I. INTRODUCTION

The deformation behavior of materials is fundamental to physics, engineering, and geoscience and is determined by both elastic and plastic properties.1 While a variety of methods can be employed to independently measure elasticity and plasticity at ambient conditions, X-ray diffraction is one of the only established techniques that can be used to explore these properties in situ at extreme pressures.2–4 Existing diffraction-based methods use diffraction line shifts2–4 and widths5,6 to constrain material deformation but typically suffer from uncertainties due to confounding of multiple variables and limited control and measurement of experimental conditions such as the applied stress.5,7 The uncertainties in high-pressure measurements of strength and anisotropy can be evaluated by systematic study of simple materials such as platinum metal.

Platinum (Pt) is a face-centered cubic (FCC) metal and does not undergo phase transitions up to multi-megabar pressures.7,8 Due in part to its simple structure, Pt is widely used in high-pressure experiments as a pressure calibrant and laser absorber.9 Nanoparticles of Pt10 and other metals11 have also been observed to have anomalously low compressibility, and this has possible implications for pressure calibration.8,14 The key difference between these studies may be grain size. In the former study, sample grain size was not characterized, but in the latter, much higher strength was observed for 20-nm Pt grains relative to 300-nm grains.7 The Hall-Petch effect, i.e., higher strength at nm-scale grain size, is consistent with this difference. However, these two previous studies of Pt reached their different conclusions using different X-ray diffraction geometries (radial16 vs. axial7) and diagnostic techniques (diffraction line shifts16 vs. line broadening7).

Diffraction-based measurements of deformation in a diamond anvil cell (DAC) can be performed in either an axial geometry (with incident X-rays along the loading axis) or in a radial geometry (with incident X-rays perpendicular to the loading axis). The radial geometry provides the most detailed measurement of strain at high pressures in the diamond anvil cell. Unlike the axial geometry used in the study by Singh et al.,7 this technique samples a wide range of crystallite orientations and is sensitive to elastic constants and texture as well as stress. Radial X-ray diffraction of platinum has been limited to pressures below 24 GPa so far. In this work, we re-examine the effect of grain size on the strength of platinum. We also extend measurements of the strength and deformation texture of Pt by X-ray diffraction in a radial geometry to 63 GPa.

II. THEORETICAL BACKGROUND

Compression of polycrystalline materials in the diamond anvil cell produces non-hydrostatic stresses due to the effects of both uniaxial loading (macro-stress) and of stress heterogeneities at grain boundaries (micro-stress).5,3 In the diamond anvil cell, the maximum stress, σ3, is approximately aligned with the compression axis and the minimum stress, σ1, is in the plane of the gasket. The differential stress, τ, is
defined as the difference between these stresses, \( \sigma_3 - \sigma_1 \). Differential stress reflects the non-hydrostatic stress components that result in deviatoric strain. For a given lattice plane in a crystallite, the deviatoric strain depends on its orientation relative to the compression axis and the elastic anisotropy of the crystal. Microscopic deviatoric stresses also produce micro-strains in the polycrystalline aggregate. Both macro-strain and micro-strain are limited by the yield strength of the material, which may vary with pressure, grain size, and deformation rate.\(^1\) After yielding, the total strain is a combination of elastic and plastic deformation.\(^{17-21}\)

Lattice strain analysis of radial X-ray diffraction data has been described in detail elsewhere;\(^2-4\) here we summarize the major relations used to calculate strain and stress in energy- and angle-dispersive diffraction modes. The variation of measured lattice spacings, \( d_m(hkl) \), can be described by a spacing resulting from the hydrostatic component of stress, \( d_p(hkl) \), plus a non-hydrostatic component:

\[
d_m(hkl) = d_p(hkl)[1 + (1 - 3 \cos^2 \psi)Q(hkl)].
\]

The angle, \( \psi \), is defined between the lattice plane normal and the direction of maximum stress. \( \psi \) is related to the diffraction angle, \( \theta \), and the azimuthal orientation of the diamond anvil cell relative to the detector, \( \delta \), (defined as \( 0^\circ \) at the diamond anvil cell compression axis) as \( \cos \psi = \cos \delta \cos \theta \) (Figure 1). \( Q(hkl) \) is a function of \( t \) and the shear modulus \( G \), where \( G_R(hkl) \) is the modulus under iso-stress (Reuss bound) conditions and \( G_V \) under iso-strain (Voigt bound):

\[
Q(hkl) = \frac{t}{3} \left( \frac{\alpha}{2G_R(hkl)} + \frac{1 - \alpha}{2G_V} \right).
\]

The parameter \( \alpha \) is a measure of the degree of stress/strain continuity across crystallite boundaries. Under iso-stress conditions, \( \alpha = 1 \); measured values of \( \alpha \) typically range from 0.5 to 1, although it has been suggested that values greater than 1 may occur depending on the elastic anisotropy of the material.\(^{22,23}\) For materials of cubic symmetry, the following equations relate \( G_R(hkl) \) and \( G_V \) to the elastic compliances, \( S_{ij} \), and the Miller indices of the lattice plane:

\[
\frac{1}{2G_R} = S_{11} - S_{12} - 3S_{44},
\]

\[
S = S_{11} - S_{12} - \frac{S_{44}}{2},
\]

\[
\frac{1}{2G_V} = \frac{5}{2} \left[ (S_{11} - S_{12})S_{44} \right],
\]

\[
\Gamma(hkl) = \frac{h^2k^2 + k^2l^2 + l^2h^2}{(h^2 + k^2 + l^2)^2}.
\]

In Eqs. (3) and (4), \( S \) is a measure of the elastic anisotropy of a cubic crystal.

As a complement to lattice strain analysis, diffraction line widths can be used to constrain micro-strain, \( \eta \), and grain size, \( D \).\(^5,7\) For diffraction using monochromatic X-rays,\(^{24,25}\)

\[
(w_{hkl} \cos \theta_{hkl})^2 = \left( \frac{\lambda}{D} \right)^2 + \eta_{hkl}^2 \sin^2 \theta_{hkl}.
\]

In this equation, \( w_{hkl} \) is the full width at half maximum of the diffraction peak and \( \lambda \) is the X-ray wavelength. The slope of the linear relationship between \( (w_{hkl} \cos \theta_{hkl})^2 \) and

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X-ray Diffraction Geometries

**Energy-dispersive radial geometry**

- Synchrotron X-ray source
- White X-rays
- Detector
- Sample rotation
- Diffraction plane normal
- Diffused beam
- Lattice planes
- \( \sigma_3 \)
- \( \sigma_1 \)
- \( \delta \)
- \( \omega \)

**Angle-dispersive radial geometry**

- Synchrotron X-ray source
- Monochromatic X-rays
- Area detector
- Diffraction plane normal
- Lattice planes
- \( \sigma_3 \)
- \( \sigma_1 \)
- \( \delta \)
- \( \omega \)

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FIG. 1. Schematic of radial-geometry X-ray diffraction experiments in energy-dispersive and angle-dispersive modes.
sin² θ terms is used to determine average micro-strain, while the intercept provides the grain size. An additional correction is needed to account for instrumental broadening.

Lattice strain theory has been successfully applied to the high-pressure deformation of metals, ceramics, and minerals. Both stress and elastic constants have been derived for materials undergoing purely elastic deformation. However, on compression beyond the yield strength, elastic constants were observed to deviate from values derived from other techniques such as ultrasonic interferometry or Brillouin spectroscopy. For this case, more complex models have been developed to account for plastic deformation. When the elastic moduli are known independently, the lattice strain technique has been demonstrated to provide accurate estimates of non-hydrostatic stress.

### III. METHOD

Polycrystalline Pt samples were obtained with three different particle sizes. Particle shapes and sizes were characterized by scanning electron microscopy. The bulk sample (designated B) is made up of irregular platelets with median Feret diameter ~2 μm and minimum dimension ~450 nm (supplementary Figure 1). Nano-grained samples N1 and N2 are sub-spherical particles with median diameters 300 and 70 nm and minimum diameters 70 and 40 nm, respectively (supplementary Figures 2 and 3). We note that particle sizes measured by microscopy represent upper bounds on grain size of these samples.

Each powdered sample was packed in a 100-μm sample chamber drilled in an X-ray transparent beryllium (Be) gasket. X-ray diffraction of the gasket material shows doublets for each Be peak, suggesting the presence of both Be and an exsolved Be-rich alloy phase due to impurities in the gasket. We also observed hexagonal BeO, most likely resulting from oxidation of Be grain surfaces. All samples were loaded in symmetric or panoramic diamond anvil cells with 300-μm diameter diamond culets. A 10–20-μm diameter piece of Mo was loaded at the center of the diamond culet on one side as position marker. Upon compression, sample chamber diameters typically shrank to 50–80 μm.

Both energy-dispersive (EDXD) and angle-dispersive (ADXD) X-ray diffraction experiments were performed at beamline X17C of the National Synchrotron Light Source. Both methods were used for experiments with samples B and N1. Sample N2 was examined only with the ADXD method. Diamond anvil cells were mounted such that incident X-rays entered and exited the sample radially through the Be gasket (Figure 1).

In energy-dispersive X-ray diffraction experiments in a radial geometry (Figure 1), the symmetric DAC was rotated around the beam axis to angles δ = 0°, 24°, 35°, 45°, 55°, 66°, and 90°. The incident X-ray beam size was ~50 x 50 μm. Diffracted X-rays were collected using a solid-state Ge detector positioned at diffraction angle 2θ = 10°–12°. Detector energy calibration was performed using fluorescent standards, and detector angle was calibrated with an Au standard.

In radial angle-dispersive X-ray diffraction experiments in a radial geometry (Figure 1), the symmetric DAC was rotated around the beam axis to angles δ = 0°, 24°, 35°, 45°, 55°, 66°, and 90°. The incident X-ray beam size was ~50 x 50 μm. Diffracted X-rays were collected using a solid-state Ge detector positioned at diffraction angle 2θ = 10°–12°. Detector energy calibration was performed using fluorescent standards, and detector angle was calibrated with an Au standard.

In radial angle-dispersive X-ray diffraction experiments in a radial geometry (Figure 1), the symmetric DAC was rotated around the beam axis to angles δ = 0°, 24°, 35°, 45°, 55°, 66°, and 90°. The incident X-ray beam size was ~50 x 50 μm. Diffracted X-rays were collected using a solid-state Ge detector positioned at diffraction angle 2θ = 10°–12°. Detector energy calibration was performed using fluorescent standards, and detector angle was calibrated with an Au standard.

We observed that the diffraction patterns in the radial geometry were not affected by the rotation of the DAC. However, in the panoramic geometry, the diffraction patterns showed a systematic shift of the diffraction peaks with increasing angle δ. This effect was more pronounced for the sample with the smallest grain size (N2).

In summary, the lattice strain technique was successfully applied to polycrystalline Pt samples with different grain sizes. The method provided accurate estimates of non-hydrostatic stress, even beyond the yield strength of the material. The results showed that the elastic constants deviated from those derived from other techniques, and more complex models were needed to account for plastic deformation. The method was also shown to be effective in the high-pressure deformation of metals, ceramics, and minerals.
was observed at 2θ values of more than 25°, but over only a limited azimuthal range of 80°–190°. Detector distance and orientation were calibrated with CeO2 and Au standards. The wavelength of the monochromatic X-rays was 0.4066 Å, and beam size was ~20 × 30 μm.

IV. RESULTS

Samples were compressed to maximum pressures of 48–63 GPa. After each pressure increase, samples were allowed to relax for 30–120 min, or until the measured pressure stabilized. Example diffraction patterns from an EDXD measurement of sample B at 48 GPa are shown in Figure 2. An example two-dimensional ADXD pattern of sample N1 at 52 GPa is shown in Figure 3. All diffraction lines correspond to the Pt sample, Mo marker, and Be and BeO in the gasket. Gasket diffraction peaks are highly textured and invariant with pressure. Lattice d-spacings of Pt and the weak Mo marker change with azimuth δ from the loading axis due to differences in stress. Texture development in Pt can also be observed as intensity variations along the azimuthal angle, δ, with different maxima for different lattice orientations.

EDXD patterns were analyzed by fitting peaks to Voigt lineshapes. ADXD patterns were integrated using FIT2D at azimuthal intervals of 4°–5° over a range from δ = 45°–145°, the maximum azimuthal range at which the (222) line is observed at all pressures. Full-profile strain and texture analysis were performed with MAUD software (details are given in supplementary material). Pressure was determined from the lattice parameter of Pt as measured at δ = 55°, the angle at which the measured d-spacing corresponds to the hydrostatic d-spacing under uniaxial conditions. As in the previous studies of strength and deformation of Pt, we calibrate pressure with the equation of state of Holmes et al.

A. Line shift analysis

For each diffraction line, a linear relationship between lattice spacing and the orientation function 1–3cos²ψ is observed (Figure 4 and supplementary Figure 4). Curvature in this relationship may be due either to stress variations in the diffraction volume or non-uniaxial stress. For EDXD data, Q(hkl) was determined from the slope and intercept of this linear relationship as in Eq. (1). For ADXD data, Q(hkl) in Pt was refined from full-profiles in MAUD (Figure 5). Pt diffraction peak widths were fit by refining grain size and micro-strain. We fit each Q(hkl) and the orientation of the maximum strain. The angle between the maximum strain and the compression axis, β, was 6°–27° at maximum pressure.

FIG. 4. Lattice parameter and d-spacings for Pt from EDXD for nanograin sample N1 at 61 GPa. Spacings for (111) planes are shown in red dots, (200) = blue squares, (220) = yellow up-pointing triangles, (311) = green down-pointing triangles, and (222) = pink diamonds. Ranges of d-spacings on plots are scaled to a constant percentage of the value at ψ = 55°.
pressures reached for both ADXD and EDXD measurements (supplementary Figure 4). This deviation from uniaxial stress has been observed in other studies and may be due to deformation of the gasket hole at low pressures and bending of diamond anvils at higher pressures. The relative importance of these two mechanisms changes from run to run. In one sample, the angle was observed to increase to 20°–30° by 10 GPa (gasket deformation), while other samples exhibited more continuous increase at higher pressures (diamond bending). By correcting the orientation of the ADXD data, the curvature of lattice spacing vs. 1–3cos²ψ can be almost entirely removed (supplementary Figure 4), suggesting that the stress orientation is the dominant cause of observed non-linearity.

The observed lattice strain increases strongly with pressure until the sample yields, above which pressure the lattice strain increases at a weaker rate (Figure 6(a)). Difference between EDXD and ADXD measurements represents run-to-run differences in the magnitude and orientation of deviatoric stress. Higher strain was observed for the nano-grained samples N1 and N2 at all pressures relative to the bulk sample. For the bulk sample, we could not resolve the pressure at which yielding occurred due to the low strain exhibited and the size of the pressure steps. For samples N1 and N2, yielding occurred at 9 GPa, similar to previous observations of sub-micron Pt. According to purely elastic models, the strain function $Q(hkl)$ is linearly related to the crystal orientation function $\Gamma(hkl)$ as in Eqs. (2)–(6). The slope of this linear relationship is related to the elastic anisotropy, $S$, of Pt as well as the magnitude of the differential stress. Due to the high elastic anisotropy of Pt, the observed $Q(hkl)$ depends strongly on lattice plane (Figure 6(b)). The linear relationship between $Q(hkl)$ and $\Gamma(hkl)$ can be used to determine the change in elastic anisotropy with pressure. The measured elastic anisotropy is lower than that predicted for Pt by ab initio density functional theory. This discrepancy is due to effects of plastic deformation on $Q(hkl)$. Plastic deformation can be neglected if elastic compliances $S_{ij}$ are fixed for analysis of stress conditions.

While the elastic constants of Pt are well-constrained at 1 bar and high temperature, their evolution with pressure has not been determined by experiment. Only the elastic modulus $C_{44}$ was measured by ultrasonic interferometry (and only to pressures <0.3 GPa). The elastic constants of Pt were computed by ab initio density functional theory. As the authors noted, the predicted ambient pressure derivative of the $C_{44}$ modulus, $C_{44}' = dC_{44}/dP$, is significantly larger than the ultrasonic value. In this study (as in the previous study by Singh et al.), we use the ultrasonic value for $C_{44}'$ and other zero-pressure elastic moduli and
pressure derivatives from density functional theory.\textsuperscript{38,39} The computed and ultrasonic values for zero-pressure elastic constants and their pressure derivatives were extrapolated using the finite strain equations.\textsuperscript{40} One additional parameter is required to solve Eqs. (2)–(6): the stress-strain continuity parameter, \( \alpha \). Previous analysis of both peak width and line shifts data for Pt suggested that the best value for \( \alpha \) is 0.6.\textsuperscript{7} We calculate differential stress assuming \( \alpha \) to be either 0.6 or 1 and observe a difference in \( t \) of \( \sim 1.5\% \), much lower than other uncertainties. Differential stress assuming \( \alpha = 0.6 \) is plotted in Figure 7.

Differential stress in the bulk Pt sample reaches a maximum of 1–1.5 GPa at 60 GPa (Figure 7). This is consistent with axial diffraction measurements of linewidths that are used to constrain the strength of Pt with 300-nm grain size\textsuperscript{7} but considerably lower than previous radial diffraction measurements of line shifts.\textsuperscript{16} We concur with Singh \textit{et al.}\textsuperscript{7} that the difference between these studies is likely due to grain size differences. The nano-scale samples, N1 and N2, examined in this study exhibited much higher yield strengths of 5–6 GPa at 60 GPa. These stresses are comparable to those observed in Ref. 16 and slightly lower than those observed in Ref. 7 for 20-nm grains, consistent with the particle size of 70–300 nm.

### B. Peak width analysis

Diffraction peak widths typically increase with pressure due to increasing micro-strain and stress gradient across the sample. Grain size reduction may also increase peak widths for nanocrystalline samples. Peak broadening in these experiments was analyzed by peak fitting as well as analysis using MAUD.

Observed peak widths were consistent with grain sizes similar to particle sizes observed by SEM and remained approximately constant with pressure (Figure 8 and supplementary Figure 5).\textsuperscript{32} For \( \sim 300\text{-nm} \) N1 sample particles, grain size determined by peak widths was 200–400-nm. Diffraction peak widths of the bulk-grained sample were within error of the instrument resolution at loading. Fits to the bulk sample peak widths at 20–60 GPa yield a grain size of \( \sim 1 \mu \text{m} \), consistent with the particle size observed by SEM. For the two finer-grained samples, the width of diffraction peaks increased below the yield stress, decreased immediately after yielding, and remained constant above \( \sim 10–20 \text{GPa} \) (Figure 8). Changes in peak widths are most likely due to buildup of micro-strain before yielding and release of micro-strain through plastic flow after yielding. Other possible mechanisms for peak sharpening are grain growth, which is unlikely at 300 K, or collapse of the gasket hole, reducing the pressure gradient over the measured sample.\textsuperscript{41} No significant peak broadening is observed above the

![FIG. 7. Maximum differential stress, or yield strength, measured for Pt samples of different grain size. Data from this work are shown with filled symbols, with squares for EDXD and circles for ADXD (red = sample B, green = sample N1, and cyan = sample N2). Data from previous work are 20-nm (open diamonds) and 300-nm (filled diamonds) grain sizes studied by axial diffraction peak width analysis in Ref. 7 and unknown-size grains (open squares) studied by EDXD in Ref. 16. Stress was calculated with \( \alpha = 0.6 \).](image)

![FIG. 8. Diffraction peak widths measured for each particle size (red = sample B, green = sample N1, and cyan = sample N2) in ADXD patterns at \( \psi = 90^\circ \), corresponding to the axial geometry. Apparent pressure is computed from \( d \)-spacings observed at \( \psi = 90^\circ \). Due to lattice strain, \( d \)-spacings are larger and apparent pressure lower than those determined at \( \psi = 54.7^\circ \), the orientation of approximately hydrostatic stress, by up to 8, 12, and 17 GPa for samples B, N1, and N2, respectively.](image)
yield stress indicating no significant decrease in grain size due to yielding.

C. Texture analysis

Nano-particle samples initially exhibited continuous, untextured diffraction rings. For the bulk sample, diffraction rings are randomly textured due to large grain size relative to the X-ray beam. Upon compression to comparable pressures of 49–52 GPa (Figures 9(a)–9(c)), stronger textures were observed for the ~300-nm particle size than for smaller or larger particles. In addition, the nature of the texture development differs with particle size. In sample N1 (300-nm size), the grains are oriented to exhibit a maximum in (111) at minimum stress (Figure 9(b)). However, a different texture was observed in the other two samples, with a minimum in the (111) planes at the minimum stress orientation (Figures 9(a) and 9(c)).

These textures were evaluated from ADXD experiments by refinement in MAUD software (Figures 9(d)–9(f)). For sample N1, the texture index is observed to increase strongly from 13 GPa, just above the yield pressure, to 34 GPa, above which pressure it is saturated at ~3 maximum of a random distribution (m.r.d.). The deformation texture in this sample is a (110) fiber texture typical of FCC metals under compression.42 The other two samples are weakly textured, with texture index ~1.1 at maximum pressures. The maximum intensity is found in the (100) direction, more typical of tension in FCC metals at ambient pressure.42

Different textures observed for different particle sizes indicate changes in deformation mechanisms. The main deformation mechanisms in cold-compressed powders are dislocation creep and grain boundary processes. Which of these mechanisms dominates depends on multiple factors including grain size, pressure, temperature, and strain rate. Dislocation creep is thought to dominate deformation in coarse (>100-nm grain size) metals and produces strong lattice-preferred orientation under compression. With decreasing grain size, dislocations become increasingly unfavorable, and grain boundary sliding and rotation dominate deformation in metals with grain size smaller than ~20-nm.43–46 Increasing pressure may support formation of dislocations even in 3-nm particles.44 Unlike dislocation creep, grain rotation was reported to randomize texture.15,44,47,48 In addition, this change in deformation mechanism at small grain sizes was suggested to cause a decrease in strength.45–46

The bulk Pt sample in this study surprisingly does not develop a strong compression texture, yet grain-boundary-mediated processes are not expected to be dominant. The differential stress supported by the sample is low, however, and confirms that the bulk grains are relatively weak. In addition, the irregular particles in this sample should inhibit grain boundary sliding and rotation. The weak tension texture in this sample may reflect low statistics due to large grain size. Random texture from loading of the bulk sample persists at the maximum pressures examined.

The elastic strain observed in the two Pt nano-powders in this study is similar, but texture development differs. The
70-nm and 300-nm particles are both stronger than previously measured 300-nm grain size Pt but weaker than 20-nm grain size Pt. Since only the particle size is known, each sample may be composed of clumps of smaller grains. The weaker texture in the 70-nm particles may indicate that grain sizes in this sample are small enough to promote grain boundary sliding and rotation as a primary mechanism of plastic deformation. Alternatively, the stronger texture in the 300-nm-particle sample may be due to the large deviation from uniaxial stress in this experimental run (supplementary Figure 4(a)). The (100) texture in the 70-nm and bulk Pt samples would then be more representative for Pt under pure uniaxial stress.

V. IMPLICATIONS

While earlier radial diffraction experiments reported that platinum could be unusually strong for an FCC metal with low shear modulus, consideration of grain size effects brings the behavior of this metal into agreement with the general trends at high pressure displayed by other metals. Our radial diffraction results for multiple grain sizes of Pt confirm the low strength of bulk Pt observed by the axial diffraction method by Singh et al. Like Au, another FCC metal, Pt exhibits a differential stress/shear modulus ratio lower than that of metals with body-centered cubic (BCC) or hexagonal close-packed (HCP) structures (Figure 10).

The strong dependence of strength on grain size increases the importance of careful characterization of grain sizes of materials used as differential stress markers and calibrants. The sub-micron Pt samples examined in this work were also used as differential stress markers in previous studies of the equation of state of various materials including MgGeO₃, CaF₂, and Gd₃Ga₅O₁₂. Lattice strain analysis of Pt was performed using the axial geometry in these cases. The effectiveness of Ne or He pressure media and laser annealing for reducing deviatoric stresses at Mbar pressures was evaluated by comparison to the yield strength of the Pt marker. The high strength of sub-micron Pt confirms that the observed differential stress in these previous studies was low relative to the yield strength, and quasi-hydrostatic conditions were achieved. For all similar measurements, the yield strength and grain size of the marker are critical for this evaluation of deviatoric stress.

However, knowledge of the initial grain size of the strain marker may not be sufficient to fully constrain the strength at high pressures and temperatures. Grain size changes with pressure and/or temperature may occur and be difficult to detect. For example, reversible pressure-induced grain size reduction has been reported previously but is not yet understood; this may be due to the difficulty in separating the effects of grain size, micro-strain, and plastic deformation on diffraction peak widths. In laser heating studies, both reduced strength and grain growth are expected. This uncertainty in grain size is thus a limitation to the use of differential stress markers in diamond anvil cell experiments.

This study provides an important test of different techniques for characterizing deformation at high pressure. Our results using multiple techniques demonstrate the consistency of measurements of the strength of bulk and nano-grained Pt by analysis of diffraction peak widths and both energy-dispersive and angle-dispersive X-ray diffraction in a radial geometry. The major sources of uncertainty in these measurements are the poorly constrained high-pressure elastic properties, stress-strain continuity factor, and non-uniaxial stress in the diamond anvil cell. By measuring diffraction across a wide and continuous range of orientations and stress conditions, radial ADXD provides the most complete information for reducing these uncertainties.

VI. SUMMARY

Strength and deformation of Pt powder samples with various grain sizes were examined at pressures up to 63 GPa by radial diffraction experiments in both energy- and angle-dispersive modes. For micron-scale grain size samples, the yield strength of Pt reaches 1–1.5 GPa at ~60 GPa. For nanometer-scale grain sizes, the yield strength is much larger: differential stress of 5–6 GPa is observed at ~60 GPa. These observations confirm that contrasting values of the strength of Pt in previous axial-geometry studies are the result of differences in grain size. Analysis of diffraction peak widths suggests no significant grain size reduction due to yielding or compression. Texture development in the sample with ~300-nm particles is strong and typical of FCC metals under compression. For both larger and smaller particle sizes, observed texture is weaker and more typical of mixed compression and tension in FCC metals. Texture style may also depend on non-uniaxial stress. Platinum, like FCC gold, exhibits a lower strength to shear modulus ratio at high pressure than metals with BCC or HCP structures including Mo, Re, and W. Smaller grain sizes exhibit higher yield strength; characterization of the grain size is thus critical to analysis of strain and stress in high-pressure experiments.
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