



Material Strength at High Pressure

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Background/Motivation

Over the past 30 years, advancements in the focusing schemes, beam quality, and X-ray brilliance at synchrotron light sources have enabled the reduction of X-ray beam dimensions from tens of micrometers into the sub-micrometer domain. These developments have enabled spatial mapping of pressure gradients and the stress state in micron-sized samples at multi-megabar pressures in the diamond anvil cell (DAC). This poster presents angle-dispersive X-ray diffraction (XRD) results collected on beamline P02.2 at the PETRA-III synchrotron using a beam size (FWHM) of 0.85 x 0.85 microns from an Ir sample in a Bi pressure transmitting medium (PTM). The use of these new sub- μm X-ray beams allows for more precise investigations of material strength. We look to apply these techniques in future experiments to obtain more accurate investigations of phase transitions as well as being able to correct for non-isotropic stress conditions generated under extreme static pressure, thereby improving material equation of state (EoS) models.

Advancements of X-ray Beam Facilities

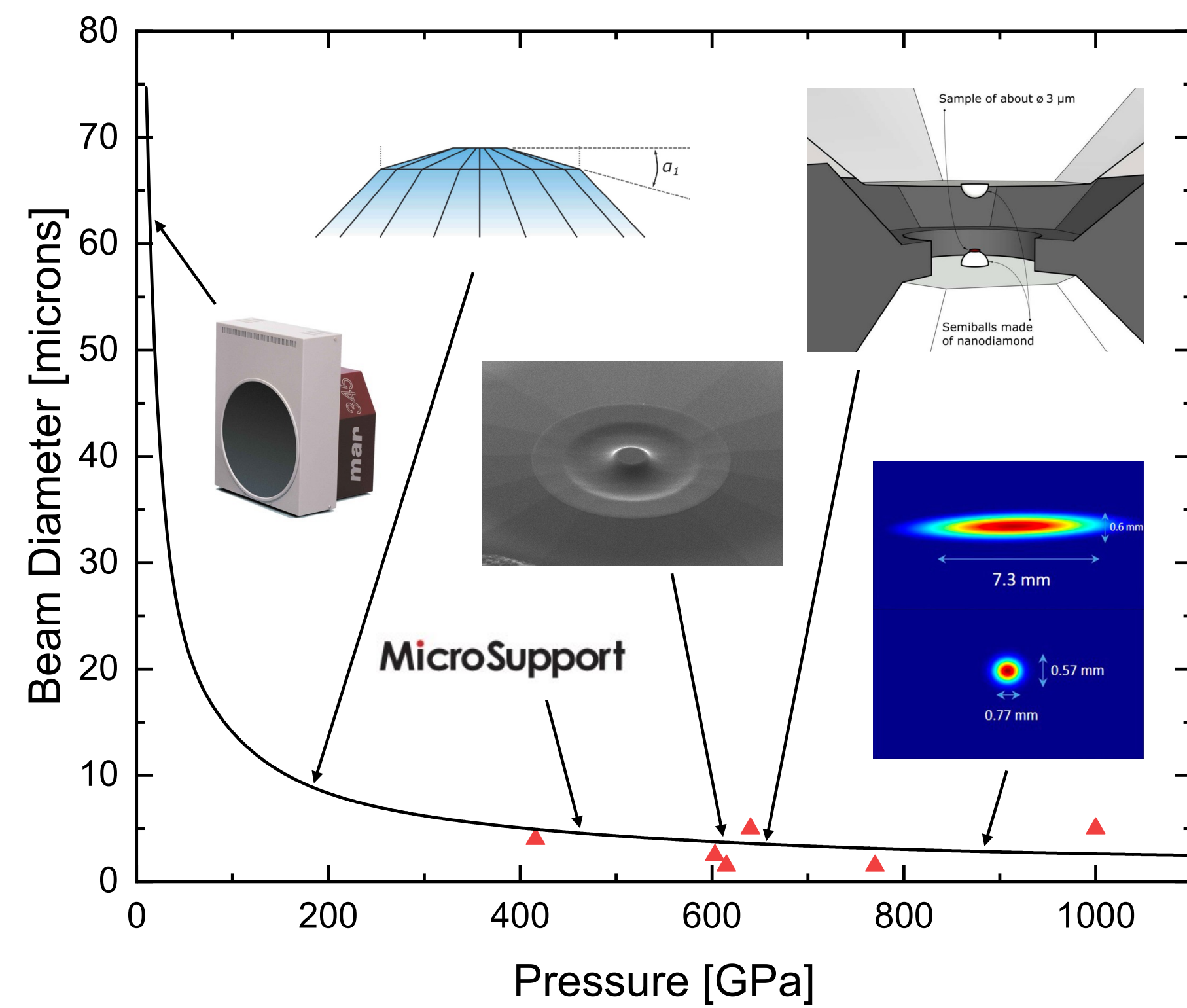


Figure 1: Beam size diameter compared to maximum pressure achieved, curve extrapolated from [1]. Figures from [2] (mar345TM), [3] (Culet), [4] (dsDAC), [5] (X-ray Beam).

The following advancements at fourth generation facilities have recently been carried out:

- Improved beam focusing capabilities has resulted in sub-micron X-ray beams [6]
- Fast Piezo motors allow reproducible positioning with minimal backlash
- Instantaneous readout detectors, such as the EIGER and DECTRIS, which have small pixel sizes and spatial distortion
- Increased brightness of X-rays beams have resulted in shorter acquisition times

Simultaneous developments to those listed above at synchrotron facilities have resulted in mapping the sample chamber of a DAC using a sub-micron beam becoming a reality. This is in stark contrast to previous single diffraction images, often containing parasitic diffraction, taking tens of minutes to collect with larger beam sizes.

Advantages of using sub-micron beams include being able to load, and thus find, smaller samples, resulting in more quasi-hydrostatic conditions, as well as effectively negating pressure gradients, by probing a smaller sample volume.

Determining Uniaxial Stress

The ‘Singh approach’ defines the uniaxial stress component $t = (\sigma_{33} - \sigma_{11})$ to be a macroscopic measure of the non-hydrostaticity in a DAC, where σ_{33} and σ_{11} are the uniaxial and radial stress components respectively [7]. The lattice parameter, $a_m(hkl)$, obtained from different diffraction peaks satisfies the following relationship

$$a_m(hkl) = M_0 + M_1(\Gamma(hkl)(1 - 3\sin^2(\theta))) \quad (1)$$

where the gradient $M_1 = -a_p(\alpha St/3)$, a_p is the lattice parameter in the absence of uniaxial stress, $a_p \approx M_0$ is the y-intercept, θ is the position of the peak and $\Gamma(hkl)$ is given as a combination of miller indices h , k and l , $\Gamma(hkl) = (h^2k^2 + k^2l^2 + l^2h^2)/(h^2 + k^2 + l^2)^2$. Plotting the linear Equation 1 allows us to determine t from the gradient and y-intercept

$$t \approx -\frac{3M_1}{\alpha SM_0} \quad (2)$$

where S is a combination of the single-crystal elastic compliance components and α is taken to be a weight between 0.5 and 1 which relates to the contribution from Reuss and Voigt shear moduli (stress and strain continuity respectively). Here we take a value of $\alpha = 0.5$.

The Diamond Anvil Cell

DACs are commonly used to reach pressures in the hundreds of gigapascal regime. Single-crystal diamonds are glued into seats before alignment, the load is commonly supplied through backing plate screws. Diamond anvils themselves can also be modified to produce smaller culet diameters (10 μm) resulting in pressures in excess of 1 TPa [4].

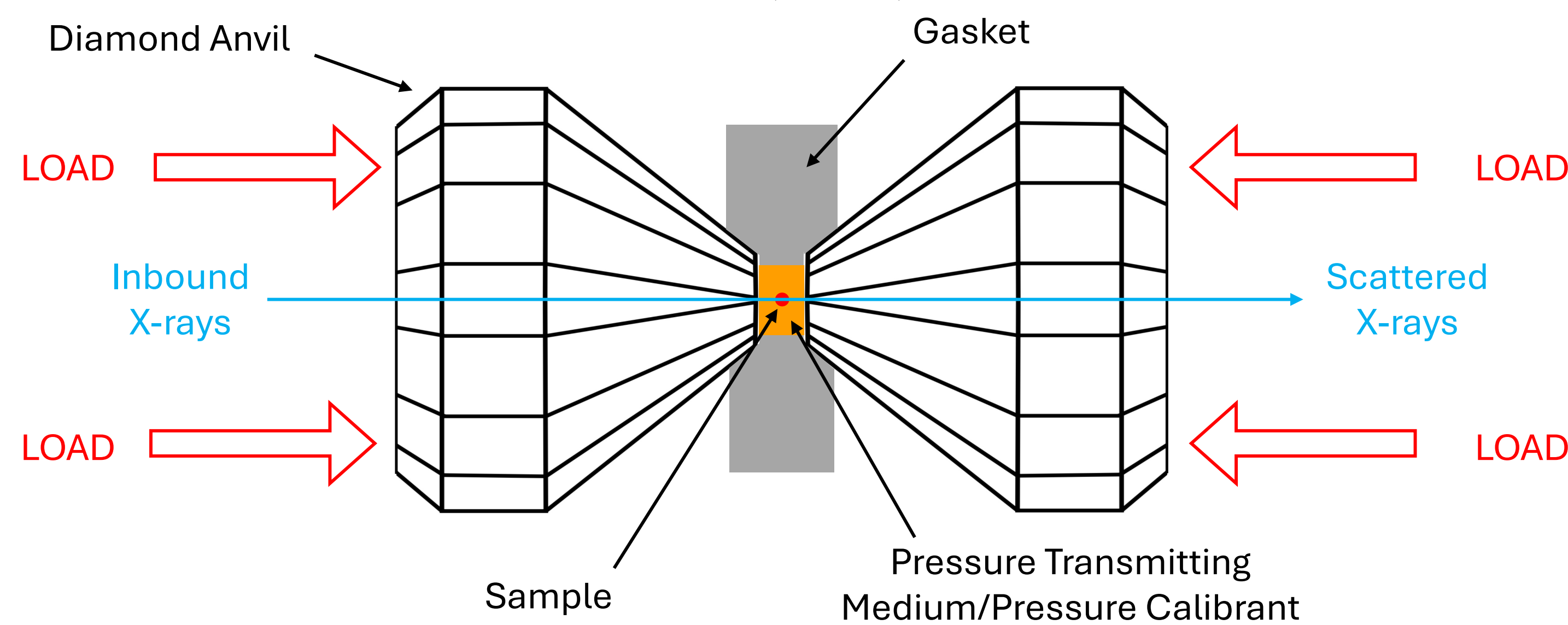


Figure 2: Schematic of a DAC used in high pressure experiments, not to scale. The sample is surrounded by a pressure transmitting medium with low shear strength, such as Bi (can also act as a pressure calibrant), to limit non-hydrostatic effects before being placed in a drilled gasket chamber (W or Re) to support the sample.

Powder X-ray Diffraction

Angle-dispersive XRD is used to probe the atomic configuration of materials. Diffraction images are integrated azimuthally before the structure is determined.

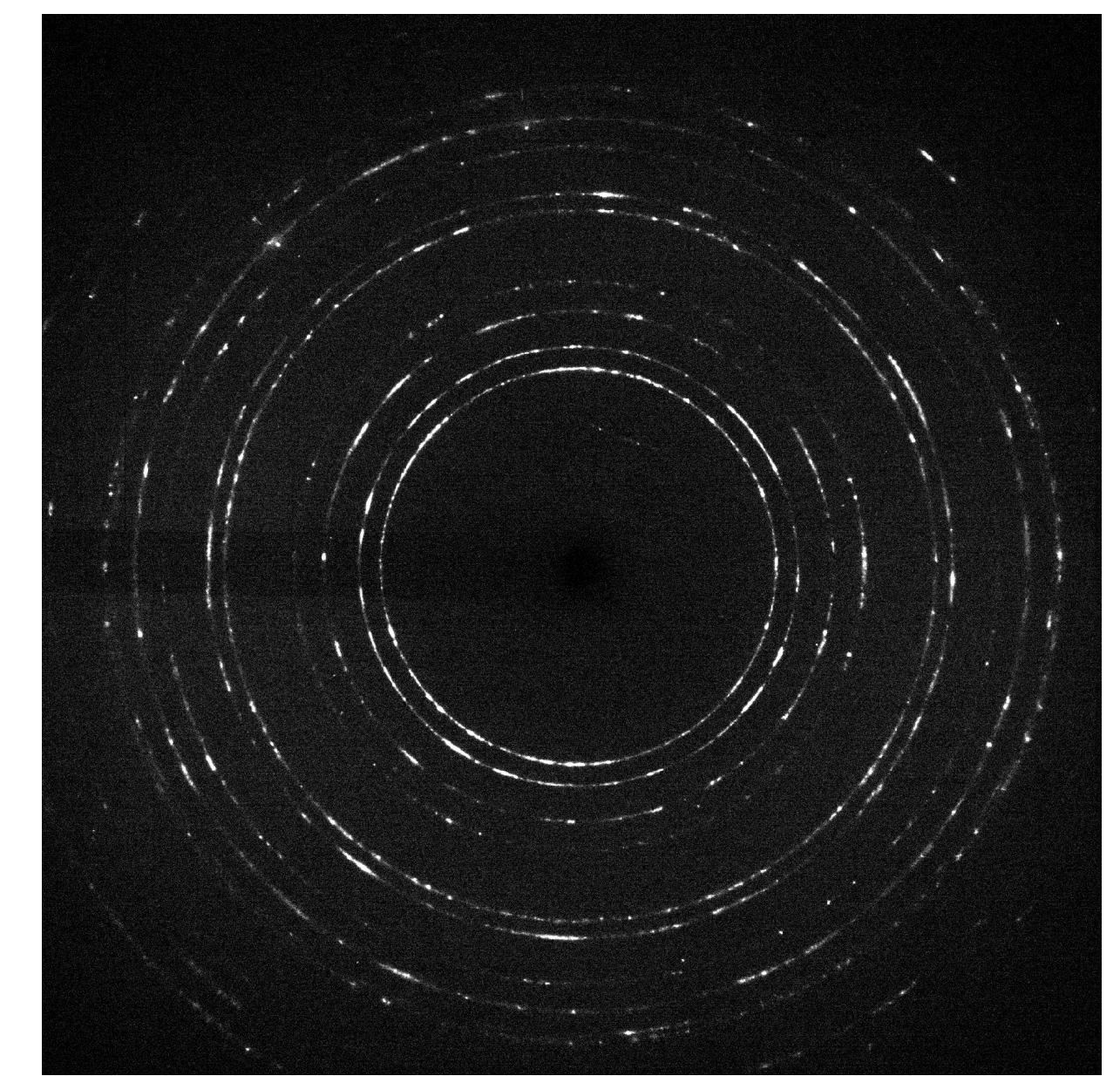


Figure 3: Powder XRD of Ir and Bi at 148 GPa collected at the P02.2 Beamline of PETRA-III in Hamburg.

Pressure Mapping

Due to the advancements at synchrotrons, it is now possible to carry out 2D grid scans and obtain pressure maps.

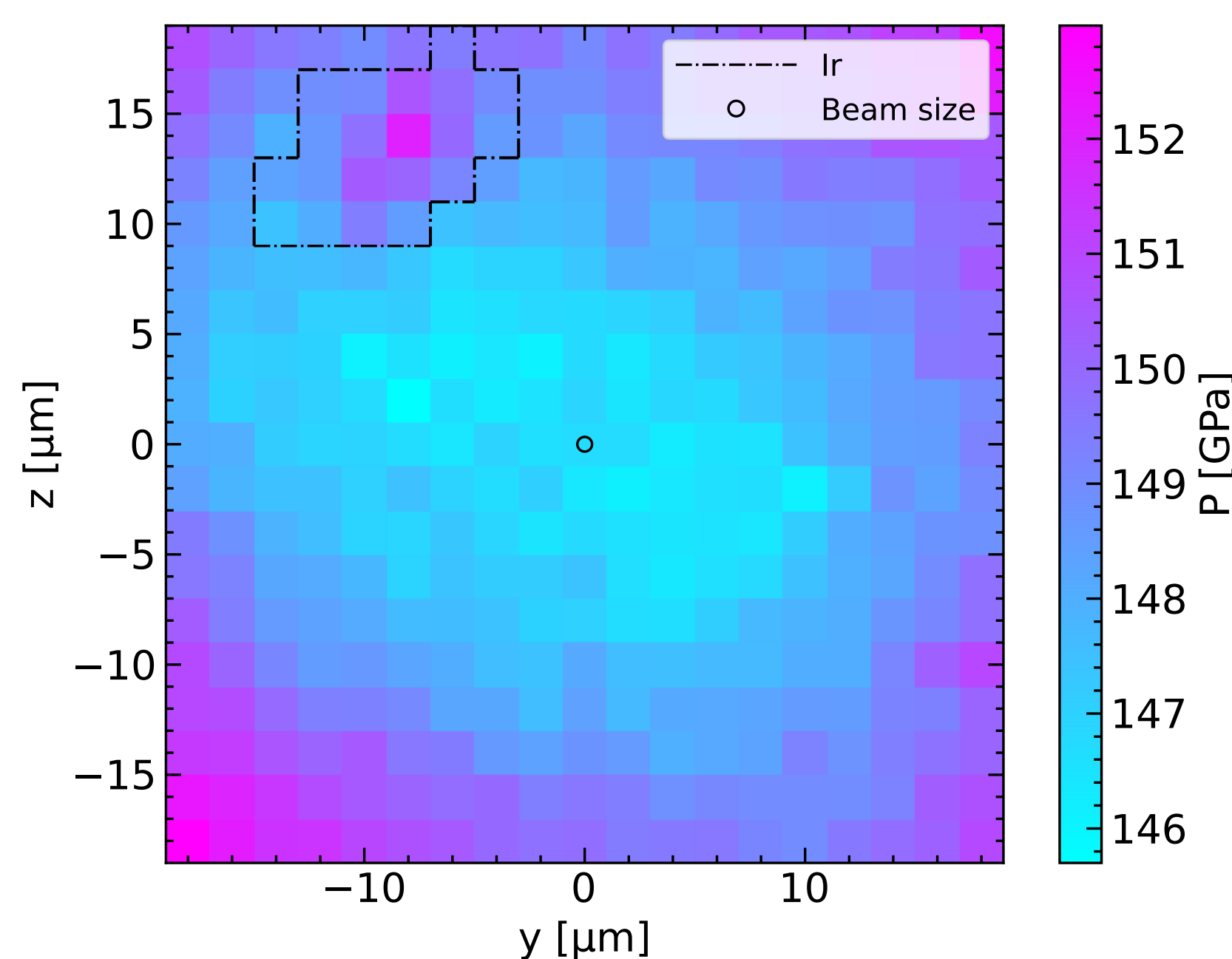


Figure 4: 19x19 grid scan using a sub-micron beam with 1s exposure per point, dashed line indicates the location of the sample. Pressure determined from a Bi EoS.

Which is Experiment, Which is Artificial?

We see random noise of t comparable to a Gaussian distribution with no correlations to the pressure distribution or location of the Ir sample in Figure 4. This highlights the limitations of Singh’s approach to determining strength.

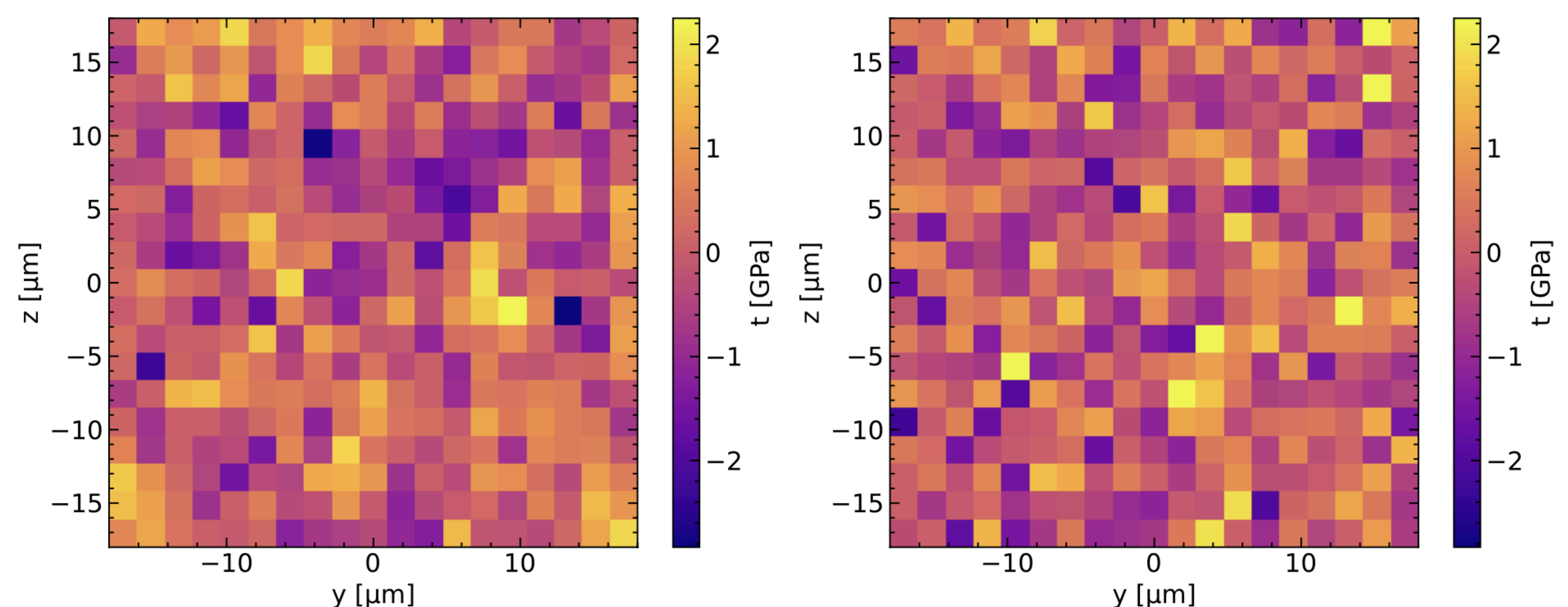


Figure 5: Comparison of a uniaxial stress map at 148 GPa in Bi (left) to a random Gaussian distribution (right) centred around the average value of t in (left) with a 95% confidence range bound by the same limits.

References

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Acknowledgements

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