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1 **A simple wire-coil resistive heater for high temperature radial X-ray diffraction in a** 2 **diamond anvil cell**

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9 Diamond anvil cells are commonly used at synchrotron X-ray diffraction beamlines
10 to study structural and thermoelastic properties of materials at high pressure. In
11 a radial geometry, where the X-ray probe is oriented perpendicular to the axis of
12 force, the deformation and strength of materials can be measured in situ. Because
13 the anelastic and failure properties of materials depend strongly on temperature,
14 many applications would benefit from the ability to measure high pressure radial
15 diffraction under elevated and accurately-controlled thermal environments. Previous
16 work to introduce high temperature to radial diamond anvil cells has been largely
17 limited to laser heating, with relatively scant efforts to resistively heat the sample.
18 Here we report a relatively straightforward adaptation of a simple wire coil heater,
19 with in situ high-temperature radial diffraction performed on tungsten carbide up to
20 573 K at beamline 12.2.2 of the Advanced Light Source. The results demonstrate
21 that the differential stress supported by WC decreases with increasing temperature:
22 the differential stress on the basal (001) and pyramidal (101) planes decreased 6.6%
23 and 5.5%, respectively, while the (100) plane only saw a 2.7% decrease, in agreement
24 with previous studies.

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25 I. INTRODUCTION

26 Diamond anvil cells (DACs) are tools widely employed to study material properties as
27 a function of pressure. When used in conjunction with a synchrotron X-ray beam, DACs
28 allow for the in situ characterization of a material's atomic structure and/or thermoelastic
29 properties via diffraction. If the DAC is deployed in radial geometry (rDAC), in which the
30 X-ray beam is perpendicular to the strain axis, information on the mechanical properties
31 of the material, including lattice strain, crystal preferred orientation, deviatoric stress, and
32 strength can be obtained (e.g., 1 and 2). This technique has long been used to investigate
33 geologic and planetary materials (e.g., 3–5), as well as in the development of ultrahard and
34 other exotic industrial materials^{6,7}.

35 To simultaneously investigate the effects of elevated temperature, some previous work
36 has developed in situ double-sided laser heating, in which high-powered infrared lasers are
37 directed onto the sample along the strain axis, normal to the X-ray beam^{8,9}. This technique
38 has been successful at reaching temperatures >3000 K, replicating conditions found deep
39 within the Earth and allowing for the study of material rheology that governs Earth processes
40 and observations, such as mantle convection and seismic anisotropy (e.g., 10 and 11). Laser
41 heating in a DAC, however, has limitations that constrain its utility for certain applications.

42 These are well known and reported; a primary issue is large temperature gradients of
43 up to 100s of K per micron¹². In both axial and radial geometries this can introduce large
44 uncertainties, but particularly for rDAC studies, the X-ray beam necessarily probes sample
45 material that is outside the laser hotspot, at temperatures potentially as low as ambient
46 temperature (Figure 1). Additionally, because laser heating relies on spectropyrrometry to
47 measure temperature, it is typically only used for experiments in which the target temper-
48 ature is in excess of 1200 K and the sample emits measurable visible or near-infrared light.
49 For some materials science research, e.g. for materials suitable for next-generation cutting
50 blades, 1200 K is near the upper limit on the temperature range of interest (e.g., 13), which
51 extends down to 500 K. This temperature range requires a different approach.

52 An alternative to laser heating is resistive heating, in which an electrical current is applied
53 across a heater that is in close proximity to the sample. Techniques that employ external
54 heaters, often a wire coil, that surround the diamond tips, or internal heaters, in which the
55 heater is itself within the sample chamber^{14,15}, have both been developed, with the former far

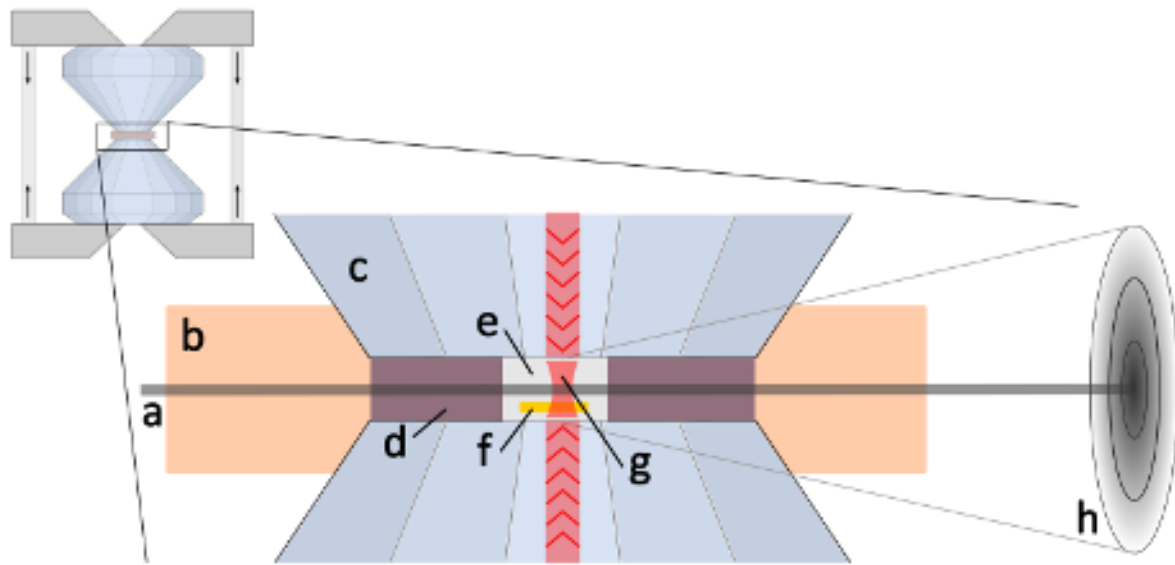


FIG. 1. Schematic of a laser heated DAC in radial X-ray diffraction geometry. a: incoming X-ray beam. b: Kapton. c: Diamond anvil. d: Boron epoxy gasket. e: Sample powder. f: gold flake (pressure standard). g: laser hotspot. h: Debye rings

more common. At synchrotrons, resistively heated DACs are widely used in axial geometry (in which the strain axis is parallel to the X-ray beam), but very few previous studies have employed the technique in radial geometry^{16,17}. This is largely because standard wire-coil heaters fully surround the diamond tips to minimize temperature variations and thus lie in the path of both the incoming X-rays and outgoing diffracted photons. Previous efforts have therefore relied on an alternative style of resistive heating, in which conductive graphite foil is used as the heating element^{16,17}. This method requires extensive, difficult sample preparation techniques, including custom-built instruments, and is consequently highly user-unfriendly, limiting its utility and reproducibility. There have also been efforts to combine resistive and laser heating¹⁸, however resistive heaters can damage DACs and diamonds when employed without an inert atmosphere, and these protective enclosures often preclude the use of laser optics due to space constraints at beamline endstations.

Here we report a modification of a simple wire-coil resistive heater, originally developed for working in axial geometry, used in combination with a commercially-available (DESY, Petra III), modified BX90 rDAC^{19,20}. Initial experiments have been performed on tungsten carbide to a peak temperature of 573 K; we were able to accurately reproduce known mate-

72 rial properties of this extremely hard abrasive, and thus demonstrate the effectiveness and
73 relative ease of the technique.

74 II. METHODS

75 The wire-coil heater was fabricated manually after the method described by Lai et al.
76 (2020) and Wang et al. (2023), using the same pyrophyllite discs, which were obtained from
77 their lab. We made some simple modifications to the pyrophyllite disc and wire placement to
78 allow for diffraction in radial geometry. Namely, in two places, the wires were rerouted into
79 arcs and sections of the pyrophyllite disc were cut away to allow incoming X-rays to reach
80 the sample and resulting diffraction cones to exit without interference, as shown in Figure
81 2. The wires were insulated with a thin coating of alumina cement (Cotronics 940HT) to
82 avoid shorting to the metallic DAC or other components. Beyond these geometric changes,
83 the technique is similar to that deployed by Lai et al. (2020) for axially-probed externally
84 heated measurements. A thermocouple was cemented to one of the diamond anvil pavilions,
85 with the hot junction as close to the culet as possible. The thermocouple leads were threaded
86 through alumina tubing to protect them from the diamonds and DAC body. Previous work
87 in axial geometry has demonstrated that this setup accurately reflects the temperature inside
88 the sample chamber to within approximately 20 degrees^{23,24}.

89 To confirm that this remains true even with large segments of the heater removed, we
90 conducted a series of experiments in which two thermocouples were employed: one with the
91 hot junction pressed between the diamonds (i.e., where a sample would be), and a second
92 with the hot junction on one of the diamond pavilions, as during a standard experiment.

93 We performed multiple of these test experiments, varying the placement of the second
94 thermocouple from the culet and the heater. We found that placing the probe thermocouple
95 far enough down the pavilion, such that it was no longer within the inner diameter of the
96 heater, led to a difference in measured temperature of 200 degrees at 300 C. We should also
97 note that an additional variable in cell geometry is the placement of the heater in relation
98 to the diamonds: it is absolutely essential that the heater effectively surrounds the diamond
99 tips (and hence the sample) and does not fall in one direction such that the diamond tips are
100 protruding above or below the plane of the heater. This happened accidentally during one
101 test experiment; in this case, the probe thermocouple was inside the heater but the sample

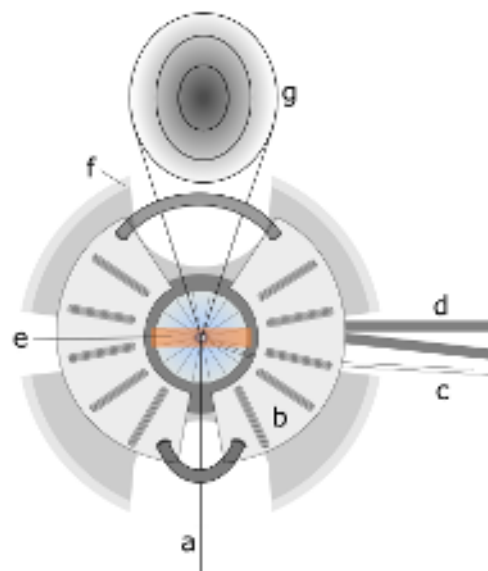


FIG. 2. Schematic of the modified wire-coil heater in a BX90 rDAC. A: incoming X-ray beam. B: pyrophyllite disc with wire coils; arched wires are directed towards the viewer. C: Thermocouple, with hot junction cemented to the diamond anvil near the sample. D: electrical leads. E: Kapton support, with boron epoxy gasket and sample at center on the diamond culet. F: rDAC body, with large portholes allowing a 30 degree opening angle for diffraction cones. G: Debye rings.

was not and the measured temperature difference was again over 200 degrees. However, as long as both the sample and the thermocouple were within the inner diameter of the heater, the measured temperatures agreed within 10 degrees. We are therefore confident that the measured temperature closely reflects the sample environment provided these conditions are met.

In terms of response time, measured temperature increases rapidly after increasing current to the heater, and equilibrates within about 10 minutes. The reproducibility of runs (power vs temperatures) when using the same heater is high. However, as the heaters are made by hand, and can be made using wires of various compositions (for this study, all heaters were made with nichrome wire, however we have also fabricated heaters with Pt and W wires), it is premature to assess the overall reproducibility of the power-temperature relation.

We note that the experiments reported here were performed in air, with no vacuum chamber or protective inert atmosphere. This limits the maximum temperature to that at which the diamonds would graphitize or the DAC itself would degrade; hence, we limited

the maximum temperature to approx. 573 K. This was done for simplicity, but we note that a few hundred degrees above ambient conditions is the peak temperature of interest for some applications, and no additional sample preparation is needed. For experiments with a higher maximum temperature, an inert gas enclosure is available at beamline 12.2.2. The upper limits of temperature possible for this setup under a controlled atmosphere will be explored in future experiments, but preliminary tests indicate at least 773 K is achievable.

For radial diffraction in a DAC, the gasket must be X-ray transparent; in this case, it was fabricated from boron epoxy, with a Kapton support. This is an increasingly common rDAC technique, the details of which have been reported elsewhere¹⁰. Polycrystalline tungsten carbide (ThermoFisher, 99 %) was added to the chamber and compressed without a pressure medium; a flake of gold was also loaded into the sample chamber to act as a pressure sensor. X-ray diffraction was performed at beamline 12.2.2 of the Advanced Light Source, Lawrence Berkeley National Laboratory. Pressure was increased with a gas membrane, and the heater was powered by a TDK-Lambda 20/20 power supply, both of which were remotely controlled from outside the experiment hutch.

The angle-dispersive diffraction patterns were converted from elliptical to rectangular coordinates using FIT2D; the converted plot of azimuthal angle (η) versus diffraction angles (2θ), known as a “cake” pattern, was then analyzed using Igor Pro (WaveMetrics, Inc.). Peak positions were determined individually for three easily resolvable diffraction peaks (001, 100, and 101) by fitting the diffraction lines in the cake pattern to a sinusoidal curve with 1° increments according to Equation 1:

$$2\theta_m(hkl) = 2\theta_p(hkl)[1 + (1 - 3\cos^2\varphi)A] \quad (1)$$

where $2\theta_m$ is the measured peak position, $2\theta_p$ is the hydrostatic peak position, φ is the angle between the diffraction normal and compression directions, and A is the amplitude of the sinusoidal variation. The stress in the sample under uniaxial compression is described by Equation 2:

$$\sigma = \begin{bmatrix} \sigma_1 & 0 & 0 \\ 0 & \sigma_1 & 0 \\ 0 & 0 & \sigma_3 \end{bmatrix} = \begin{bmatrix} \sigma_p & 0 & 0 \\ 0 & \sigma_p & 0 \\ 0 & 0 & \sigma_p \end{bmatrix} = \begin{bmatrix} -t/3 & 0 & 0 \\ 0 & -t/3 & 0 \\ 0 & 0 & -2t/3 \end{bmatrix} \quad (2)$$

where σ_1 is the minimum stress along the incident X-ray beam, σ_3 is the maximum stress in the compression direction, σ_p is the hydrostatic stress component, and t is the differential

stress, which gives a lower-bound estimate of yield strength. The d-spacing is calculated with equation 3:

$$d_m(hkl) = d_p(hkl)[1 + (1 - 3 \cos^2 \varphi)Q(hkl)] \quad (3)$$

where d_m is the measured d-spacing, d_p is the d-spacing at the hydrostatic peak position, and $Q(hkl)$ is the lattice strain under the uniaxial stress condition. The quasi-hydrostatic diffraction pattern as a function of 2θ was obtained at the magic angle ($\varphi = 54.7^\circ$, effectively the hydrostatic condition). The differential stress, t , is directly related to the differential strain, $t(hkl)/G(hkl)$, by:

$$t(hkl) = 6G(hkl)Q(hkl) \quad (4)$$

where $G(hkl)$ is the shear modulus of the specific lattice plane.

III. RESULTS

To demonstrate the effectiveness of the technique, we collected radial X-ray diffraction patterns of tungsten carbide (WC), a material that has been previously well characterized at high temperatures²⁵ and high pressures²⁶, and compared our results. The pressure-temperature path involved compressing to a given pressure and collecting data at each temperature (ambient pre-heating, 100 °C, 200 °C, 300 °C and ambient post-heating) before increasing pressure again. The P-T path of the experiment is shown in Figure 3. Note that at higher pressures, the pressure does not return to the same point upon cooling. We attribute that to small changes in the distance between the anvils resulting from the thermal expansion of the various DAC components.

A set of representative 1-dimensional full integration and 2-dimensional “cake” patterns are shown in Figure 4. The cake patterns show a clear shift to higher 2-theta values with increasing pressure and sinusoidal variations of the diffraction lines along the azimuthal direction as a result of the anisotropic stress state created by the uniaxial compression. These effects also appear in the 1D integrations as peaks shift to higher 2-theta values and peak broadening at higher pressures since the integration is averaged over varying stress states. Visually, there is substantially less difference between the highest pressure XRD patterns at 30 °C and 300 °C, as expected for a refractory ceramic like WC for which the strength increases with pressure.

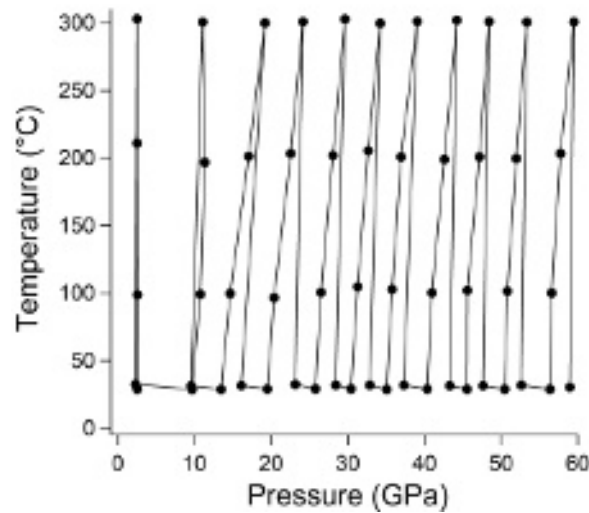


FIG. 3. Pressure-temperature path for this experiment. Each temperature point was collected without manually increasing pressure at each pressure step.

To determine the effect of temperature on the yield strength of WC, the d-spacing for each lattice plane of interest was plotted against the orientation function $(1 - 3 \cos^2 \varphi)$. In good agreement with lattice strain theory^{27,28}, a linear correlation is shown at the highest pressure (Figure 5). Pseudo-hydrostatic d-spacing values, $d_p(hkl)$, can be determined from the zero-intercept of the plot and the lattice strain under uniaxial stress conditions, $Q(hkl)$, is calculated from the ratio of the slope and zero-intercept according to Equation 3. The pressure dependence of the pseudo-hydrostatic d-spacing is plotted in Figure 5(b) and shows a continuous, linear decrease for all temperatures, indicating no phase transitions.

Using the $Q(hkl)$ calculated above, and an aggregate shear modulus under iso-stress (Reuss) conditions for WC²⁹, the differential stress for a given lattice plane $t(hkl)$ can be calculated according to Equation 4. As shown in Figure 6, the measured differential stress increases rapidly as the pressure increases at low pressures, followed by a decrease in slope at higher pressures. This change in slope is indicative of the transition from elastic to plastic deformation under the nonhydrostatic pressure. The differential stress at the plateau corresponds to the maximum elastic deformation that the lattice can withstand before bond breaking, allowing us to directly correlate the plateau (or in this case, near plateau) to the intrinsic hardness of the material via the flow strength^{28,30,31}. While the flow strength estimated from the plateau value of the differential stress is not the same as the hardness, the values have been shown to be very strongly related³²⁻³⁷.

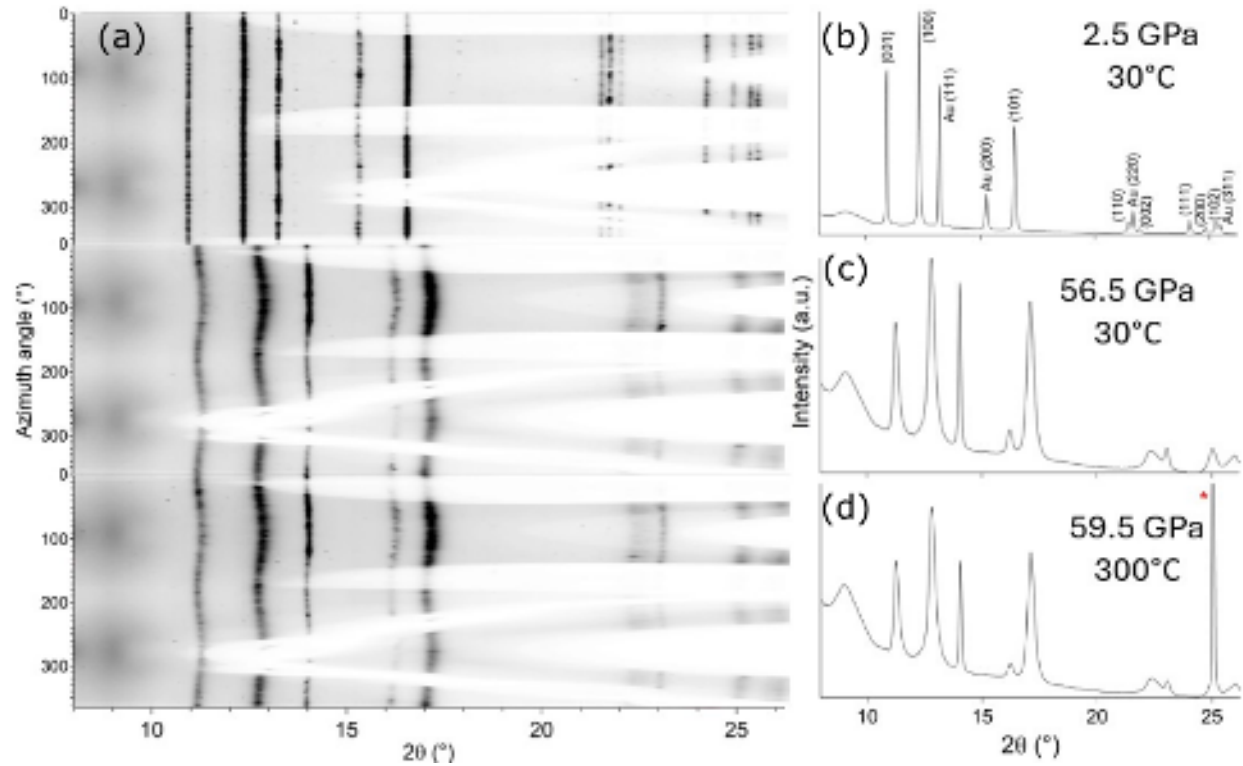


FIG. 4. Representative X-ray diffraction patterns in the form of 1-dimension full integrations (right) and 2-dimensional “cake” patterns (left). Note the 2-theta shift and sinusoidal diffraction lines in the cake patterns and 2-theta shift and peak broadening in the 1D integrations as a result of the increased pressure. The white stripes in the left panel correspond to regions of the X-ray detector that are eclipsed by the heater.

As shown in Figures 6 and 7(a), the differential stress that is supported by the WC sample, averaged over the highest 15 GPa region, decreases as the temperature increases in all studied lattice planes, indicating softening at the high temperatures. The stress for the sample at ambient temperature after the heating cycle seems to show a very similar trend to that demonstrated by the sample at high temperature, as is expected from the relaxation of the sample at high temperature. However, while all planes softened at elevated temperatures, they did not all soften equally. The basal (001) and pyramidal (101) planes saw a decrease of about 6.6% and 5.5% respectively, both of which are in good agreement with temperature dependent Knoop hardness data for polycrystalline WC reported by Lee as shown in Figure 7(b)²⁵. Interestingly, the (100) plane shows much less change at high temperatures than the other studied planes, with only a 2.7% decrease in its plateau value

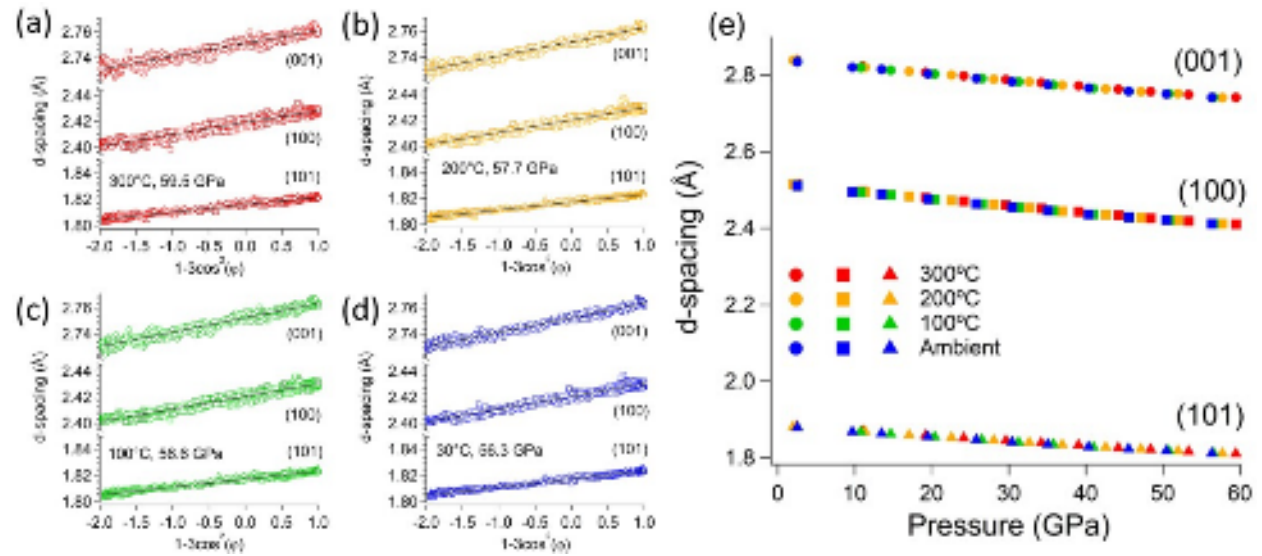


FIG. 5. (a) d-spacing versus the orientation function $(1 - 3\cos^2\varphi)$ for the (001), (100) and (101) planes at (a) 300 °C, (b) 200 °C, (c) 100 °C, and (d) 30 °C. Note the breaks in the y-axes to facilitate visualizing the small changes. The linear trend indicates good agreement with lattice strain theory. (e) Pressure dependent pseudo-hydrostatic d-spacing measured at the magic angle ($\varphi = 54.7^\circ$, where $(1 - 3\cos^2\varphi) = 0$).

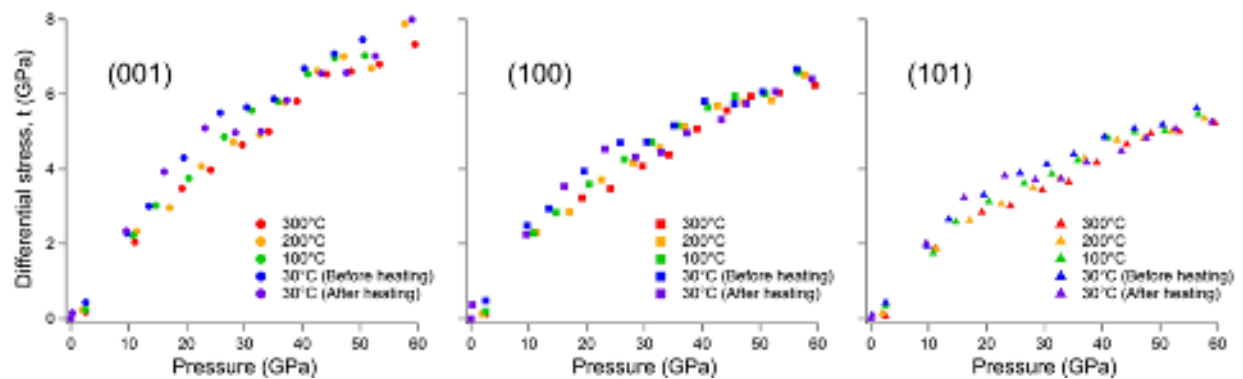


FIG. 6. Differential stress as a function of pressure for the (001), (100), and (110) lattice planes at 300 °C, 200 °C, 100 °C, and 30 °C.

of differential stress. This anisotropic effect of temperature on the yield stress of different lattice planes can also be seen in the high-temperature Knoop hardness measurements. We note, however, that additional analysis, such as texture analysis and plasticity simulations, would be required to fully understand the effect of this anisotropy, and how we would expect it to impact the elastic and viscoplastic deformations of the material at these temperatures.

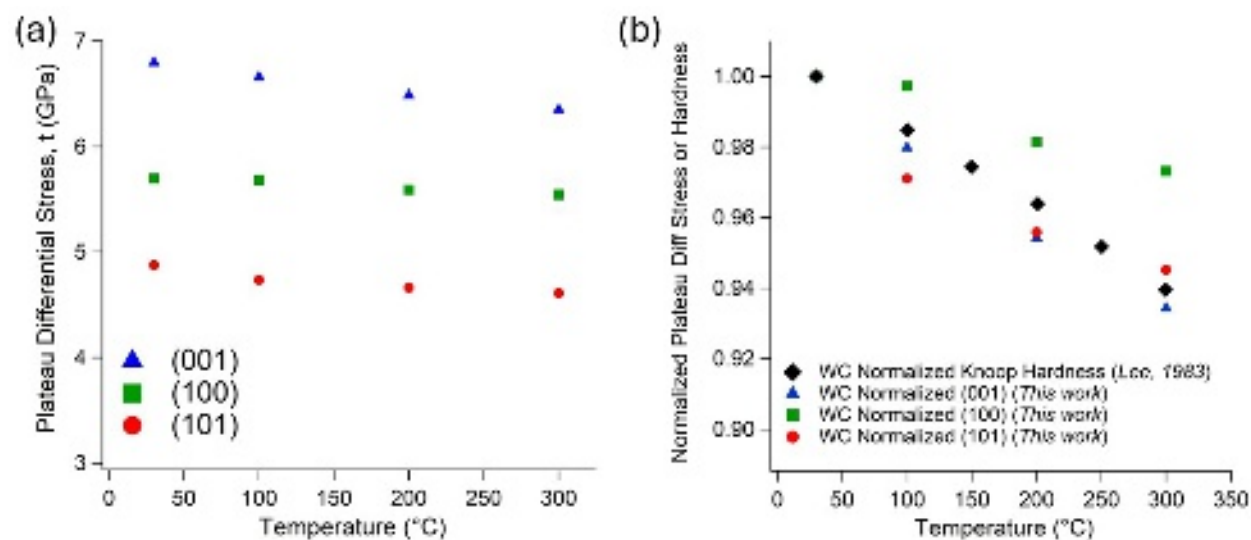


FIG. 7. (a) Plateaued differential stress, calculated as the average differential stress between 45-60 GPa, as a function of temperature. (b) Normalized differential stress vs temperature from this work compared to normalized Knoop hardness vs temperature from Lee, 1983.

IV. CONCLUSIONS

Overall, the results on the high-temperature strength of tungsten carbide and their consistency with previously reported data support the effectiveness of the resistive heating technique as a viable means for studying the temperature dependent mechanical properties of materials in a diamond anvil cell. The relative ease of setup and availability of the technique for synchrotron users, including its straightforward deployment within a commercially available cell make this resistive heating method an attractive option for high-temperature radial diffraction studies. Future experiments to higher temperatures will be explored by using this technique in conjunction with an inert-atmosphere enclosure available to general users of Advanced Light Source beamline 12.2.2.

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