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Instrumental modifications for compressive testing under hydrostatic confining conditions

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Rsum. — L'appareil de dformation sous pression de confinement conu par D. T. Griggs [1] pour l'tude des roches et des minraux a t modifi et adapt l'tude de la plasticit des alliages mtalliques et des semi-conducteurs. Ces modifications utilisent les possibilits d'une machine de dformation commerciale et augmentent la rigidit du montage. En outre, grce une diminution des frottements, nous pouvons dtecter des contraintes d'coulement d'un ordre de grandeur plus faible. De plus, une microvanne de fuite asservie une rampe permet une dpressurisation lente et rglable de l'enceinte de confinement de manire liminer la tendance la fissuration en fin d'exprience.

Abstract. — The device originally designed by D. T. Griggs [1] in order to deform minerals in compression under a confining pressure has been modified and adapted to the study of the plasticity of metal alloys and semi-conductors. The changes take advantage of a commercial testing machine, they also ensure an improved rigidity of the machine. In addition, friction has been reduced to such a level that the minimum flow stress which is now detectable has dropped by one order of magnitude. In complement, a servocontrolled microleak permits an adjustable low depressurization of the confining medium and eliminates the destruction of the samples by fracture which occurs usually at the end of a standard test.

1. Introduction.

Amongst all available techniques, uniaxial compression under normal pressure has proved to offer the largest convenience and potentialities for testing the mechanical properties of the widest range of materials. However, in the case of many materials of interest, as for example semi-conductors, some ambiguity remains after conventional compression tests on the extent of the role played by dislocation climb as an additional degree of freedom during deformation. This is mostly because of their brittleness which prevents the temperature of the tests from being lowered enough to allow for extensive motion of dislocations freed from their interactions with diffusing point defects. More generally, there is a strong demand for characterizing the plastic behaviour of brittle materials at low temperatures where the plasticity is governed by parameters strictly related to the crystal structure without being altered by diffusion.

It is well established that the temperature below which a brittle crystal fails by fracture can be significantly lowered through the superimposition of an

hydrostatic pressure to the applied stress. It has been shown, for example, that the plasticity of sapphire, spinel and silicon could be investigated at temperatures unusually low for these crystals using Knoop or Vickers microindentation tests [2-5]. However, an indentation test remains intrinsically limited, at least in the way the hydrostatic and shear components of the stress are interrelated, together with associated uncertainties on the determination of the activation parameters of deformation. Furthermore, the indentation test is known to minimize fracture but in the case of very brittle materials, it does not prevent cracks from appearing and propagating during the application of the load. Thus, it is highly preferable to make use of devices which allow for separate controls both of the uniaxial stress and external pressure. This has already been achieved by geologists whose designs were built with the aim of simulating in the laboratory, the plastic behaviour of rocks under geological conditions. Amongst all variants, two distinct classes of techniques are offered, the distinction between which relies on whether the confining medium is gaseous or solid. As far as friction is concerned and

compared to the latter solution, the former has the advantage of optimizing the overall resolution of the test. However, since solid medium confinements are drastically easier to work up than gaseous ones, at a comparable level of pressure, they are often preferred for the sake of simplicity.

The most versatile version of solid medium systems is the so-called Griggs machine [1] which was originally designed 30 years ago and subsequently adapted to a range of moderate temperatures (20-950 °C) [6]. The potential this machine offers to more conventional materials science applications has only been explored during the last few years. Significant permanent strains have been obtained by uniaxial compression at temperatures well below the minimum temperature of deformation attainable by deformation at room pressure, where dislocation multiplication operates exclusively by glide. For instance, the lowest deformation temperature in sapphire, spinel and silicon are room temperature [6], 100 °C [7] and 275 °C [8, 9] instead of 1 400 °C, 1 600 °C and 420 °C under normal pressure, respectively. In addition, a further improvement one expects from Griggs-type experiments is the dramatically large level of stress which can be applied to « classical » ductile metals or alloys in which very high dislocation densities may be introduced [10]. Indeed, one can reasonably anticipate to apply axial compressive stresses as large as 5 to 10 GPa under a confining pressure of 2 GPa at room temperature and thus to modify the controlling deformation mechanism as predictable for example from inspection and extrapolation of deformation maps. At last, this device has proved very powerful when applied to the study of phase transition in minerals. Similar work on metals or compounds, as for example shear transformations associated with volume changes could be carried out. This requires the accuracy of the measurements to be improved, in particular in the way the applied load is discriminated from the signal arising from the confining pressure as well as in the way the confining pressure is adjustable. However, the standard Griggs machine suffers the limitations of its age.

This paper deals with several alterations or improvements which we have brought to the early Griggs design in order to take advantage of modern equipment and to render the environmental conditions less critical for the samples. In fact, we have observed that fast depressurization rates resulted in damaged samples mostly sliced perpendicular to the load axis (Si, spinel, GaAs). Therefore, the application and control of the pressure, especially during the pressurization and depressurization procedures, together with the configuration of the solid confinement, have received particular attention with the aim of providing transmission electron microscope (TEM) thin foils as free of cracks as possible. In addition, the control of the mobile crosshead speed, i.e. of the deformation rate, takes advantages of all the options of a commer-

cial deformation device. Hence, the design which we describe in this paper offers the advantage of being adaptable as a standard accessory to common laboratory equipment.

Schematically, the Griggs type apparatus involves three distinct functional members (Fig. 1) :

(i) the pressure chamber inside of which the sample is deformed in compression under a confining solid medium at varied temperatures.

(ii) the mechanical application of the uniaxial stress by means of a mobile crosshead and of a set of pistons and end pieces.

(iii) the hydraulic part which controls the pressure in the confining solid medium.

In the following, these members will be described in separate sections. The reason for which some elements have received no alterations as well as the improving modifications which we have carried out will be discussed in details.

2. The pressure chamber (Fig. 2).

The pressure vessel (2), the base plate (1) and the pressure piston (3) have not suffered major modifications : steels employed for the cylinder and the safety ring have been changed into their european correspondances (819 B and Marval 18, Aubert et Duval) and their dimensions have been rounded to SI units. The sample dimensions (10) have been reduced from $9.6 \times 3.7 \times 3.7 \text{ mm}^3$ to $8 \times 3 \times 3 \text{ mm}^3$ (in some instances to $5 \times 2 \times 2 \text{ mm}^3$). Indeed, as far as glide line trace analysis and/or subsequent transmission

Fig. 1. — Schematic view of the deformation device : (i) : pressure vessel, (ii) : uniaxial force rod attached to the mobile crosshead, (iii) : hydraulic jack fixed on the pressure frame.

Fig. 2. — Pressure chamber. 1. Base plate. 2. Pressure vessel. 3. Pressure piston. 4. Force piston. 5. Lead cylinder. 6. Copper ring. 7. Upper alumina piston. 8. Graphite furnace. 9. Sample jacket. 10. Sample. 11. NaCl confining medium.

12. Lower alumina piston. 13. Pyrophyllite outer assembly. 14. Copper ring. 15. Pyrophyllite lower ring. 16. WC lower piston. 17. Thermocouple. 18. 19. Cu-Be seals.

electron microscopy are concerned, this volume of materials remains a reasonable one to work with. Although no control measurements with sufficient accuracy can be performed to prove it, it is obvious that, since the total dimensions of the solid assembly together with these of the furnace (8) have not been noticeably modified, the pressure and temperature variations on smaller samples are reduced. In addition, given the maximum load measurable with the cell (10^5 newtons in the present configuration), the reduced section of the samples permits to increase by a factor 1.5 to 3.4 the level of the applicable engineering stress. The samples (10) are jacketed in a metal cylinder (Ag, Al $\phi = 5$ mm) inside of which a square hole (3×3 mm²) has been carefully bored by extrusion or electrosparking. The choice of the metal out of which the jacket (9) is machined is governed, at the temperature of the test, by its undesired diffusion into the sample during the course of the experiment and by its ductility, i.e. its ability to transmit the environmental pressure.

The uniaxial compressive load is applied to the sample through alumina end pieces (AF 997-Desmarquets, (7) and (12) in Fig. 2). Their diameter has been reduced accordingly to 5 mm in order to lower the resistance to their displacement in the solid confinement. The graphite furnace (8) is embedded in between an inner (11) and an outer (13) tube which both ensure electrical insulation of the furnace and transmit the

external confining pressure when the pressure piston (3) is lowered. In fact, we have checked that, in order to transmit the lateral pressure correctly, the outer tube must not be made of sodium chloride. Therefore, for sake of simplicity, the outer cylinder (13) has been systematically machined out of fired pyrophyllite. In addition, the most satisfactory configuration we have tested corresponds to an inner tube made of sodium chloride with an external diameter of 12 mm. Boron nitride has also been tested and revealed unadapted to low temperature deformation under pressure especially with regards of friction.

The temperature is measured in the vicinity of the sample with a thermocoax or a thermocouple (17) located as close as possible to the lateral surface of the jacket and electrically insulated with an alumina tube. Two different couples are used (chromel-alumel and Pt-PtRh 10%), the temperature is monitored to within ± 0.5 °C using a Drusch control with small derivation and integration time constants (1 s and 10 s respectively). Indeed, the nature of the assembly provides a very fast response of the thermocoax temperature to a pulse of electrical power (for 350 W, a temperature of 400 °C could be reached in 5 min).

Friction depends critically on the diameter of the carbide force piston (4) whereas it is not affected significantly by changes in diameter of alumina rods (7, 12). Accordingly, we have reduced the former diameter to 3 mm. In addition the sealing rings (18, 19)

have been machined out of beryllium-bronze which has a very low friction coefficient compared to stainless steel (see § 6).

More details concerning the assemblies as well as the experimental procedure to assemble them are given in [6].

3. The application of the uniaxial load.

The frame which provides the pressure into the vessel has been fitted in an Instron testing machine (Model 1195, 10^5 newtons). Therefore, the application of the load at a given strain-rate takes full advantage of the flexibility brought by the commercial electronic controls instead of the limited set of fixed hand-operated gears of the original machine. The strain-rate $\dot{\epsilon} = v_T/l_0$ is then adjusted by varying the crosshead speed v_T . The smallest strain-rate accessible with a sample length l_0 of 8 mm is about $2 \times 10^{-6} \text{ s}^{-1}$. As described below in the experimental procedure, a servocontrol of the stress or of the strain has to be used in addition to the usual Instron control units, in order to operate the machine once the vessel is being pressurized or depressurized.

The load is measured with a 10^5 newtons gauge (G in Fig. 3b, maximum absolute stress 11 GPa, on a sample with 9 mm^2 section). It is applied on the sample through a tungsten carbide piston (4) and through the alumina rods (7, 12) describes in the previous section (Fig. 2). The high density alumina rods (AF 997) behave satisfactorily even at the maximum applicable load.

In the original Griggs machine, the mobile loading piston (LP) moves through the inner hole of a hydraulic jack (J), itself attached to the upper fixed crosshead (FC). Insofar as the experimental procedure is concerned, this configuration is probably the most convenient to use because the force piston does not move with respect to the sample during pressurization (Fig. 3a, see also § 5).

However, since small misalignments and since slight deviations from perfect axially are practically unavoidable, the longer the loading piston, the more likely its lateral motion by flexion at very high loads. We have thus chosen to reduce the total length of the upper (mobile) loading piston (Fig. 3b). The maximum gain in length is obviously obtained once the pressure vessel is held upon the jack, that is in between the gauge and the jack (Fig. 3).

The consequence this modification has upon the experimental procedure is important and will be discussed below.

4. The control of the pressure in the vessel.

The pressure is applied on the sample through the solid medium, itself confined inside the chamber delimited by the pressure vessel (2), the base plate (1) and the annular pressure piston (3) (Fig. 2). The pressurization operations start by lifting the whole

Fig. 3. — Schematic view showing the two distinct basic configurations, 3a : original version, the load piston (LP) goes through the jack (J), 3b : present version, the jack is attached to the lower part of the rigid frame. The length of the mobile loading piston is that between the gauge (G) and the vessel (V), through the fixed frame crosshead (FC) only. The gain in loading piston length, $h_a - h_b$ is roughly $h_a/2 \approx 40 \text{ cm}$ (mobile crosshead : MCH).

assembly at a fast speed with the hydraulic jack. Once the pressure piston is in contact with the fixed crosshead of the pressure frame, the latter reacts against the force provided by the jack. The upward motion of the

Fig. 4. — Pressure circuit. — air, — oil. R9 and R126.5 are pneumo-hydraulic pumps with low and high strengths

respectively. R1502 : hydraulic jack. R : oil tank. C : pressure gauge.

jack together with that of the mobile crosshead are then carefully monitored in order to slowly increase the pressure in the cell (more details on the experimental procedure are given in section 5).

The hydraulic jack is operated by pneumohydraulic pumps according to the circuit schematized in figure 4. The upward and downward motions of the jack piston are operated by two separated pumps with different strenghts.

One of the most important experimental precautions one has to take in the course of a test consists in reducing the potential pressure heterogeneities which invariably take place in the solid confinement during the depressurization procedure. The solid medium transmits but imperfectly the pressure, then, the time constants required to homogeneously reequilibrate pressure drops are very long (several minutes) especially at moderate temperatures. As mentionned in the introduction, the occurrence of heavily fractured and sliced samples resulting from poorly controlled depressurizations can be avoided by decreasing the pressure as slowly as possible. Particular attention has thus been paid to the control of the hydraulic leak designed to allow for the pressure drop. The hydraulic circuit is equipped with a microleak valve which has been partly rebuilt to be operated by a stepped electric motor (Fig. 5). The rotation of the valve is assigned to the linear variation of an adjustable D.C. signal. A command electronic device has been especially designed which drives the valve such that any deviation from the

Fig. 5. — Detail of the depressurization function. VHP : microleak valve actionned by a stepped motor (RM). ET : crosshead electronic control. CH1 and CH2 : hydropneumatic valves. Pressurization : CH1 open, CH2 closed. Depressurization : CH1 closed, CH2 open, VHP monitored.

linear slope is corrected proportionally to the error signal and to its derivative (time constant ≈ 10 min). The resulting variation of the pressure is shown in figure 6 (part E). Typically, the time for the pressure to

return from 1.5 GPa to ambient (0.1 MPa) can be delayed up to 10 hours. The slope of the pressure *versus* time is satisfactorily linear over 3/4 of the depressurization procedure. The initial drop is almost unavailable since it depends upon built-in uncertainties of the closed position of the microleak which, indeed, depends upon the hydraulic pressure to which it is submitted.

The principal advantages of the improvements we have brought to the hydraulic part are to dampen the pressure variations and to render the leak both adjustable and linear.

The pressure measurement has been calibrated to several pressure standards (NH_4F , RbCl) at room temperature. Both check points are consistent and reproducible.

5. Experimental procedure (Fig. 6).

The description of the experimental procedure is restricted to these operations which are specific to the present version of the Griggs machine.

Once the whole assembly is set upon the hydraulic jack, it is pneumohydraulically lifted up towards both the fixed and mobile crossheads. The present configuration has the inconvenience that the sample moves towards the piston attached to the mobile crosshead. There is therefore a serious risk of deforming or breaking the sample during this operation. Thus, provided the mobile piston has been appropriately raised, the pressure piston lying on the assembly is brought in contact with the fixed crosshead. Before keeping on lifting the hydraulic jack, the mobile crosshead is moved down to contact with the assembly. Then, the mobile crosshead keeps on moving in register with the hydraulic jack motion. This is

Fig. 6. — Typical force-time and pressure-time curves recorded in the course of the deformation test. (a.u. : arbitrary units). The full line represents the pressure, the dashed line is the signal delivered by the force gauge. A : pressurization. B : the mobile crosshead is lowered, the discontinuity results from and is a measure of friction ; the deflection from linearity at the end of stage B (HP : hit point) corresponds to the situation where lead has disappeared between the carbide and the alumina pistons. C : stress-strain curve followed by a stress relaxation. D : the force on the sample is adjusted such that σ is slightly positive (see text). E : servo-controlled depressurization (inset : details of the variation of the force applied on the sample (a) compared to the driving ramp signal (b), see text).

accomplished by assigning the output signal of a LVDT transducer measuring the distance between the mobile crosshead and the vessel to a constant value (strain control mode of the strain/stress control Instron facility). Once the desired temperature and pressure are set up (A in Fig. 6), the strain control is switched off and the determination of the point at which the mobile piston impinges the alumina rod (i.e. the hit point, HP) is carried out in the usual way (disappearance of a lead disc which is previously inserted in between the two parts). The deformation test is then carried out, which we end up with a relaxation experiment (C). The data are continuously recorded with a computer.

At the end of the experiment, the sample is brought back to ambient pressure and room temperature in such a way that the deviator $\sigma = \sigma_1 - \sigma_3$ is positive and proportional to σ_3 (typically $\sigma \simeq 0.3 \sigma_3$). The axial stress σ_1 on the sample is decreased to the desired value $\sigma_3 + \sigma$ (D) and then monitored by entering $\sigma_3 + \sigma$ through a spare input of the stress/strain control unit working now in the stress control mode (E).

Thus, the load on the sample is servocontrolled proportionally to the pressure signal which itself decreases linearly at the rate of the monitored leak.

6. Results.

It is not the aim of this paper to account for new results on plastic deformation. Rather, selected examples are given in order to illustrate the improvements of the machine and of the control of the environmental conditions for the samples. Experiments have been carried out on equimolar MgAl_2O_4 oriented for easy glide at 400 °C and on silicon at 300 °C on the present version of the device ; they have yielded experimental determinations of the flow stress for these which are in excellent agreement with identical tests carried out on the original Griggs machine [7, 8]. In addition, we have successfully reproduced the experiments with the largest $\sigma_1 - \sigma_3$ values which shows that there is no potential restriction to the field of applications of the modified system.

The careful control of the depressurization conditions has permitted, for the first time, to carry out deformation of GaAs at room temperature without dramatic fracture being present in the sample [11]. This allows to cut thin foils for post-mortem TEM observations in any plane of interest, i.e. slip plane, cross-slip plane... An illustration is given in figure 7, where wavy slip plane lines can be observed within a modest density of cracks ; these were produced after uniaxial deformation was stopped since the slip lines are in register across the cracks. When no special care is paid during depressurization, the sample turns into powder, which prevents any further TEM work from being carried out.

Presently friction has been reduced from 5 ± 1 kN to approximately 0.6 kN by means of the modifications

Fig. 7. — Slip lines on a lateral face of GaAs deformed at room temperature, $\dot{\epsilon} = 2 \times 10^{-5} \text{ s}^{-1}$ and $\sigma_3 = 1\,400 \text{ GPa}$ [11].

described in section 2. This enabled us to deform silicon at 550°C under 0.6 GPa with the excellent resolution shown in figure 8. In contrast, given the same temperature, strain-rate, confining pressure and confining assembly, the signal does not emerge from friction when both a piston with a diameter of 5 mm and stainless steel seals are employed.

7. Conclusion.

The device presented in this paper is an adaptation of the original Griggs machine which takes advantage of a commercial strain load control system. The reduction of the dimension of the samples together with the subsequent changes in the environmental configuration reduces the large uncertainties in temperature and pressure known to be present in the former design. Friction has been reduced by one order of magnitude at 550°C , this reduction depends on the temperature

Fig. 8. — Corrected deformation curve of silicon deformed at $T = 550^\circ\text{C}$, $\sigma_3 = 600 \text{ MPa}$ and $\dot{\epsilon} = 2 \times 10^{-5} \text{ s}^{-1}$ showing the sensitivity of the apparatus. $S_0 = 9 \text{ mm}^2$, $l_0 = 8 \text{ mm}$.

of the test. The inversion of the application of the pressure with respect to that of the load has reduced by a factor of about 2 the length of the outer piston, hence the risk of mechanical misalignment of the load has been minimized. At last, the successful design of the pressurization and, more important, of the depressurization controls has permitted to study almost any brittle material with the open possibility of further TEM investigation. Experiments are presently being done to apply this technique to the plasticity of selected metals and alloys.

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