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Method for correction of differential stress calculations from experiments using the solid salt assembly in a Griggs-type deformation apparatus

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ABSTRACT

It is necessary to perform deformation experiments in appropriate temperature and pressure conditions equivalent to the inside of the earth to characterize rheological properties of rocks. There are several types of deformation apparatus using different confining media such as gases or weak solids. Solid medium apparatus is suitable to generate high pressures and temperatures to investigate mechanical properties of rocks in crust or mantle. However, stress accuracy of solid medium apparatus was not high. Recent calibration allowed us to obtain steady-state stresses accurately within ±30 MPa. However, it was not able to reproduce initial linear region, transient and post-yield behaviors because the calibration was obtained from the comparison of stresses only at 5% strain. In this study, we performed several axial compression experiments on metals to measure differential stresses using a Griggs-type deformation apparatus with solid salt assembly (SSA). Measured stresses are consistent with results of the prior research within ±30 MPa under the identical conditions, while the measured stresses increased with the confining pressures. Obtained mechanical data were analyzed based on the viscoelastic constitutive law. Master curves were constructed from identical materials between the Griggs and gas apparatuses under normalized temperatures, strains, and confining pressures. Therefore, it is considered that differences between both master curves are derived from distinction of rheology components of two apparatuses. From the comparison between master curves, a correction method for the stresses obtained in the Griggs apparatus with SSA was derived. Applying the correction method to stress measurements of metals using the Griggs apparatus with SSA, it became possible to reproduce the stress equivalent to ones measured using gas apparatus not only at steady-state but also at initial linear region, transient and post-yield behaviors within an error of ±30 MPa. Moreover, the correction can be extended to higher confining pressure up to 1500 MPa.

1. Introduction

Mechanical behavior of rocks and minerals change dramatically at pressures and temperatures (e.g., Paterson, 1958; Griggs et al., 1960). It is necessary to perform deformation experiments in appropriate temperature and pressure conditions equivalent to the inside of the earth to characterize rheological properties of rocks. Viscoelastic structure of the crust and the upper mantle has been inferred by mechanical data of rocks obtained in laboratory so far (e.g., Kirby, 1983; Kirby and Kronenberg, 1984; Shimamoto, 1993; Kohlstedt et al., 1995; Muto, 2011; Muto et al., 2013).

There are several types of high temperature and high pressure rock-deformation apparatus using different confining media such as gases, liquids or weak solids (e.g., Tullis and Tullis, 1986). Gas medium apparatus has the highest accuracy for stress measurements because of using an internal force gauge (Paterson, 1970). However, experiments are typically restricted to confining pressures less than 400 MPa. Liquid medium apparatus has a disadvantage that it cannot be used for temperatures above 300 °C because of prevention from alteration of oil. Solid medium apparatus are comparatively easy to run and can be used at high temperatures (~1200 °C) and pressures (~2.0 GPa) and for the longest time (Tullis and Tullis, 1986). However, the accuracy for stress measurements is low mainly because of friction between the confining media and samples or loading piston (e.g., Tullis and Tullis, 1986).

Solid confining media initially thought to be weak, talc and pyrophyllite, and porous ceramics were used in experiments (e.g., Griggs and Blacic, 1965; Griggs, 1967; Blacic, 1972). However, such media were not as weak at elevated pressures as originally thought (Edmond and Paterson, 1971). Stewart et al. (2013) reported that mechanical data from talc assemblies were extremely higher than data from solid salt assemblies. Solid medium assemblies were developed for piston–cylinder apparatus using much weaker solid salts (Mirwald et al., 1975; Tullis and Tullis, 1986) or eutectic salts that melt at the experimental conditions (Green and Borch, 1989; Gleason and Tullis, 1995), providing improvements in differential stress measurements. However, deformation experiments in molten salt cell (MSC) are difficult at low strain rates and elevated temperatures, because molten salts are highly reactive with other components, such as sample jackets and graphite furnace, of sample assemblies.

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Recently, comparisons of stress measurements of axial compression experiments using a Griggs-type apparatus with solid salt assemblies (SSA) and a gas apparatus were performed (Holyoke and Kronenberg, 2010). This comparison provided a correction of differential stress measurements for Griggs apparatus with SSA. This calibration allowed us to measure steady-state stresses accurately within ±30 MPa. However, it was not able to reproduce full stress–strain curves from initial linear region to transient and post-yield behaviors because the calibration method was obtained from the comparison of stresses measured only at 5% strain. Moreover, since the calibration was carried out in low confining pressure, influence of confining pressure to stress measurements of Griggs apparatus is not clear.

Correction method for Griggs apparatus in various deformation conditions is required for revealing detailed rheological properties of the lower crust and upper most mantle rocks. Especially, the usage of SSA is suitable for doing long, low strain rate experiment because of stability of the assembly. In this study, we performed axial compression experiments on metals, then we derived a new correction method for stress measurements of Griggs apparatus by the master curve method.

2. Materials and experiments

2.1. Experimental apparatus

Axial compression experiments were performed in a solid medium Griggs-type piston cylinder deformation apparatus (Griggs apparatus). Solid salt assembly (SSA) was used as sample assembly (Fig. 1). Samples were jacketed in silver and nickel cylinders to avoid contact with NaCl pressure medium. High temperatures were achieved by the use of graphite furnace. Temperatures of samples were measured with a R-type thermocouple. Sample assemblies were brought into a pressure vessel, and a confining pressure piston ($\sigma_{2,3}$ piston) was driven by controlling a hydraulic pressure ram by a hand pump, and confining pressure was controlled. Moving rate of a loading piston (σ_1 piston, WC load piston) inside of the confining pressure piston was controlled by a motor and gears with a constant rate. Loads of samples were measured by a load cell arranged outside of the pressure vessel. Displacement of a loading piston was measured by displacement transducers fixed on an immovable platen.

2.2. Axial compression experiments

Experiments were performed on high-purity metals such as nickel and molybdenum. Similar to previous studies (Holyoke and Kronenberg, 2010), high purity metals were chosen because of their uniform properties and high thermal conductivity. Some nickel and molybdenum were purchased from Nilaco (Japan) and some nickel were same stocks of samples used in Holyoke and Kronenberg (2010). Experimental conditions are shown in Table 1.

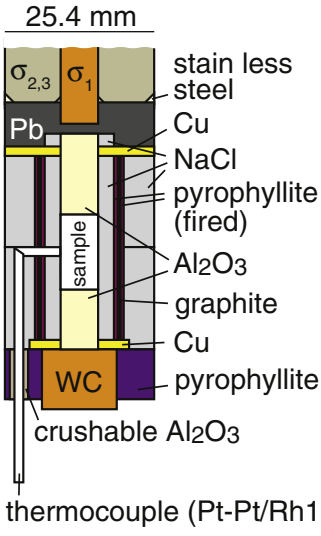

Fig. 1. Schematic of the solid salt assembly (SSA) used for axial compression experiments in this study.

Recorded load–displacement charts basically exhibit the following curves with several stages (Fig. 2). First, the axial piston begins to move and load increases rapidly related to overcome the static friction between the assembly or the lead and the axial piston (stage 1). Then, the axial piston moves into the lead and load increases linearly with gentle slope (run-in slope; stage 2). When the lead becomes very thin between the pistons, the slope of the load curve becomes steeper and penetrates the lead (stage 3). The axial piston contacts the sample and the load virtually increase linearly (stage 4). After the transition from linear to non-linear deformation (stage 5), sample exhibits creep behavior (stage 6) (e.g., Holyoke and Kronenberg, 2010; Druiventak et al., 2011; Tarantola et al., 2012).

3. Results

Obtained stress–strain curves of metals were shown in Fig. 3. Samples were shortened to strains of over 5% well beyond the linear response. Metals have yielded at about 2% strain in almost conditions. Yield stresses decrease with increase in temperatures and decrease in strain rates. Measured differential stresses increase with confining pressures (Fig. 3) at the same temperature and the strain rate. All experiments had no systematic difference in initial loading slopes (Fig. 3). Pure elastic slopes were one or two orders bigger than what is observed in the Griggs apparatus (Fig. 3a and b). Differential stresses measured at $\varepsilon = 5\%$ in all of the strain steps performed in the Griggs apparatus using the SSA were considerably higher than those measured in experiments performed at the identical conditions in gas apparatus (Fig. 4). In all experiments performed using the gas apparatus, the differential stress rises rapidly to the yield point (Fig. 4). The measured differential stresses performed by using the Griggs apparatus with the SSA did not rise as rapidly after the hit point as those results by the gas apparatus, generally reaching the yield point between $\varepsilon = 1$–2%. Yield stresses by using the Griggs apparatus with the SSA were always higher than those observed in gas apparatus experiments. After yielding, the nickel strain hardens in a similar manner as observed in the gas apparatus experiments (Fig. 4).

4. Derivation of the correction method

4.1. Outline of the analysis

In this section, we derive a new calibration method between the Griggs apparatus with SSA and the gas apparatus by applying the viscoelastic constitutive law which includes transient mechanical behavior of the entire system of apparatuses. We first introduce the secant modulus,

Table 1
Experimental conditions.

Experiment	Sample material	Confining pressure (MPa)	Strain rate (/s)	Temperature (°C)	Strength at $\varepsilon = 5\%$ (MPa)
N130403700	Nickel	1200	2×10^{-4}	700	211
N130403600	Nickel	1200	2×10^{-4}	600	294
N130416700	Nickel	300	2×10^{-4}	700	189
N130416600	Nickel	300	2×10^{-4}	600	247
N130604800	Nickel	300	2×10^{-5}	800	119
N130604700	Nickel	300	2×10^{-5}	700	132
N130604600	Nickel	300	2×10^{-5}	600	202
N130821700	Nickel	1200	2×10^{-6}	700	182
M140324700	Molybdenum	1500	2×10^{-4}	700	502
M140324600	Molybdenum	1500	2×10^{-4}	600	659
N140819700	Nickel	1500	2×10^{-4}	700	267
N140819600	Nickel	1500	2×10^{-4}	600	328