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Compression and shear on lead in a rotational diamond anvil cell

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ABSTRACT
By application of large plastic shear on a lead sample in a rotational diamond anvil cell, we studied the pressure self-multiplication and the stress deviation phenomena, along with the consequential effects on a phase transformation of lead. It is indicated that pressure can be promoted by the gradual addition of shear. The stress deviation in the sample along different Chi angles is minimal and within the systematic error range. It is thus specified that a quasi-hydrostatic condition is generated in the sample chamber. Moreover surprisingly, under such shear-controlled pressure elevation, the lead fcc-to-hcp phase transformation pressure is found to initiate and complete, respectively, at 12.8 and 18.5 GPa, which is identical to those observed in hydrostatic compressions. The phenomena of the so-launched quasi-hydrostatic pressure, the self-multiplication, along with the consequential effects on the phase transformation properties by shear at pressures are expected to lead to further understanding of materials as well as to potential new technologies at extremes.

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1. Introduction

A rotational diamond anvil cell (RDAC) generates large plastic shear under high pressure and provides a means to fully explore the properties of materials under a unique stress distribution.[1–13] By far, a number of research works have been preceded in RDAC and interesting experimental and theory results have been achieved. Especially, primary understanding regarding materials under shear has been continually established. One observation is that the pressure in the sample chamber could be promoted by shear.[8–13] The other is that the shear-promoted pressure has an exceptional impact on structural phase transformation of a material; can either reduce the phase transformation pressure,[8–12] substitute a reversible phase transformation with an irreversible one,[10] or lead to new phases.[5] Analysis seems to point to a conclusion that shear-induced phase transformation follows strain-induced kinetics.[12,13]

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Although the application of an RDAC for shearing materials at pressures has been done over decades, critical *in situ* analysis is much more recent.[3,8] Systematical experimental studies and characterization are far from sufficient to build a theoretical framework. While most former works focus on hard materials with a few exceptions, this paper presents our investigation in application of shear to lead, one of the other kind – the soft most metallic solid material. And based on which the characterization and distinction of the pressure self-multiplication effect, the stress and strain deviation, and the phase transformation under high pressure condition. We also discuss the potentials of new physics and technologies introduced by shear.

2. Material and methods

A pair of composite anvils with 3 mm culet size was used in our rotational cell (details published in [10]). A cubic boron nitride gasket with a hole of 300 μm in diameter and 100 μm in depth for the sample chamber was selected. In one of the experiment, only lead powder sample was loaded into the sample chamber. In the other experiment, platinum powder was added to the center of the lead sample for pressure verification. The lead sample has a purity of 99.999% and the size is 325 mesh and platinum sample is 99.999% and 325 mesh, respectively. *In situ* synchrotron angle-dispersive X-ray diffraction measurements were carried out at 18.5 GPa in the radial-diffraction geometry [14] at the X17C beam line of the National Synchrotron Light Source. X-ray spectrum was collected with a CCD detector. The X-ray wavelength was 0.40765 Å.

During the experiment, one anvil was rotated with designed angles, after each of which an XRD pattern was taken. Selected XRD patterns are shown in Figure 1. Using the XRD and the equations of state (EOS) of Pt and Pb, we determine the corresponding pressure after each operation. For Pt, $K_0 = 273$ GPa, $K_0' = 5.20$ by Matsui et al. [15], and the Vinet EOS which was used to achieve these values are adopted. For Pb, $K_0 = 43.1 \pm 1.6$ GPa, $K_0' = 4.6 \pm 0.3$ of Pb reported by Mao and Bell [16] along with the first-order Murnaghan EOS they used to fit their data were adopted. We use the identical EOS to eliminate the possible additional calculation error.

3. Results and discussion

3.1. The pressure self-multiplication

In the experiment where platinum powder was added to the center of the lead sample, the load on the sample was added to 1.5 GPa before application of anvil rotation. During anvil rotation, the promoted pressure (calculated by both P_{Pb} and P_{Pt} discussed above) by shear corresponding to anvil rotation angle is shown in Figure 2, along with resultant errors from measure unit cell volume of Pt. It is found that the pressure in the sample increased with the angles of anvil rotation. Note that only 1.5 GPa load was initially applied on the sample without any addition later on in our experiments. It is the application of shear (by anvil rotation) that induces pressure elevation, which is termed pressure self-multiplication. Before anvil rotation of 40°, where pressure reaches 11.6 GPa, the pressure increases almost linearly with anvil rotation. After this point the pressure elevates with a lower rate until the rotation to 55° with pressure reaching 13.1 GPa. After 55° rotation, the

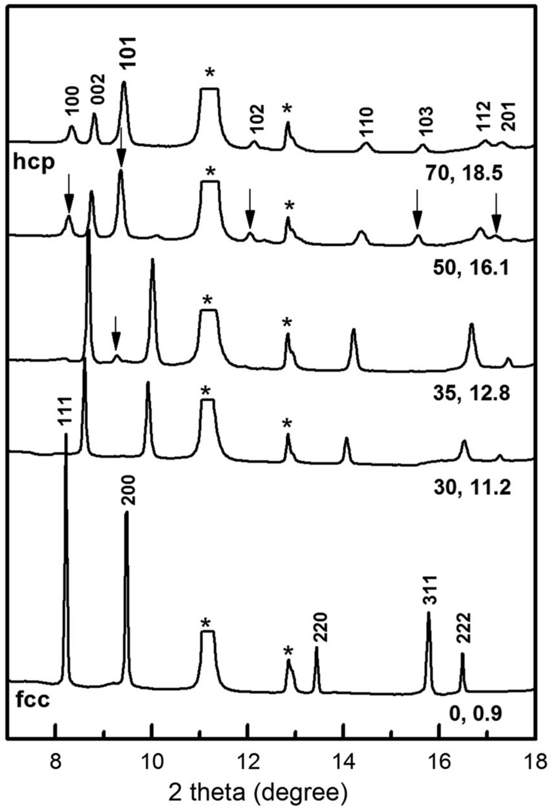

Figure 1. Selected XRD pattern of Pb at pressure and shear in RDAC. The arrows point out the appearance of the peaks of the hcp phase. The asterisks mark the peaks from the gasket. Angles of anvil rotation (in degree) and pressure (in GPa) are noted below the patterns.

pressure increases with a higher and invariable rate again. From the XRD result in Figure 1 it can be seen that in such a pressure scale there is a phase transformation of Pb from the fcc to hcp structure. Therefore, we ascribe the decreased pressure elevation rate to the phase transformation of the Pb sample. Another fact to point out is that for Pt, Pb in the sample chamber can be reasonably considered as a good pressure-transmitting medium that generates highly quasi-hydrostatic condition, therefore the pressure error by Pt can reasonably considered minimal. In addition, the calculated pressures from Pb are highly consistent with that from Pt within the experimental error range. Such a consistency of calculated pressures indicates the suitability and flexibility to use each one of the elements for pressure calibration in our experiments. It also reflects the high quasi-hydrostaticity in the sample chamber, which is further discussed next.

3.2. Minimal strain and stress deviation in Pb

Figure 3(a) shows the representative cake plots of the XRD diffractions of the fcc phase at 9.8 GPa and the hcp phase at 18.5 GPa. It can be seen that the *d*-spacing of crystal planes, both from the fcc and the hcp phases, does not display clear azimuthal dependency,

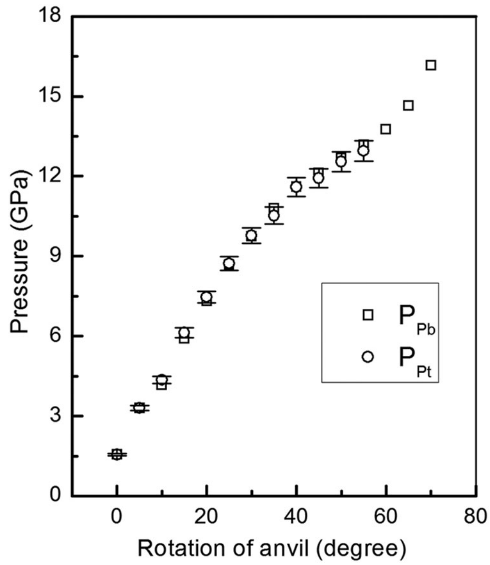

Figure 2. The pressure attained at each anvil rotation. P_{Pb} and P_{Pt} are pressures calculated from EOS of Pb and Pt, Error of P_{Pt} is determined by the error of Pt's unit cell volume.

indicating that the deviatoric stress in the sample chamber is minimal. The *d*-spacing variations of the (111) and (200) planes of the fcc phase with the azimuth angles at pressures are analyzed to further determine the state of strain in the sample. Both (111) and (200) planes show identical trends and Figure 3(b) displays the plots from the (111) plane. By linear fitting the *d*-spacing at different detector azimuths, it can be obtained that the

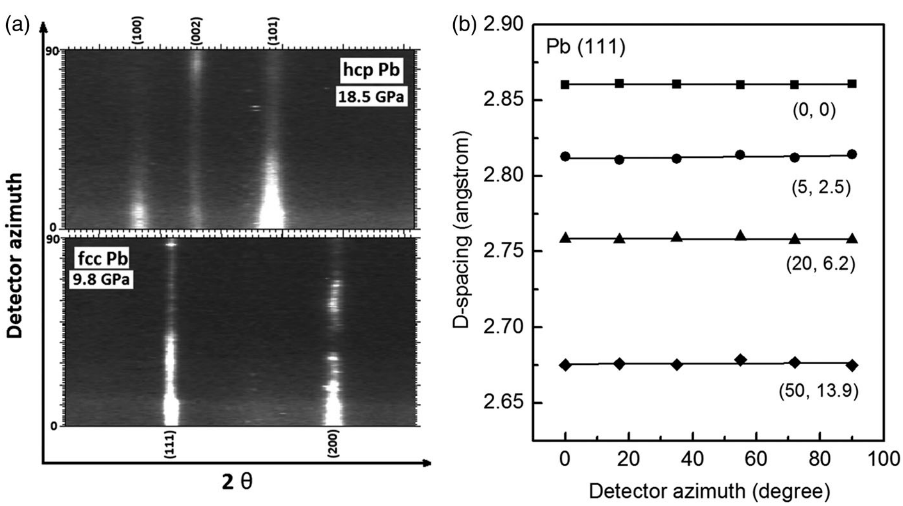

Figure 3. (a) Representative X-ray diffraction patterns by plotting 2θ (horizontal axis) versus azimuth angle (vertical axis). Deviation from a straight line is a measure of lattice strain. (b) The *d*-spacing variations of the (111) planes of the fcc Pb with the azimuth angles at pressures. Solid lines are the linear fit to the data points. Numbers below the plots are the anvil rotation angles (in degree) and pressure (in GPa).

d-spacing variation from the average is in the rate below 10^{-3} range, which is lower than the resolution of the X-ray system. It may be thus considered that *d*-spacing does not vary with the azimuths (within the limit of the X-ray system). This is consistent with the observation in the cake patterns in Figure 3(a). With the change of the detector azimuth from 0 to 90°, the *d* spacing of Pb (111) at each pressure maintains a constant value. From the definition of strain, $\varepsilon = (\Delta d/d_0)$, it can be determined that the strains in the sample also has very high isotropy. It is a novel phenomenon that a quasi-hydrostatic condition is generated in the RDAC chamber after large mechanical shear operation was applied to the sample. Due to such minimal observed strain, it is not meaningful anymore to use the recent popular models based on observable strain divergence at azimuths, for instance by Singh [17] and Xu et al. [18], to do further analysis.

From *d*-spacing (111), (200) and (220), the lattice parameter *a* of the fcc Pb can be calculated, we may use the variation of this parameter to represent the average strain in the sample to evaluate the homogeneity of the shear-promoted pressure. We calculated the strain and the pressure (stress) in the sample along different directions by the integration of the XRD images in different chi angles. The strain–stress relations along 0°, 17°, 35°, 55°, 72°, 90° of chi angles are shown in Figure 4(a). The overlap of the six curves indicates that among different chi angles the strain–pressure relations are similar. In Figure 4(b), through the linear fitting of average strains at the six chi angles, the biggest slope of the lines is 6.0E−5, giving a maximum strain difference of ~5E−3, which is in the experimental resolution range. The minimum strain- and stress deviation indicates that a quasi-hydrostatic condition has been maintained in the application of shear to the Pb sample. Such a novel property of Pb along with the pressure self-multiplication capability presents us with an exceptional potential to use such a material as a pressure-transmitting medium to

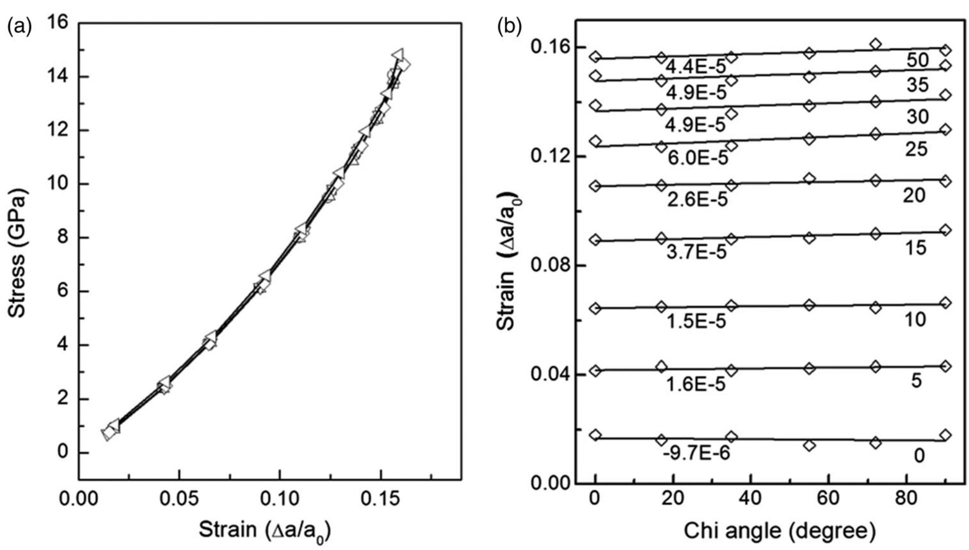

Figure 4. (a) The strain-stress relation in the Pb sample along different chi angles. (b) The strain deviation along different chi angles in the sample. Solid lines are the linear fit to the data points. The numbers below the lines are slopes of the fitted lines. Numbers on the right are the anvil rotation angles (in degree).

generate acceptable homogeneous conditions at high pressures promoted from shear. We expect this to lead to interesting high pressure technology.

3.3. Phase transformation of Pb

In the experiment where only lead powder was loaded into the sample chamber, the pressure of the sample was first ramped up to 0.9 GPa before application of anvil rotation. Initially, we observed five diffraction peaks from the sample that can be indexed to the (111), (200), (220), (311) and (222) planes of fcc Pb (Figure 1). With rotation, these five peaks commonly shift to higher 2θ angles. This phenomenon means the pressure in the sample increased with the relative rotation of the anvils. When the rotation reached 35° corresponding to 12.8 GPa, a new peak at 2.5063 Å appeared. This new peak can be indexed to the (101) peak of the hcp phase of Pb. This turned out to be the beginning of the fcc-hcp phase transformation in the sample. When the rotation reached 40° and the pressure correspondingly reached 14.1 GPa, three other diffraction peaks indexed to the hcp (100), (102) and (201) were also observed. In succession, with an increase in the rotation angle, these new appeared peaks became stronger; at the same time the intensity of the original fcc peaks was getting weaker. At 18.5 GPa all fcc peaks disappeared and thus Pb completely transformed into its hcp structure. Our experiment therefore showed that the fcc–hcp transformation of Pb by shear in an RDAC starts at 12.8 GPa and completes at 18.5 GPa. Some experimental and theoretical results of Pb's phase transformation pressure [16,19–21] are listed in Table 1. Compared with the precious results, the hcp-phase-in pressure of the present Pb induced by the application shear and by hydrostatic compression is surprisingly identical. When Pb was compressed in the nonhydrostatic condition with solid as the pressure-transmitting medium, the start of the fcc-hcp transformation was at a relatively lower pressure because of the concentration of stress. [13] We may therefore further believe that a quasi-hydrostatic pressure condition has been generated and maintained in all of our operations. Furthermore, the stress condition of such attained pressure in the sample chamber is equivalent to that from compression.

4. Conclusions

In conclusion, the application of a large plastic shear leads to the successive pressure promotion on the subjected sample. Soft materials such as lead can regenerate homogeneous pressure by shear, that is, they can be considered as good pressure-transmitting medium in shear. With such a medium, the high pressure properties, such as the transformation

Table 1. Fcc-hcp phase transformation pressures from the present Pb sample compared with previous experimental and theoretical results. The referred experiments were executed in DAC with different pressure transmitting media.

	hcp in (GPa)	fcc out (GPa)
Pb alone (present)	12.8	18.5
Pb alone [16]	11.0	15.0
Methanol–Ethanol [16]	13.1	15.5
NaCl [19]	12.0	20.0
Theoretical work [20]	13.7	–
Theoretical work [21]	16.5	–

pressure, can be identical to hydrostatic compression even with large plastic shear. Such a novel behavior, for example, the quasi-hydrostatic pressure launched by shear, and the pressure self-multiplication phenomenon, presents us with a new potential of future high pressure technology.

Disclosure statement

No potential conflict of interest was reported by the authors.

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