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### ADVERTISEMENT





### Axial temperature gradient and stress measurements in the deformation-DIA cell using alumina pistons

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The deformation-DIA apparatus (D-DIA) coupled with synchrotron X-rays allows investigating materials elastic and plastic properties at high pressure. Most D-DIA deformation cells use alumina pistons that can also be used for measurement of the differential stress in the compression column by in situ X-ray diffraction. Here, we quantify the axial temperature (T) gradient in the D-DIA deformation cell and better constrain stress measurements in its compression column by studying an alumina specimen compressed and deformed at pressure P in the range 3.9–5.5 GPa and nominal temperature  $T_o = 1673$  K. The axial T gradient, obtained from alumina equation of state, is ~155 K/mm at the centre of the cell and does not vary significantly during deformation to 20% specimen strain. This T gradient, if not taken into account when measuring the experimental pressure in the alumina pistons, leads to significantly overestimating pressure. Unlike pressure, stress measurements in alumina are weakly sensitive to temperature. During deformation, the "true" differential stress in the compression column is evaluated at 596  $\pm$  20 MPa using an elastoplastic self-consistent model, while raw uncertainties on experimental differential stresses reach 84 MPa. A comparison between the simulated and experimental data allows to conclude that, although dislocation glide in the basal plane is the primary slip system at run condition, with an estimated critical resolved shear stress (CRSS) of 120 MPa, prism plane slips and pyramidal plane slips also contribute significantly to the aggregate homogenous deformation and texture development, with CRSS on the order of 280 MPa. © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4801956]

#### I. INTRODUCTION

The D-DIA, coupled with synchrotron radiation (Wang et al., 2003), has been extensively used over the past decade to investigate high-pressure materials rheological properties (a review in Raterron and Merkel, 2009; see also Li and Weidner, 2010; Hunt et al., 2010; Raterron et al., 2011; Bollinger et al., 2012; Girard et al., 2012; Raterron et al., 2012; Yu et al., 2012; Girard et al., 2013) and elastic properties (e.g., Li and Weidner, 2008; Li and Weidner, 2012). In typical D-DIA deformation cells (Figure 1), a sleeve of graphite in contact with both vertical anvils is used as furnace to heat up the specimens; it is isolated from the pressure medium-made of a mixture of boron and epoxy, or mulliteby an alumina sleeve. One or several specimens can be loaded in the compression column along the axis of the furnace in between crushable alumina cylinders. Crushable (polycrystalline) alumina ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) has numerous advantages: (i) it is easily machinable; (ii) it accommodates part of the strain during cold compression, limiting specimen damages; (iii) it is a stable phase at high pressure and temperature, and does not react easily with other materials; (iv) at run conditions, crushable alumina cylinders become fully dense and can be used as pistons to transfer load onto the specimen(s) by advancing or retracting the D-DIA vertical anvils; (v) sintered alumina can be used to evaluate the stress applied on the compression column using X-ray diffraction (details below). This latter point is particularly critical when deforming hard amorphous materials or single crystals (details in Girard *et al.*, 2010) from which no diffraction data can be used for stress measurement.

One disadvantage of using alumina is its relatively high thermal conductivity at high temperature (6–8 W m<sup>-1</sup> K<sup>-1</sup> in the range 1100-1700 K, e.g., Munro, 1997) which is about three times that of zirconia (e.g., Raghavan et al., 1998; Schlichting et al., 2001), another common material used as end plugs in high pressure assemblies. The high conductivity enhances heat loss from the cell toward the vertical anvils which are directly in contact with the alumina cylinders, causing a marked axial thermal gradient, although no quantification of this gradient has been reported. Measuring thermal gradients in high-pressure cell assemblies is not straightforward since introducing extra thermocouples in the assembly significantly changes its thermal properties (thermocouple wires are heat sinks). As for stress measurements, despite on-going improvements in the diffracted X-ray detection system (Weidner et al., 2010), micro-stress and micro-strain heterogeneities in deforming aggregates translate into large uncertainties on the macroscopic differential stress. This issue can be addressed by introducing elastoplastic self-consistent (EPSC) models for data analysis (Li et al., 2004; Burnley and Zhang, 2008; Merkel et al., 2009), although no such modeling has been reported for alumina.

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FIG. 1. Schematic cross section of the D-DIA deformation cell assembly used in the present study. The dimensions (to scale) are those before cold compression.

We report here the measurement of axial thermal gradients in the D-DIA cell at a nominal temperature of 1673 K, from temperature quantification in hydrostatic condition based on alumina equation of state (EoS). We also report an EPSC modeling for alumina aggregates which allows better quantifying the differential stress in D-DIA compression column when using alumina pistons. This EPSC modeling also gives some insight on the relative activities of alumina dislocation slip systems at high pressure and temperature.

#### II. EXPERIMENT, RUN-PRODUCT MICROSTRUCTURES AND DATA TREATMENT

#### A. Experimental protocol

For the present run AL009, one 3-mm long cylindrical sample of crushable alumina (1.4 mm in diameter) was loaded in between two porous alumina piston in the cubic deformation cell (6.15 mm on edge, Figure 1). Specimen and pistons were prepared from the same starting material (Rescor 960) manufactured by Cotronics Corp. (NY, USA). This material consists of 96% alumina mixed with 4% mixture of oxides used as a binder which is composed primarily of SiO<sub>2</sub> and MgO and minor amounts of CaO, Na<sub>2</sub>O, K<sub>2</sub>O. Once loaded, the assembly was compressed to 50 tons oil pressure in the D-DIA located at the X17B2 beamline of the National Synchrotron Light source (NSLS, Upton, NY, USA). Temperature (T) was then ramped up for 50 min to nominal  $T_o = 1673$  K. Despite failure of both thermocouples during the ramp to final temperature, a temperature vs. power relation T = f(W) was recorded up to  $T \sim 1173$  K. The T = f(W) relation of this experiment was identical to calibrations recorded from previous experiments, which allowed using the previous calibrations to control temperature during the experiment. Power was maintained at a constant value until the experiment was quenched by cutting off the power supply at the end of run.

Diffraction data collection was performed in three steps. During STEP 1, hydrostatic conditions were maintained (fixed anvils) at pressure and temperature, and diffraction spectra were collected from the alumina specimen and pistons along the axis of the furnace in order to quantify the axis temperature gradient from alumina EoS. During this step,



FIG. 2. Run STEP 2 rheological data. (a) Specimen strain versus time: a steady-state strain rate (indicated) is achieved after ~5% strain; the error bars are smaller than the size of the symbols (crosses). (b) Differential stress versus specimen strain as obtained experimentally using the indicated alumina hkl lines; the actual experimental points at given strains are shown for 012 line, together with the large uncertainties affecting them (crosses); the large dashed line shows the "true" flow stress as further obtained from the EPSC modeling (Sec. IV). (c) Pressure versus specimen strain as measured *in situ* by X-ray diffraction at the centre of the cell (i.e., at  $T_o = 1673$  K).

spectra were collected over 600-s live time to obtain wellresolved diffraction data. During STEP 2, D-DIA top and bottom vertical anvils were advanced at constant oil pump rate (0.007 mm/s), eventually resulting in mechanical steady-state deformation for the specimen, i.e., constant differential stress and strain rate. During this step, the specimen was axially shortened by ~20% (Fig. 2(a)) while 200-s live-time spectra were collected from the centre of the specimen, i.e., from the hottest location within the cell at  $T_o = 1673$  K. These spectra were then used for EPSC modeling of alumina deformation, in order to better quantify the differential stress during STEP 2. During STEP 3, D-DIA vertical anvils were stopped resulting in the restoration of hydrostatic conditions within 30 min, as verified by X-ray diffraction. The 600-s livetime spectra were then collected from the specimen and the top piston along the axis of the furnace to quantify the axial T gradient. The essential difference between STEP 1 and STEP 3 was the distance between D-DIA vertical anvils (5.29 mm and 4.82 mm, respectively). Comparing the respective temperature gradients allows quantifying the effect of shortening the assembly on its thermal properties.

At the end of the run, the cell assembly was queched in temperature under pressure. Pressure was then decreased within 1.5 h by gradually decreasing the main-ram oil pressure. Specimen length was maintained constant during decompression (verified by radiography) by controlling manually top and bottom vertical anvil positions.

#### **B.** Run-product microstructures

Run AL009 product was impregnated with epoxy and cut along a plane containing the compression direction. It was mechanically polished using a succession of 6  $\mu$ m, 1  $\mu$ m, and 0.1  $\mu$ m diamond powder slurries and chemically polished to enhance grain boundaries using colloidal silica. Grain size measurements were collected from three locations along a polished sample half: centre of the alumina sample, 100  $\mu$ m from the interface between the sample and piston and halfway through the piston. Samples were imaged using standard secondary electron (SE) imaging techniques on a Quanta 600 FEG-SEM with the sample tilted  $70^{\circ}$  to the electron beam to enhance the topographic effects of the chemical polishing (Fig. 3). All images were corrected for distortion due to tilting by software in the SEM during image collection and were collected using the same magnification ( $\sim 6400 \times$ ). Grain boundaries in these images were traced and digitized. Measurements of long axes, short axes, total area, and orientation of long axes relative to the compression direction were determined using Image SXM and are reported in Table I. Zones of binder, primarily located along grain boundaries and as small  $<1 \,\mu$ m, and inclusions were not included in grain size analyses. The average grain size ( $\sim 1.5 \pm 1.0 \ \mu$ m) and orientations within the investigated zones are all the same within the error of measurement (Table I).

#### C. X-rays imaging and diffraction collection

The techniques used here for specimen imaging, diffraction data collection and data treatment have been de-

TABLE I. Run-product grain-size characterization.



FIG. 3. SE-SEM image collected after run AL009 at the centre of the alumina specimen. The average grain orientation is roughly perpendicular to the compression direction (Table I).

scribed elsewhere (Vaughan *et al.*, 2000; Chen *et al.*, 2004; Raterron and Merkel, 2009; Weidner *et al.*, 2010; Bollinger *et al.*, 2012). A summary of these techniques is provided below.

At the NSLS X17B2 beamline, a polychromatic hard X-ray beam is generated by a superconducting wiggler. In imaging mode, the front slits are removed and the large incident beam goes through the D-DIA anvil gap, across sample and cell assembly. Specimen radiographs are collected downstream on a YAG crystal fluorescing under the X-rays, and recorded with a CCD camera. Specimen length is measured on the radiographs from the distance between the images of X-ray absorbent metal-foils (here made of Re) placed at sample ends (Vaughan et al., 2000). When deforming the specimen, images are recorded as a function of time. Specimen strain at a given time  $\varepsilon = \ln(l_o/l)$  is measured from the radiographs, where *l* is the length of the specimen and  $l_o$  is its length at time t = 0; here  $\varepsilon$  is >0 in compression. Deducing specimen strain rate  $\dot{\varepsilon} = d\varepsilon/dt$  is straightforward, from the slopes obtained on  $\varepsilon = f(t)$  plots.

In diffraction mode, the front slits reduce the cross section of the incident beam, here to a window  $150-\mu$ m high and  $160-\mu$ m wide. By moving up, down, and side ways the D-DIA, one can choose the volume of interest for X-ray

Zone	Image	Grain number	Long axes ( $\mu$ m)	Short axes ( $\mu$ m)	Angle of long axes to compression (deg)
Specimen centre	1	89	$2.0 \pm 1.5$	$1.2 \pm 0.5$	$87 \pm 55$
	2	123	$1.8 \pm 1.3$	$1.1 \pm 0.8$	$105 \pm 52$
	3	93	$2.0 \pm 1.4$	$1.1 \pm 0.7$	$104 \pm 52$
Average			$1.9 \pm 1.4$	$1.1 \pm 0.8$	$98 \pm 53$
Specimen ends	1	156	$1.8 \pm 0.9$	$1.1 \pm 0.6$	$93 \pm 53$
	2	169	$1.7 \pm 1.0$	$1.0 \pm 0.6$	$89 \pm 53$
Average			$1.8 \pm 0.9$	$1.0 \pm 0.6$	$90 \pm 53$
Piston	1	91	$2.2\pm1.2$	$1.2\pm0.7$	$89 \pm 59$

diffraction. The dimension of the front-slit window imposes the uncertainty on Z, the vertical position of the diffracting volume (here,  $\delta Z = 75 \ \mu m \sim 0.08 \ mm$ ). Diffracted X-rays are collected using a 10-element energy-dispersive multi-detector (Weidner et al., 2010) placed behind a conical slit which imposes the diffraction angle  $\theta$  (here,  $2\theta =$  $6.4^{\circ}$ ). At pressure, the X-rays diffracted toward the two vertical detector elements (numbered 1 and 9) are directly collected trough the gap between D-DIA lateral anvils, while those diffracted laterally (detector elements 2-8, and 10) travel through D-DIA (X-ray transparent) back anvils made of sintered-diamond and are consequently attenuated. Since only one transparent back anvil was used during run AL009, no data were collected at pressure with detector element 10, which was blocked by an opaque tungsten-carbide (WC) anvil; and no data were collected with detector element 2 which was not functional during the experiment. The other detector elements were calibrated before the experiment at room conditions (open press spectra) using standard alumina-powder diffraction lines and the  $\gamma$  rays (14.4 keV and 122 keV) emitted by a 57Co radioactive source placed in the vicinity of the multi-detector. For each investigated diffraction volume at a given time, 8 diffraction spectra were collected simultaneously using the 8 available detector elements (1, and 3-9).

#### D. X-ray diffraction data treatment

The alumina diffraction spectra were calibrated and fit using *Plot85* (http://www.mpi.stonybrook.edu/NSLS/X17B2/ Support/Plot85/plot85.htm), leading to *d*-spacing quantification of alumina 012, 104, 110, 113, 024, 116, 214, 300 first order diffraction lines. For energy calibration of detectorelement channels, the maximum allowed error was typically <10 eV which corresponds to a maximum relative error <0.02% on *d*-spacing. Each diffraction line of each spectrum corresponds to alumina crystallographic plane in a specific orientation with respect to the vertical direction. The angle  $\psi$ between the normal of a given diffracting plane and the principal stress direction  $\sigma_3$ —which during STEP 2 of the present run roughly corresponds to the vertical direction—is related to the detector-element azimuth angle  $\eta$  of a given element by the relation:

$$\cos\psi = \cos\theta \times \cos(\eta - \eta_o), \tag{1}$$

where  $\eta_o$  is the principal-stress direction azimuth angle (~90° during STEP 2). This relation allows measuring variations in *d*-spacing of given crystallographic planes as function of their orientations with respect to  $\sigma_3$  direction. Such data are critical when evaluating the principal-stress direction and quantifying the differential stress experienced by the sample. These data carry information about the flow stress experienced by alumina grains in given orientations in the deforming aggregate, and can be used to modeling the aggregate plasticity using EPSC models (see Sec. IV). Besides information about stress, information about sample texture can be deduced from the comparison of peak intensities in the spectra collected with different elements of the multi-detector (Bollinger *et al.*, 2012). This requires, however, normaliza-

tion of the diffracted intensities to account for the sensitivity of each element to the X-rays and for the back-anvil attenuation effect. Finally, by averaging alumina *d*-spacing over the sampled crystallographic-plane orientations, the hydrostatic pressure within the assembly can be quantified from the unit cell volume at given *T* using alumina EoS. This later property is used here during the hydrostatic STEP 1 and STEP 3 to, conversely, measure the temperature at various positions along the cell-assembly vertical axis, i.e., to quantify the axis temperature profile knowing the temperature  $T_o = 1673$  K (hence the pressure) at the centre of the assembly.

To calculate pressure (or temperature) and experimental stress values, the measured *d* spacing was fitted using the *Polydefix* software (http://merkel.zoneo.net/Multifit). *P* at given *T* (or *T* at given *P*) is obtained from the measured unitcell volume and alumina Birch-Murnaghan EoS using the parameters as reported by Anderson (1995) (see also Anderson and Isaak, 1995). The fit for stress measurement—detailed in Bollinger *et al.* (2012)—is based on the model reported by Singh *et al.* (1998) which proposes that the lattice elastic strains vary with  $\psi$  according to the equation:

$$d_{hkl}^{m} = d_{hkl}^{0} [1 + (1 - 3\cos^{2}\psi)Q], \qquad (2)$$

where  $d_{hkl}^m$  is the measured *d*-spacing of a given {hkl} plane at angle  $\psi$ ,  $d_{hkl}^0$  is the hydrostatic *d*-spacing, and Q is the lattice strain parameter for given hkl lines. Q and  $d_{hkl}^0$  are fitted to the data. Residual stresses in the alumina polycrystal can then be evaluated using the elastic model of Singh et al. (1998), the fitted Q parameters, and the single crystal elastic moduli of the material. In this calculation, we use the ambient pressure/high temperature  $C_{ii}$  of Anderson *et al.* (1992) along with the *ab initio* pressure derivatives of Duan et al. (1999). Experimental pressure derivatives of Gieske and Barsch (1968) could also be used without affecting the actual values of the deduced differential stresses. The elastic residual stress model of Singh et al. (1998) does not account for stress heterogeneities between grains in different orientations and, hence, each lattice plane provides a different estimate of the differential stress. This will be addressed below using EPSC modeling (Sec. IV). The *Polydefix* software was also used to calculate each detector-element (intensity) correction factors, and extract the normalized intensities for the diffraction lines collected during deformation (STEP 2). The normalized intensities-interpolated every 5°, in-between or at detector-element positions-were input within the BEAR-TEX software (Wenk et al., 1998) to extract orientation distribution functions (ODFs) from the diffraction data, and ultimately calculate the inverse pole figures (IPFs) of the compression direction representative of alumina texture development during run STEP 2.

#### III. HYDROSTATIC T-GRADIENT MEASUREMENTS

The present work is focused on the T gradient along the vertical axis of the cell assembly. The radial T gradient, which necessarily exists in the cell, is beyond the topic of this article. The temperature values obtained here using X-ray diffraction are, thus, averaged over the diffraction volume which is

Point #

1

2

3

STEP 1

TABLE II. P, T, and weighted-mean differential stress  $\langle \sigma \rangle$  along the cell axis.

	4	+0.6	1673	5.4	$-117 \pm 65$	5.1	1616	$-118\pm 66$
5 6 7 8	5	-0.6	1673	5.6	$-101 \pm 51$	5.1	1583	$-102\pm52$
	6	-1.2	1673	6.9	$-104\pm 66$	5.1	1377	$-107\pm67$
	7	-1.7	1673	8.4	$-113 \pm 62$	5.1	1134	$-120\pm65$
	8	0	1673	5.0	$-120 \pm 48$	5.1	1679	$-120\pm52$
	9	+2.3	1673	9.0	$+20 \pm 20$	5.1	1034	$+21\pm21$
STEP 3 10 12 13 14 14 14 14 15	10	0	1673	3.9	$-77\pm84$	3.9	1673	$-77\pm84$
	11	+0.3	1673	3.9	$-96 \pm 48$	3.9	1669	$-96\pm48$
	12	+0.6	1673	4.1	$-99\pm53$	3.9	1640	$-100\pm53$
	13	+0.9	1673	4.7	$-111 \pm 68$	3.9	1550	$-113\pm68$
	14	+1.4	1673	5.9	$-113 \pm 64$	3.9	1341	$-117\pm 66$
	15	+1.7	1673	6.9	$-139\pm 64$	3.9	1183	$-146\pm67$
	16	+2.0	1673	7.7	$-4 \pm 27$	39	1060	-4 + 29

<sup>a</sup>T assumed constant, P ( $\pm 0.1$  GPa), weighted-mean  $\langle \sigma \rangle$  obtained from *Polydefix*.

<sup>b</sup>P assumed constant,  $T(\pm 1 \text{ K})$ , weighted-mean  $\langle \sigma \rangle$  obtained from *Polydefix*.

sub-parallel to the alumina specimen diameter. During run STEP 1 and STEP 3, X-ray diffraction data were collected from several Z positions along the cell vertical axis. In the following, the vertical position of the specimen centrewhich experiences the highest temperature  $T_o = 1673$  K is set at Z = 0; axial positions above are measured with Z > 0. During STEP 1, diffraction data were collected from 9 axial positions ranging from Z = -1.7 mm to Z = +2.3 mm, covering 4.0 mm of the 5.3 mm gap separating D-DIA vertical anvils after compression. During STEP 3, diffraction data were collected from 7 axial positions between Z = 0 and Z = +2.0 mm, hence scanning the top half of the 4.8 mm of the cell assembly height after deformation. At each given Z position, 8 diffraction spectra were collected simultaneously for 600 s live time. The diffraction spectra were fitted leading to *d*-spacing quantification for the following alumina crystallographic planes:  $\{012\} \equiv \{01\overline{1}2\}, \{104\}$  $\equiv \{10\overline{1}4\}, \{110\} \equiv \{11\overline{2}0\}, \{113\} \equiv \{11\overline{2}3\}, \{024\} \equiv \{02\overline{2}4\}, \{02\overline{$  $\{116\} \equiv \{11\overline{2}6\}, \{214\} \equiv \{21\overline{3}4\}, \text{ and } \{300\} \equiv \{30\overline{3}0\}.$  The obtained *d*-spacing were then computed using *Polydefix* to quantify unit cell parameters, thus pressure (or temperature), and experimental differential stress along the cell-assembly axis.

The pressure, temperature, and experimental differential stress ( $\sigma = \sigma_3 - \sigma_1$ , where  $\sigma_3 \equiv \sigma'_{33}$  is the principal stress component) were obtained by fitting alumina d-spacing (Table II). We used the diffracted data collected from Z = 0, i.e., from the diffracting volume of known temperature  $T_o = 1673$  K, to quantify  $P_o$  at the centre of the cell. We obtained  $P_o = 5.1$  GPa during STEP 1 (before deformation), and  $P_o = 3.9$  GPa during STEP 3 after deformation and stress relaxation. T and  $\sigma$  values at  $Z \neq 0$  were obtained either assuming a constant  $T = T_o$  along the cell axis (isothermal values), or a constant pressure along the axis (hydrostatic values). Assuming a constant  $T = T_o$  along the cell axis—an unrealistic assumption since the cell ends in contact with D-DIA

anvils are much cooler than its centre-we obtain a steep apparent pressure gradient, i.e., a ~4 GPa increase between the cell centre and its ends (Table II). Yet, we obtain low stresses along the cell axis which are not consistent with the apparent large *P* gradient, because of the wrong isothermal assumption. Assuming now hydrostatic conditions within the assemblyi.e., adjusting temperature in *Polydefix* to obtain a constant pressure  $P = P_{o}$  along the cell axis—also results in low stress conditions along the cell axis (Table II), which are however consistent with the hydrostatic assumption. The hydrostatic T reported in Table II is thus representative of thermal conditions within the cell.

Plots of the hydrostatic temperature obtained during STEP 1 and STEP 3 versus the vertical Z position reveal steep temperature gradients from the hot cell centre (at Z = 0 and  $T_o = 1673$  K) to its cold ends in contact with D-DIA vertical anvils (Figure 4). The average temperature at the anvil contacts was  $T \sim 596$  K during STEP 1 when the vertical-anvil gap was 5.29 mm, and  $T \sim 745$  K during STEP 3 when the anvil gap was only 4.82 mm. Closing the vertical-anvil gap during compression of the cell assembly (STEP 2) resulted in increasing the anvil temperature. Along the whole compression column, average temperature gradients are ~400 K/mm. During both STEP 1 and STEP 3, the temperature gradient over the 2 mm at the centre of the cell (for  $-1 \le Z \le +1$ ) was fairly constant:  $\sim 155 \ (\pm 5) \ \text{K/mm}$ .

#### **IV. STRAIN AND STRESS MEASUREMENT DURING ALUMINA DEFORMATION**

In order to avoid confusion between the differential stress values obtained experimentally using different methods, we use here the following convention: the stress measured using one given hkl line is simply called "differential stress"; that obtained by averaging the experimental values obtained with different hkl lines is called "average differential stress,"

 $-4 \pm 29$ 



FIG. 4. Corrected temperature (crosses) versus Z (vertical position) during (a) run STEP 1 and (b) run STEP 3. Uncertainties of T and Z are smaller than the symbol size. Z = 0 corresponds to the centre of specimen and cell assembly. The vertical lines show the position of the Re foils at specimen ends. The curve is a polynomial fit through the corrected temperatures, with  $T_o = 1673$  K at Z = 0 (equations and R<sup>2</sup> coefficient indicated). The open squares show the positions and temperatures of the contacts with D-DIA vertical anvils, as observed on the radiographs and extrapolated from the polynomial fits, respectively.

and that obtained from the Polydefix program with different hkl lines taking into account their respective uncertainties is called "weighted-mean differential stress.

During run STEP 2, axisymmetric compression of the cell assembly promoted  $\sim 20\%$  strain for the alumina specimen. Specimen radiographs were regularly recorded in between collecting 200-s live-time X-rays spectra from the centre of the cell (at Z = 0 and  $T_o = 1673$  K). The time-resolved *P* and weighted mean differential stress  $\langle \sigma \rangle$  obtained from *Polydefix*, as well as the corresponding specimen strains, are reported in Table III.

## A. Steady-state deformation conditions and rheological observations

Steady-state conditions of deformation were reached after ~5% specimen strain, i.e., differential stress and specimen strain rate remained then approximately constant for STEP 2 remaining duration (Figs. 2(a) and 2(b)). The sample was deformed at a constant specimen strain rate  $\dot{\varepsilon}$  ~ 4.8 (5) × 10<sup>-5</sup> s<sup>-1</sup> over ~15% strain (Fig. 2(a)). Differential stresses

TABLE III. P, weighted-mean differential stress and strain during STEP 2.

Time (s)	Point #	P (GPa)	$\langle \sigma \rangle$ (MPa)	$\langle \sigma \rangle^{a}$ corrected (MPa)	ε <sup>b</sup> (%)
- 37	1	5.23	$91\pm57$	$72 \pm 58$	0
313	2	5.3	$184\pm60$	$215\pm74$	0.9
594	3	5.36	$320\pm94$	$307\pm67$	1.7
921	4	5.45	$513\pm75$	$462\pm75$	2.7
1542	5	5.49	$685\pm75$	$612\pm76$	4.6
1789	6	5.44	$714\pm82$	$628\pm77$	5.8
2193	7	5.35	$673\pm81$	$596\pm74$	7.9
2505	8	5.24	$668\pm84$	$610\pm82$	9.5
2751	9	5.09	$687\pm80$	$603 \pm 81$	10.8
2979	10	4.98	$679\pm78$	$595\pm83$	11.8
3228	11	4.91	$718\pm79$	$617\pm77$	12.9
3472	12	4.87	$688\pm81$	$604 \pm 77$	14.1
3817	13	4.75	$673\pm76$	$605\pm80$	15.7
4094	14	4.64	$724\pm76$	$622\pm77$	17.0
4330	15	4.54	$646\pm80$	$585\pm75$	18.2
4569	16	4.49	$624\pm77$	$567\pm77$	19.3
4801	17	4.28	$627\pm79$	$554\pm68$	20.5

<sup>a</sup>Corrected  $\langle \sigma \rangle$  obtained without 012 and 024 *d*-spacing.

<sup>b</sup>Strain calculated using the  $\varepsilon = f(t)$  data obtained from radiographs.

deduced using alumina hkl lines are evenly distributed in the range 0.5-0.9 GPa during steady state deformation (Fig. 2(c)). Differential stress fluctuations with strain are representative of uncertainties on the measurement, as illustrated for 012 line (grey dotted line) for which experimental points and corresponding large uncertainties are indicated. The 012-line stress uncertainties, partly due to low diffraction counts, explain the discrepancy between 012-line and 024line differential stresses (black dotted line), which should be equal and are not: 024-line stress is significantly lower. Using all alumina hkl line differential stresses after 5% strain, the weighted-mean differential stress during STEP 2 steady-state condition is estimated from *Polydefix* to  $\langle \sigma \rangle \sim 624$  (80) MPa (Table III). Over the first 5% of specimen strain, P increased from 5.2 GPa to 5.5 GPa, then decreased steadily to reach 4.5 GPa at 20% specimen total strain (Fig. 2(c)). Variations in pressure within 1 GPa are often observed during deformation in the D-DIA; they are related to piston force (oil pressure) adjustments and cell-assembly plastic response to the varying mechanical conditions. The average pressure during STEP-2 steady state deformation was  $\langle P \rangle \sim 5.0$  (5) GPa. Finally, based on the temperature profiles measured before and after deformation (Figure 4), the average temperature over the whole specimen length can be estimated to  $\langle T \rangle \sim 1540$ (130) K during run STEP 2.

Comparison of the lattice strain parameters Q (Eq. (2)) versus specimen strain, obtained experimentally from alumina *d*-spacing during STEP 2 allows constraining the EPSC modeling outputs, according to the procedure developed in Merkel *et al.* (2009, 2012), (Fig. 5, see below). The 012 data are not included since its Q parameter is better constrained by 024 line parameter. The hkl-line Q parameters are evenly distributed in the range  $5 \times 10^{-4}$  to  $9 \times 10^{-3}$ , with the lowest values for 104 and 116 data (mean Q  $\sim 5.5 \times 10^{-4}$  and 6.1  $\times 10^{-4}$ , respectively) and the maximum values for 024 data ( $\sim 8.3 \times 10^{-4}$ ). The Q parameters obtained for 110 and 300



FIG. 5. Run STEP 2 data analysis. (a) Lattice strain parameter Q (see Eq. (2)) versus specimen strain as obtained from *Polydefix* for different alumina hkl lines (indicated). (b) Lattice strain parameter Q versus specimen strain as obtained from the EPSC modeling using the best-fit parameters reported in Table IV, for the indicated hkl lines.

data are fairly high  $\sim$ 7.6 × 10<sup>-4</sup>. Lower (intermediate) values are obtained for 214 and 113 data ( $\sim$ 6.5 × 10<sup>-4</sup> and  $\sim$ 7.2 × 10<sup>-4</sup>, respectively).

Figure 6(a) shows alumina inverse pole figure obtained *in situ* during deformation from alumina hkl-line intensities at ~16% specimen total strain, using the protocol reported by Bollinger *et al.* (2012). The alumina developed a texture with a marked maximum sub-parallel to  $001 \equiv 0001$  pole, usually interpreted as resulting from the high activity of alumina basal-plane dislocation slip at high temperature (see below).



FIG. 6. Inverse pole figures of the compression direction, (a) during experimental deformation at 16% specimen strain, as obtained by implementing the corrected alumina hkl-line experimental intensities into the BEARTEX software; (b) as obtained at 20% from the EPSC modeling using the best-fit parameters reported in Table IV. The simulation reproduces fairly well the experimental strong maximum sub-parallel to 0001 pole, yet it fails to reproduce the experimental minimum near  $11\overline{20}$  pole. See text for further explanation.

This inverse pole figure allows constraining the EPSC model proposed below for alumina.

## B. Alumina deformation mechanisms at high temperature

Because of its good mechanical properties and stability at high-temperature, with numerous technical applications, alumina plasticity has been studied extensively since the 1960s. Alumina dislocation slip systems and twinning are reviewed by Heuer and Castaing (1985) and Heuer et al. (1998) (see also Lagerlöf et al., 1994). Alumina basal and rhombohedral twinning has been well characterized (e.g., Heuer, 1966; Pirouz et al., 1996; Castaing et al., 2002). Twinning is a very active mechanism at low to moderate temperatures (e.g., Castaing et al., 2004), particularly rhombohedral twinning. Twinning is, however, a minor deformation mechanism at high temperature (T > 1273 K) as dislocation slip systems are more easily activated, and twinning is more difficult due to its interactions with the dislocation forest (Castaing et al., 2002). At high temperature and low stress conditions, fine-grained aggregates deform by grain-size sensitive creep, likely involving grain-boundary sliding accommodated by dislocation slip (review in Ruano et al., 2003). Under high differential stress, as is the case here, coarse-grained aggregates (typically  $>2 \ \mu m$  for  $\sigma \sim 600$  MPa) mostly deform by dislocation glide (Heuer et al., 1980).

The alumina slip systems which have been clearly identified at high temperature are:  $(0001)1/3(2\overline{1}\overline{1}0)$  basal slip,  $\{\bar{1}2\bar{1}0\}$  (10 $\bar{1}0$ ) prism plane slip, and  $\{10\bar{1}1\}$  1/3 ( $\bar{1}101$ ) (or possibly  $\{\bar{1}012\}\ 1/3\langle\bar{1}101\rangle$  or  $\{2\bar{1}\bar{1}3\}\ 1/3\langle\bar{1}101\rangle$ ) pyramidal plane slips.  $\{01\overline{1}0\}\ 1/3\langle 2\overline{1}\overline{1}0\rangle$  prism plane slip has also been observed at high temperature (Klassen-Neklyudova et al., 1970). Although  $(10\overline{1}0)$  prismatic dislocations are the most active at low temperature (typically <973 K)—due to the dislocation dissociation into three low-energy  $1/3(10\overline{10})$ partials-the basal slip is the primary slip system at high temperature. This is because basal and prismatic dislocation glides are controlled by Peierls mechanisms (double kink formation and diffusion, see Mitchell et al., 1999) with different activation energies. At 1.5 GPa pressure and  $T \sim 925$  K, basal plane slip and prism plane slip exhibit the same critical resolved shear stress (CRSS ~700 MPa, Lagerlöf et al., 1994), while at 1673 K the basal plane slip CRSS is  $\sim$ 20 MPa and about 5 times lower than that of the prismatic slip CRSS ( $\sim$ 100 MPa). The low activity of prism plane slip at high T (with respect to basal-slip activity) has been attributed to easy cross slip of (1010) dislocations which favors their dissociation into two basal dislocations, i.e., limits the dissociation in low-energy  $1/3(10\overline{1}0)$  partials (e.g., Lartigue *et al.*, 2003). Pyramidal slips are the hardest slip at high temperature, yet they are active under high differential stress (Heuer et al., 1980) and their contribution to sample strain is required to satisfy the Von Mises criterion for homogeneous aggregate deformation by dislocation slip. Pyramidal slips were clearly activated at high temperature under moderate confining pressure (Snow and Heuer, 1973). Little is known on the effect of high pressure (P > 3 GPa) on the above mechanisms since, to our knowledge, no steady-state deformation of alumina at pressure > 1.5 GPa has been reported.

## C. EPSC modeling of alumina high-temperature plasticity

In order to better constrain the differential stress during steady-state deformation (STEP 2), we used a modification of the EPSC model proposed for aggregate plasticity by Turner and Tomé (1994) that allows for grain rotation induced by slip and stress relaxation due to twinning (Clausen et al., 2008; Neil et al., 2010), which we adapted to alumina plasticity. This mean-field scheme yields information about the absolute strength of the involved deformation mechanisms, stress distribution among grains in the deformed specimenresulting in the differential stress and Q parameter distributions showed in Figures 2(b) and 5(a)-and "true" differential stress values for the polycrystal (large dashed line in Figure 2(b)). The inputs in the model are alumina crystal data and elastic constants (see above), a list of alumina active deformation mechanisms and their geometry (i.e., dislocation slip systems and twinning), and a set of parameters specifying each mechanism strength, as for instance slip-system CRSS at deformation conditions. Strain hardening parameters can also be input, although in the present case no strain hardening is observed (Table II and Figure 2(b)); we thus set to zero the model hardening parameters. In the model, only intracrystalline deformation mechanisms are considered; they are activated into modeled spherical "grains" of a given orientation with respect to a mean homogeneous stress field, which is calculated iteratively by averaging the contributions of all other grain orientations to the aggregate plasticity. At the onset of deformation, the modeled aggregate consists of 3000 random grain orientations. While deformation proceeds, lattice preferred orientations (LPOs) develop within the modeled aggregate, gradually changing its rheological properties. Outputs of the model are the hkl lattice-strain parameters Q (Eq. (2)) which are directly related to stress distribution within the aggregate, the corresponding "true" differential stress and resulting aggregate LPOs, as well as evolution of these parameters with specimen strain. These can be compared with the measured Q parameter distributions (Figure 5(a)) and aggregate LPO (Figure 6(a)) to adjust and verify the modeling.

Since dislocation slip dominates alumina deformation at high T, no twinning is implemented in the present model. In the model, T is set to 1673 K—that of the volume at the centre of the cell from which diffraction data were collected during STEP 2—while the variation of P with strain matches the observed one (Figure 2(c)). The only adjustable parameters here are, thus, the CRSS of the implemented dislocation slip systems. We implemented in the model the six dislocations slip systems mentioned above and listed in Table IV, i.e., the basal plane slip, both prismatic slips, and the three known pyramidal slips. To test and set the model parameters within the framework of previous works on alumina plasticity, we imposed the four following rules. (1) The basal plane slip is always activated, together with at least one prism plane slip (active at high T) and one pyramidal plane slip for homogeneous deformation of the aggregate. (2) The basal plane slip

TABLE IV. EPSC model slip systems.

Slip system	CRSS best fit (MPa	
Basal plane slip		
(i) $(0001)1/3(2\bar{1}\bar{1}0)$	120	
Prism plane slips		
(ii) $\{\overline{1}2\overline{1}0\}\langle 10\overline{1}0\rangle$	280	
(iii) $\{01\overline{1}0\} 1/3 \langle 2\overline{1}\overline{1}0 \rangle$	280	
Pyramidal plane slips		
(iv) $\{10\overline{1}1\} \frac{1}{3} \langle \overline{1}101 \rangle$	280	
(v) $\{\bar{1}012\} 1/3 \langle \bar{1}101 \rangle$	280	
(vi) $\{2\overline{1}\overline{1}3\} 1/3 \langle \overline{1}101 \rangle$		

has the lowest CRSS, to account for its high activity at high T. (3) When activated together, both prism plane slips (numbered (ii) and (iii) in Table IV) have, arbitrarily, the same CRSS. (4) The CRSS of the pyramidal plane slips (hardest slip systems at high T) are equal to or greater than that of the prismatic slips. Testing the model, we noticed that the second and third pyramidal plane slips (numbered (v) and (vi) in Table IV) have comparable effects on hkl Q parameter outputs and on the resulting specimen LPO; when activated together with identical CRSS, their respective activities are also similar and about half that of one of them activated alone. Therefore, to limit the model input parameters, the last pyramidal slip ((vi) in Table IV) is not activated, i.e., it is represented in the modeling through the second pyramidal plane slip ((v) in Table IV). Within the above set of rules and choices, adjusting the model parameters consists of choosing which prism-plane and pyramidal-plane slips are activated (from (ii) to (v) in Table IV), and setting four CRSS values: that of the basal slip, that of the prism plane slips, and two values for the activated pyramidal slips. These parameters were, thus, adjusted to best fit the khl Q parameters (Figure 5) observed in steady state condition of deformation, as well as the LPO measured in situ in the deforming specimen (see below and Figure 6).

Testing the model led to the following conclusions: the lowest Q-parameter values (104 and 116 data) are very sensitive to the basal-plane slip CRSS, and are well accounted for when the basal slip CRSS is  $\sim$ 120 MPa; the Q-parameter for 214 and 113 data only show the appropriate values when both pyramidal slips ((iv) and (v) in Table IV) are activated with the same CRSS than that of the prismatic slip ((ii) and/or (iii) in Table IV); this also stabilizes the simulated LPO (Figure 6(b)). The prism plane slip CRSS must then be set to  $\sim 2$  to 3 times that of the basal plane slip. With the above setting, 110 and 300 Q parameters are also reasonably accounted for. The Q parameter obtained from the EPSC model for 012 and 024 data is, however, always lower than the observed ones, although it agrees with the experimental values within their uncertainties. Figure 5(b) shows the lattice-strain Q parameters obtained from the EPSC modeling of run STEP 2, using the best-fit parameters reported in Table IV. It shows that alumina Q-parameter values are reasonably reproduced by the modeling. Slight variations in Table IV parameters are possible, although significant changes in the reported CRSS dramatically affect the model outputs, i.e., the modeled Q parameters and/or LPO. The model is, thus, fairly

discriminative regarding CRSS inputs. In the absence of solid (theoretical or observational) criteria guiding the modulations of the pyramidal-plane slip CRSS, with respect to each other and to that of the prism plane slips, we report here the simplest solution where all prism-plane slips and pyramidal-plane slips have the same 280-MPa CRSS.

Using the best fit parameters reported in Table IV, the IPF predicted by the model does not reproduce the pole figure obtained experimentally (Figures 6(a) and 6(b)): there is an important predicted fiber between the  $10\overline{10}$  and  $11\overline{20}$  poles which is absent in the experimental IPF. The LPO outputs of the model are very sensitive to prism and pyramidal plane slip CRSS. Slightly changing their relative values significantly affects the simulated LPO. Testing the model, we concluded that a strong maximum sub-parallel to 0001 pole observed experimentally (Figure 6(a)) can only be obtained when all prism plane and pyramidal plane slips are activated with comparable CRSS. Yet, in this configuration, a maximum between 1010 and 1120 poles is unavoidable in the simulated IPF. This discrepancy between experimental and simulated LPOs may result from oversimplifications in the model, which in particular does not consider twinning as an active deformation mode at high pressure and temperature (see Sec. V). An interesting result from the model, however, is that the classic alumina LPO (Figure 6(a)), often attributed to the activity of the basalplane, may also result from the equal activation (same CRSS) of all prism and pyramidal plane slips which combined activation stabilizes alumina 0001 pole direction.

The differential stress at steady-state conditions (at strain >5%) given by this modeling, i.e., the "true" flow stress, is  $\sigma \sim 596 \pm 20$  MPa; the reported  $\sigma$  value and uncertainty were here obtained by averaging the "true" flow stresses resulting from varying the model slip-system CRSS without significantly changing the model outputs (Q parameter values and 0001 maximum in the inverse pole figure). The obtained "true" flow stress is lower than the experimental weighted-mean differential stress (Table III,  $\langle \sigma \rangle$ ). However, the weighted-mean differential stress obtained by averaging d-spacing contributions without that of 012 and 024 data (Table III, noted corrected  $\langle \sigma \rangle$ ) are in good agreement with the "true" flow stress. This is not surprising given that 012 and 024 differential stresses are not consistent, likely due to their large uncertainties (see above). Let us emphasize here that using the EPSC modeling, the obtained "true" flow stress value is based on a physical understanding of the aggregate heterogeneous stress state, and the resulting uncertainty is much lower (here  $\sim 20$  MPa) than that obtained by averaging alumina hkl differential stress values (up to 84 MPa, Table III).

#### V. DISCUSSION AND CONCLUSION

We report measurements of the axial temperature profile in a standard D-DIA deformation cell, using alumina end plugs as pistons during deformation. The reported T profiles show a gradient ~155 K/mm at the centre of the cell. This gradient, which is fairly constant up to 20% specimen strain, may depend on specimen thermal conduction, i.e., it may be lower with specimens of low thermal conduction. To limit the thermal gradient within the specimen, one should use as short a specimen as possible: a 1-mm specimen placed at the centre of the cell experiences a *T* gradient <80 K, i.e., its temperature is known to  $\pm$ 40 K. Another way to reduce specimen *T* gradient would be to place better thermal-insulator materials (such as zirconia) in the compression column; such modifications of the D-DIA deformation cell are currently under investigation.

From the temperature profiles reported in Figure 4, we estimated the heat flux driven through alumina toward D-DIA cold vertical anvils, before and after specimen deformation. For this calculation, we assumed thermal steady state and used Fourier's one-dimensional equation for heat conduction which here involved alumina thermal conductivity, the T gradient between the centre of the cell and the anvils, and the alumina piston cross section. Since alumina conductivity depends on temperature, we calculated the average conductivity over the observed T profiles ( $\sim 7.5 \pm 0.5$  W m<sup>-1</sup> K<sup>-1</sup>, see Munro, 1997 and Figure 4); the piston cross sections were estimated to 1.79 mm<sup>2</sup> and 1.96 mm<sup>2</sup> before and after deformation, respectively, assuming that alumina volume remained constant during compression. We obtained 11.4 W and 11.1 W for the total heat flux through the alumina pistons toward both anvils before and after deformation, respectively. These estimates show that the heat flux through the alumina pistons toward D-DIA anvils remains fairly constant during deformation of the specimen. It has been reported that several extra watts must be supplied to the D-DIA cell in order to maintain constant its temperature during deformation (e.g., Amiguet et al., 2009). Shortening of the alumina in the compression column, thus, cannot explain the extra heat loss causing the need for extra power supply. The reported extra heat loss may be due to other causes, such as the shortening of the graphite furnace itself and/or of the boron nitride (BN) sleeve around the specimen (Figure 1), which thermal conductivities are much higher than that of alumina.

Our results show that measuring pressure in the alumina pistons can lead to significantly overestimating P, when the axial temperature gradient is not taken into account. Indeed, Table III shows that assuming an isothermal cell at T = 1673 K, the P values obtained for instance above and below a 2 mm long specimen (at position  $Z = \pm 1$  mm) could be overestimated by as much as 1.5 GPa. However, applying the proper T correction using the temperature profiles reported in Figure 4 leads to an accurate measurement of the cell pressure. Unlike pressure, stress measurements in alumina are weakly sensitive to temperature. Measuring stress in the alumina pistons can be quite convenient when deforming materials from which diffraction data are not suitable for accurate stress analysis (single crystals, large-grain or complex multiphase aggregate, amorphous materials, etc.). We show here that the "true" flow stress applied on the specimen is easily estimated by the weighted-mean differential stress  $\langle \sigma \rangle$  obtained from *Polydefix* by averaging alumina first-order diffraction lines. It is, however, recommended to ignore alumina 012 and 024 data when calculating  $\langle \sigma \rangle$ : they give inconsistent results and show large uncertainties. A more accurate stress measurement can be obtained by using the EPSC model reported here for alumina high-temperature deformation. Yet, adjusting the model is time consuming and may not be done in the short

time frame of experiments carried out at synchrotron beamlines. Averaging alumina 104, 110, 113, 116, 214, 300 differential stresses is, thus, a simple "rule of thumb" giving a good estimate for the "true" flow stress experienced by deforming specimens.

The EPSC model for alumina reported here gives information about alumina deformation at high pressure and temperature. The model shows that, to account for rheological observations, all basal, prismatic, and pyramidal slips must contribute to the aggregate deformation. Given the grain size of our aggregate ( $\sim 1.5 \ \mu m$ ) and the stress level during deformation ("true" flow stress  $\sigma \sim 596$  MPa), this observation is consistent with the deformation map reported by Heuer et al. (1980). The EPSC model also provides CRSS estimates at run conditions for the active dislocation slip systems (Table IV). It confirms that the basal plane slip dominates high-temperature alumina deformation; however, because of its high efficiency, a marked LPO with 0001 pole sub-parallel to the principal stress direction is quickly produced, which geometrically limits basal-slip activity in the textured aggregate. This explained the significant activity of the harder prism plane slips and pyramidal plane slips, which allows homogeneous deformation of the aggregate and stabilizes its LPO. Yet, the discrepancy between experimental and simulated LPOs (Figure 6) suggests that other deformation mechanisms (e.g., twinning), not accounted for in the present modeling, are at play. Indeed, the texture observed in Zn and Os deformed at high pressure (Kanitpanyacharoen et al., 2012), which are consistent with combined basal slip and tensile twinning activity, are similar to the experimental texture reported here for alumina. Lebensohn et al. (2007) also predict such a texture for ice (hcp) deformed in compression using only basal slip, yet accounting for the corresponding non-negligible intragranular stress fluctuations across the grain. One or both of the above mechanisms may be present in the case of alumina, and should be implemented and tested in further more sophisticated modeling for this material. Finally, another interesting observation is the CRSS value obtained here for the prism plane slip (280 MPa, see Table IV), which is 7/3 that of the basal plane slip (120 MPa): both basal-plane slip CRSS and prism-plane slip CRSS, obtained here at  $T_o = 1673$  K and  $P_o = 5$  GPa, roughly corresponds to those reported for these slip systems at  $P \sim 1.5$  GPa and  $T \sim 1313$  K (Lagerlöf et al., 1994, their Figure 2). This observation supports Mitchell et al.'s (1999) interpretation of a thermally activated process for alumina dislocation slips (kink diffusion), since the CRSS obtained at high P and high T are comparable to those obtained at lower P and lower T. We, thus, conclude that the thermally activated process must be sensitive to pressure with a non-zero activation volume in the corresponding activation enthalpy.

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