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## Measurement of Body-Centered Cubic Gold and Melting under Shock Compression

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We combined laser shock compression with in situ x-ray diffraction, to probe the crystallographic state of gold (Au) on it's principal shock Hugoniot. Au has long been recognized as an important calibration standard in diamond anvil cell experiments due to the stability of its face-centered cubic (fcc) structure to extremely high pressures (P >600 GPa at 300 K). This is in contrast to density functional theory and first principles calculations of the high-pressure phases of Au that predict a variety of fcc-like structures with different stacking arrangements at intermediate pressures. In this work, we probe high-pressure and high-temperature conditions on the shock Hugoniot and observe fcc Au at 169 GPa and the first evidence of body-centered cubic (bcc) Au at 223 GPa. Upon further compression, the bcc phase is observed in coexistence with liquid scattering as the Hugoniot crosses the Au melt curve before 322 GPa. The results suggest a triple point on the Au phase diagram that lies very close to the principal shock Hugoniot near  $\sim$  220 GPa.

8 of the noble metals. It is one of the least reactive chemical 9 elements and the face centered cubic (fcc) crystal structure is predicted to be stable over hundreds of GPa [1-5]. Its chemcal inertness and the stability of its crystal structure, along with its scattering efficiency, makes Au a particularly useful pressure standard for high-pressure diamond anvil cell (DAC) experiments [6, 7]. Shock compression experiments up to 10 TPa (1 TPa = 1000 GPa = 10 MBar) showed no obvious discontinuities in shock velocity / particle velocity data, which has been used to suggest the absence of solid-solid phase transitions and a very small volume change on melting [8–13].

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A wide range of *ab initio* calculations first predicted structural transformations in Au at pressures ranging between 151 and 620 GPa from the fcc crystal structure (with ABC stacking) to a variety of close-packed structures with alternate stacking of the Au atoms in the close packed direction: doule hexagonal close packed (dhcp) with ABAC stacking [1, 2], nexagonal close packed (hcp) with AB stacking [1–5], and also an ABCACB stacking structure [2]. In 2002, a body centered cubic (bcc) structure was proposed to be a stable at high pressure, due to hybridization of the 5p states with the 5d band [4]. More recently, first principles calculations predicted a transformation to the *hcp* and *bcc* structures at 255 and 480 GPa, respectively, along the 300 K isotherm [14].

The theoretical prediction for phase transition along the 300 K isotherm were in good agreement with conventional DAC data that used *in situ* heating with x-ray diffraction to observe the appearance of new Bragg peaks that could be indexed to hcp structure, at  $\sim 248$  GPa and 860 K [1]. Upon cooling slowly to room temperature, those new peaks increased in intensity as the fcc peaks decreased. Recent double-stage DAC and toroidal DAC 300 K isotherm measurements show only the fcc structure at all pressures up to 1065 and 603 GPa respectively [7, 15]. Due to these discrepancies between 42 different static measurements and theoretical predictions, our

Gold (Au) is perhaps the most recognizable and precious 45 principles calculations. Shock compression provides an al-46 ternative pathway to probing materials at high pressures and 47 temperatures beyond the reach of static compression in the <sup>48</sup> DAC. Recent advances in user facilities, such as the Dynamic 49 Compression Sector (DCS) at the Advanced Photon Source 50 (APS) synchrotron, have allowed high-quality synchrotron x-51 ray diffraction (XRD) to be collected under shock compres-52 sion [16].

> In this letter, we present our study of the crystal structures 54 of Au up to a maximum pressure of 322(27) GPa along the 55 principal shock Hugoniot. The fcc phase is observed up to 56 169(8) GPa. Coexistence of fcc with bcc Au is observed at 57 223(11) and 253(19) GPa. The fcc to bcc phase transition is 58 completed by 262(10) GPa, where we observe a coexistence 59 of bcc Au with liquid scattering on the Hugoniot, suggesting 60 only a small pressure range on the shock Hugoniot where the 61 bcc phase alone is stable. At the maximum pressure obtained, 62 322(27) GPa, only liquid Au is observed and melting on the 63 Hugoniot is complete. The results indicate the presence of a 64 triple point near the principle shock Hugoniot at  $\sim 220(20)$ 65 GPa. These measurements improve our understanding of the 66 Au phase diagram and bolster the recent theoretical predic-67 tions [14].

> Experiments were carried out at DCS [16], using the geom-69 etry shown in Figure 1a. A high-energy laser (351 nm) [17], <sub>70</sub> focused to a 500  $\mu$ m diameter focal spot, sent an ablatively-71 driven shockwave through the sample package. As shown in 72 Figure 1b, shock targets consisted of 50  $\mu$ m polyimide with <sub>73</sub> an Al flash coating ( $\sim 0.1 \ \mu \text{m}$ ) on one side and a deposition <sub>74</sub> of a 6.8  $\mu$ m thick Au layer on the other. A 500  $\mu$ m thick sin-75 gle crystal [100] Lithium Fluoride (LiF) window was glued to <sub>76</sub> the polyimide/Au with an estimated glue thickness of 1-3  $\mu$ m. 77 The LiF was coated with  $\sim 0.1~\mu \mathrm{m}$  of Al to enhance reflec-78 tivity for velocity measurements. Further information can be 79 found in the Supplementary Material [18].

The drive laser and x-rays were  $7^{\circ}$  and  $52^{\circ}$  from sample 43 current understanding of the Au phase diagram is inadequate 81 normal, respectively (Figure 1a). Laser energies of up to 44 and further experimental data is needed to benchmark first  $_{82} \sim 75 \text{ J}$  (using a 10 ns flat top pulse shape) and  $\sim 63 \text{ J}$  (using a 10 ns flat

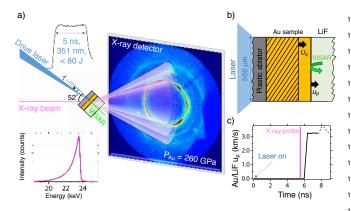


FIG. 1. a) Schematic overview of experimental setup at the Dynamic Compression Sector, APS. The x-ray beam (spectral flux shown) probes the sample at an angle 52° from sample normal, with the diffracted x-rays from the sample recorded on the detector  $\sim 100 \ \text{mm}$ away. b) Sketch of the target assembly. The x-ray beam probes the full sample at a time equal to or before the shockwave (traveling at shock velocity U<sub>S</sub>) reaches the LiF window. The compressed region behind the shock front is highlighted by the shaded region in the Au sample. c) The measured Au/LiF particle velocity history  $(u_p)$  from VISAR for the compressed data are also shown; the dashed portion of the VISAR trace is related to the reverberation/release wave interactions that occur several nanoseconds after the sample probe time.

ing a 5 ns flat top pulse shape) sent shocks of up to  $\sim 322$ GPa through the Au. The distinct pressure states that could be accessed were dependent on the discrete laser intensities available. Pressure was determined using a point VISAR Velocity Interferometer System for Any Reflector) and from

A pink x-ray beam from a U17 undulator, shown inset to Fig- 151 surements is observed (green triangles). ure 1a, provided a quasi monochromatic x-ray flux spectrum 152 Gauss') to fit the experimental data [18].

GPa. The lowest pressure obtained in this study (P = 169(8) 166 tectable solid at 322 GPa). GPa) was obtained using the maximum energy of the laser 167

with a 10 ns laser pulse length ( $\sim$ 75 J); the remaining data used a 5 ns pulse length. Figure 2 shows a series of integrated XRD profiles from 0 GPa (ambient foil before compression) up to maximum pressure. At 169 GPa, a shift of the ambient <sub>116</sub> fcc peaks to higher Q (where  $Q = 4\pi \sin \theta / \lambda$  and  $\lambda$  is defined as the peak of the spectral flux from Figure 1a) is observed as the unit cell is compressed. Least squares fitting of all diffraction peaks, using the 'Exp-Gauss' function, shows a good fit to expected positions of ambient fcc Au and compressed <sub>121</sub> fcc Au (purple triangles in Figure 2). Due to the highly tex-122 tured nature of the deposited Au sample, intensity variations are observed around the Debye-Sherrer rings. Textured peaks from uncompressed material ahead of the shockwave are highlighted by white ellipses in Figure 2b; see supplemental material for full x-ray diffraction image of the uncompressed foil 127 [18]. There is a change in texture of the fcc Au as the compressed fcc region becomes more powder-like due to dislo-129 cation formation and plastic flow during shock compression [22, 23]. The peak positions of fcc Au agree well with the ex- $_{131}$  pected shift in d-spacing along the principal Hugoniot (purple triangles and solid lines in Figure 3).

The next pressure state on the Hugoniot was P = 223(11)134 GPa. Faint peaks were visible that do not fit to the ambient 135 or compressed fcc Au; these new peaks are shown by green 136 triangles in Figure 2. There is clear texture of the compressed 137 fcc peaks at similar azimuthal angles as the pre-shot ambi-138 ent fcc Au peaks, and also observed is a much less textured 139 powder ring in close proximity to the fcc (111) peak. The 140 three peaks found at  $Q = 3.112 \text{ Å}^{-1}$ , 4.391 Å<sup>-1</sup>, and 5.361  $^{141}$  Å $^{-1}$  (*d*-spacing values of 2.019 Å, 1.431 Å, and 1.172 Å) 142 can be indexed to a body centered cubic structure. At slightly higher pressure, P = 253(19) GPa, both compressed fcc Au impedance matching of the Au sample and LiF window [18]. 144 and bcc Au peaks are observed but the relative intensity of X-ray diffraction measurements were collected on a Ray- 145 bcc / fcc is higher. The positions of the bcc (110), (200), and onix SX165 flat panel CCD detector. A single x-ray pulse of 146 (211) peaks are plotted in Figure 3 (green dashed lines) us-100 ps duration was isolated from the APS Hybrid filling 147 ing a previously determined Au Hugoniot  $P-\rho$  path to determode using a high-speed chopper system. The x-rays were 148 mine the d-spacings for all the fcc and bcc reflections over timed to probe the Au sample just before shock entry into the 149 our detectable range [13]. An excellent agreement with the LiF window, so as to avoid late time pressure release states. 150 d-spacing values determined from our x-ray diffraction mea-

At 262(10) GPa and above, fcc Au is not observed and inwith a peak energy of 23.54 keV ( $\lambda = 0.5266 \text{ Å}$ ) [18]. The <sub>153</sub> stead the *bcc* Au is observed with a significant increase in difsample target package was placed in the vacuum chamber in 154 fuse scattering signal indicative of liquid Au; we note that this transmission geometry, with the sample to detector distances 155 is not a broadening of the bcc or fcc peaks and is clearly disdetermined using a polycrystalline silicon x-ray standard [19- 156 tinct scattering from partially melted Au. At 322(27) GPa, 21]. Two sample-detector distances of 111.75 mm and 92.96 157 only liquid scattering signal is observed, indicating the shock mm were used during the experiments. Due to the asymmet- 158 Hugoniot has left the solid/liquid coexistence along the meltric spectral flux of the x-ray source (Figure 1a), standard peak 159 ing curve. A large coexistence range (~116 GPa) of solid Au fitting functions (Gaussian, Lorentzian, or Pseudo-Voigt etc.) 160 and liquid Au was predicted by the multi-phase EOS by Kerare not suitable to fit the diffraction peaks. Here, we use a 161 ley [24], with melting beginning at 212 GPa and completing convolution of a Gaussian peak with an exponential tail ('Exp- 162 at 328 GPa. In this work, we observe diffraction patterns with 163 clear solid and liquid diffraction at pressures of 262 and 298 X-ray diffraction (XRD) measurements were collected on 164 GPa, suggesting a coexistence of at least 36 GPa and no more the shock Hugoniot up to a maximum pressure of 322(27) 165 than  $\sim 72$  GPa (no detectable liquid at 250 GPa and no de-

From our x-ray diffraction data, we are able to calculate

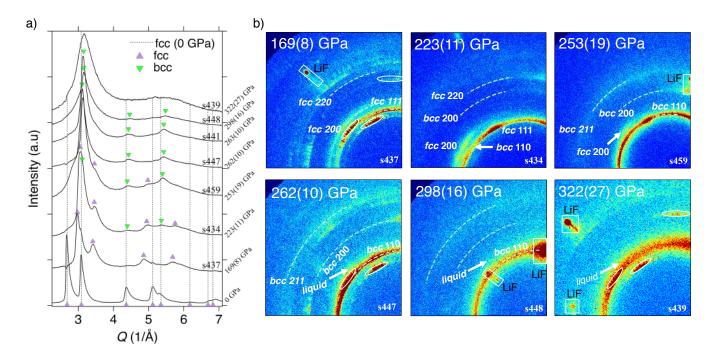


FIG. 2. a) Radially integrated x-ray diffraction profiles for shock compressed fcc (purple triangles), fcc + bcc (purple and green triangles), bcc(green triangles) + liquid, and liquid only regions along the shock Hugoniot. Ambient fcc peaks were also measured in a pre-shot exposure and have been subtracted from each compressed profile (the dotted lines represent ambient fcc Au peak locations). The intensity of the liquid only shot at 322 GPa was increased ( $\times$ 2) to a similar scale as all other data. b) A selection of the raw x-ray diffraction images with fcc, bcc or liquid scattering labelled. Strong single crystal Bragg reflections from the LiF window are highlighted by white boxes. Ambient fcc peaks, due to the uncompressed Au ahead of the shockwave, are highlighted by white ellipses. These features are masked in the integrated patterns in Figure 2a. All raw data can be found in the Supplementary Material [18].

atom. The density, calculated from x-ray diffraction, is plotted 196 sented here and used for illustrative purposes only. against pressure, determined from velocimetry measurements, in Figure 4. Our solid Au data points agree well, within experimental error, with the Yokoo Hugoniot data (black line and open circles) [13], the Kerley multi-phase EOS (blue and pink lines) [24], and the SESAME 2705 EOS (red dashed line) [25]. Whilst there have been recent advancements in characterizing the structure of shock compressed liquids [26], extracting accurate density measurements of the shocked liquid sample from diffraction data alone has not yet been demonstrated. 181

By using the Rankine-Hugoniot equation, based on con-182 servation of energy,  $E=\frac{1}{2}P(\frac{\rho-\rho_0}{\rho\rho_0})$  [28], where P is the pressure determined from impedance matching,  $\rho$  is the density from x-ray diffraction measurements, and  $\rho_0$  is the initial density (19.3 g/cm<sup>3</sup>), we can compare our measured Hugoniot data with the calculated Hugoniot and melting curve, and proride a useful description of the Au phase diagram in pressureenergy space (Figure 5). The gradient of the fcc and bcc melting slopes are estimated from the Clapeyron slope of the predicted melting curve, shown inset to Figure 4 [14], and from 216

the density of the phase from the volume of the unit cell. We 193 The dashed lines in Figure 5 represent proposed phase boundtake an average of the lattice parameter, a, calculated from the 194 aries that must encompass the experimental data (within exd-spacing of each observed peak to determine the volume per 195 perimental bounds) and are estimated based on the data pre-

> Since no measure of temperature is made during these shock experiments, we rely on theoretical calculations to de-199 termine the Hugoniot path in P-T space. A recent first principles prediction of the phase diagram of Au, that found bcc Au was a stable phase at high-pressure & high-temperature, 202 is plotted as an inset to Figure 4 [14]. The calculated shock  $_{203}$  Hugoniots are also plotted in P-T space, the blue-to-pink dashed line represents the melt line between the Kerley solid 205 and liquid EOS. All Hugoniots are plotted using an initial den-206 sity of  $\rho_0 = 19.3 \text{ g/cm}^3$  and our measured data show good <sup>207</sup> agreement with the principal Hugoniot in  $P - \rho$ . We note that the uncertainty in the measurement of the initial foil density could suggest porosity of the starting foils, which would result in higher temperatures along the Hugoniot and shock melting 211 at lower pressures. Based on the uncertainty in the initial den-212 sity measurement ( $\sim 3.6$  %, resulting in  $\rho_0 = 18.6$  g/cm<sup>3</sup>),  $_{213}$  melting on the porous Hugoniot would begin  $\sim 50$  GPa lower 214 than observed in this work due to a sharper increase in tem-215 perature [28].

In our experiments, a rapid entropy increase that is generthe volume change on melting from the shock Hugoniot [13]. 217 ated under shock compression, causes a significant increase in

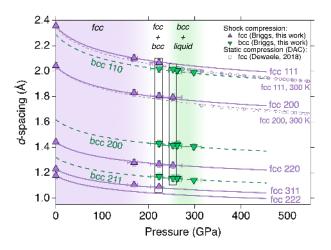


FIG. 3. Lattice d-spacing vs. pressure plot for fcc (upward triangles) and bcc (downward triangles) Bragg peaks. The shaded regions represent the fcc, fcc + bcc, and bcc + liquid phases observed in this work. The two square boxes represent data collected from the same shot, which are also shown in Figure 4. Solid purple lines represent the fcc Au, along the Hugoniot (from Ref. [13]), dashed green lines represent bcc Au. The dashed purple line with open symbols represents static DAC measurements of fcc Au that extend beyond 500 GPa [15].

temperature, allowing us to probe a small region of the phase diagram predicted by Smirnov [14]. In static compression experiments above 230 GPa, heating of the fcc phase is required to change the crystal structure [1]. These results emphasize the important effect of temperature on the structural stability of Au at high-pressure. The melting curve in the Smirnov predicted phase diagram was estimated using the Lindemann criterion [29] and the phase diagram suggests that Au along the Hugoniot would melt from the fcc phase [14]. In this study we only saw bcc Au in coexistence with the fcc and with the liquid phases. The close vicinity of phase coexistence of fcc-g bcc and bcc-liquid suggests a triple point in Au that lies very close to the principle shock Hugoniot at  $P \sim 220 \pm 20$  GPa (grey shaded region in Figure 5). Whilst we cannot determine the temperature of the triple point in this work, its pressure is very close to the value predicted from first principle calculations of  $P \sim 235$  GPa (in that work the temperature of the triple point was calculated at  $\sim 6600$  K).

In this study, we observe the first evidence for *bcc* Au on the principal shock Hugoniot. The Hugoniot follows the *fcc* 1298 + *bcc* phase region between 225 and 253 GPa, after which the Hugoniot passes through a small region of *bcc*-only phase 1240 space before very quickly reaching the melt curve. We observe *bcc* Au in coexistence with the liquid between 262 and 1242 298 GPa, after which only liquid Au is observed. This work 1243 highlights the requirement for further first principle calculations, at finite temperatures, to determine a new multiphase 1245 EOS of Au that considers the *fcc* to *bcc* phase transition we 1246 have discovered in this work.

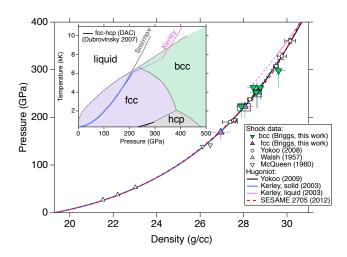


FIG. 4. Pressure vs density for Au. Open symbols represent shock Hugoniot data [8, 12, 27], with calculated Hugoniot curves plotted as solid lines [13, 24, 25]. Inset shows the proposed phase diagram of Au based on first principles calculations [14]. Our data (solid triangles) overlay well with the existing Hugoniot curves [13, 24, 25]. The dashed lines are extrapolations to regions where the Kerley EOS suggests melting should occur [24].

We thank Xiaoming Wang, Nicholas Sinclair, Adam Schutemperature, allowing us to probe a small region of the phase
diagram predicted by Smirnov [14]. In static compression experiments above 230 GPa, heating of the fcc phase is required to change the crystal structure [1]. These results emphasize

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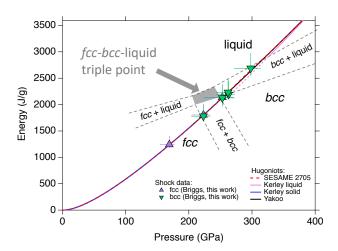


FIG. 5. Energy vs pressure plot for Au. Calculated Hugoniot data are plotted as solid lines, with the blue and pink dashed lines representing the extension of the Kerley solid EOS and liquid EOS, respectively, [24] to the pressures where only solid or solid-liquid data are observed. The dashed black lines are representative phase boundaries in energy-pressure space, with the gray shaded region representing the region of the *fcc-bcc*-liquid triple point.

work performed under the auspices of the U.S. Department 294 of Energy by Lawrence Livermore National Laboratory un- 295 [12] R. G. McQueen and S. P. Marsh, J. Appl. Phys. 31, 1253 (1960). der LLNL's Laboratory Directed Research and Development 296 [13] M. Yokoo, N. Kawai, K. G. Nakamura, K. I. Kondo, Y. Tange, (LDRD) Program under grant numbers 18-ERD-001 and 18-ERD-012. Lawrence Livermore National Laboratory is operated by Lawrence Livermore National Security, LLC, for the U.S. Department of Energy, National Nuclear Security Administration under Contract DE-AC52-07NA27344. This 302 [16] publication is also based upon work performed at the Dy- 303 namic Compression Sector supported by the DOE/NNSA under Award No. DE-NA0002442 and operated by Washington State University. This research used resources of the Advanced Photon Source, a DOE Office of Science User Facility operated for the DOE Office of Science by Argonne National 309 Laboratory under Contract No. DE-AC02-06CH11357.

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