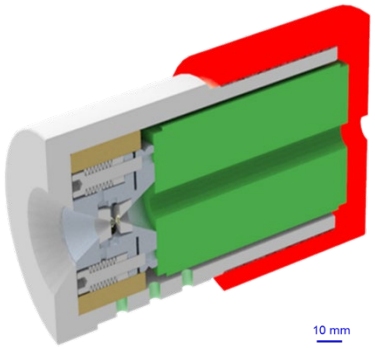
BOOK OF ABSTRACTS



Two Decades of Rapid Compression Diamond Anvil Cell Research: Present and Future Developments

21st - 23rd of July 2025

The invention of the dynamic diamond anvil cell dDAC (Evans et al. 2007) has created a new field of research aiming at bridging the gap between traditional static DAC and dynamic compression experiments. Since the first publication of the original design, almost 20 years have passed, and many new variations of the original design have transpired, including high and low temperature dDACs as well as rapid compression membrane driven DACs and combinations thereof. In addition, analytical techniques used to characterize and quantify compression dependent behavior, have expanded from pure observational work, based on rapid optical cameras, to include time resolved X-ray diffraction and spectroscopy (fluorescence and Raman) at both large scale facilities (Synchrotrons and XFELs) and home laboratories. The science that is being pursued with this new dDAC technology covers the study of kinetic effects during crystallization and melting when crossing liquid-solid, solid-solid and even liquid-liquid phase boundaries. By using different compression rates, we can explore different pathways for the synthesis of new compounds. Thus, it seems timely to gather and review technological advances and more important advances in the field rapid compression science to assess and discuss where this still relatively new field will develop. For this reason, we are organizing a workshop on rapid compression technology and science at DESY in Hamburg.

PROGRAM

Monday July 21st		
13:00 – 13:10	Welcome	O. Seeck (assistant deputy director photon science, DESY)
Session 1: T*echnical overview of rapid compression techniques at different facilities/laboratories** ***Chair: H. P. Liermann		
13:10 – 13:40	P02.2 LTP project: Combining dDAC with XRD, X-ray Imaging and high temperature techniques	E. O'Bannon (LLNL)
13:40 – 14:10	Recent developments at the dynamic diamond anvil cell platform at European XFEL	R. Husband (DESY)
14:10 – 14:40	High-Pressure Non-Equilibrium Phase Transition Dynamics and Physical Properties: Time-Resolved Probing, Rapid Loading Techniques and Applications.	C. Lin (HPSTAR)
14:40 – 15:10	Dynamic diamond anvil cell for time-resolved study: Application on Water	F. Dembele (CEA)
15:10 – 15:40	Coffee Break	
15:40 – 16:10	Developments in High Pressure Platforms Integrated with Advanced X-ray Techniques at HPCAT	C. Park (APS)
16:10 – 16:40	dDAC Research and Development at Los Alamos National Laboratory	G. Zeff (LANL)
16:40 – 17:10	Dynamic compression in dDAC for molecular crystals	J. Yan (Edinburgh Uni.)
17:10 – 17:40	Discussion	H. P. Liermann (DESY)
17:40 – 18:40	Poster Session and Discussion with Beer and Pretzel	
19:00	Self-Paid Dinner (Pizzeria Rolatino)	

Tuesday July 22rd		
Session 2:* Studying kinetics of phase transitions during rapid compression in elements** ***Chair: St. McWilliams		
9:00 – 9:30	Rapid dDAC compression of Bi at ms and µs timescales	E. O'Bannon (LLNL)
9:30 – 10:00	Rapid Compression of Gallium: A Pandora's Box	E. Ehrenreich-Petersen (DESY)
10:00 – 10:30	Coffee Break	
10:30- 11:00	Streamlining x-ray diffraction data process using MILK	A. Berlin (Uni. Utah)
11:00 – 11:30	Using rapid compression DAC for forming amorphous state of materials	G. Yang (Institute of Chemistry, Chinese Academy of Sciences)
11:30 – 12:00	Discussion	St. McWilliams (Uni. Edinburgh)
12:00 – 13:00	Lunch	
Session 3:* Rapid compression in earth and planetary science** ***Chair: K. Appel		
13:00 – 13:30	Dynamic Compression in the DAC to Explore Earth's Deep Interior	H. Marquardt (Uni. of Oxford)
13:30 – 14:00	Cyclic loading experiments in the dynamic DAC: Strains and stresses in polycrystalline MgO	J. Buchen (BGI)
14:00 – 14:30	Laser-heated dDAC: Fe and SiO_2 phase transitions on the millisecond timescales	S. Pandolfi (IMPMC - Sorbonne University)
14:30 – 15:00	Rapid compression of single crystals of quartz in the membrane-driven DAC and implications for the formation of shock-metamorphic effects	C. Otzen (Uni. of Freiburg)
15:00 – 15:30	Coffee Break	
15:30 – 16:00	Insight into impact mineralogy from time-resolved diffraction experiments during fast compression or decompression	L. Ehm (Stony Brook Uni.)
16:00 – 16:30	Compressional rate-dependent stability of ammonia hydrates upon crystallization of water-rich ammonia-water solutions	A. Mondal (ESRF)
16:30 – 17:00	Using rapid compression for measuring the Grüneisen parameter and studying of the equation of state	Lei Su (Shanghai Advanced Research in Physical Sciences)
17:00 – 17:30	Discussion	K. Appel (EuXFEL)
17:30 – 19:00	Poster Session & Discussion	
19:00 – 21:00	Workshop Dinner (BBQ)	

Wednesday July 23rd		
Session 4:* Studying kinetics of phase transitions in molecular solids** ***Chair: N. Giordano		
9:30 – 10:00	Structure Evolution and Kinetics of Phase Transitions in Dense Molecular Solids under Rapid Compression	S.-C. Yoo (Washington State Uni.)
10:00 – 10:30	Study of multiple crystallization pathways via metastable phases in ice VI phase by using dynamic diamond anvil cell	G. Lee (KRISS)
10:30- 11:00	Coffee Break	
11:00 – 11:30	H_2-CH_4 van der Waals compounds under rapid compression	M. P. Alvarez (Edinburgh Uni.)
11:30 – 12:00	Closeout discussion: Which directions is the field or rapid compression developing?	N. Giordano (DESY)
	Farewell	

TALKS

P02.2 LTP project: Combining dDAC with XRD, X-ray Imaging and high temperature techniques

Earl F. O'Bannon III[1] (on behalf of co-authors)

[1] *Lawrence Livermore National Laboratory, Livermore, USA*

The development of new dynamic DAC techniques and diagnostics has occurred over more than 8 years' worth of long term project proposals at the Extreme Conditions beamline P02.2 PETRA-III. This has been a collaboration with scientists from Lawrence Livermore National Laboratory, Los Alamos National Laboratory, the University of Edinburgh, and the ECB. During these multiple projects we have implemented several new capabilities, a faster dynamic diamond anvil cell (dDAC) which was coupled with fast GaAs X-ray detectors we added X-ray phase contrast imaging (PCI) to this fast diffraction setup so we could image opaque metallic samples, and we have combined all the above with two high temperature techniques resistive heating and laser heating. The resistive heating dDAC has been used to dynamically compress a melted Bi sample into the solid Bi-V phase. For higher temperatures, we combined the fast XRD and PCI setup with the existing laser heating setup at ECB. We benchmarked this new technique by investigating the high pressure melting of Pt. We find good agreement with the melting points obtained from this technique with those obtained from more traditional approaches such as X-ray diffraction. These multiple long-term proposals have been very successful in developing new capabilities which have already been utilized by the high-pressure community. Additionally, some of this technology has been transferred to other facilities such as the HED instrument at the EuXFEL.

Recent developments at the dynamic diamond anvil cell platform at European XFEL

Rachel J. Husband[1], Cornelius Strohm[1], Minxue Tang[2], and Zuzana Konôpková[2]

[1]Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany
[2]European XFEL, Schenefeld, Germany.

The structural response of materials over a wide range of strain rates is of interest to a wide range of scientific and engineering disciplines ranging from materials physics to geoscience. The so-called intermediate strain-rate regime (up to 10^3 s^{-1}) can be accessed using piezo-driven dynamic diamond anvil cell (dDACs), which have been shown to be capable of subjecting materials to pressurization rates up to ~100 TPa/s[1] using compression timescales of <1 ms. Since their development almost two decades ago, these devices have been used to study non-equilibrium phenomena at high pressures, particularly relating to phase transition kinetics and the formation of metastable phases. However, limitations in the temporal resolution offered by optical and Synchrotron X-ray structural probes have meant that the majority of studies have been restricted to pressurization rates below ~1000 GPa/s. The unique time structure of the European XFEL bunch pattern, in which MHz pulse trains are produced at a frequency of 10 Hz, makes it the ideal facility to perform time-resolved measurements during dynamic compression on sub-millisecond timescales. The dDAC compression platform[1] at the High Energy Density (HED) Instrument allows for the structural response of the sample to be studied using MHz X-ray diffraction, offering sufficient temporal resolution to accurately pinpoint phase transition pressures at the fastest compression rates achievable with the latest generation of dDAC. Following its commissioning in 2021 (community experiment #2592), this set-up has been offered as a standard platform which has been utilized by a range of user groups studying a diverse set of sample systems.
This talk will present an overview of the current capabilities of the dDAC platform, outlining recent developments and future plans to implement high temperatures capabilities. Aspects which are specific to XFEL research, such as X-ray heating, will be discussed. In addition, we will present some recent scientific highlights from recent experiments performed by selected user groups.

[1]R. J. Husband et al., *'A MHz X-ray diffraction set-up for dynamic compression experiments in the diamond anvil cell.' J Synchrotron Radiat* **30**, 671–85 (2023). doi:10.1107/S1600577523003910

High-Pressure Non-Equilibrium Phase Transition Dynamics and Physical Properties: Time-Resolved Probing, Rapid Loading Techniques and Applications

Chuanlong Lin[1], Yanchun Li[2] and Xiaodong Li[2]

[1]Center for High Pressure Science & Technology Advanced Research (HPSTAR), China
[2]Institute of High Energy physics (IHEP), Chinese Acadmy of Science, China

Changes in crystal structure and material properties are widespread in nature, with nearly all materials undergoing phase transitions when subjected to changes in pressure or temperature. This phenomenon is a significant research subject across multiple disciplines, including condensed matter physics, geoscience, materials science, and chemistry. In high-pressure scientific experiments, the rate of pressure change over time varies depending on the loading method, ranging from nanosecond timescales in shock wave experiments, to millisecond and microsecond scales in rapid loading experiments, and even to minutes or longer in diamond anvil cell (DAC) experiments. This diversity in compression timescales implies that pressure-induced phase transitions and changes of physical properties may exhibit distinct non-equilibrium behaviors across different timescales. The structural evolution pathways and mechanisms of materials transforming from one equilibrium state to another can vary significantly depending on the timescale. Under non-equilibrium conditions, materials may exhibit unique physical phenomena and possess potential application value.

This report focuses on transition kinetics (e.g., Si, Ge, PbS) and mechanoluminescent materials as examples to introduce how our research groups at HPSTAR and IHEP utilizes time-resolved probe and rapid loading techniques to study the kinetics of structural phase transitions and the evolution of material properties under high pressure on different time scales. Through these studies, we reveal the unique dynamic response mechanisms of materials under high-pressure non-equilibrium states across different timescales.

Dynamic diamond anvil cell for time-resolved study: Application on Water

Florian Dembele[1,2] , Charles Pépin[1,2] , Florent Occelli[1,2] , Andre Ramesh[1,2] , Matteo Levantino[3] , Paul Loubeyre[1,2]

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[2]Université Paris-Saclay, CEA Laboratoire matière en conditions Extrêmes, 91680 Bruyères-le-Châtel, France
[3]ESRF, ID09 , 38000 Grenoble, France

Dynamically compressed and statically compressed matter can exhibit different behaviours. Such differences may be related to mechanisms of phase transitions, nucleation kinetics[1], or competition between metastable states[2]. We have developed a dynamical Diamond Anvil Cell to probe compression rates ranging from those obtained by quasi-static second timescale to dynamic nanosecond timescale pressure ramps. Three time-resolved measurements have been developed: angular dispersive X-ray diffraction on the ID09 beamline; optical imaging using a Phantom camera; and fluorescence and Raman spectroscopy.
Initial work on the dynamic compression of water has been focused on solidification of the metastable overcompressed fluid[3]. Recently, we performed a systematic study of dynamically decompressed ice VII. The d-DAC enables a controlled isothermal decompression ramp. The essential output is summarized in the figure below (Fig 1). The boundaries of the phase diagram of H_2O are dramatically shifted. For high compression rate ice VII can be recovered at ambient pressure. The strong shift of the melting line of water questions fundamental issues of how a solid melt.

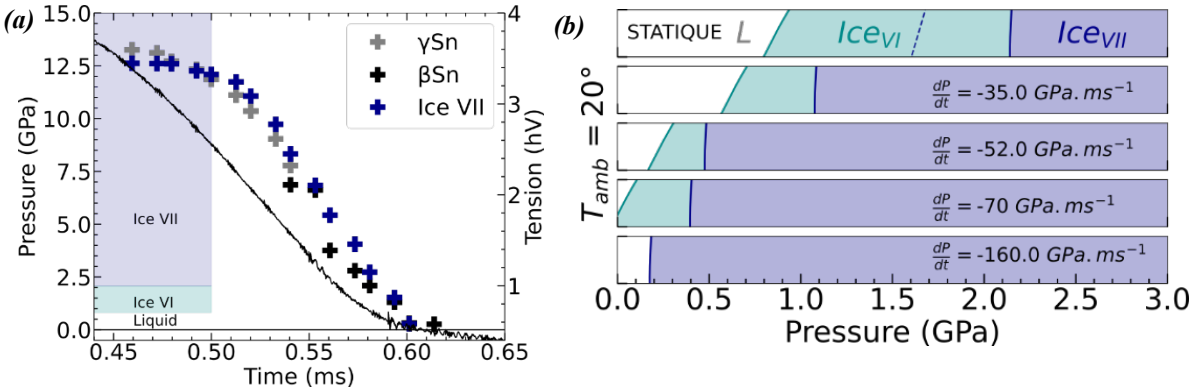

Fig 1. ***(a)*** *The configuration of the dDAC bench for time-resolved X-ray diffraction on ESRF's ID09 beamline enables the acquisition of micro-structural information with a temporal resolution of up to 250 kHz. An example of a decompression process is presented, demonstrating the metastability of ice VII at high decompression rates. Phase identification is obtained from diffraction patterns, and pressure is determined using lattice parameters and the Birch-Murnaghan equation of state.* ***(b)*** *Evolution of the water diagram phase close to ambient in function of the compression rate, data from the dDAC bench and ID09. This study highlights a new paradigm in dynamic exploration of phase diagrams with transition line displacements, due to competition between thermodynamic stability and kinetics nucleation of phase*

[1]Geun Woo Lee et al. "Crystallization of water in a dynamic diamond-anvil cell: Evidence for ice VII like local order in supercompressed water". Phys. Rev. B 74 (13 2006), p. 134112.
[2]Charles M. Pépin et al. "Kinetics and structural changes in dynamically compressed bismuth". Phys. Rev. B 100 (6 2019), p. 060101.
[3]Charles M. Pépin et al., « Metastable Water at Several Compression Rates and Its Freezing Kinetics into Ice VII », *Nature Communications* 15, nº 1 (2024)

Developments in High Pressure Platforms Integrated with Advanced X-ray Techniques at HPCAT

Changyong Park[1]

[1]HPCAT, XSD, Argonne National Laboratory

HPCAT has been a hub for active instrumental and platform development, including hydrostatic or uniaxial compression devices, laser and resistive heating, cryogenic cooling, and dynamic diamond anvil cell (d-DAC), for investigations of the structural, vibrational, electronic, and magnetic properties of materials over a wide range of pressure-temperature conditions. With one of the HPCAT-U beamlines, 16-ID-B, fully operationalized after the APS-U, and with more being commissioned with newly enabled X-ray capabilities, it is imperative to resume existing and new experimental platform developments. Recently, we successfully commissioned a laser-heated d-DAC experiment and introduced a couple of new experimental platforms, including multi-axis diamond anvil cell (MDAC) and high-speed rotational diamond anvil cell (HS-RDAC). These new devices are designed to finely control the deviatoric stress upon compression and decompression, and to induce shear deformation with a controlled rate in azimuthal direction with respect to the probing X-ray beam direction, respectively. While d-DAC is aiming at observing the strain-rate related phenomena, these new strain-tailoring devices complement the study of deformation-related phenomena. With multiple new X-ray capabilities planned to be added, including scanning probe X-ray diffraction imaging (SP-XDI), Bragg coherent diffraction imaging (BCDI), phase contrast imaging (PCI), time-resolved XRD, and combinations of these, HPCAT is enriching the field of high-pressure research with new platforms for deformation- and rate-controlled compression. In this presentation, we will highlight the past and present developments of the experimental platforms at HPCAT and discuss the advanced X-ray techniques that are being integrated with these high-pressure platforms.

*HPCAT operations are supported by DOE-NNSA's Office of Experimental Sciences. The Advanced Photon Source is a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

dDAC Research and Development at Los Alamos National Laboratory

Garrett Zeff[1]

[1] *Los Alamos National Laboratory*

This talk highlights recent and ongoing efforts in dDAC research and development at Los Alamos National Laboratory (LANL). Preliminary dDAC research efforts at LANL began ~10 years ago with dynamic compression experiments employing user facility instrumentation at the PETRA-III Extreme Conditions Beamline (ECB, P02.2) and HPCAT (Argonne National Laboratory). Based on advantages of dDACs at these facilities, as well as some in the literature, we began the development of a LANL dDAC in ~2019. Key features of the LANL dDAC include radial X-ray diffraction capabilities (developed in collaboration with the ECB and the University of Utah) and the implementation of nested piezoelectric actuators to enable measurements during sequential compression and active decompression in a single experiment. We are currently focused on the development of high-temperature resistive heating capability and the deployment of the dDAC in time-resolved Raman spectroscopy experiments. This talk will focus primarily on the technical aspects of the dDAC R&D, using the dynamic compression of various metals and oxides in X-ray diffraction experiments to highlight the scientific advancements enabled by these techniques.

Dynamic compression in dDAC for molecular crystals

Jinwei Yan[1], Miriam Pena Alvarez[1], Phillip Dalladay-Simpson[2], Eugene Gregoryanz[1]

[1]University of Edinburgh
[2]Center for High Pressure Science & Technology Advanced Research

Dynamic diamond anvil cells (dDACs) have enabled precise and rapid pressure modulation far beyond traditional static DACs, reaching compression rates up to hundreds of TPa/s[1,2]. This advancement opens new opportunities to explore non-equilibrium phenomena in materials under extreme conditions, bridging the gap between static compression and shock experiments while avoiding excessive heating or diagnostic limitations[2,3]. Unlike static loading, dynamic compression has been shown to alter melting curves, shift solid–solid phase boundaries, and induce rate-dependent transitions—including pressure-induced amorphization—by kinetically hindering equilibrium pathways and stabilizing metastable structures. We systematically evaluate and apply three types of dDAC systems—piezoactuator-driven (P-dDAC), gas membrane-driven (M-dDAC), and stepper motor-driven (S-dDAC)—to the compression of molecular systems including nitrogen and CH_4–H_2 mixtures[4-6]. We analyze their respective advantages across different pressure ranges, compression rates, and temperature regimes. P-dDACs achieve rates up to 6.8 TPa/s by optimizing input current, while M-dDACs are well-suited for cryogenic conditions (10–250 K), maintaining rates above 400 GPa/s and enabling integration into cryostats. S-dDACs offer precise control at moderate rates and wide compatibility[3]. The application of dDACs on condense molecules reveal that compression rate plays a critical role in determining molecular crystal structure and stability. For instance, rapid compression promotes the formation of γ-N_2[5], while suppressing the formation of hydrogen-rich van der Waals compounds such as $(CH_4)_3(H_2)_{25}$[6]. These findings underscore the kinetic influence of dynamic loading in stabilizing or inhibiting specific phases. By tuning the compression rate and thermal environment, we access metastable states and uncover distinct transformation pathways, demonstrating the unique capabilities of dDAC techniques in high-pressure molecular science.

[1]J.S. Smith, et al.,Rev. Sci. Instrum., 96: 072208 (2015)
[2]Z. Jenei, et al.,Rev. Sci. Instrum., 90: 065114 (2019)
[3]W.J. Evans, et al.,Rev. Sci. Instrum., 78: 073904 (2007)
[4]J. Yan, et al., Rev. Sci. Instrum., 93: 063901 (2022)
[5]J. Yan, et al., Sci. Reports, 14,16394 (2024)
[6]J. Yan, et al., Phys. Rev. B 110, 224109 (2024)

Rapid dDAC compression of Bi at ms and µs timescales

E.F. O'Bannon III[1], C.M. Pepin[2], and co-proposers from Exp. 3076

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Bismuth (Bi) has a rich phase diagram at relatively low pressures and temperatures with a variety of crystal structures accessible below 10 GPa. The high-pressure behavior of Bi has been extensively studied using a variety of high-pressure techniques and diagnostics. Static DAC techniques combined with x-ray diffraction, dynamic diamond anvil cell (dDAC) with x-ray diffraction, and dynamic laser ramp and shock compression with x-ray diffraction and /or VISAR. Early shock compression studies found that the phase transition pressures were consistent with the phase transition pressures reported from static compression studies. However, these studies did not utilize techniques which yielded crystal structure information. Structural determination under dynamic compression has been carried out recently using time resolved X-ray diffraction techniques and significant deviations from the equilibrium phase diagram have been reported. Interestingly Bi-III was not observed in these experiments on compression, and instead Bi-V was observed alongside an unidentified metastable phase Bi-M at pressures as low as 3 GPa. Additionally, the observation of Bi-V at lower pressures in shock experiments when compared to static compression experiments is surprising, since kinetic hinderance typically results in over-driving of the phase transition boundary.

To resolve these discrepancies, we carried out a series of dDAC experiments at the High Energy Density (HED) instrument at the European X-ray free electron laser (EU-XFEL) facility. We utilized the long pulse trains where we collected data for 550 µs at 0.56 MHz. This mode allowed us to collect diffraction data across the entire compression ramp of the dDAC. Before each compression ramp, we carried out runs without compression to assess X-Ray heating of the sample. We chose an X-ray fluence that minimized X-ray heating. Using the data collected during these runs without compression we used a Cu thermal EOS to assess heating as well as finite element modelling and found that the temperature increase was less than 30 K. In all the runs we observe the phase transition sequence that has been established from static compression experiments. Hence, we do not skip Bi-III in these runs and our preliminary analysis suggests that the Bi-III stability field may be shrinking at these compression rates. Notably, the compression rates we achieved in these runs are much slower than those reported in.

Rapid Compression of Gallium: A Pandora's Box

Emma Ehrenreich-Petersen[1], Olivia S. Pardo[2], Thea Engler[1], Bernhard Massani[3], Konstantin Glazyrin[1], Daniel Sneed[2], Johannes Hagemann[1], Mario Wendt[1], Sergej Wenz[1], Garrett Zeff[4], Mathias Trabs[5], Blake T. Sturtevant[4], Christian Schroer[1], R. Stewart McWilliams[3], Zsolt Jenei[2], Hanns-Peter Liermann[1], Earl F. O'Bannon[3]

[1]Deutsches Elektronen-Synchrotron DESY
[2]Lawrence Livermore National Laboratory
[3]University of Edinburgh
[4]Los Alamos National Laboratory
[5]Karlsruher Institut für Technologie

Single elemental systems under high pressure and temperature conditions can exhibit either simple or complex behavior. In this work, we study pure Ga, one of the single elemental systems that has very complex behavior under varying pressure and temperature conditions. Gallium is a crystalline solid at ambient conditions crystallizing in the base centered orthorhombic structure. Ga will melt if the temperature is raised to just above room temperature (~302.9 K) at ambient pressure, and in fact, the heat generated from a human hand will melt it. Also, with just the application of only ~1 GPa at room temperature, Ga will also melt. The pressure-induced solid-liquid transition is particularly interesting as it can be followed during reasonable time scales using a dynamic diamond anvil cell. This means that one can extract the phase fraction using a variety of techniques such as X-ray diffraction (XRD), or X-ray phase contrast imaging (XPCI). The analysis of XRD data and processing of the XPCI data using principal component analysis and machine-learning-assisted segmentation of images gives us the phase fraction as a function of time. Using these data, we can fit various kinetics models to extract different kinetic parameters of the solid-liquid phase transition in Ga. Here, we study how the compression rate and the inclusion of a pressure marker influences these kinetic parameters. Applying increasingly higher pressures disclose a complex nature of this seemingly simple element, where we use XRD to uniquely identify which phases are present at a given time during (de)compression.

Streamlining x-ray diffraction data process using MILK

Audrey Berlin[1]

[1]*University of Utah*

Processing diffraction data is a time-consuming and tedious task for synchrotron users. As such, researchers often opt to use software with easily accessible user interfaces such as MAUD, GSAS, and TOPAS. However, with recent advances in diffraction imaging technology and the development of experimental techniques such as the dynamic diamond anvil cell (dDAC), our capability to generate data has increased exponentially. This "data avalanche" necessitates a new approach to data processing, for which we present a use of the Materials Analysis Using Diffraction (MAUD)[1] Interface Language Kit (MILK)[2]. MILK is a Python scripting interface for the automation of Rietveld analysis, which was initially developed for analysis of neutron diffraction data. Instead of analyzing diffraction image files by hand using MAUD, users can use MILK to programmatically analyze images via sequential and parallel computing, dramatically reducing the amount of time that users spend analyzing data. In this presentation, we will discuss limitations and potential uses for MILK. X-ray diffraction in radial geometry permits measurement of the sample's texture and lattice strain, and our refinements must account for these parameters. We modified MILK's code to process xray diffraction data collected in a radial dynamic diamond anvil cell (RdDAC)[3] experiment at the PETRAIII Extreme Conditions Beamline, P02.2[4]. In this experiment, we compressed iron powder to ~36 GPa while taking in-situ x-ray diffraction images and simultaneous optical images. We will present the use of MILK for creating consistent, reproducible refinements that account for phase change, texture, and lattice strain analysis of RdDAC x-ray diffraction data. MILK has the potential to streamline data processing for users as we enter a new era of experimentation in dynamic compression and advanced data collection.

[1]Lutterotti, L. Maud: a Rietveld analysis program designed for the internet and experiment integration. Acta Crystallogr A 56, s54–s54 (2000)

[2]Savage, D. J. et al. MILK: a Python scripting interface to MAUD for automation of Rietveld analysis. J Appl Crystallogr 56, 1277–1286 (2023)

[3]Huston, L. Q. et al. New dynamic diamond anvil cell for time-resolved radial x-ray diffraction. Rev Sci Instrum 95, (2024)

[4]Liermann, H. P. et al. The Extreme Conditions Beamline P02.2 and the Extreme Conditions Science Infrastructure at PETRA III. J Synchrotron Radiat 22, 908–924 (2015)

Using Rapid compression DAC for Forming Amorphous State of materials

Guoqiang Yang[1]

[1]*Institute of Chemistry, Chinese Academy of Sciences, University of Chinese Academy of Sciences Beijing 100101, China*

In recent years, dynamic diamond anvil cell with the compression/decompression rates between the static high pressure and shock-wave high pressure has attracted great attention of researchers in the field of high pressure science and technology. This technique is regarded as a powerful tool for studying time-dependent high pressure non-equilibrium dynamics, including phase transition kinetics, metastable phase formation and interception, crystal growth, chemical reaction, strain and stress processes, and has broad application prospects. In this report, a rapid compression DAC with different detection system were presented and studies in the amorphous materials formation and others were discussed.

Dynamic Compression in the DAC to Explore Earth's Deep Interior

Hauke Marquardt[1]

[1]*University of Oxford*

Processes in Earth's deep mantle govern our planet's inner dynamics and impact on surface plate tectonics. A quantitative understanding of the physical and chemical properties of the deep mantle is thus pivotal to model Earth's dynamic evolution, including the long-term chemical interactions between mantle and atmosphere that are vital to the development of habitability on Earth, and possibly other planets. In particular, a limited understanding of the effects of major lower mantle phase and spin transitions on physical properties hampers our ability to converge towards a consistent interpretation of seismic observations and evaluate their relevance for mantle dynamic processes.

In this contribution, I will discuss possible applications of time-resolved Synchrotron x-ray diffraction in the dynamic Diamond-Anvil Cell (dDAC) to explore phase transitions in the deep mantle, focusing on the iron spin crossover in (Mg,Fe)O and the post-stishovite transition in SiO_2. Experiments in both axial and radial diffraction geometry will be discussed. The experimental findings allow us to quantify the impact of phase transitions on physical properties as well as their seismic signature, and the results can guide our interpretation of mantle seismic observables and help to quantify the geodynamic impact of mantle phase transitions.

Cyclic loading experiments in the dynamic DAC: Strains and stresses in polycrystalline MgO

Johannes Buchen[1], Biao Wang[2], Niccolò Satta[2], Viktoria Trautner[2], Giacomo Criniti[3], Alba San José Mendéz[4], Hanns-Peter Liermann[4], Hauke Marquardt[2]

[1]University of Bayreuth
[2]University of Oxford
[3]Earth & Planets Laboratory
[4]Deutsches Elektronen-Synchrotron DESY

The propagation of seismic waves through the Earth's interior is controlled by the elastic properties of the rock-forming minerals and by the resultant partitioning of elastic strains and stresses between the grains of these minerals. We present cyclic loading experiments in the dynamic DAC (dDAC) to resolve and quantify the partitioning of elastic strains and stresses in polycrystalline MgO at high pressures and at frequencies close to those of seismic waves. Making use of the dDAC setup at the Extreme Conditions Beamline (ECB, P02.2) at PETRA III, we performed cyclic loading experiments on polycrystalline MgO at pressures between 40 and 70 GPa and at frequencies between 10 and 100 mHz. To capture the time-resolved response of polycrystalline MgO to the applied loading cycles, we recorded time series of X-ray diffraction (XRD) patterns with an acquisition rate of one XRD pattern per second. XRD patters were recorded in radial diffraction geometry to allow for the analysis of lattice strains in terms of volume and elastic shear strains. Our analysis of the oscillating volume and shear strains revealed different modes of the partitioning of elastic strains and stresses between the grains of MgO that arise from the elastic and plastic anisotropy of MgO at high pressures. We further propose a simple way to describe the experimentally observed partitioning of elastic strains and stresses quantitatively. In combination with our analysis approach, cyclic loading experiments will be used in future studies to investigate variations in the dynamic elastic response of polycrystalline materials as a function of frequency, pressure, and temperature.

Laser-heated dDAC: Fe and SiO_2 phase transitions on the millisecond timescales

Stella Chariton[1], Arianna Gleason[2], Maxime Juin[3], Wendy Mao[4], Francesca Miozzi[5], Silvia Pandolfi[3], Vitali Prakapenka[1], Matthew Ricks[4]

[1]GSECARS - University of Chicago
[2]SLAC National Laboratory
[3]IMPMC - Sorbonne University
[4]Stanford University
[5]Carnegie Geophysical Laboratory

We present the setup for dynamic compression in a diamond-anvil cell (DAC) at the 13-ID-D beamline at the Advanced Photon Source in Argonne National Laboratory[1]. Using fast loading in a membrane-DAC we demonstrate compression rates up to 1 TPa/s, coupled with double-sided laser heating and in situ x-ray diffraction with ms temporal resolution. These novel capabilities, bridging previous static- and shock-compression experiments, are used to investigate iron and α-quartz SiO_2 phase transitions. Investigation of SiO_2 transition under dynamic loading aims at reconciling previous shock and static compression experiments, e.g., gas gun or laser ablation[1-4], which fail to explain or reproduce the naturally-occurring impact metamorphism of SiO_2[5,6]. Here, we investigate for the first time the phase transitions of SiO_2 over the millisecond timescales at high temperature (above 100K), reporting stishovite crystallization that was not observed at 300 K[6].

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Rapid compression of single crystals of quartz in the membrane-driven DAC and implications for the formation of shock-metamorphic effects

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Meteorite and asteroid impacts produce extreme pressure and temperature conditions that induce distinct structural changes in quartz, including the formation of planar deformation features (PDFs, i.e. amorphous lamellae) and high-pressure silica polymorphs. Despite their diagnostic importance in identifying impact sites on the Earth, the mechanisms underlying these transformations are poorly constrained. To identify the phase transformation mechanisms, we have carried out rapid, non-hydrostatic compression experiments on single-crystal quartz samples using the membrane-driven diamond anvil cell (mDAC) at pressures up to 35 GPa. Using two crystallographic orientations of the single-crystal quartz samples ((0001) and (10-11)), in-situ synchrotron X-ray diffraction, combined with post-experimental transmission electron microscopy, enabled time-resolved tracking of phase transitions during compression and decompression. In the experiments with quartz cut parallel to (0001), we identified the formation of a previously unknown metastable silica phase isostructural to the mineral rosiaite ($PbSb_2O_6$), occurring via a diffusionless, displacive mechanism. During decompression, this phase transformed into amorphous lamellae resembling PDFs observed in naturally shocked quartz. Besides this, the formation of nanocrystalline stishovite was also observed for the (10-11)-orientation, indicating a further solid-state nucleation pathway. With the remarkable similarity of our final, fully decompressed samples with observations in naturally and experimentally shocked quartz, the findings provide new insights into the atomic-scale processes responsible for the formation of shock-metamorphic effects in quartz. The experiments contribute to bridge the gap between static and dynamic high-pressure experiments and highlight the relevance of rapid compression in the mDAC as a tool for simulating and understanding geological shock phenomena.

Insight into impact mineralogy from time-resolved diffraction experiments during fast compression or decompression

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Impact cratering is recognized as an important process in the formation and evolution of planetary bodies[1]. Impactites and meteorites are records of the aggregation process of asteroids and planetesimal material, the collision of planetary bodies, and ejection processes from the surface of planets and their satellites[2-4]. Our knowledge of impact processes has significantly improved over the past decades through a combination of field studies, experiments and numerical simulations and allows us to infer the peak pressure and temperature conditions during the impact from a combination of petrographic signatures, mineralogical phase transitions and the occurrence of high pressure polymorphs in the impactite or meteorite[2-5]. The current working hypothesis for interpretation of results of long shock duration events is that these are consistent with results from static pressure and temperature experiments. However, our recently conducted fast compression and decompression experiments coupled with time-resolved powder diffraction measurements suggest that phase boundaries and transitions sequences are dependent on compression and heating rates, which would lead to significantly modified phase maps compared to the phase diagrams determined at equilibrium conditions. We will present the results from experiments on key minerals found in common meteorites, which are broadly used in impact barometry, such as the plagioclase solid solution endmembers albite and anorthite, as well as enstatite. The influence of compression/decompression rates and temperature on the structural phase transitions in enstatite and on the formation of maskelynite, amorphous plagioclase will be discussed.

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Compressional rate-dependent stability of ammonia hydrates upon crystallization of water-rich ammonia-water solutions

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Understanding how water-rich ammonia-water (NH_3-H_2O) solutions crystallize and the phase stability of resulting ammonia hydrates, is key to unraveling the complex hydrogen-bonding networks that govern their physical and chemical behavior. Yet, results on the crystallization pathways at room temperature remain controversial[1,2], reporting either the formation of ammonia hemihydrate (AHH, $2NH_3$:$1H_2O$) + ice VII or ammonia monohydrate (AMH, $1NH_3$:$1H_2O$) + ice VII at similar conditions. Here we investigate the role of compression rate, a previously overlooked factor, to resolve discrepancies in the reported P-T phase diagrams and phase stability. We take advantage of the diamond anvil cell setup for dynamic compression (dDAC) facilities available at P02.2 DESY to follow the crystallization of 25 wt% NH_3-H_2O solutions at compression rates ranging between 0.005 to 4 GPa/sec. Compression rates exceeding 0.5 GPa/sec promote direct crystallization of a body-centered cubic (bcc) phase (DMA') with possible AMH stoichiometry coexisting with H_2O ice VII, while compression rates below 0.2 GPa/sec stabilize monoclinic NH_3-rich AHH-II and ice VII phases. In contrast, intermediate rates produce a mixture of both hydrates alongside ice VII. The compression behavior and phase stability of these two distinct phase assemblies are investigated furthermore, which warrant further discussion on the composition of the DMA' phase, the effect of ice VII on the compressibility of ammonia hydrates and the possibility of incorporating NH_3 impurities within the ice VII bcc crystal lattice.

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Using rapid compression for measuring the Grüneisen parameter and studying of the equation of state

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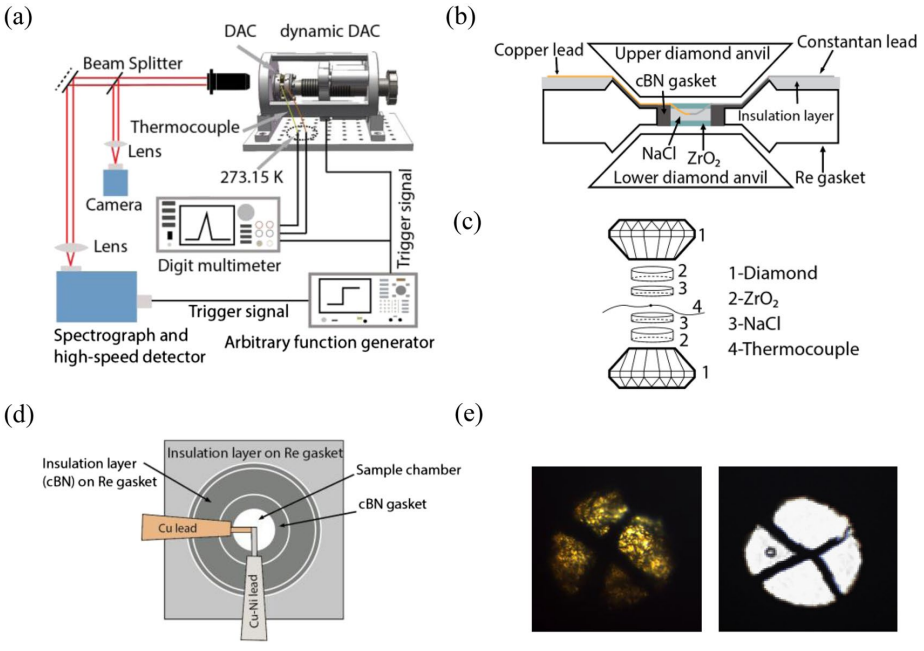
Equation of state is a thermodynamic equation relating state variables, which describe the state of matter under a given set of physical conditions. The Grüneisen parameter (γ) is crucial for determining many thermal properties, including the anharmonic effect, thermostatistics, and equation of state of materials. However, the isentropic adiabatic compression conditions required to measure the Grüneisen parameter under high pressure are difficult to achieve. Thus, direct experimental Grüneisen parameter data in a wide range of pressures is sparse. We developed a new device that can apply pressure (up to tens of GPa) with an extremely short time of about 0.5 ms, confidently achieving isentropic adiabatic compression. Then, we applied our new technique to sodium chloride and measured its Grüneisen parameter, which conforms to previous theoretical predictions. According to our obtained sodium chloride Grüneisen parameters, the calculated Hugoniot curve of the NaCl B1 phase appears up to 20 GPa and 960 K. Our results suggest that this new method can reliably measure the Grüneisen parameter of even more materials, which is significant for researching the equation of state in substances.



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Structure Evolution and Kinetics of Phase Transitions in Dense Molecular Solids under Rapid Compression

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Understanding the structure evolution and kinetics of phase transitions of simple molecular solids such as H_2O and N_2 under dynamic compression has been an active area of research for many decades. Primarily, these studies have used either gas guns or lasers to drive the sample with high strain rates to high pressure-temperature (PT) conditions. Relatively recent development of piezo-electric actuator driven dynamic-diamond anvil cells (d-DAC) can bridge the strain rate gap between traditional static DAC and shock compression studies, ultimately in an emerging phase space. Coupling with the 3G(synchrotron) and 4G(XFEL) X-rays and time-resolved (TR) optical and spectroscopic probes, d-DAC is now capable of probing time-evolution of crystal structure and chemical bonding under dynamic loading to strain rate of 103 /sec and, thereby, providing fundamental insights into kinetics and mechanisms governing phase transitions of dense solids at extreme PT conditions. The study in this intermediate strain rate regime is important, not only to complement conventional static (105 /s) experiments along nearly isothermal compression pathways, but also because many diffusion-limited phase transitions of dense fluids and solids occur in this time scale of μs to ms. In this talk, following a brief description of technology developments amid d-DAC and various TR spectroscopic and X-ray diffraction probes, we will describe our recent efforts to investigate structure evolutions and chemical mechanisms governing the phase transitions in H_2O and N_2 under rapid compression and offer a few insights into future dynamic research direction using d-DAC

Study of multiple crystallization pathways via metastable phases in ice VI phase by using dynamic diamond anvil cell

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Dynamic pressurization has revealed various metastable phases of H_2O due to slow kinetics at low temperature, which yields diverse crystallization pathways. However, the crystallization pathways is rather simple at elevated temperature, since atoms and molecules are thermally activated. However, if physical changes (e.g., (de)compression rate, driving force, growth rate) can significantly affect the individual and collective motion of atoms or molecules, we may have a chance to find the metastable phases and multiple crystallization pathways. Here, we study the crystallization pathways of ice VI by controlling the metastability and detecting fast kinetics. We combine dynamic diamond anvil cell (dDAC) technique and X-ray free electron laser (XFEL) which can achieve the metastability of liquid water and detect the fast kinetics of the crystallization with a short time-resolution of a few microseconds. This technique reveals at least *five different pathways of freezing-melting process* at ROOM Temperature which are hidden within stable ice VI pressure regime. This occurs via metastable ice VII and a new metastable ice which exist both within the stable ice VI pressure range. We identify the structure of the new metastable ice and call ice XXI. Moreover, molecular dynamics simulation shows that supercompressed water evolves from high-density water (HDW) to very high-density water (VHDW) as pressure increases. This structural evolution of supercompressed water affects nucleation of the competing metastable crystalline phases and thus the multiple transition pathways in the stable ice VI pressure regime, which is in line with Ostwald's step rule. These findings provide new insights to find more metastable ice phases and their transition pathways at room or elevated temperatures on icy planets.

H_2-CH_4 van der Waals compounds under rapid compression

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Under high pressure, methane and hydrogen form $(CH_4)_2$ H_2, $CH_4(H_2)_2$, and $(CH_4)_3(H_2)_{25}$ van der Waals compounds with stoichiometries determined by the H_2 concentration. This study investigates the effect of compression rates (up to 183 GPa/s) on the formation and morphology of these compounds at pressures up to 60 GPa utilizing dynamic diamond anvil cells combined with time-resolved Raman spectroscopy.

POSTERS

Poster 1: The lattice parameter development of Zinc (Zn), during fast compression in the dDAC

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The work presented here was collected at intermediate compression rates leading to effective strain rates of 2×10^{-2} s^{-1} to 2.7×10^{2} s^{-1} and using the dynamic diamond anvil cell at P02.2, PETRA III, DESY. The evolution of the lattice parameter of Zinc (Zn) depends on the sample environment and, consequently, the pressure transmitting medium. Nevertheless, we observe a significant change in Zn's non-hydrostatic lattice parameter evolution, when compressed at strain rates of 2×10^{-2} s^{-1}, or 2.7×10^{2} s^{-1}. The *c/a* ratio at 2.7×10^{2} s^{-1} does not correlate with the results of quasi-static or slow, non-hydrostatic experiments. Instead, the observed lattice parameter shows higher *c/a* ratio, which is the characteristic of hydrostatic conditions. The deviation indicates that the strain rate drastically alters the development and predominance of the different deformation mechanisms.

Poster 2: Rapid Compression at the DanMAX beamline

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Herein rapid compression capabilities at the DanMAX beamline at the MAX IV synchrotron are shown. Compression rates up to 7 GPa/s are demonstrated on different systems including Sb_2Te_3, Bi_2Se_3, Bi_2Te_3 as well as elemental Sb, giving an overview of the attainable data quality and possibilities of rapid compression at the DanMAX beamline. The obtained data are furthermore compared with published high-pressure studies of the compounds showing whether dynamic compression has an effect on observed high-pressure polymorphs and crystal structures in these compounds.

Poster 3: Present and Future Extreme Conditions Research at Low (PETRA III) & Ultra-Low (PETRA IV) Emittance Synchrotron Sources at DESY

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With the advent of high energy ultra-low emittance synchrotron sources extreme conditions research at high-pressure and high/low temperatures has enter a new realm. While at 3rd generation sources mostly static high-pressure experiments have been performed, the 4th generation sources will enable the exploration of hierarchical structures both in space and time, making optimal use of the low emittance and even more important the superb coherence of these new sources.

In this presentation we demonstrate the current status of Diamond Anvil Cell (DAC) research capabilities at the Extreme Conditions Beamline P02.2 at PETRA III and high-light the new developments. Particular emphasized will be the combination of time resolved X-ray diffraction and coherent imaging that has been developed at P02.2 in order to study hierarchies in space and time, which will be the major focus of the Extreme Conditions Time Resolved XRD & Imaging Microscope (ExTReM) at PETRA IV. Thus, ExTReM will combine the unprecedented brilliance and coherence of PETRA IV to perform static (min) to fast (MHz) high-P and high/low-T X-ray diffraction and imaging experiments at high energies to create the ultimate microscope that enables the study of 5 dimensions spanning 6–7 orders of magnitude in time, spatial resolution, and compression rate.

Poster 4: Time-resolved x-ray imaging and diffraction in the dynamic diamond anvil cell at extreme conditions

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Through collaboration between LLNL and the Extreme Condition Beamline at PETRA-III, new technical advancements have enabled time-resolved, simultaneous x-ray imaging and diffraction in the diamond anvil cell. Combined with in-situ diffraction, we achieve ultra-fast propagation-based phase-contrast imaging of materials, including those opaque to visible light, on millisecond to minute time-scales in a variety of pressure-temperature environments. We utilize both static and dynamic diamond anvil cells to characterize the kinetics of high-pressure phase transitions, including visualization of melt and crystallite growth regions, observation of nucleation sites, and evolution of crystallite morphology during compression and decompression following a user-defined voltage waveform driven by a piezo-actuator. We discuss the current capabilities of this experimental set-up, present examples of sample chamber imaging during ramp compression, and highlight updates in utilizing machine-learning for image processing to extract valuable kinetic parameters for our understanding of phase transformations within materials under extreme conditions.

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Poster 5: High-pressure time resolved X-ray diffraction experiments of lanthanides

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Both Cerium and Praseodymium undergo a volume collapse under compression that originates from the same underlying behavior of the 4f electron(s). Yet the outcome could not be more different: In the case of cerium with one affected 4f electron the volume collapse leaves the crystal symmetry intact whereas for Praseodymium with two 4f electrons the crystal symmetry changes from a high symmetry dfcc to a low symmetry α-U. Our dynamic compression experiments in a diamond anvil cell now also reveal kinetic differences despite the same fast electronic mechanism. The transition cannot be overdriven in pressure in cerium which indicates a fast kinetic process whereas fast compression rates in Pr lead to a pressure increase for the phase boundary pointing to slower kinetics possibly due to the realization of a new crystal structure.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344

Poster 6: A thin Dynamic Diamond Anvil Cell: Proof of Concept Experiments

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It is well-known that the current versions of the piezo-driven dynamic diamond anvil cell (dDAC) are very demanding on the space required. This means that previously one has been restricted to only temperatures obtainable by resistive heating and to rather simple experimental setups.

Therefore, we present a recent advancement to overcome these issues with the fabrication of a thin dDAC. This new development opens up for several new experiments. The thin length of the dDAC provide enough space to drive in mirrors for laser-heating without changing the laser-heating setup. As a result, we are able to use this new dDAC to expand the range of temperatures that can be reached using a piezo-driven dDAC. However, not only those experiments requiring high temperatures, but also experiments with more complicated setups can be probed, for instance multimodal imaging.

Here, we show an example from the laser-heating table at the Extreme Conditions Beamline, P02.2, at PETRA III, DESY and one from the general-purpose table. The former, for testing the capabilities and heating profiles achievable whereas the latter uses an intricate setup using an optical microscope together with X-ray phase contrast imaging (XPCI) and diffraction – a setup enabled by the new LAMBDA 9M detector which features a hole in the direct beam path for XPCI and a new objective with a hole placed upstream of the sample. This is used to study and compare the information one gets from optical micrographs versus what one gets from XPCI.