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Diamond anvil cell measurement of high-pressure yield strength of vanadium using *in situ* thickness determination

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The pressure dependence of the quasistatic yield strength of vanadium in polycrystalline foils and powders has been measured up to 90 GPa at room temperature using an implementation of a nonhydrostatic diamond-anvil cell technique, an extension of the technique of Meade and Jeanloz [C. Meade and R. Jeanloz, J. Geophys. Res. **93**, 3261 (1988)]. A feature present in this work is the use of an *in situ* determination of the sample thickness and pressure profile using synchrotron x-ray techniques, allowing a determination of the strength at each point across the sample. Following an initial increase in the strength with pressure, a decrease in the strength of vanadium was observed starting at ~40–50 GPa. The softening of the yield strength of vanadium at high pressure is quite surprising and unusual, following the trend of an unusual softening of the shear modulus associated with a subtle phase transition from body-centered-cubic-to-rhombohedral structure in a recent x-ray diffraction experiment.

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I. INTRODUCTION

Vanadium exhibits unusual mechanical properties under pressure as a result of subtle electronic structure effects. Its unique properties make it particularly attractive for the study of the effects of elastic anisotropy and weak martensitic phase transformations on material strength. Vanadium is a nonmagnetic group Va transition-metal possessing a body-centered-cubic (bcc) crystal structure and refractory properties at ambient pressure. It has technological applications, including structural members in fusion reactors, a bonding agent for titanium and steel, and it is an important component of specialty steel alloys. Pure vanadium exhibits an elastic anisotropy due to pressure softening of the C_{44} shear modulus,[1–3] which is common to the group Va elements. However, only in vanadium has C_{44} been calculated to become negative in the zero-temperature bcc phase (at ~140 GPa). The pressure softening is of interest because of its impact on strength and other mechanical properties[3] and also because it enhances the electron-phonon coupling leading to an anomalously high superconductivity transition temperature.[4] The negative modulus calculated in the theoretical studies[1] suggests a pressure-induced structural instability in vanadium. This prediction was confirmed by recent x-ray diffraction studies, which observed a structural transformation in vanadium from a bcc phase to a rhombohedral phase at about 69 GPa.[5]

This unusual behavior of C_{44} may also impact the pressure dependence of the yield strength of vanadium. The yield strength is the minimum stress necessary to induce plastic deformation. Lower stresses only cause reversible elastic deformation. The shear modulus sets a natural scale for material strength,[6] so it may be expected that the extraordinary softening of the shear modulus will lead to an effect on the strength at pressure, perhaps even a reduction of the strength at pressure. This decreased strength would be unusual since, as Bridgman observed, metals commonly show enhanced strength and ductility by applying small amounts of load/pressure.[7] Several mechanisms, applicable to conventional bcc metals, have been proposed to account for the pressure dependence of the yield strength.[8] The strength may also be affected by the pressure dependence of the order parameter of the rhombohedral phase at pressures above the transition. Here we report our use of the diamond-anvil cell (DAC) to measure the strength of pure vanadium at room temperature at pressures up to 90 GPa. The results provide experimental data to challenge and validate theoretical models and develop insights into the fundamental mechanisms governing the pressure-dependent yield strength of vanadium.

The DAC is a useful and extremely versatile device for high-pressure experimental studies.[9] The capabilities of the DAC to rapidly access high pressures and the availability of modern 3rd generation synchrotron x-ray sources have enabled studies of the mechanical properties of various materials at high pressures.[10–12]

Previously, Meade and Jeanloz reported yield strength measurements of MgO in a DAC based on measurement of the sample thickness and radial pressure gradient, determined using a ruby fluorescence method.[13] The radial pressure gradient dP/dr is related to the shear stress σ_{rz} (and strength) of the material through the mechanical equilibrium equations.[14] In an axially symmetric configuration, such as in an ideal DAC sample, this relationship is given by $\sigma_{rz} \approx (h/2)(dP/dr)$ where h is the sample thickness. Throughout a large region of the sample, starting from a distance of approximately one sample thickness from the center and extending to approximately one sample thickness from the edge, the yield stress Y of the sample can be approximated as twice the shear stress σ_{rz} neglecting other components of the stress under two assumptions:[15] (1) that the sample does not slip at the diamond surface and (2) that the radial and azimuthal stresses are approximately equal,[11,16]

$$Y = 2\sigma_{rz} \approx h(dP/dr). \qquad (1)$$

The factor of 2 here comes from using the Tresca yield criterion.[15] Finite element modeling of this DAC configuration conducted in separate studies supports our analysis using Eq. (1).[11,17] We determined the radial pressure gradient (dP/dr) by measuring the pressure distribution across the vanadium sample in the DAC. Energy- and angle-dispersive diffraction with an x-ray beam focused to a spot size of ~10 μm were used to determine the crystal structure and lattice parameters. Using these parameters combined with the known equation of state (EOS) of vanadium yielded the radial pressure profile. The sample thickness h was determined using the following *in situ* x-ray absorption technique. When an x-ray is transmitted through the sample, the intensity of the transmitted x-ray decreases exponentially with the thickness. This effect can be used to determine the thickness, h according to

$$h = \log(I_0/I)/(\mu \bullet \rho) \qquad (2)$$

where I is the transmitted intensity, I_0 is the incident intensity, μ is the mass absorption coefficient, and ρ is the sample density. The values of I and I_0 were measured. μ is assumed to be pressure independent and was calculated from the slope of the absorbance ($A = -\log I/I_0$) as a function of foil thickness for a set of reference foils at ambient conditions. We also corrected for the effect of the absorption due to the diamond anvils themselves. Before applying pressure the initial absorption was measured, which corresponded to the sum of the absorption due to both the diamond anvils and the foil. This initial measurement served as a calibration to correct for the diamond absorption is subsequent measurements. The sample density, ρ, was determined by *in situ* x-ray diffraction. The sample thickness, h, can then be obtained from Eq. (2), from the known values of μ and ρ and the measured absorbance.

II. EXPERIMENTAL

We used two experimental approaches for our measurements. In one case we used an energy dispersive x-ray diffraction (EDXD) system (16BMB at HPCAT/Advanced Photon Source/Argonne National Laboratory). This experiment was performed with 200 μm diameter diamonds mounted in a DAC. A rhenium gasket, preindented to a thickness of 25 μm and drilled with a ~80-μm-diameter hole, was packed with vanadium powder (99.99% Alfa Aesar ~325 mesh, ~36 μm grain size). EDXD patterns for pressure determination were collected at a spatial resolution of ~10 μm [full width at half maximum (FWHM)] and a Ge point detector positioned at 12° from the direct beam ($E \bullet d = 59.3073$). Incident and transmitted intensities of the x-ray beam were measured using two MIC-205 ion chambers from Advanced Design Consulting USA, Inc. Data were collected up to a pressure of 90 GPa. To estimate the sample thickness, absorption of the rhenium gasket was measured. A number of rhenium foils indented to various thicknesses were used to get an effective-mass absorption coefficient for a white beam. In this case the sample thickness was assumed to be uniform, independent of the radial location along the sample. X-ray diffraction patterns and the absorption were collected at several points separated by 10 μm along the gasket hole diameter and perpendicular to the diamond culet.

The second experimental approach utilized an angle-dispersive x-ray diffraction (ADXD) system (16BMD at HPCAT/Advanced Photon Source/Argonne National Laboratory). This experiment was carried out with 300 μm culet diamonds. Instead of studying vanadium powder packed in a gasket hole, a 100-μm-thick vanadium foil was squeezed between the 300 μm culet diamonds. A series of vanadium foils of 100, 75, 50 and 25 μm thicknesses (99.8% purity with <50 ppm Al, Ca, Cr, Cu, Ni, Sn and 150 ppm Mo, Si and Ti and 200 ppm Fe, ESPI metals) was used to calibrate the absorption measurements with PDC254d-PN diodes (Detection Technology). We used an incident monochromatic x-ray beam with a size of ~10 μm (FWHM), $\lambda = 0.387451$ Å and an image plate detection system (MAR345 imaging plate detector). Incident and transmitted intensities of the x-ray beam were measured as described above. One difference in this second set of experiments was the use of a series of vanadium foils instead of rhenium foils to calibrate the sample thickness measurement as a function of pressure. This approach allowed us to directly measure the thickness of the sample as a function of position, $h(r)$. The pressure in the DAC was incrementally increased. Following each pressure increment, x-ray diffraction patterns and the absorption scans were collected at several points spaced by 30 μm across the diameter of the diamond culet to determine the pressure, radial pressure gradient, and thickness across the sample. The maximum pressure for this set of data was 65 GPa. X-ray diffraction patterns were converted from two-dimensional images to intensity versus 2θ with the FIT2D program[18] and then the structure and lattice parameters were determined using the XRDA (Ref. 19) program.

The vanadium EOS reported by Ding *et al.*[5] was used to calculate the pressure from our measurement of the lattice parameters of vanadium. It is important to note that in the work of Ding *et al.* the rhombohedral and cubic phases lie, within experimental accuracy, on a single pressure-volume EOS curve. Over a broad range following the bcc-rhombohedral transition, the diffraction patterns of these phases are very similar; the diffraction pattern of the high-pressure rhombohedral phase differs from the bcc phase in line broadenings rather than well resolved peak splittings.[5] Thus we have made all of our pressure and density evaluations from the diffraction measurements using the nonhydrostatic EOS of Ding *et al.*[5] In this way, we avoid complications related to identifying the precise transition pressure, which is difficult to discern given the resolution of our diffraction measurements. The analysis methodology for the yield strength is not affected by this simplification, since it does not depend on the crystal structure, only the thickness and pressure gradient.

III. RESULTS

Selected EDXD patterns for the vanadium powder up to 80 GPa are stacked in Fig. 1(a). Asterisks in the figure denote

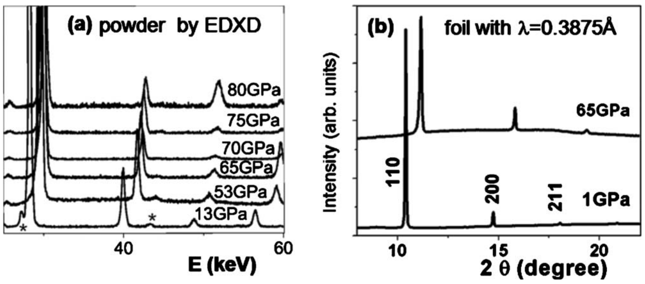

FIG. 1. (a) Stack plot of selected energy dispersive powder x-ray diffraction patterns of vanadium powder. The asterisks indicate diffraction peaks from the Re gasket. (b) Angle dispersive powder x-ray diffraction patterns of vanadium foil with λ=0.3875 Å. Top and bottom lines correspond to the patterns at 65 and 1 GPa, respectively.

small peaks from the rhenium gasket. Angle-dispersive powder x-ray diffraction patterns for the vanadium foil at 1 and at 65 GPa with λ=0.38745 Å are shown in Fig. 1(b). When the cubic phase $(\boldsymbol{a}_C, \boldsymbol{b}_C, \boldsymbol{c}_C)$ distorts to the rhombohedral phase $(\boldsymbol{a}_R, \boldsymbol{b}_R, \boldsymbol{c}_R) = (-\frac{1}{2}\boldsymbol{a}_C + \frac{1}{2}\boldsymbol{b}_C + \frac{1}{2}\boldsymbol{c}_C, \frac{1}{2}\boldsymbol{a}_C - \frac{1}{2}\boldsymbol{b}_C + \frac{1}{2}\boldsymbol{c}_C, \frac{1}{2}\boldsymbol{a}_C + \frac{1}{2}\boldsymbol{b}_C - \frac{1}{2}\boldsymbol{c}_C)$, the $(110)_C$ line splits into $(100)_R$ and $(\bar{1}10)_R$, and the $(211)_C$ line splits into three lines $[(110)_R$, $(2\bar{1}0)_R$ and $(2\bar{1}\bar{1})_R]$, whereas the $(200)_C$ line remains a single line $(\bar{1}11)_R$. We do not observe the (110) or (211) peaks of the bcc phase to split into multiple peaks up to 65 GPa in the ADXD data.

Since pressure was applied very slowly, the strain rates were very small ($\sim 10^{-3}$ to $\sim 7 \times 10^{-2}$ s^{-1}).[13,20] We assume the yield strength (Y) is equivalent to the sample thickness multiplied by the radial pressure gradient as in Eq. (1).[13] Figure 2(a) shows the ADXD measured pressure (filled squares) and sample thickness (open squares) as a function of radius along the horizontal diameter of the 300 μm culet for a given DAC load. To obtain the yield strength of the given DAC load, the data were analyzed conventionally following the approach of Meade and Jeanloz:[13]

(1) the average radial pressure gradient at the given DAC load was obtained from the average slope of the pressure versus radius plot;

(2) the pressure at the center was used to represent the given load; and

(3) the thickness at the given pressure load was taken from the value at the sample center.

Using Eq. (1), the yield strength of vanadium was calculated by multiplying the average radial pressure gradient and the thickness at the pressure of the center of the sample and plotted in Fig. 2(b). For the example shown in Fig. 2(a), the yield strength at the pressure 39.8 GPa is 3.633 GPa inferred from the average dP/dr, 0.194 GPa/μm, and the thickness at the center, 18.7 μm. From the contribution of the deviation in dP/dr and the variation in sample thickness over the points, the error of the strength is estimated as ±0.8 GPa.

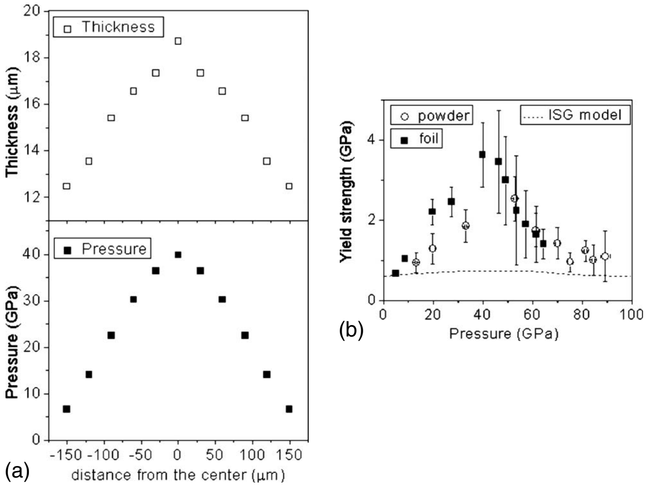

FIG. 2. (a) For a given DAC load condition, the pressure (GPa) and thickness (μm) as a function of the distance from the center (μm). The pressure and thickness are represented by filled and open squares, respectively. (b) The yield strength as a function of pressure obtained by multiplying the average dP/dr and the sample thickness at the center. Solid squares and open circles represent the points from ADXD with foil and from EDXD with powder, respectively. The dashed line indicates the calculated yield strength as a function of pressure based on the ISG model (see text) which also increases to ~45 GPa and then decreases. However, the magnitude of the predicted changes is much smaller than the measured strength.

Our experimental yield strength values at ambient condition are 0.336 GPa for ADXD and 0.488 GPa for EDXD. The yield strength is very small compared to that of high strength steels at ambient pressure, 2.5 GPa. This softness is consistent with the use of vanadium as bonding material. Steinberg *et al.* developed a model in which the pressure dependence of the yield strength is assumed to be the same as that of the shear modulus: $Y/Y_0 = G(P)/G(0)$ where Y and G are the yield strength and the polycrystalline shear modulus at a given pressure P, and Y_0 is the yield strength at ambient pressure.[21] By using reported values for Y_0 (Ref. 22) and the calculated pressure-dependent shear modulus model[3] $G(P)$ for vanadium, the improved Steinberg-Guinan yield strength $Y_{\mathrm{ISG}} = Y_0 \bullet G(P)/G(0)$ is shown for reference as a dashed line in Fig. 2(b). Our measured vanadium yield strength values are notably higher than the theoretical predictions.

We observed the powder sample has lower strength than the foil up to ~50 GPa. The powder becomes more efficiently packed and achieves the maximum density above 10 GPa. However, the foil is a compact form of the polycrystalline material. The strength difference may be caused by the different pressure-strain paths as is earlier work on measuring strength as a function of pressure.[11,13] Moreover, the fabrication process (e.g., rolling) introduces residual stresses into the sample. Here we attribute the difference in strength of the powder and foil samples in Fig. 2(b) to both initial conditions of strain and residual stress.

IV. DISCUSSION

We observed two distinct regimes in the strength of vanadium under pressure. The strength increases with pressure up to ~40 GPa; as the pressure increases further, the strength decreases until ~80 GPa and then remains approximately constant up to the highest pressure achieved in our experiment, 90 GPa. Extrapolation of the low-pressure data to ambient pressure gives a yield strength value that is consistent with published values.[22] The increase in strength with pressure at low pressures is consistent with pressure strengthening observed in many materials, including other bcc metals.[7] The shear modulus sets a natural scale for the strength, as mentioned in the discussion of the SG model above. Several authors have argued that the pressure dependence of the strength should follow that of the shear modulus[6,21] and many papers have reported results from strength experiments on materials that exhibit this relationship.[23] There are materials that do not exhibit a simple relationship between strength and the shear modulus,[11,13] and we have seen above that our measured strength for vanadium is one such case. In these cases, this result is attributed to strain hardening since the DAC sample must undergo significant shear strain as it is pressurized to 10s of GPa. In most materials both the strength and the shear modulus increase monotonically with pressure within a single phase. The decrease in strength above ~40–50 GPa observed in our vanadium study is unusual. Kinsland and Bassett reported that the strength of MgO plateaus at ~5 GPa,[24] and Weir *et al.* reported that the strength of Ta increases up to ~40 GPa and then decreases from ~80 to ~100 GPa (Ref. 11) in contrast to other experiments that have found the Ta strength increases smoothly with pressure to ~100 GPa, as discussed below. This decrease is attributed to "strain softening," citing earlier work by Bridgman showing softening due to inhomogeneities[7] and by Meade and Jeanloz discussing preferred grain orientations.[13] In those materials the shear modulus increases monotonically with pressure. In vanadium the situation is quite different. A second-order phase transition of vanadium was reported at 65–69 GPa (Ref. 5) and more recently as low as 35 GPa.[25,26] This bcc-to-rhombohedral phase transition is expected to be a martensitic transformation in which the cubic symmetry is reduced to a trigonal symmetry through a diffusion-free distortion of the lattice.[3,27,28] It is widely believed that particular features of the Fermi surface (FS) could cause a premartensitic phonon softening and lead to the martensitic transformation.[29] Recent *ab initio* calculations on vanadium elastic constants and shear modulus[1,3] support the hypothesis that vanadium undergoes a mechanical instability in C_{44} due to a FS effect. The shear modulus of polycrystalline vanadium has been calculated to increase up to ~45 GPa, and then to decrease with further increase in pressure up to ~120 GPa.[3] The strength of vanadium shows very similar trends, peaking at 40–50 GPa. However, the strength is superelastic: the magnitude of the increase and decrease in strength is much greater than that of the shear modulus. The decrease in the calculated shear modulus is a precursor to the rhombohedral transformation. Unfortunately our work was not able to resolve the subtle changes in the diffraction pattern associated with the bcc-to-rhombohedral transition.

The unusual decrease in the strength with pressure motivates us to conduct the analysis of the strength using a second approach for comparison. The pressure varies throughout the sample and this variation is used to determine the strength through the radial pressure gradient. In the approach used above, the strength was determined from a linear fit to the varying pressure, and that value was assigned to a single pressure, the peak pressure in the sample, when reported in Fig. 2(b). In principle the foil data sets, $h(r)$ and $P(r)$, contain more information than just the average slope and peak thickness as seen in Fig. 2(a). The stress equilibrium relationship that leads to Eq. (1) can be applied at each point in the material, provided the underlying assumptions are valid [see Fig. 3(a)]. So at each value of the radius, we can calculate piecewise values for the yield stress, $Y(r) \approx h(r) \bullet (dP(r)/dr)$. Here the values near the center ($r \approx h$ plus beam size) and near the edges are excluded since other components of the shear stress dominate. These areas are shown as the crosshatched regions in Fig. 3(a). All of the data for the pressures calculated in this piecewise manner are shown by the filled squares in Fig. 3(b). Using this alternate approach we find the pressure corresponding to the maximum yield strength to be lower pressure by ~5 GPa compared to the data analyzed by the conventional method of Meade and Jeanloz in Fig. 2(b). This shift to lower pressure is a result of the fact that the center pressure is higher than the corresponding piecewise pressures. Moreover, the yield strength measurements using the piecewise approach, below the pressure of the maximum yield strength, lie higher than those of the conventional approach, while those above the pressure of the maximum yield strength coincide in both approaches. This result is attributed to the fact that the tangent, $dP(r)/dr$, is higher than the average dP/dr obtained from the slope and that the pressure decreases faster than the thickness as r increase (r is the distance away from the center). Overall, the trends of the yield strength as a function of pressure are very similar between the two analysis methods. The piecewise approach has the advantage of removing the ambiguity in the pressure to which the strength should be assigned.

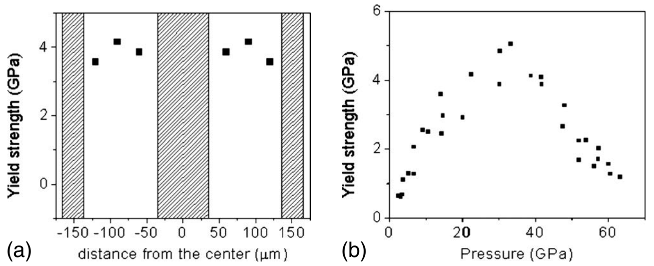

FIG. 3. (a) The yield strength as a function of the distance for the foil sample is calculated by multiplying the pressure gradient and thickness at each position according to Eq. (1) from Fig. 2(a) and is represented as filled squares. The crosshatched regions represent the excluded areas in which stress components other than σ_{rz} are expected to dominate due to symmetry and/or boundary conditions. (b) The strength calculated by the piecewise approach over all the load pressures.

Changes or nonuniform distributions of the intensity around Debye-Scherrer diffraction rings are indicators of texture/preferred orientation. The ADXD experiment allowed us to observe this texture/preferred orientation effect under uniaxial compression. From our studies, Fig. 4 shows selected images of the two-dimensional diffraction patterns of a vanadium foil in a DAC at the sample center ($r=0$). Sev-

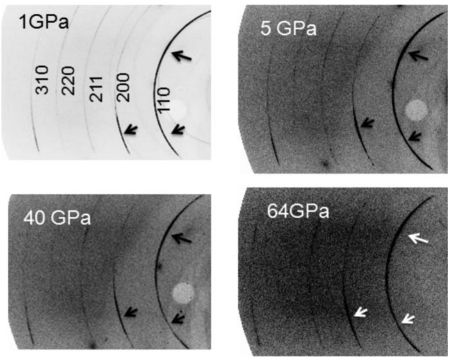

FIG. 4. X-ray diffraction patterns of vanadium foil sample at (a) 1, (b) 5, (c) 40, and (d) 64 GPa on MAR3450 image plates. Arrows are used to indicate the arcs of the (110) and (200) rings which show intensity changes.

eral arcs of the (110) and (200) diffraction rings of the vanadium foil are indicated with arrows. Their intensities relative to the rest of the arcs change significantly as pressure increases from 1, to 5, to 40 GPa. This trend suggests that the degree of preferred crystallographic orientation in the sample is enhanced as pressure increases. Some researchers propose that this texture/preferred orientation of the sample may cause strain softening in the yield strength,[11,13] an effect that may be particularly strong in vanadium at high pressure due to its elastic anisotropy.[2]

Tantalum (Ta), in the same periodic table group Va as vanadium, is also predicted to exhibit an anomaly in C' and C_{44} at high pressure in which the shear moduli are insensitive to pressure in the range of 1 to 2 Mbar.[30] The yield strength of Ta was reported in various experiment conditions; nonhydrostatic conditions using powders by Cynn[31] and hydrostatic conditions with single crystals by Dewaele.[10] The yield strength in both experiments is found to be 2.6–3 GPa at 50 GPa, which is comparable to that of vanadium. Furthermore, these experiments show a smooth increase in the tantalum yield strength as the pressure increases up to ~100 GPa, while vanadium shows a decrease at 40–50 GPa. This difference in properties may originate from the structural stability differences between tantalum and vanadium. Tantalum is stable as bcc up to a few hundred GPa and higher while vanadium transforms to a rhombohedral structure at much lower pressure.

V. CONCLUSIONS

We have determined the yield strength of vanadium up to 90 GPa. In this study, we used x-ray diffraction and absorption techniques to make *in situ* measurements of the pressure gradient and the sample thickness. These measurements were used to evaluate the pressure dependence of the material strength. We observed an initial increase in strength with pressure followed by a decease in the yield strength of vanadium starting at ~40–50 GPa and continuing until 80 GPa and then roughly constant up to 90 GPa. Our experimentally determined pressure dependence of the strength of vanadium behaves similarly to the predicted shear modulus pressure dependence.[3] The decrease in the yield strength as a function of pressure appears to occur prior to the bcc-to-rhombohedral phase transition and is followed by a slow increase in rhombohedral stability region. We have demonstrated a new piecewise analysis approach that removes the ambiguity in assigning a particular pressure to a given measured value of the strength. This method yields results that are consistent with the conventional approach of Meade and Jeanloz.[13]

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