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Deformation of single crystal sample using D-DIA apparatus coupled with synchrotron X-rays: In situ stress and strain measurements at high pressure and temperature

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ABSTRACT

We present a technique for high pressure and high temperature deformation experiment on single crystals, using the Deformation-DIA apparatus at the X17B2 beamline of the NSLS. While deformation experiments on polycrystalline samples using D-DIA in conjunction with synchrotrons have been previously reported, this technical paper focuses on single crystal application of the technique. Our single crystals are specifically oriented such that only [1 0 0] slip or [0 0 1] slip in (0 1 0) plane is allowed. Constant applied stress (sigma <300 MPa) and specimen strain rates were monitored using in situ time-resolved X-ray diffraction and radiography imaging, respectively. Rheological properties of each activated slip system in the crystals can be revealed using this technique. In this paper, we describe the principle of sample preparation (e.g. $[1\,1\,0]_c$ and $[0\,1\,1]_c$ orientations) to activate specific slip systems (i.e. [1 0 0](0 1 0) and [0 0 1](0 1 0), respectively), stress measurement and procedures of the deformation experiments.

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1. Introduction

Strain rate and stress are two critical parameters for high pressure (P) and high temperature (T) deformation experiments. Before in situ synchrotron high pressure experiments the maximum pressure for such deformation experiments was limited up to 3 GPa. Recently, developments of the Deformation DIA (D-DIA) and in situ X-ray measurements at synchrotron beamlines [2,5,14–16] have expended the pressure range beyond 10 GPa [11]. The geometry of the D-DIA is well adapted to bring the X-ray beam onto the sample and for stress and strain measurements. Sample dimensions and hence strain can be measured from time resolved X-ray radiography imaging and stresses are determined using multiple X-ray diffraction of polycrystalline sample. The X-ray diffraction defines the elastic strains of a particular subset of grain in the sample oriented along the diffraction vector. The differential stress is derived from the elastic strains of two subsets of grains with different orientations relative to principle stress and elastic constants [6]. This type of experiments required a bright high energy X-ray source only delivered by synchrotron to penetrate through the pressure medium of D-DIA.

Deformation experiments on single crystals offer the possibility to reveal flow properties of individual slip systems. Such information is critical for modeling polycrystalline aggregate. However it is not trivial to derive sample stress from single crystals diffraction. Therefore, the deformation cell assembly has to be modified to include a polycrystalline stress sensor in the deformation column.

In this paper we describe the technique for single crystal deformation experiment at synchrotron X-ray beamline.

2. Samples orientation and preparation

To be able to activate and study a particular slip system, we have to deform the single crystal along the corresponding orientation. Fig. 1 shows the geometrical relation between a slip system and the principal stress. The Schmid factor $S_{schmid}=\cos\chi\cos\alpha$ represents the coefficient of the applied stress on the slip direction in the given plane. In order to have the highest activity of the slip system, during a uniaxial deformation experiment, the slip plane and the slip direction have to be oriented at 45° to the compression axis (Fig. 1). At this

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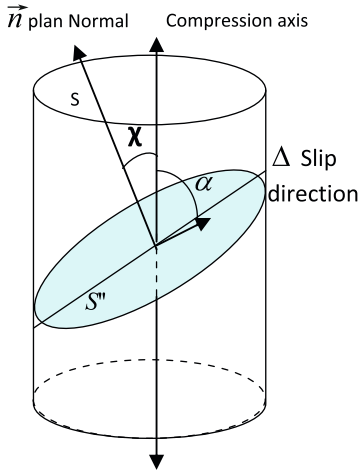

$$\sigma_{applied} = F$$
$$\tau = \sigma_{applied} \cos\chi \cos\alpha$$
$$S_{schmid} = \cos\chi \cos\alpha$$

Fig. 1. Schmid factor calculation. The total stress is vertical, S'' plane (in blue) is the slip plane and, τ is the stress projected in the slip plane and slip direction. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

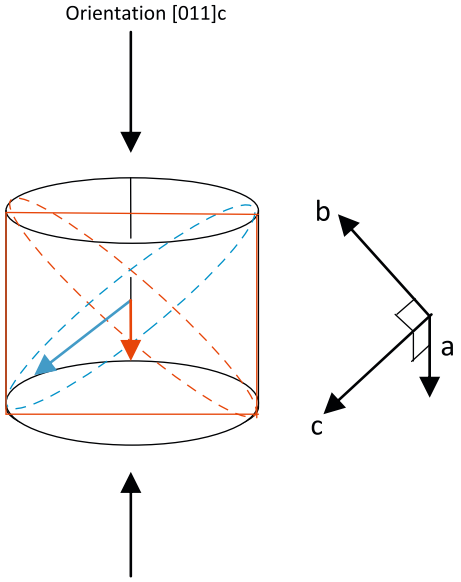

Fig. 2. Representation of the orientation $[0\,1\,1]_c$ with the different slip systems studied. The slip system represented in blue is [0 0 1](0 1 0), which is mainly activated in this orientation. In red are represented the slip plane and slip direction for [1 0 0](0 1 0), [100](0 0 1) and [0 0 1](1 0 0). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

condition the slip system experiences the maximum shear stress and the Schmid factor S reaches its maximum value of 0.5.

In our experiments we use the single crystal olivine sample, which has an orthorhombic structure (*Pbnm*). In olivine, the most active dislocation slip systems are [1 0 0](0 1 0) and [1 0 0](0 0 1), i.e. slip along the a direction in (0 1 0) and (0 0 1) planes, together with [0 0 1](0 1 0) and [0 0 1](1 0 0), i.e. slip along the c direction in (0 1 0) and (1 0 0) planes [9–11]. Slip along *b* direction is impossible because *b* vector is too long to be a Burgers vector. In the olivine, three different orientations can be used to activate the above slip systems. These orientations are $[1\,1\,0]_c$, $[0\,1\,1]_c$ and $[1\,0\,1]_c$, which represent the direction of the single crystal along the compression axis in a cubic structure for simplified notations. Here we illustrate how to activate only the [1 0 0](0 1 0) slip system and prohibit all the other three. As shown in Fig. 2, when the crystal is orientated in the direction such that the $[1\,1\,0]_c$ is

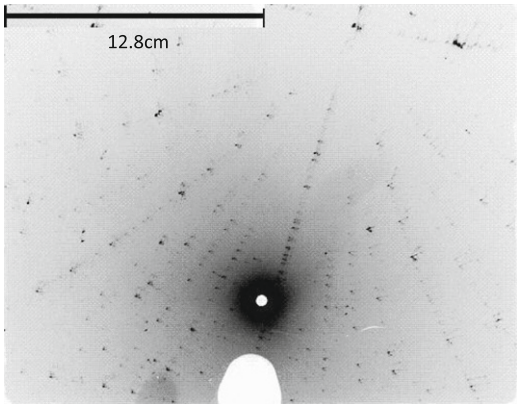

Fig. 3. Example of X-ray diffraction pattern for the orientation $[1\,1\,0]_c$.

along the compression axis, the Schmid factor for [1 0 0](0 1 0) slip system is equal to 0.5 because the angle between the compression axis and the slip direction α is equal to 45° and the angle between the normal of the slip plane and the compression axis χ is equal to 45°. However for [0 0 1](0 1 0) slip system the angle between the compression axis and the slip direction α is equal to 90°; therefore the Schmid factor is equal to zero. For the [0 0 1](1 0 0) slip system, the slip plane is at 90° of the compression axis therefore the Schmid factor is equal to zero. For [1 0 0](0 0 1) the slip plane is at 45° of the compression axis but the slip direction is at 90°; therefore the Schmid factor is equal to zero. Similarly, when the crystal is orientated in the direction such that the $[0\,1\,1]_c$ is along the compression axis, the Schmid factor for the [0 0 1](0 1 0) slip system is equal to 0.5. However when the crystal is orientated in the direction such that the $[1\,0\,1]_c$ is along the compression axis, the Schmid factors for [1 0 0](0 0 1) and [0 0 1](1 0 0) slip systems are both equal to 0.5. These two slip systems are activated simultaneously. In order to study and to activate the respective properties of *a* and *b* slips, we chose to study first the orientation $[1\,1\,0]_c$, and then $[0\,1\,1]_c$.

The orientation of the single crystal is determined by X-ray diffraction using an in-house X-ray tube in reflection mode. The current and voltage of the X-ray tube are purposely set low enough to avoid the characteristic X-ray emission of the Mo target so that a white X ray source is used for diffraction. Diffraction patterns are collected on flat imaging plates (Fig. 3). The indexing and the orientation are solved with the software Orientexpress [7]. Allowable uncertainty of orientation in the program is set to 2°. The oriented single crystal is sliced while it is held on the goniometer head. Then cylindrical samples are obtained by core drilling, and both ends of the cored cylindrical sample are polished.

3. High pressure deformation of single crystal using Deformation-DIA press

The D-DIA press [14] is a cubic type multi-anvil apparatus. The top and bottom anvils can be driven independently to the main ram by two differential pumps (see Fig. 4). For deformation experiments at high pressure the six anvils are driven simultaneously by the main ram to achieve the desired pressure. Then the top and bottom anvils are forwarded independently to apply a uniaxial stress on the pressured cell and the four lateral anvils move backward to maintain a constant pressure in the cell.

The cubic pressure medium is made of a mixture of amorphous boron and epoxy resin (4:1 in weight) with 6.15 mm edge. The single crystal sample is loaded between two crushable alumina pistons in the pressure medium (Fig. 5). A cylindrical graphite furnace is used to heat up the sample and the temperature is

measured with two lateral thermocouples (W3%Re–W25%Re). The crushable alumina pistons accommodate the initial deformation during cold compression. The pistons are hardened at high temperature so that they can deliver the differential stress generated by the motion of top and bottom anvils.

4. Rheological properties of single crystal measured using X-ray synchrotron beam

The deformation experiments are conducted at the superconductor beamline (X17B2) [2] of the National Synchrotron Light Source (NSLS, Brookhaven National Laboratory, New York). High energy white X-rays are used to measure the sample stress and strain rate (Fig. 7).

5. Stress determination

To derive the stress applied on the single crystals a powdered material is needed in the deformation column as stress sensor to perform diffraction during the deformation at high pressure. The cell assembly is shown in Fig. 5. There are two polycrystalline alumina pistons, two single crystal samples and one layer of powder stress sensor in the compression column. The alumina pistons can also serve as stress sensors to measure the stress at different points in the assembly.

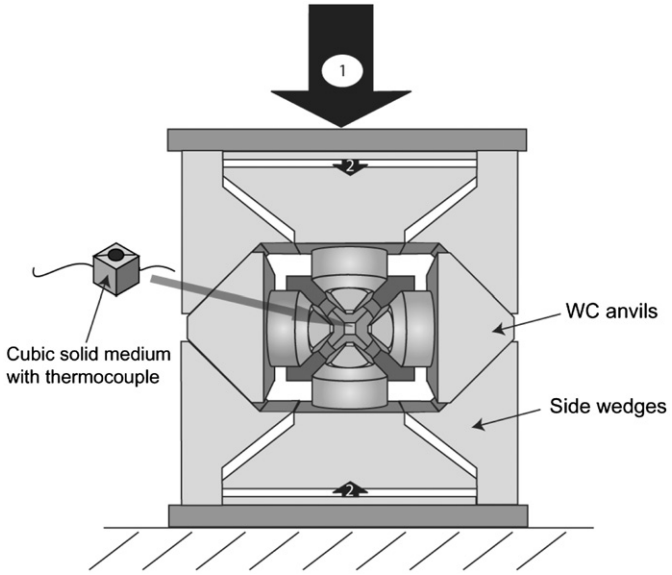

Fig. 4. Schematic in 3D of the Deformation-DIA press (Courtesy to Hélène Couvy).

The size of the incident X-ray beam is defined by a set of upstream slits to 100 μm × 100 μm. X-ray beam illuminates the sample through the front anvil gap. Four diffraction patterns are collected simultaneously at different azimuths angles of the diffraction cone (Fig. 7). Two detectors are located along the vertical anvil gap (at $\varphi \approx 0$ and 180° azimuths) and two are located in the horizontal plane (at $\varphi = 90$ and 270° azimuths). In order to receive the diffraction signal in the horizontal plane, the two back anvils have to be transparent to X-rays (high energy). Regular anvils for cubic type apparatus are made of WC. Therefore the back anvils in our experiments are replaced by those made of sintered cubic BN. The diffraction cone is collimated through a conical slit with a gap of 50 μm, which ensures the four detectors sample the same volume in the polycrystalline stress sensor. Technical details are presented in Chen et al., [2].

During deformation, the stress field along the deformation column is assumed to be uniform. Therefore the stress experienced by the stress sensor represents that of the sample. Differential stress induces different microscopic lattice strains along different orientations in the stress sensors. By measuring X-ray diffraction in different azimuths we can derive the differential stress. In the D-DIA the deformation is along the vertical axis. The principle stress is σ_3, parallel to the compression axis. σ_1 and σ_2 are in the horizontal plane, normal to the compression axis. Assuming the stress field is cylindrical symmetry then σ_1 and σ_2 are equal. Thus the stress field in the D-DIA cell (pressure cell) can be expressed as

$$\sigma_{ij} = \begin{bmatrix} \sigma_1 & 0 & 0 \\ 0 & \sigma_2 & 0 \\ 0 & 0 & \sigma_3 \end{bmatrix} = \begin{bmatrix} \sigma_p & 0 & 0 \\ 0 & \sigma_p & 0 \\ 0 & 0 & \sigma_p \end{bmatrix} + \begin{bmatrix} -t/3 & 0 & 0 \\ 0 & -t/3 & 0 \\ 0 & 0 & 2t/3 \end{bmatrix}$$

$$\sigma_p = \frac{1}{3}(\sigma_1 + 2\sigma_3) \text{ and } t = \sigma_3 - \sigma_1$$

where σ_p is the hydrostatic pressure and t is the differential stress.

The lattice strain induced by the deviatoric stress can be determined by measuring the d spacing $d_m(h\,k\,l)$, and is given by the following relation:

$$(d(hkl)_m - d(hkl)_p)/d(hkl)_p$$

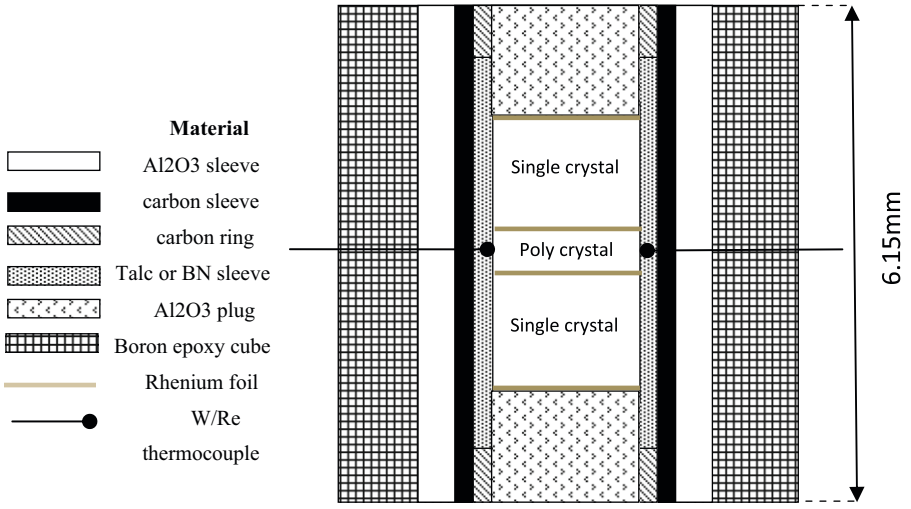

Fig. 5. Schematic cut of a D-DIA cell.

where $d(h\,k\,l)_p$ is the d spacing under pressure σ_p, $d_m(h\,k\,l)=d_p(h\,k\,l)(1+(1-3\cos^2\varphi)Q(h\,k\,l))$ with φ being the angle between the diffraction vector and σ_3, $Q(h\,k\,l)=(t/3)(2G(h\,k\,l))^{-1}$.

$G(h\,k\,l)$ is the effective elastic modulus calculated for a $(h\,k\,l)$ plane at the high pressure and temperature conditions [12]. For detectors along the vertical and horizontal plane, $\varphi\approx 0$ and $\varphi=90$, respectively. Thus

$$t=2\times G(h\,k\,l)\times\frac{(d(h\,k\,l)_{vertical}-d(h\,k\,l)_{horizontal})}{d(h\,k\,l)_p}$$

where $d(h\,k\,l)_{vertical}$ and $d(h\,k\,l)_{horizontal}$ are the d spacing measured in X-ray diffraction spectra collected by the vertical detectors and by the horizontal detectors, respectively. The stress applied on the sample can be calculated for each $(h\,k\,l)$ diffraction plane and then, an average value of the applied stress is obtained. By measuring the d spacing at φ_p such that $(1-3\cos^2\varphi)$ we can calculate the volume of the lattice unit cell under σ_p and therefore determine the pressure using the Birch Murnaghan equation. This method is accurate only under the assumption of elastic stress propagation in the stress sensor. An alternative technique based on elasto-plastic self-consistent models (EPSC models) can be used to derive the sample stress when significant plastic deformation occurs in the stress sensor [3]. This last method derives the stress applied on the samples by considering the elastic and plastic behaviors of large numbers of grains treated individually and deformed in non-hydrostatic conditions at high pressure [1]. This technique has been demonstrated for a couple of polycrystalline samples (quartz, MgO, cobalt) [1,5,8], but not yet for the stress sensor (alumina powder) we used.

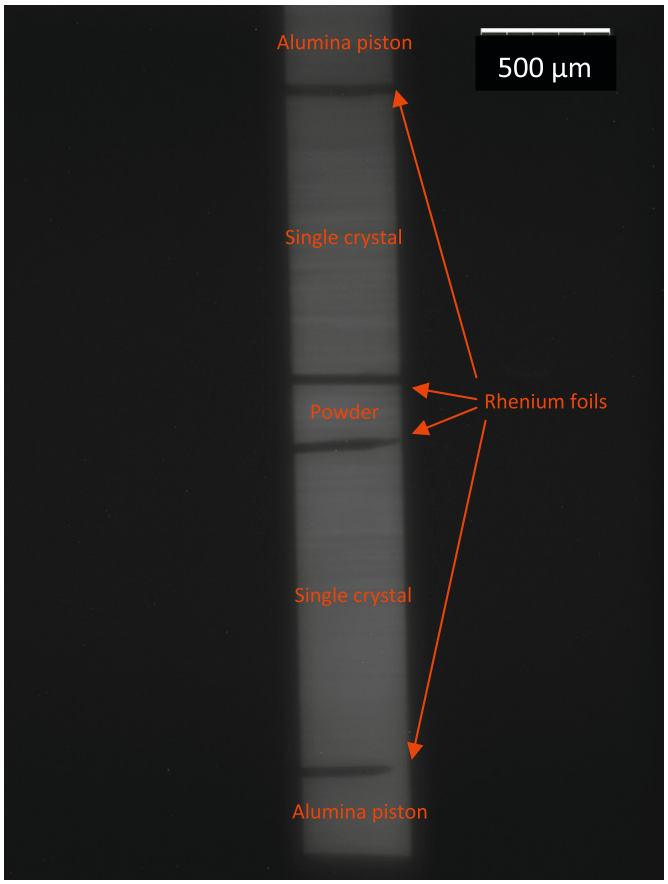

Fig. 6. X-ray radiography image recorded during the deformation of two single crystals delimited by rhenium foils. The black shadow on each side of the picture are the anvils.

6. Strain measurement

A CCD camera is used to record the image of the single crystal sample in visible light using a YAG crystal fluorescent screen. Slits define a 2 mm × 2 mm window for radiography imaging (Fig. 7).

The strain rate can be determined using sample images. The evolution of the sample length can be followed and measured during the deformation on the X-ray radiography images using the rhenium foil, which delimitate the sample length [13]. See in Fig. 6 an example of radiography image of two single crystals. With the relation below we obtain the total strain during the deformation:

$$\varepsilon(\%)=\ln(l_0/l_{(t)})\times 100$$

where l_0 is the initial length of the single crystal (before the deformation), and $l(t)$ the length of the single crystal at a given time t.

Thus, by plotting the total strain versus time for each sample we obtain a curve with a slope corresponding to the strain rate of the samples. From this measure the strain rate can be calculated with an accuracy of $10^{-6}\,s^{-1}$. In Fig. 8 we can see an example of a curve total strain versus time obtained during the deformation experiment of two single crystals $[1\,1\,0]_c$ and $[0\,1\,1]_c$. The slope of each curve gives the respective strain rate for each single crystal.

7. Experimental procedure and examples

The D-DIA cell containing the sample and the stress sensor is placed in the press and then the press is closed. The pressure is slowly increased up to the desired pressure condition. The pressure is monitored using the X-ray diffraction and the equation of state of the stress sensor. The temperature is subsequently

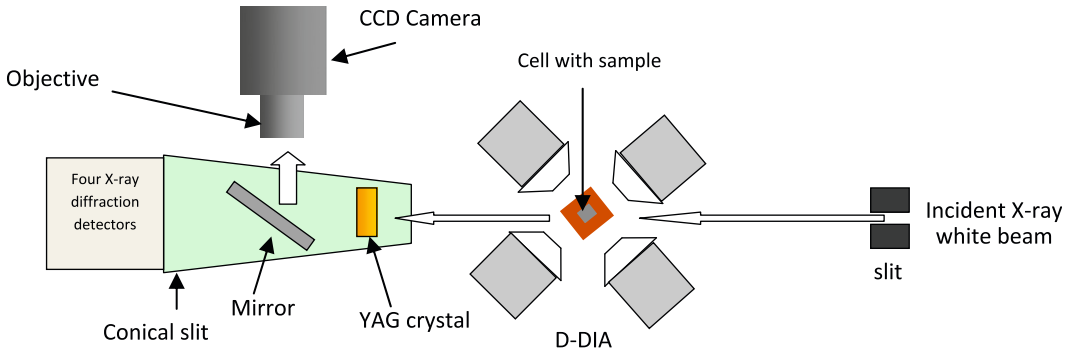

Fig. 7. Schematic of the D-DIA press on the synchrotron beamline.

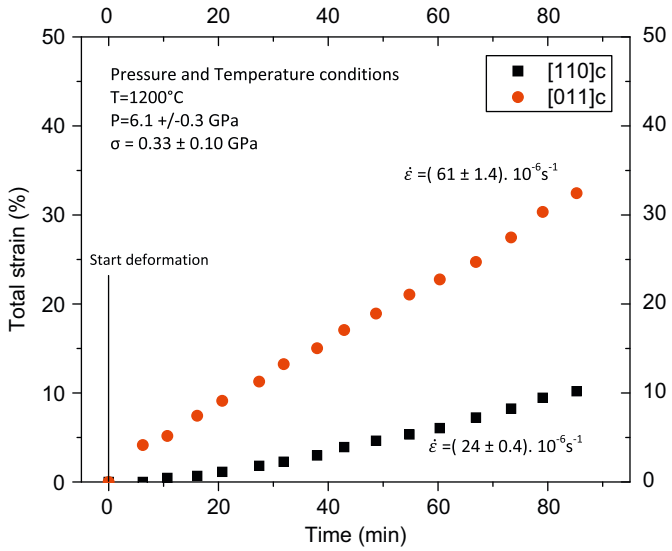

Fig. 8. Example of curve total strain versus time for two single crystal orientations ($[1\,1\,0]_c$ and $[0\,1\,1]_c$) deformed simultaneously at 6 GPa.

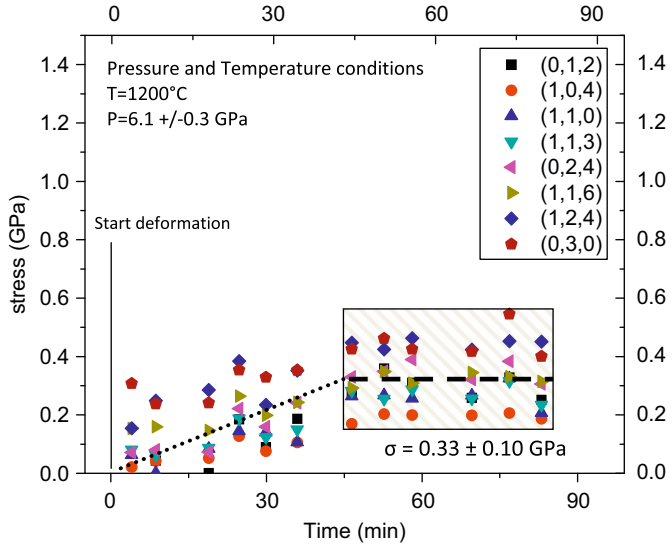

Fig. 9. Example of graph stress evolution for each ($h\,k\,l$) diffraction plane versus time.

increased to anneal the sample. Deformation starts when both desired pressure and temperature are reached. The samples and the stress sensor are deformed in the same column of the cell so that they experience the same conditions of pressure, temperature and stress. X-ray diffraction can be performed on the stress sensor placed in the middle of the cell assembly and on the top/bottom alumina pistons. Diffraction spectra are collected as a function of time during the deformation. Between each X-ray diffraction pattern of the stress sensors, X-ray radiographies of the single crystals are collected to follow the evolution of the sample length during the deformation. To make sure we reach the steady state (constant stress and constant strain rate), we deformed the sample for at least 5 percent strain. The X-ray diffractions and radiography images can be briefly analyzed to ensure the constant stress and strain rates. After the deformation temperature is decreased first, both the main ram and differential rams are retracted simultaneously for decompression. All the deformed samples are recovered for microstructure investigation.

The X-ray diffraction spectra are analyzed to determine the d spacing for each ($h\,k\,l$) diffraction plane along different orientations with regard to the principle stress. The stress is then derived from the variation of the d spacing as described earlier. Fig. 9 shows an example of the evolution of the stress during a deformation experiment. The stress increases during the transient stage and then reaches a steady state. The shaded area on the graph indicates the stress value obtained during the steady state. A mean value of the applied stress is calculated from the data in this area.

8. Conclusion

We have demonstrated that the essential deformation data needed for constructing rheological flow law for single crystals can be obtained from the experimental system previously applied to polycrystalline material. With a modified high pressure cell assembly, we can obtain the stress and strain rate at a similar accuracy to that in the polycrystalline deformation experiment. For a given crystal symmetry, a single crystal can be oriented such that only selected slip systems are active. Thus a rheological law can be determined for this activated slip system using the system we described here [4]. Therefore rheological behavior of a material can be predicted through modeling using the properties of individual slip systems.

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