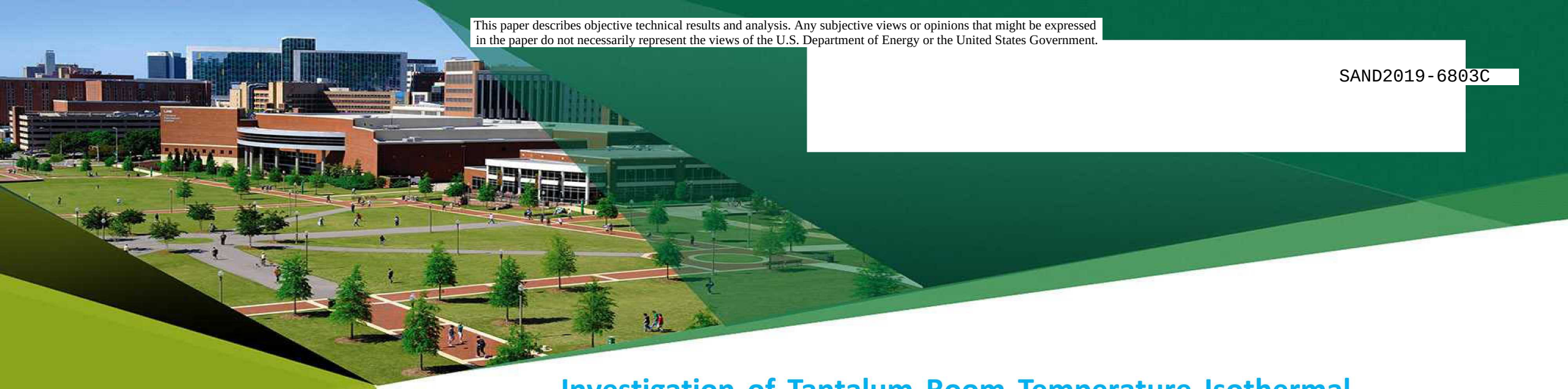

SAND2019-6803C

Investigation of Tantalum Room Temperature Isothermal Compression to Multi-megabar Pressures Using Two-stage Diamond Anvils

SCCM June Meeting

Wednesday, June 19, 2019

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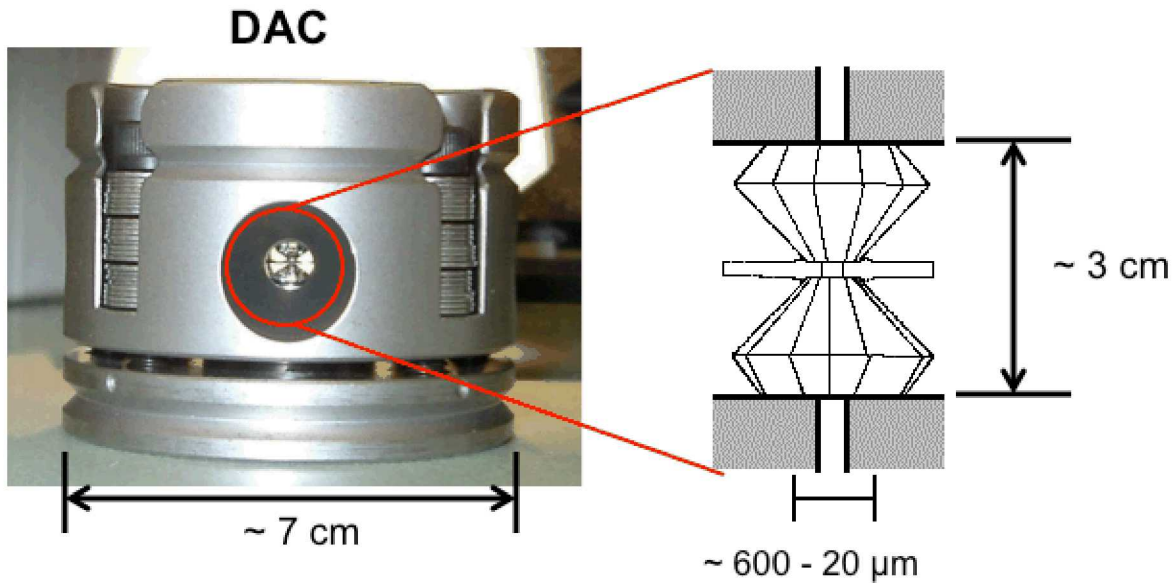
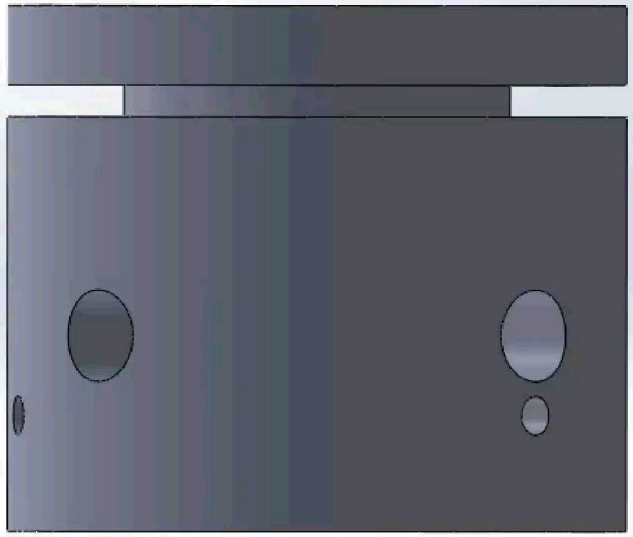
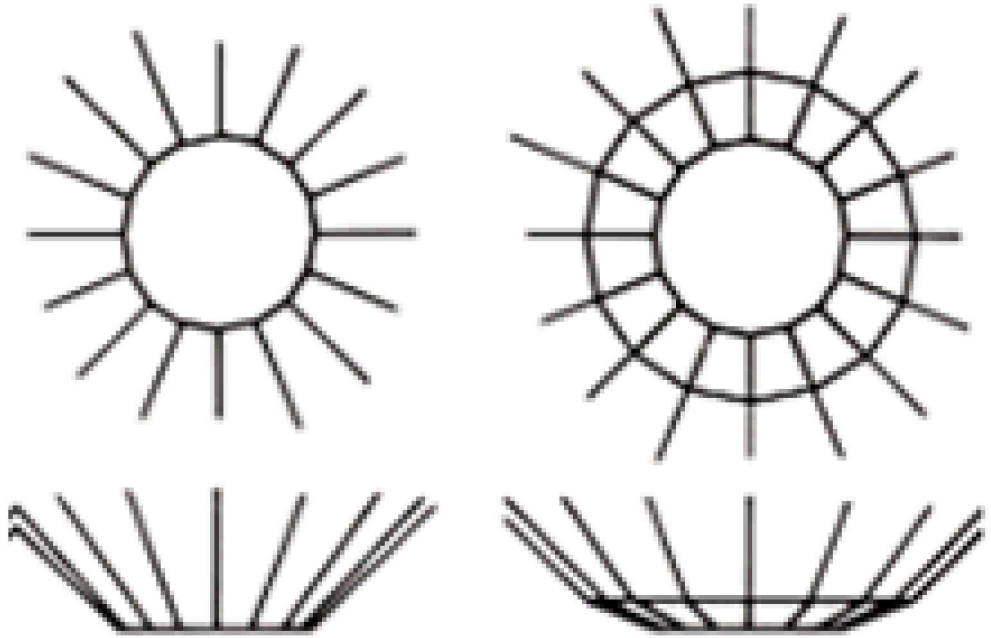
Outline

- Overview of DAC
- Single and Two stage DAC
- Experimental measurements with X-ray Diffraction
- Recent Tantalum measurements



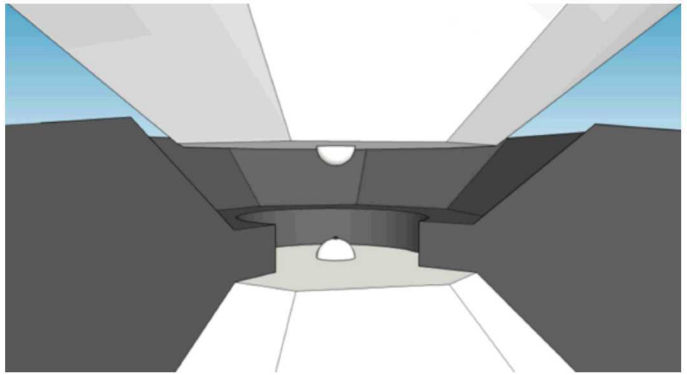


Brief Overview of Single Stage DAC

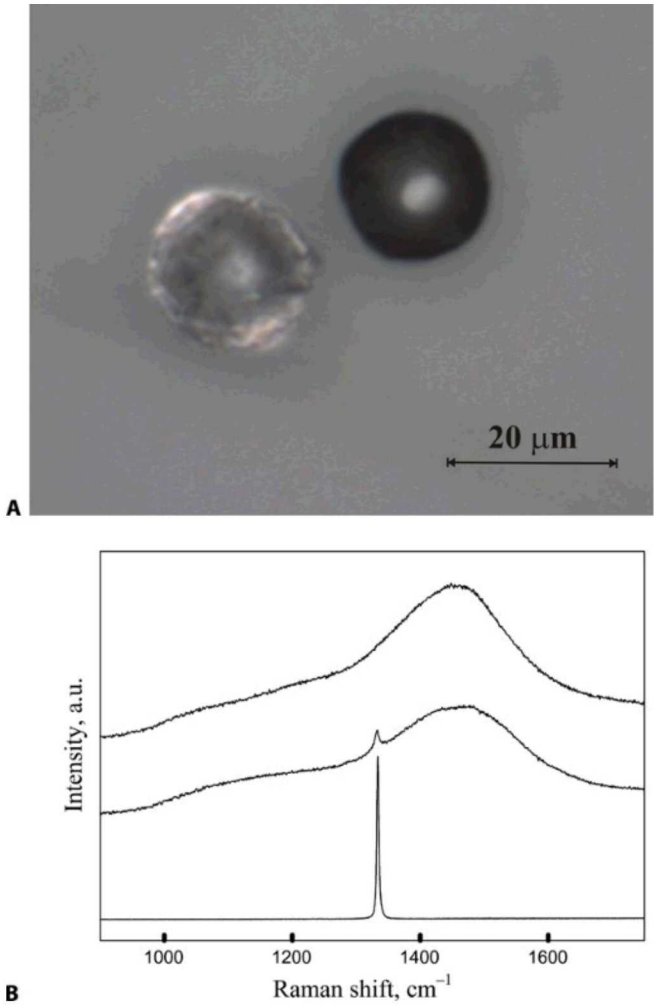


- Typical bevel angle 7°-8.5° (up to 12° used)
- Culet size limits maximum reachable pressure (P=F/A)
- Culet diameter of 300 μm → 100 GPa
- Beveled anvils allow for pressure greater than 100 GPa to be reached

Two stage Anvils for Multi- Mbar Pressures

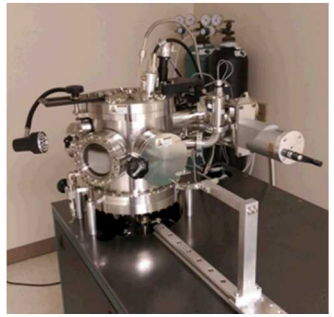


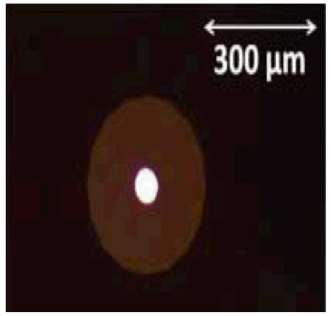
- Dubrovinsky et al converted glassy carbon balls into NCD via anvil press and high temperature
- Pressures reached a claimed 1 TPa in *Science Advances* 20 Jul 2016
- Reproducibility is difficult with manually placing and aligning NCD microballs

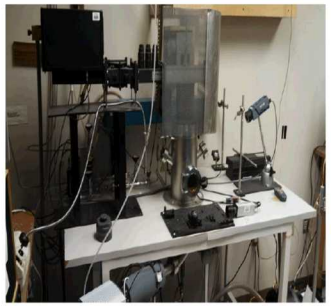


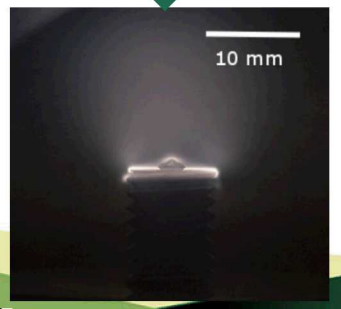
N. Dubrovinskaia, *Science Advances* 20 Jul 2016

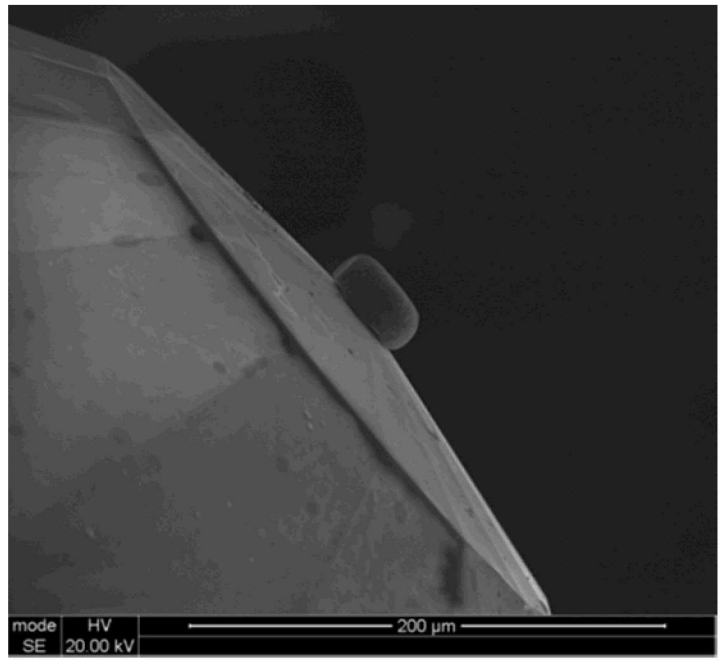
Two Stage Diamond Anvil Cells via Mask-less Lithography





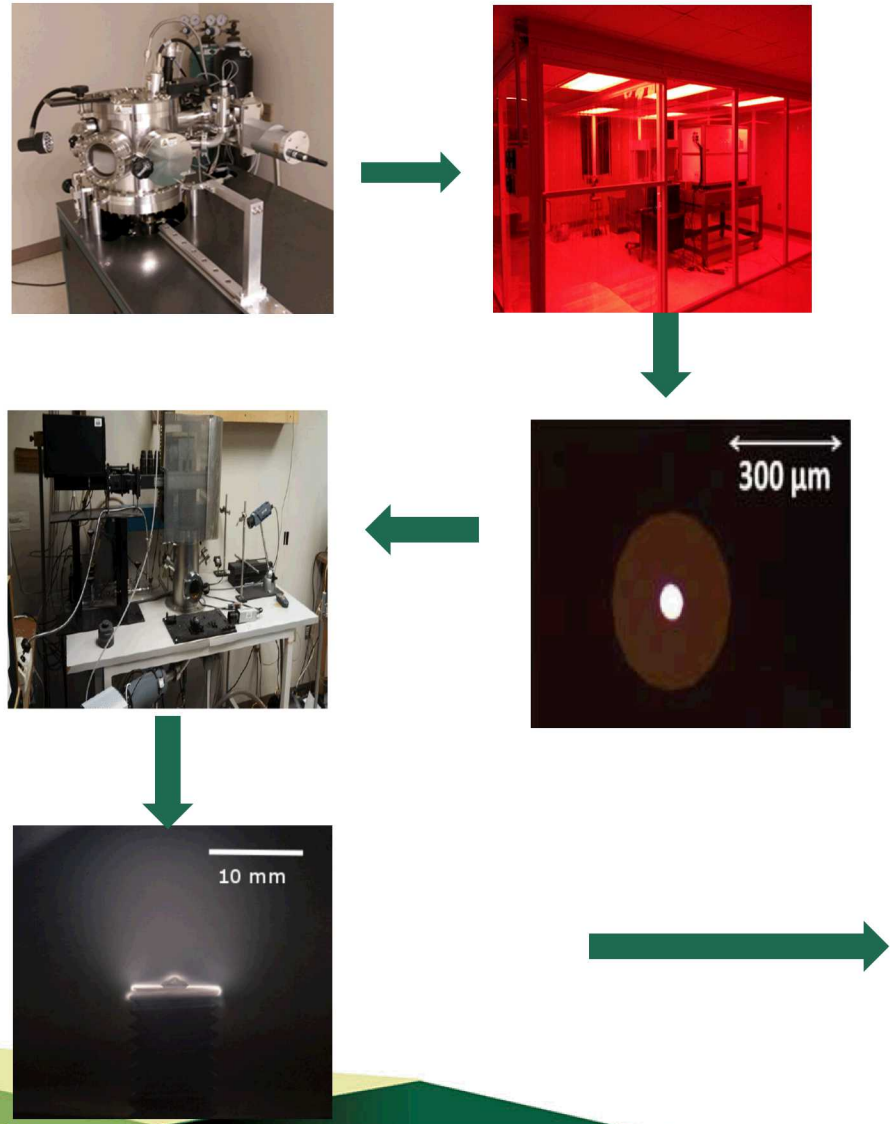






- The flow chart outlines the microfabrication process of two-stage diamond anvils.
- Highly reproducible diamond placement on culet

Two Stage Diamond Anvil Cells via Mask-less Lithography



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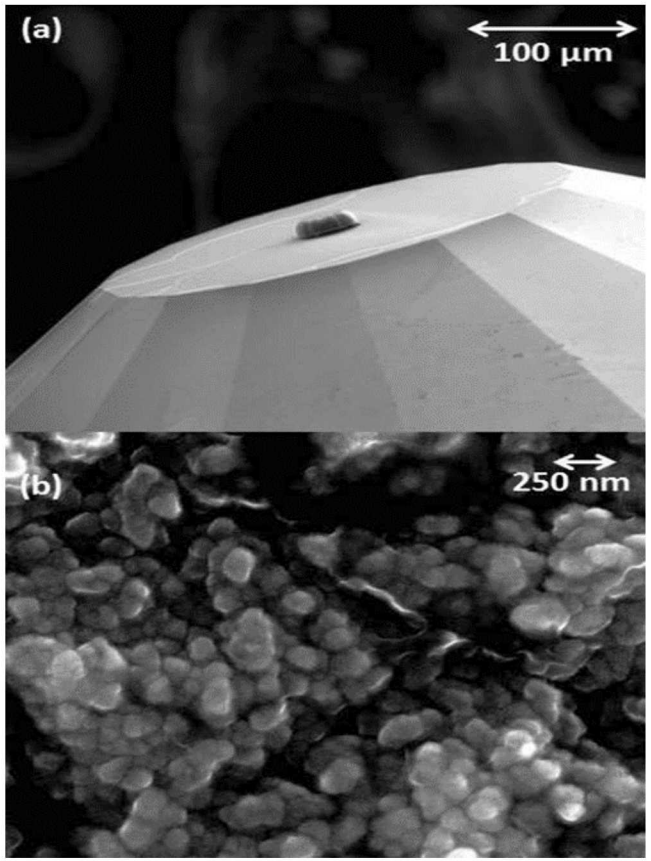
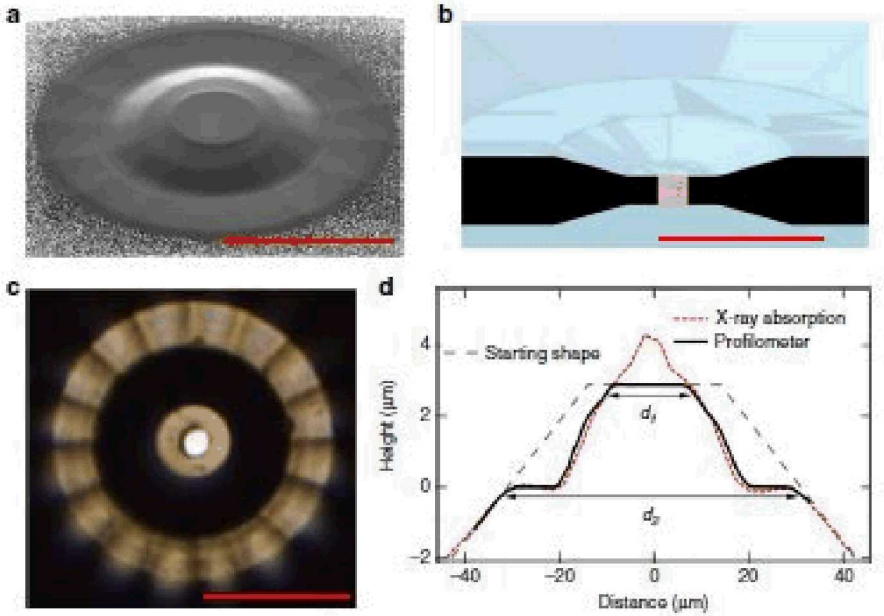


Focused Ion Beam Machining of Single Crystal DAC

- New approach to two stage diamond anvil cell was pioneered very recently
- Toroidal anvil cell was created via Focused Ion Beam Machining onto single crystal diamond substrate
- Toroid design is made to circumvent problems arising from large elastic deformation under multi-megabar pressures

Dewaele *et al*	**~400 GPa**
T. Sakai *et al*	**~400-600 GPa**
Zs. Jenei *et al*	**~600 GPa**

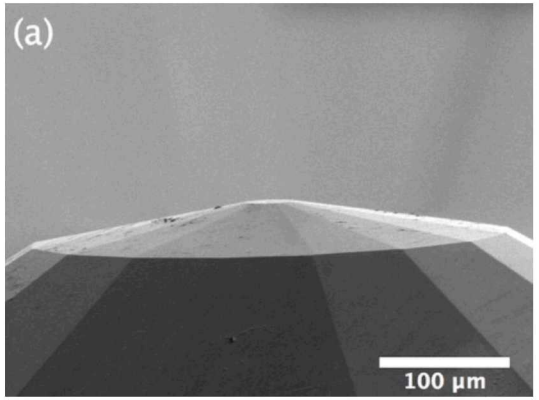
A. Dewaele, *Nature Communications* 2018



Experimental Details on Ta EOS Measurement With Toroidal DAC

- Two stage toroidal diamond anvils were machined from the beveled design of single crystal diamond utilizing the Focused Ion Beam (FIB) technique. Two toroidal designs were employed with central flats of 8 microns in diameter.
- Rhenium metal was utilized as an internal pressure standard in the multi-megabar range
- X-ray diffraction experiments under high pressure on polycrystalline samples were carried out at the beam-line 16-ID-B and 16-BM-D, HPCAT, Advanced Photon Source at Argonne National Laboratory
- For lower pressures below 80 GPa, gold utilized as an internal pressure standard and neon as pressure medium

Our recent experiments with 2stage FIB anvil – Tantalum sample



Side-view of the starting beveled diamond anvil

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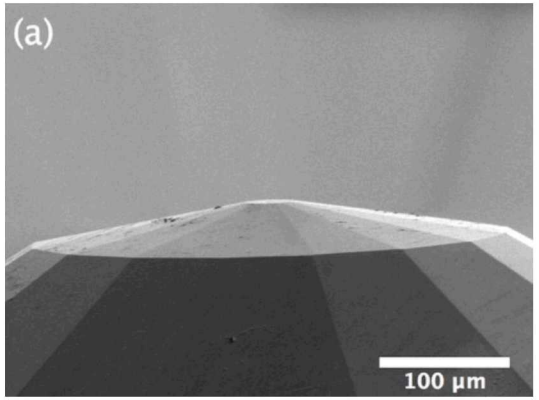

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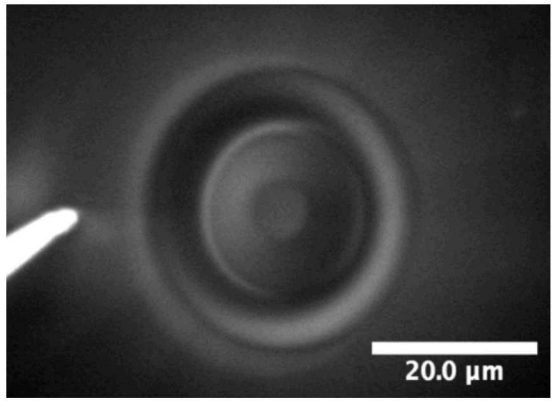

Top-view of the FIB machined toroidal anvil with a central flat region of 8 microns in diameter.

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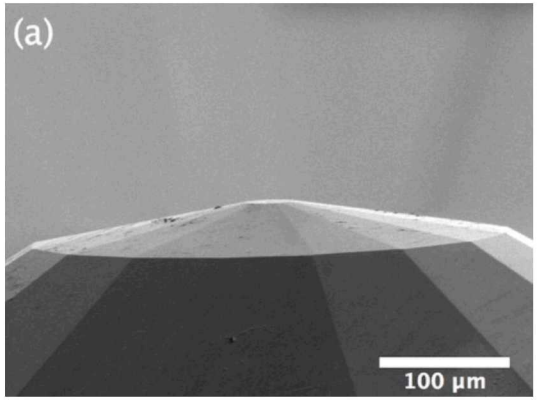

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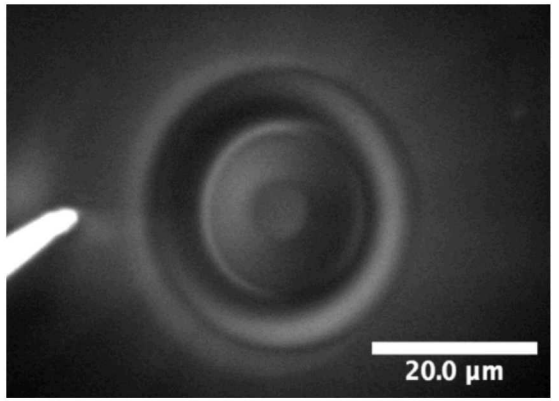

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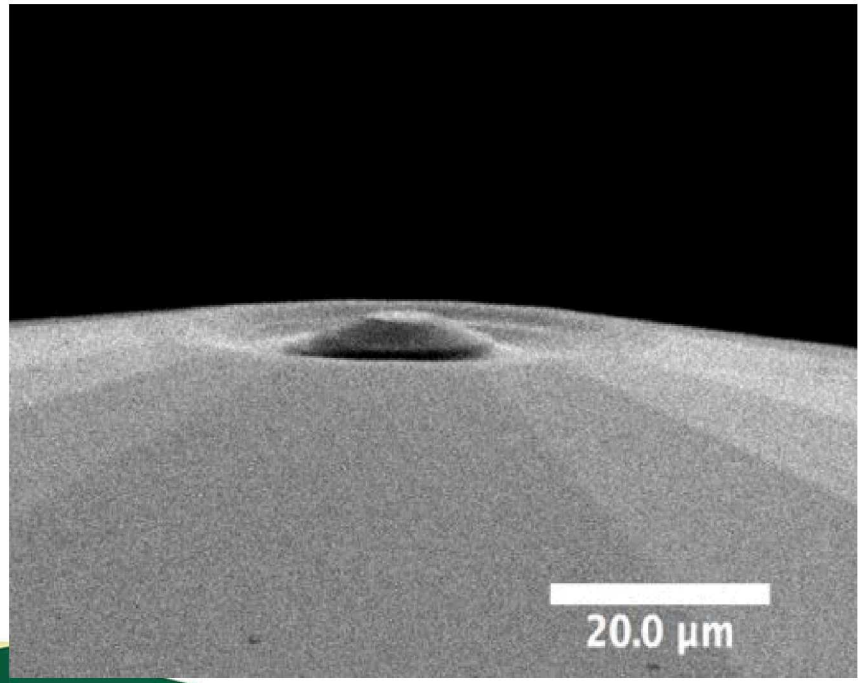
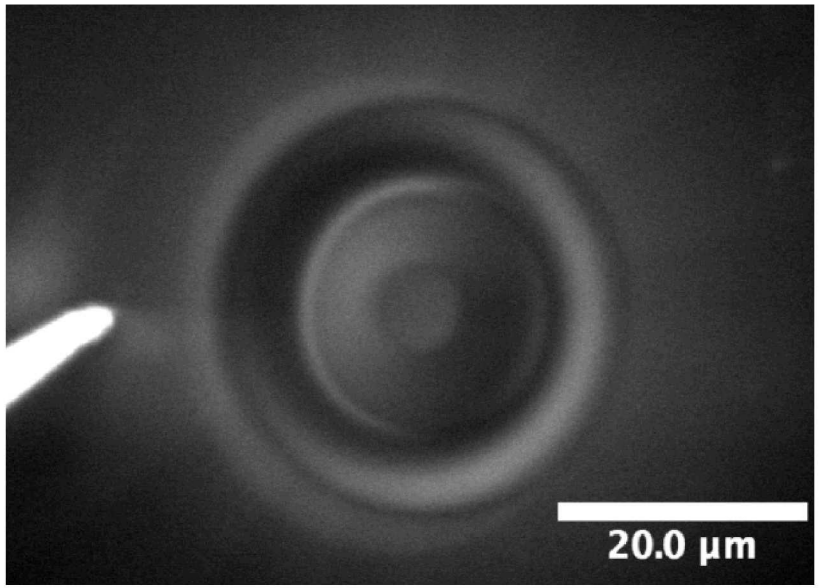
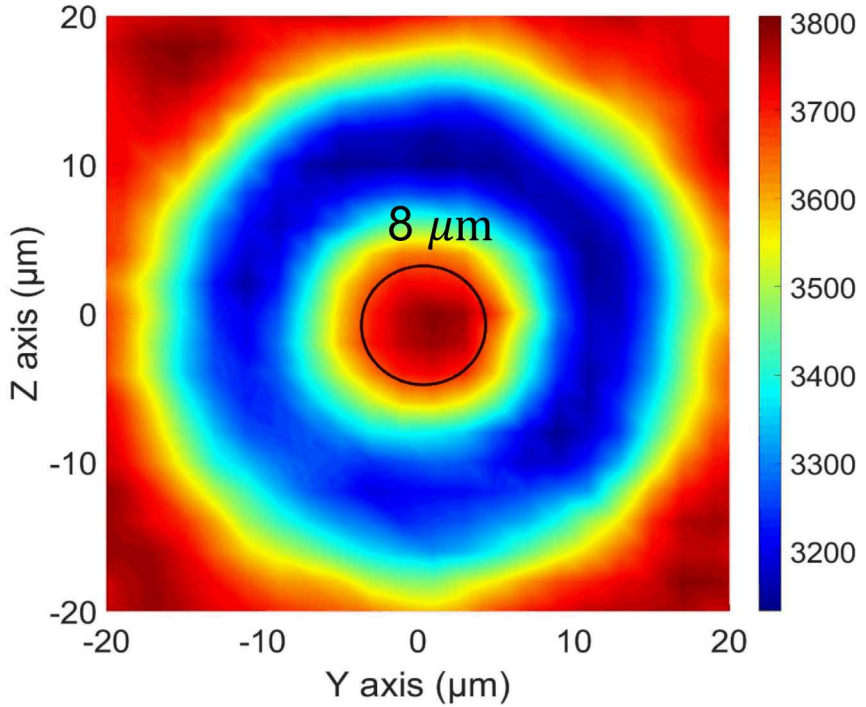


Close-up side-view of the toroidal shape of the diamond anvil showing height of toroid anvil tip is 5 microns.

Ultra High Pressures with Toroidal Anvils

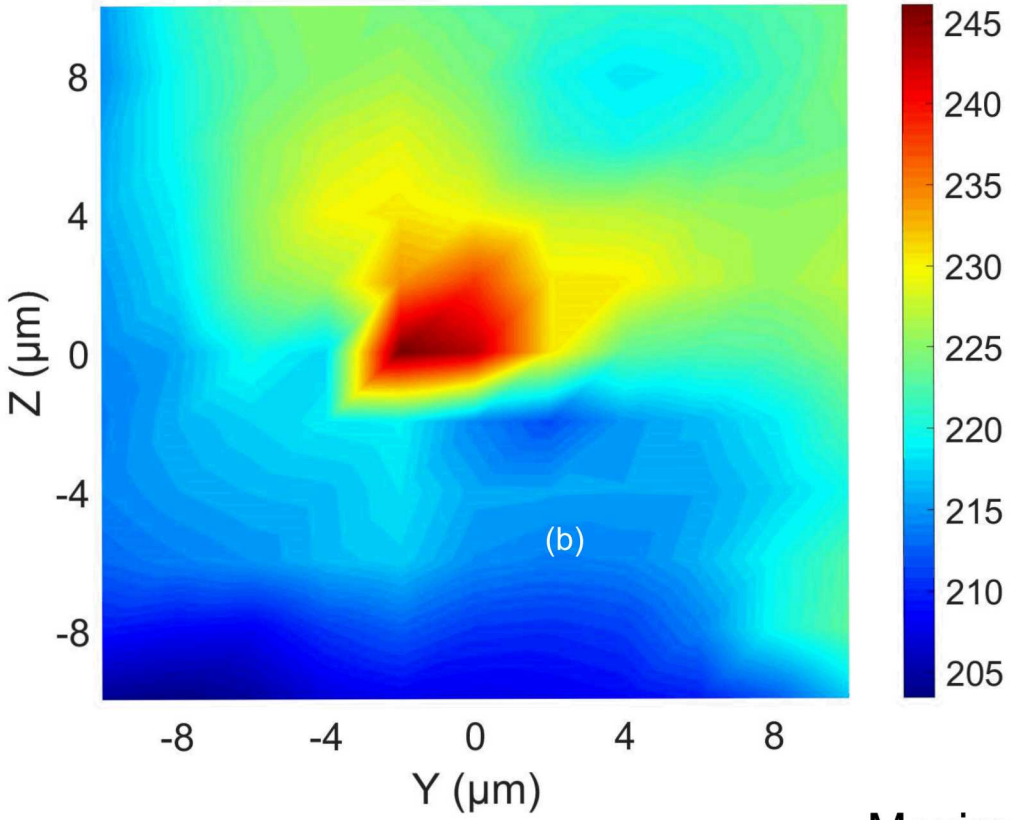


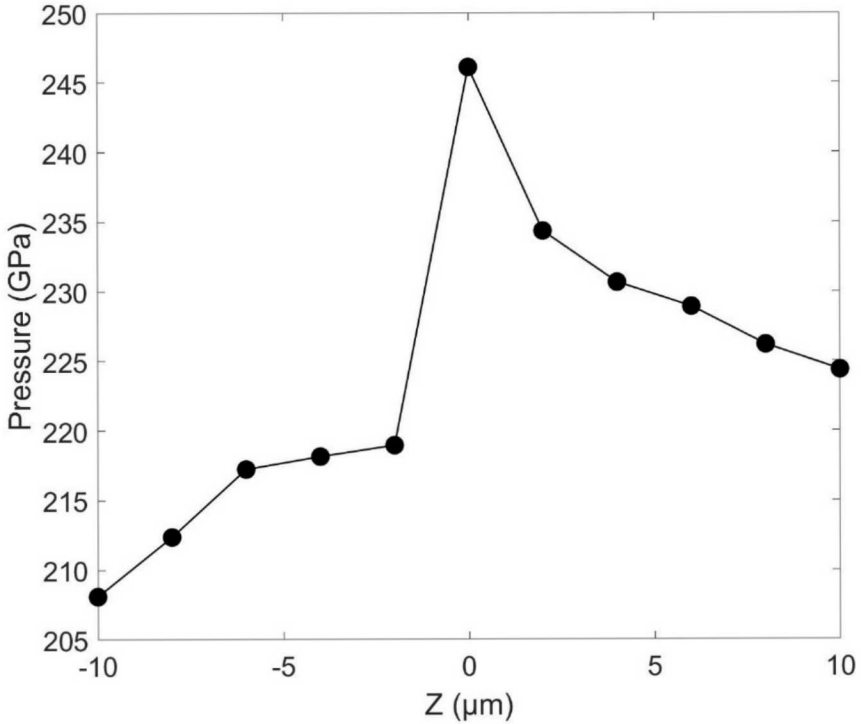


- The maximum transmission area indicated by a circle at the center represents the toroidal anvil central flat of 8 microns in diameter.

- Thinning of gasket material on outer periphery is consistent with DAC failure at 310 GPa

Toroid Pressure Distribution



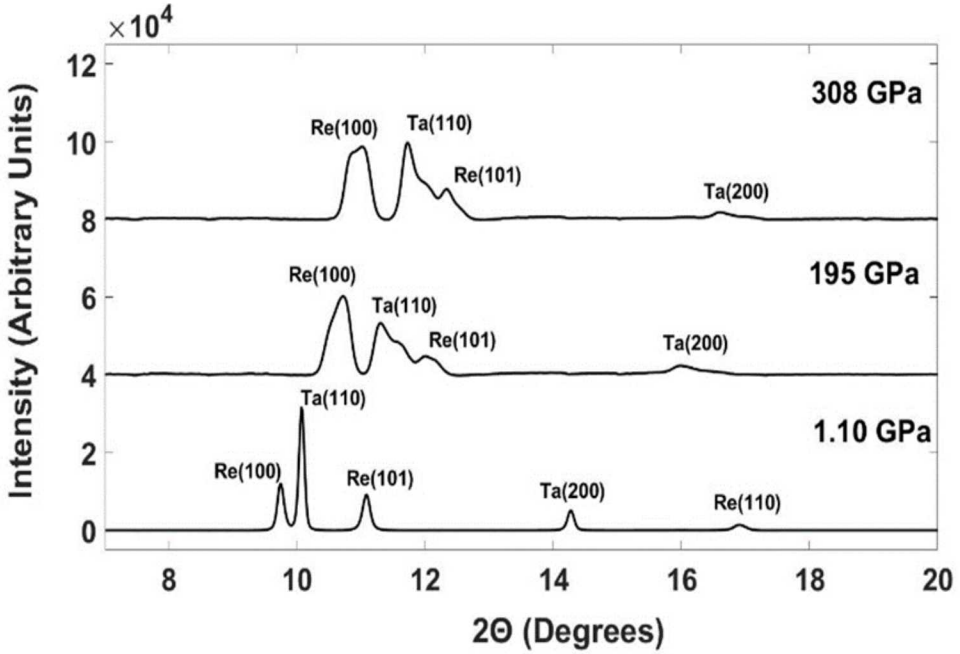


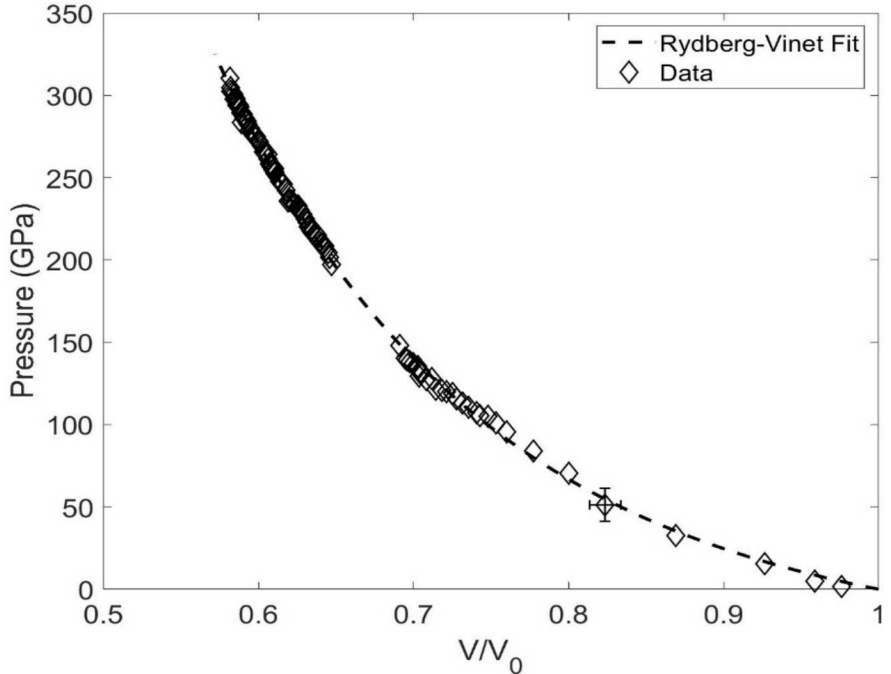
Maximum Pressure Gradient: 13.6 GPa/μm

Pressure (GPa)

- Pressure distribution capability at HPCAT, Argonne National Laboratory exhibits low pressure gradient

Integrated X-ray diffraction profile and Rydberg-Vinet Fit for Ta-Re Sample Mixture





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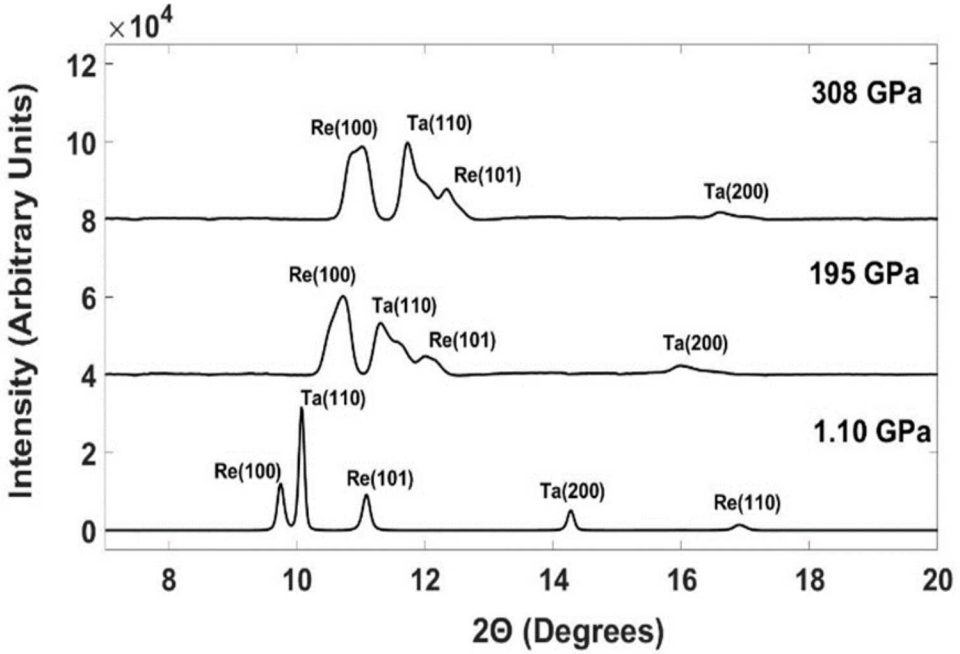
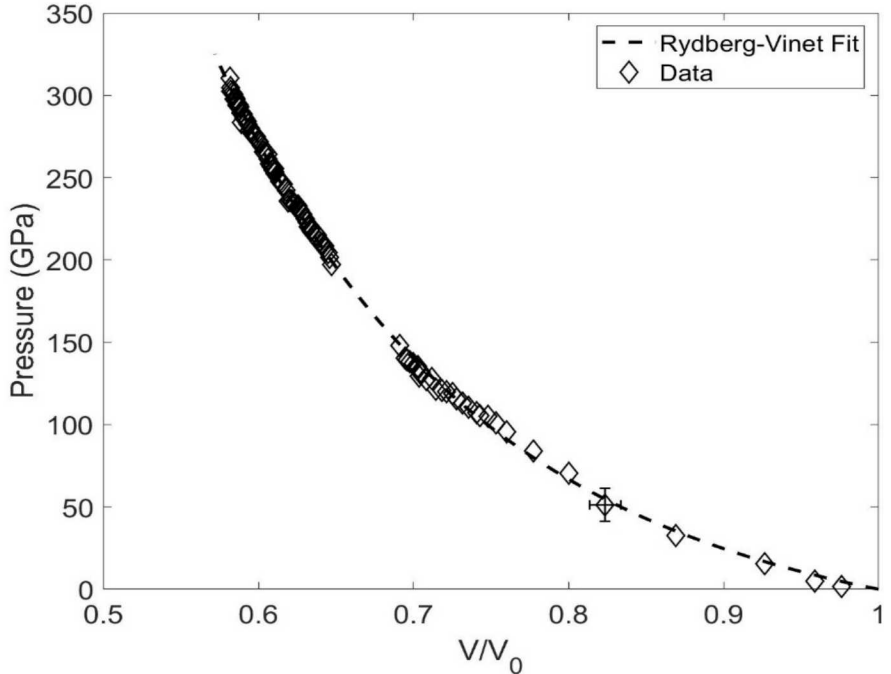


- X-ray diffraction pattern for tantalum-rhenium (*Ta-Re*) mixture. The *Ta*-diffraction peaks are indexed to a body-centered-cubic phase while the *Re*-diffraction peaks are indexed to a hexagonal close-packed phase.



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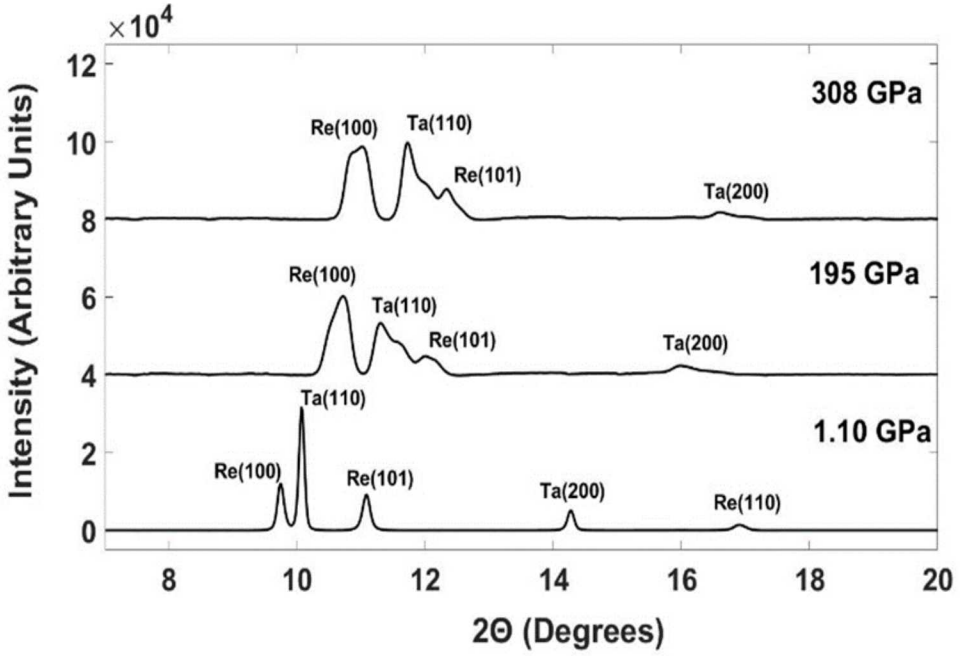

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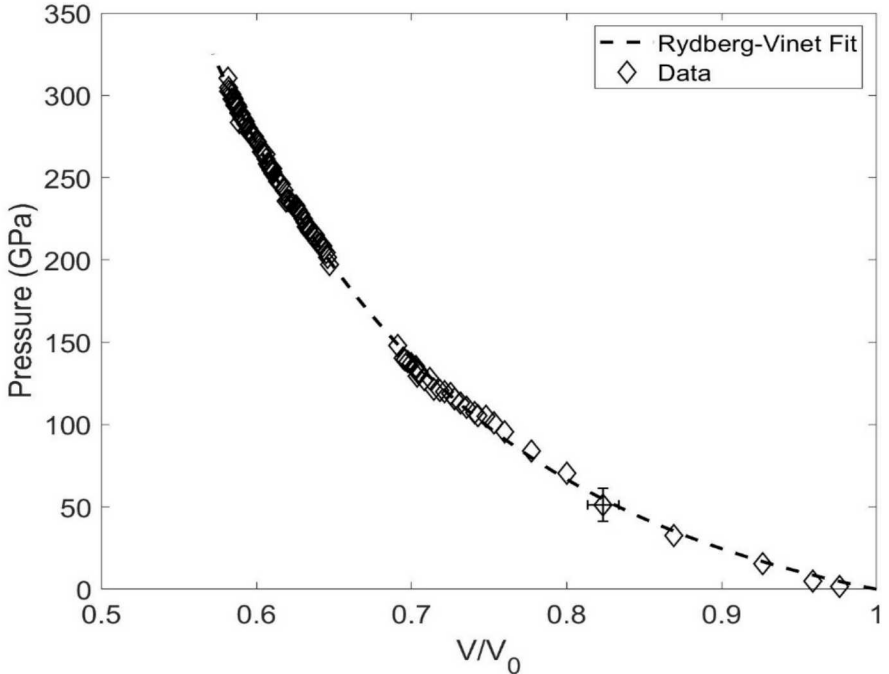
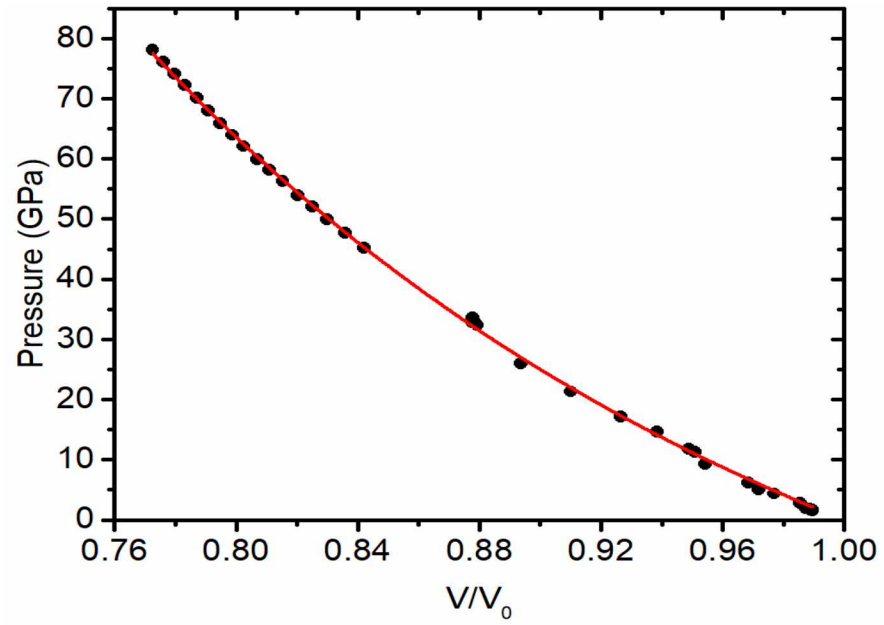
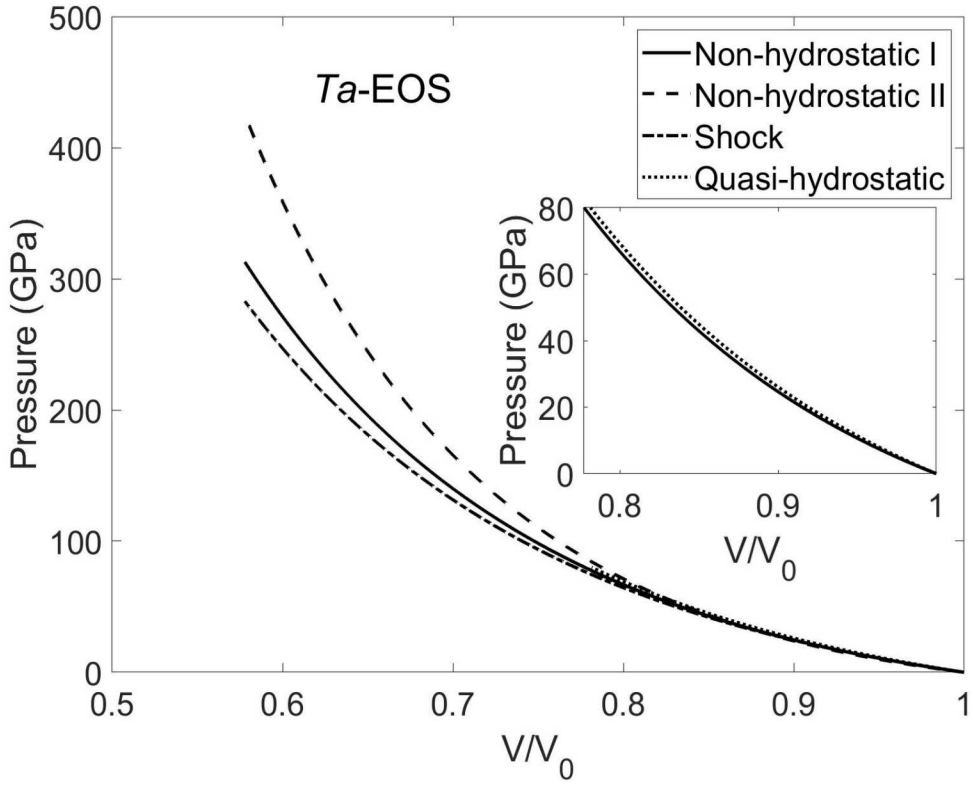
- The measured non-hydrostatic equation of state of *Ta* to 310 GPa ($V/V_0 = 0.581$) using *Re* as a pressure marker and using equation of state parameters by Anzellini *et al*



Comparison of Rydberg-Vinet Equation of States of Tantalum



- The measured quasi-hydrostatic equation of state of *Ta* to 80 GPa using *Au* and *Ne* as a pressure marker



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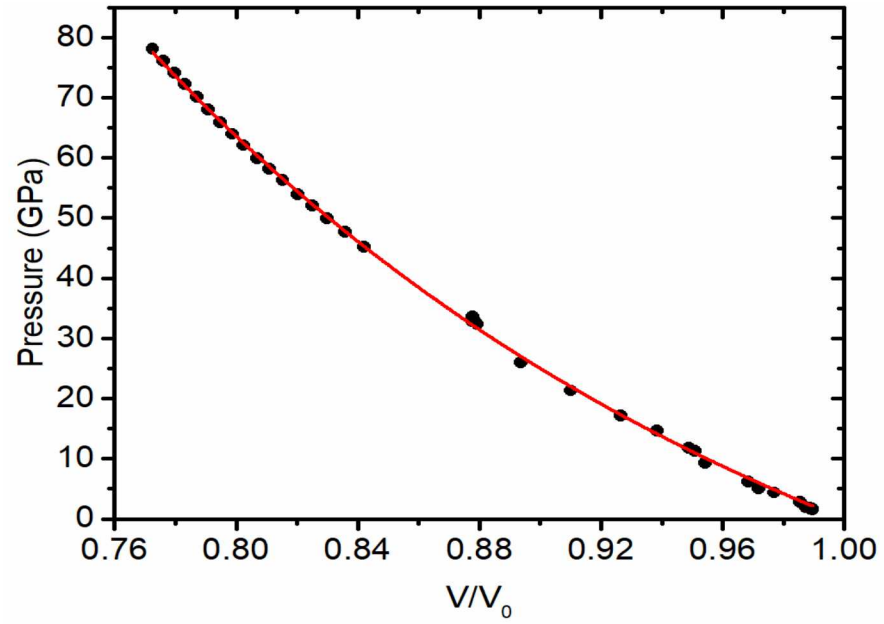

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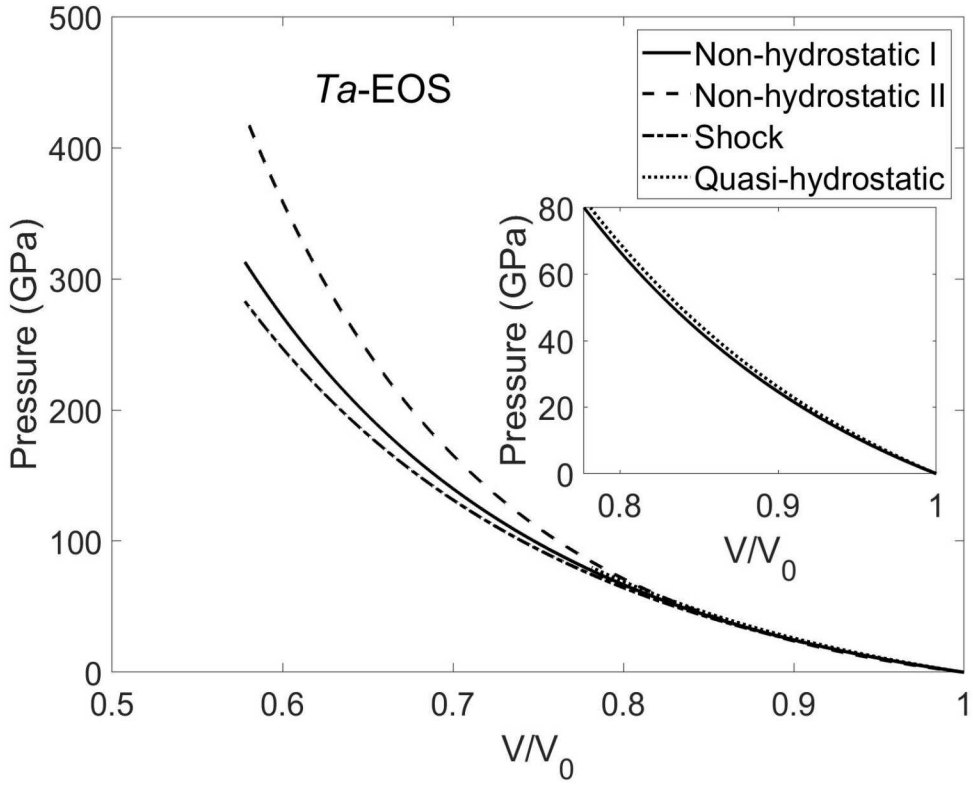

- The comparison of measured equation of state of *Ta* with two different EOS of *Re* by Anzellini *et al*[12] and Dubrovinsky *et al*[3] (Non-hydrostatic I and Non-hydrostatic II).

EOS Fit Parameters

Experimental Description	Pressure Marker	Pressure Range (GPa)	Bulk Modulus K_0 (GPa)	Pressure Derivative K'_0	Reference
Non-hydrostatic I using Anzellini et al[12] EOS for Re	Rhenium	0 - 310	186.6±2.0	4.36±0.05	This study
Non-hydrostatic II using Dubrovinsky et al[3] EOS for Re	Rhenium	0 – 422	166.1±1.4	6.05±0.01	This study
Shock Compression	-	0 – 230	189.4±1.8	3.90±0.05	R. G. McQueen, S. P. Marsh, J. W. Taylor, J. N. Fritz, and W. J. Carter, *High Velocity Impact Phenomenon* (Academic, New York, 1970)
Quasi-hydrostatic (neon-medium)	Gold	0-80	201.0±2.4	3.21±0.11	This study
Ultrasonic Data	-	0-0.5	194	3.83	K. Katahara, M. Manghnani, and E. Fisher, "Pressure derivatives of the elastic moduli of niobium and tantalum", J. Appl. Phys. **47**, 434 (1976)

Conclusions and Future Work

- The toroidal diamond anvils can generate near Terapascal pressures (TPa) pressures, however, nano-beam x-ray diffraction capability at synchrotron facilities would be required to get structural data at TPa pressures

- Static pressure compression of Tantalum to > 300 GPa using Rhenium as a internal x-ray pressure standard. The body-centered-cubic phase is stable to volume compression $V/V_0 = 0.58$.

- Future developments into increasing achievable pressure thresholds may lie in optimizing toroidal DAC design

- Decreasing grain size of CVD grown NCD may increase strength of NCD to withstand higher pressures

Acknowledgements

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