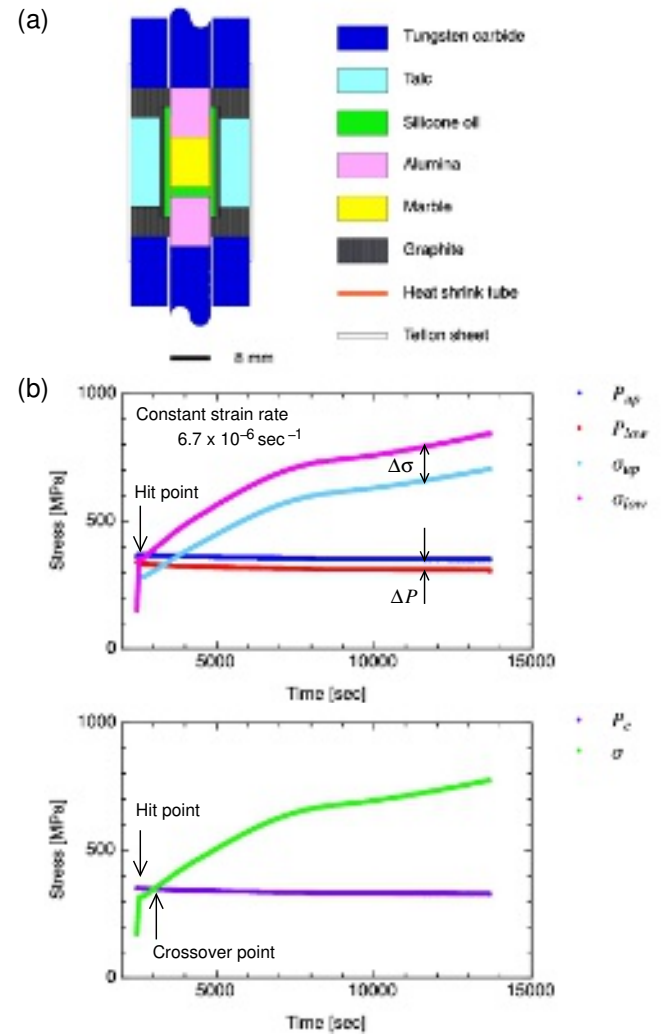


FIG. 11. A constant strain-rate test (CIV14) on Sivec marble conducted at room temperature using a silicone oil cell. (a) Sample assembly. A heat-shrink tube was used to center the sample within the assembly. (b) Mechanical data.

the entire loading system. If the machine stiffness is constant, the sample strain is derived as

$$\epsilon = \frac{1}{L_0} \left(\Delta x - \frac{A_0}{G} \sigma_{\text{diff}} \right) \quad (13)$$

where A_0 is the initial cross-sectional area of the cylindrical sample. However, the machine stiffness determined from uniaxial compression tests of a WC rod changes nonlinearly with axial force, as described above. For convenience, appropriate ranges of axial force were selected for each experiment, and G was determined by linear regression.

The Young's modulus (E) of the WC rod used for the calibration test is much higher than that of the alumina pistons used in the sample assembly, but the influence of this difference in E is negligibly small compared with the total deformation of the apparatus (Figure 6) and was therefore not considered in the stiffness correction. As the axial stress on the

WC rod varied nonlinearly with displacement, an appropriate value of machine stiffness was selected for each experiment based on linear regression within a selected stress range.

During deformation experiments, the cross-sectional area of the sample increases, leading to apparent strain hardening in the stress–strain curves. Based on a simple shape-change model, the differential stress was corrected as

$$\sigma_{\text{diff}}^* = (1 - \varepsilon)\sigma_{\text{diff}} \quad (14)$$

where σ_{diff}^* is the area-corrected differential stress (Appendix A).

Using Eq. (14), stress–strain curves of Sivec marble deformed at room temperature and 360 MPa confining pressure, using an all-talc (+NH₄F) assembly (Figure 7a) and a silicone oil + talc assembly (Figure 11a), are constructed (Figure 12a). In the experiment using silicone oil, the measured value of P_c was slightly larger than σ , and hence the differential stress given by Eq. (12) was not zero at the hit point (Figure 11b). Therefore, stress–strain curves were constructed in two ways: one assuming that the differential stress was zero at the crossover point of P_c and σ , and the other assuming that σ_{diff} was zero at the hit point. The strength of the heat-shrink tube covering the sample was neglected.

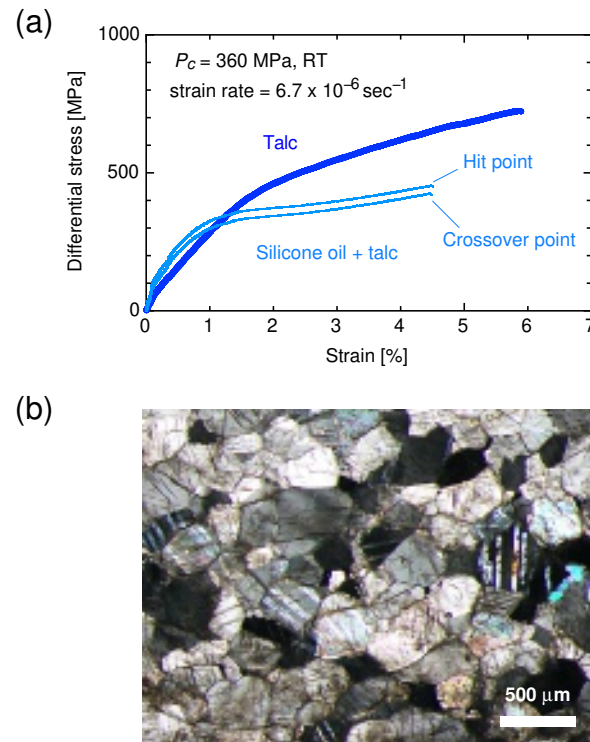


FIG. 12. (a) Stress–strain curves of Sivec marble obtained from constant strain-rate tests at room temperature using talc (CIV03) and silicone oil assemblies (CIV14). (b) An optical micrograph of deformed Sivec marble taken under crossed polarized light. The compression direction is vertical. Intra- and transgranular cracks are abundant. Twins and undulose extinction are present.

B. Interpretation of mechanical data

The stress–strain curve of Sivec marble obtained from the all-talc assembly exhibited continuous strain hardening, with the yield stress (σ_Y) around 400 MPa. The thin section of the deformed sample shows semi-brittle deformation characterized by microcracking, broad twins possibly of deformation origin, and undulose extinction (Figure 12b). In contrast, Sivec marble deformed in the silicone oil + talc assembly exhibited clear yielding at slightly lower stress (~ 300 MPa), and the differential stress remained nearly constant up to 3% strain. A shear fracture cutting through the specimen was visible after the experiment. Although talc is moderately weak, its yield strength ($\sigma_Y \simeq 150$ MPa at $P_c = 200\text{--}400$ MPa^{47,50}) is not negligible compared with the room-temperature strength of calcite rocks. It is therefore likely that shear failure of Sivec marble in the all-talc assembly was suppressed by plastic resistance of the talc inner sleeve, and that the apparent strain hardening was partly due to deformation of the talc confining medium.

The strength of calcite rocks such as marble and limestone has been extensively studied in laboratory experiments using oil- and gas-medium deformation apparatuses. At room temperature, the mechanical behavior of Carrara marble systematically changes from strain weakening ($P_c < 100$ MPa) to strain hardening ($P_c > 100$ MPa).^{50,51} The yield strength is about 350 MPa at $P_c = 400$ MPa. Our results on Sivec marble obtained at $P_c = 360$ MPa are broadly consistent with these previous studies.

VI. SUMMARY AND FUTURE PERSPECTIVES

In this paper, we described the design of the Kumazawa apparatus and its stress measurement system, and presented results of preparatory experiments obtained using a new Kumazawa-type apparatus. Two pairs of load cells placed above and below the pressure vessel revealed complex behaviors of axial stresses and confining pressures during pressurization and axial deformation. These behaviors can be explained by a spring–slider model of shear forces acting on the sample column. Generation of thermal stresses at elevated temperatures, associated with a reduction in frictional force within the solid assembly, was also captured by the real-time stress measurement system. Preliminary results of deformation experiments on Sivec marble at room temperature were discussed to demonstrate the capability of differential stress measurements using the Kumazawa-type apparatus.

In deformation experiments using the Griggs apparatus, NaCl is most commonly used as the confining medium because of its weakness at high temperatures.⁵⁰ Holyoke and Kronenberg²¹ performed calibration tests on NaCl and molten-salt cell assemblies using high-purity metals as standard samples, and compared the results with those obtained from gas-medium apparatuses at 300 MPa confining pressure to correct for the effects of internal friction. Subsequent experiments using talc as a confining medium yielded extremely overestimated stresses.⁵² Our results for the all-talc assem-

bly, showing apparently very high stresses at the loading side (σ_{low}), are consistent with these previous findings.

By using upper and lower load cells, the influence of friction was successfully reduced in the present study. However, a significant difference still remains between the mechanical data obtained from the all-talc assembly and those obtained using a liquid cell composed of silicone oil. Therefore, further improvement of sample assemblies employing weaker confining media is essential for the effective use of the Kumazawa-type apparatus. To this end, we are currently conducting calibration tests on talc, NaCl, and other confining media at high pressure and high temperature using the new Kumazawa-type apparatus.⁵³ Detailed results will be reported elsewhere.

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VII. AUTHOR DECLARATIONS

A. Conflict of Interest

The authors have no conflicts to disclose.

VIII. DATA AVAILABILITY

All data will be made available upon request to the corresponding author.

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Appendix A: SAMPLE AREA CORRECTION

Using the real-time stress measurement system, the axial stress σ is calculated from the initial cross-sectional area, A_0 , of the cylindrical sample as

$$\sigma = \frac{F}{A_0} \quad (\text{A1})$$

However, deformed samples after experiments are generally barrel-shaped and the cross-section area at the center of the sample is increased (Figure 13a). To correct the effects of sample deformation on the mechanical data, we simply assume that the volume of the sample is preserved during deformation, which is commonly assumed for plastically deforming rocks and minerals, and approximate its shape as a cylinder (Figure 13b). The corrected cross-section area, A^* , is given as

$$A^* = A_0 + \Delta A \quad (\text{A2})$$

where ΔA is an increase in cross-section area of the cylindrical sample. The axial shortening strain (ϵ , positive for shortening) is defined by

$$\epsilon = \frac{L - L_0}{L_0} \quad (\text{A3})$$

where L_0 and L are the sample length before and after deformation, respectively. Conservation of the sample volume gives

$$A_0 L_0 = A^* L \quad (\text{A4})$$

Combining Eqs. (A3) and Eq. (A4), we have

$$\frac{A_0}{A^*} = 1 - \epsilon \quad (\text{A5})$$

In the case of a uniaxial compression test ($P_c = 0$), the axial stress, σ^* , acting on the sample area A^* is balanced with the axial stress on the inner piston given by Eq. (A1) (Figure 13b, middle). Hence,

$$\sigma^* = \frac{A_0}{A^*} \sigma \quad (\text{A6})$$

Using Eq. (A5), we obtain

$$\sigma^* = (1 - \epsilon) \sigma \quad (\text{A7})$$

In the case of triaxial deformation experiments under confining pressure ($P_c > 0$), we must note that a part of the sample bulging out from the inner piston is subjected to vertical force as well as horizontal one from the confining medium (Figure

13b, right). Hence, direct application of Eq. (A7) underestimates the axial stress. The force balance on the upper half part of the deformed sample yields

$$\sigma^* A^* = \sigma A_0 + P_c \Delta A \quad (\text{A8})$$

Using Eqs. (A2) and (A5), we obtain

$$\sigma^* = (1 - \epsilon) \sigma + \epsilon P_c \quad (\text{A9})$$

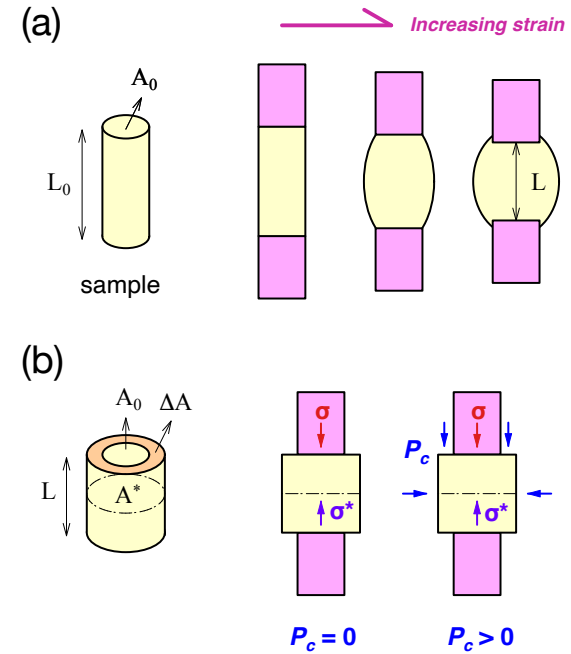


FIG. 13. Correction of the sample cross-section area for stress-strain curves. (a) Schematic illustration of sample deformation in axial compression tests. (b) A simple cylinder model used for the sample-area correction. See text for detailed explanation.

The differential stress after sample-area correction is given by

$$\sigma_{diff}^* \equiv \sigma^* - P_c \quad (\text{A10})$$

From Eq. (A9), we have

$$\sigma_{diff}^* = (1 - \epsilon)(\sigma - P_c) \quad (\text{A11})$$

Using Eq. (12), Eq. (A11) is rewritten as

$$\sigma_{diff}^* = (1 - \epsilon) \sigma_{diff} \quad (\text{A12})$$