# <u>**4th Worshop on Studies of**</u>

# Dynamically Compressed Matter with X-rays

<u>CoMax</u>

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ESRF

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STREAMLINE

### **Invited Speakers**

Mandy Bethkenhagen, LULI, France Erik Brambrink, European XFEL, Germany Pinaki Das, Washington State University, USA Adrien Descamps, Queen's University Belfast, UK Jean-Alexis Hernandez, ESRF, France Mikko Hokka, Tampere University, Finland Andy Krygier, Lawrence Livermore National Laboratory, USA Bratislav Lukic, ESRF, France Malcom McMahon, University of Edinburgh, United Kindom Ivan I. Oleynik, University of South Florida, USA Silvia Pandolfi, Sorbonne Université, France Arnaud Sollier, CEA, France

### **Organisers**

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Michel Arrigoni, ENSTA, France Simon Bland, ICL, UK Jon Eggert, LLNL, US Marion Harmand, IMPMC, France Dominik Kraus, HZDR, Germany Paul Loubeyre, CEA, France Sandro Scandolo, ICTP, Italy Cornelius Strohm, EuXfel, Germany Tommaso Vinci, LULI, France

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### Dynamically Compressed Matter with X-rays

12 March to 14 March 2024

#### Tuesday 12 March – ESRF Auditorium

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14:00	Welcome: R. Torchio and M. Mezouar	
	Session I: Dynamic compression at the ESRF - Chair: O.	Mathon
14:15	Status and first results from the High Power Laser Facility	J-A. Hernandez ESRF, France
14:40	Experimental platforms for dynamic studies with MHz X-ray radioscopy at ID19 of ESRF	<b>B. Lukic</b> ESRF, France
15:05	Development of a laser-driven shock compression platform at the ID09 beamline of the European Synchrotron Radiation Facility	<b>A. Sollier</b> CEA, Arpajon, France
15:30	Coffee break	
	Session II: Matter under high strain rate - Chair: S. E	lland
15:50	XPCI Observations of Dynamic Compressive Fracture of Fiber Reinforced Polymer Composites and Granite Rocks	<b>M. Hokka</b> Tampere University, Finland
16:20	Pores collapse and spall fracture: a direct observation using fast ultra-high speed X-ray Phase Contrast Imaging	<b>T. Virazels</b> University Carlos III of Madrid, Spain
16:40	Dynamic compaction under shock loading experiments using ultra- high speed X-ray radiography	<b>M. Arrigoni</b> ENSTA Bretagne, France
17:00	Probing the evolution of solid microjets from grooved Sn samples and their impact on Asay foil and piezoelectric mass diagnostics using X-ray radiography.	<b>JR. Burie</b> CEA Arpajon, France
	Session III: Sponsor	
17:20	High Power Nanosecond Laser for Dynamic Shock Compression and roadmap to high peak power laser at high repetition rate	<b>O. Zabiolle</b> Sponsor: Amplitude, France
17:40	Break	
18:00	Beamlines visits	
18:30	Poster session & Wine and cheese	



#### Wednesday 13 March – ESRF Auditorium

	Session IV: Phase diagrams and Warm Dense Matter - Ch	nair: D. Kraus
8:30	X-ray absorption spectroscopy of dynamically compressed matter	<b>A. Krygier</b> LLNL, USA
9:00	Ultrafast X-ray Absorption Spectroscopy on Fe and Cu up to shock melting collected at HPLF (ESRF)	S. Balugani ESRF, France
9:20	X-ray diffraction measurements across the melt line in shocked nickel	K.A. Pereira University of Massachusetts, USA
9:40	Femtosecond structural probing of warm dense matter with Betatron laser-based X-ray source	<b>F. Dorchies</b> Univ. Bordeaux, CNRS, CEA, CELIA, France
10:00	A pulsed power facility for studying the warm dense matter regime	<b>B. Jodar</b> CEA, DAM, France
10:20	Coffee break & Group Photo	
	Session V: Dynamic compression for Planetary Science - Ch	air: S. Scandolo
10:50	Exploring the deep interior of ice giant planets with density functional theory and shock-compression experiments	M. Bethkenhagen LULI, Ecole Polytechnique, France
11:20	Combining ultrafast X-ray spectroscopy and diffraction to investigate deep planetary interior conditions	<b>S. Pandolfi</b> Sorbonne Université, France
11:50	Time-resolved X-ray absorption spectroscopy of shocked magnesiosiderite: possible implications for the origin of magnetite in Martian meteorite ALH84001	<b>A.P. Dwivedi</b> European XFEL, Germany
12:10	Phase transitions and electronic properties of Fe2O3 under laser compression by ultrafast in-situ X-ray absorption spectroscopy	J. Pintor IMPMC, Sorbonne University, France
12:30	Lunch	
13:40	Beamlines visits	
14:30	Roundtable & Coffee	
	Session VI: New instrumental opportunities - Chair:	T. Vinci
16:25	The HED instrument at European XFEL: Unique capabilities to study material properties of laser-compressed matter	<b>E. Brambrink</b> European XFEL, Germany
10.25	First results obtained using the DiPOLE laser at the European XFEL	<b>M. McMahon</b> University of Edinburgh, UK
17:10	Towards the direct measurement of bulk temperature in shock- compressed matter using inelastic X-ray scattering at an XFEL	<b>A. Descamps</b> Queen's University Belfast, UK
	Single pulse extended x-ray absorption fine structure capability at	<b>P. Das</b> Washington State University,
17:40	the Dynamic Compression Sector - ZOOM Presentation	USA
17:40 18:25		-

#### Thursday, 14 March – ESRF

	Session VII: Matter under high strain rates - Chair: A	. Rack
9:00	Ductile failure in metals: How does void growth rate depend on loading rate?	<b>G. C. Ganzenmüller,</b> Albert-Ludwigs Universität Freiburg, Germany
9:20	Dynamic Shear Localization and Vortical Flow during Shock Wave Induced Pore Collapse in Sucrose	<b>S. Neogi</b> Brown University, USA
9:40	A new approach for shockwave experiments in arbitrary geometries: pulsed power driven exploding wires and foils	J. Strucka Imperial College London, UK
10:00	Measuring material strength in laser compressed Ta via in situ X- ray diffraction at the Dynamic Compression Sector	<b>D. Mcgonegle</b> AWE, UK
10:20	Coffee break	
	Session VIII: Phase transitions, phase diagrams - Chair: N	M. Levantino
10:40	Phase transitions in amorphous materials under shock compression	I. Oleynik University of South Florida, USA
11:10	Macroscopic and microscopic study of Glassy GeO <sub>2</sub>	<b>A. Benuzzi</b> LULI, Ecole Polytechnique, France
11:30	Dynamic behaviour of zirconium-based metallic glasses under laser shock compression	Y. Raffray Institute of Physics Rennes, UMR CNRS, France
11:50	Studies of Dynamically Compressed Methane-Hydrogen with Raman Spectroscopy	J. Yan University of Edinburgh, U.K.
12:10	Conclusion	
12:30	Lunch	- -
13:40	Beamlines visits	
14:30 - 17:30	Instrumental Training Session	

#### X-ray Absorption Spectroscopy coupled to laser-driven dynamic compression at ESRF on ID24-ED with HPLF

J.-A. Hernandez<sup>1</sup>, N. Sevelin-Radiguet<sup>1</sup>, R. Torchio<sup>1</sup>, S. Balugani<sup>1</sup>, G. Berruyer<sup>1</sup>, S. Chazalette<sup>1</sup>, C. Clavel<sup>1</sup>, D. Bugnazet<sup>1</sup>, D. Lorphèvre<sup>1</sup>, F. Perrin<sup>1</sup>, S. Pasternak<sup>1</sup>, F. Villar<sup>1</sup> and O. Mathon<sup>1</sup>

<sup>1</sup> European Synchrotron Radiation Facility

The High-Power Laser Facility is a laser-driven dynamic compression platform (drive laser and optical diagnostics) coupled to the energy-dispersive X-ray absorption spectroscopy beamline ID24-ED at the ESRF and offers *in situ* high quality single-bunch (100 ps) XANES and EXAFS measurements between 5 and 28 keV. In this presentation we will provide an overview of the facility and its evolution over the past couple of years as well as some scientific results obtained during the 13 user experiments already performed.

HPLF drive laser currently delivers 55 J in 4 to 15 ns square pulses, producing ablation pressures up to 140 GPa in black kapton. The facility is equipped with two line-imaging VISAR and soon with a line-imaging streak optical pyrometry system (mid-2024).

#### Experimental platforms for dynamic studies with MHz X-ray radioscopy at ID19 of ESRF

B. Lukić<sup>1</sup>, A. Rack<sup>1</sup>, A. Cohen<sup>2</sup>, A. Sollier<sup>3</sup>, S. Bland<sup>4</sup>, W. Proud<sup>5</sup>, D. Eakins<sup>6</sup>

<sup>1</sup>ESRF –The European Synchrotron, 71 Avenue des Martyrs, Grenoble, France; <sup>2</sup>Nuclear Research Center – Negev (NRCN), Beer Sheva, Israel; <sup>3</sup>CEA, DAM, DIF, Arpajon, France; <sup>4</sup>Plasma Physics Group, Imperial College London, UK; <sup>5</sup>Institute of Shock Physics, Imperial College London, UK; <sup>6</sup>Department of Engineering Science, University of Oxford, Parks Road, Oxford, UK; **lukic@esrf.fr** 

3rd and 4th generation high-energy synchrotron photon sources present unparalleled capabilities for investigating material responses during transient dynamics at high spatiotemporal scales using hard X-rays. The combination of high brilliance, short bunch duration (down to 60 ps) and high energy, such as of the extremely brilliant source at ESRF-EBS [1], provides an avenue for exploring materials under extreme conditions of shock and highstrain rate loading, coupled with *in-situ* subsurface ultra-high-speed X-ray radioscopy measurements. These capabilities, together with the high imaging sensitivity and beam size of the ID19 beamline, facilitate the sub-surface visualization of engineering-scale structures and natural systems at representative scales (i.e., micro- to meso-scale). In addition, this has spurred the development of material loading platforms within the beamline setting such as the Split-Hopkinson Pressure bar (SHPB), single-stage gas launcher, ns-pulsed laser shock, pulsed power-driver, and a chamber compatible with energetic materials. These platforms, open to the user community, enable the study of materials under a diverse array of extreme loading scenarios. ESRF has recently introduced new access modes [2], such as block allocation groups (BAG), aim at building a collaborative community and providing regular access to the shared pool of these cutting-edge installations. Naturally, the so-called "Shock" BAG was formed, bringing together experts in shock physics and dynamic behaviour of materials, building upon the installations available at the beamline. The resulting community-driven scientific topics tackle the growing demand for developing novel engineering materials with the ability to sustain the high strain rate and shock as well as fundamental physical questions of material phase change and instabilities of shocked matter. Some of the recent experimental campaigns have been successfully conducted using the SHPB, gas gun and pulsed laser installations, with applications ranging from reproducing earthquake scenarios, dynamic fracture of novel composite and additively manufactured materials, to shock propagation and dynamically driven cavity collapse, of which selected examples will be showcased. Finally, future outlooks and developments will be provided, focusing at firm establishment of the Shock physics user program at ESRF.

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- [3] M. Olbinado, et al., Optics Express, 25(12), 13857-13871, (2017).

<sup>[1]</sup> P. Raimondi, et al., Communications Physics, 6, 82, (2023).

#### Development of a laser-driven shock compression platform at the ID09 beamline of the European Synchrotron Radiation Facility

A. Sollier<sup>1,2</sup>, C. Pépin<sup>1,2</sup>, M. Kozhaev<sup>3</sup>, C. Mariette<sup>3</sup>, W. Reichenbach<sup>3</sup>, M. Levantino<sup>3</sup>

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Over the past decade the coupling of new dynamic compression platforms with large scale X-ray facilities [1-2] has led to a revolution in the field of high-energy-density physics (HEDP), which studies the properties of materials under extreme conditions of pressure and temperature. It is now possible to probe matter in situ, with temporal and spatial scales commensurate with those of molecular dynamic (MD) simulations [3], therefore providing a direct mean for verification and validation of their predictions. At ESRF, we have demonstrated the possibility of studying laser-shocked Fe [4] and Ta [5] samples up to pressures of several Mbar in the Warm Dense Matter (WDM) regime, through single-pulse synchrotron X-ray absorption spectroscopy (XAS) experiments performed on the ID24 beamline with the GCLT portable high power laser system. These experiments were the first to deliver high-quality data in both the XANES and the EXAFS range for materials under dynamic compression. Their success led to the launch of the High Power Laser Facility (HPLF) project [6] on the ID24 beamline. The phase 1 of this project (HPLF-1), which aims at coupling a 100 J laser to the ID24 beamline to perform laser shock and ramp-compression experiments probed by time-resolved XAS is now completed, and several experiments have now already been performed on this facility. The second phase of the project (HPLF-II) foresees the extension to other X-ray techniques, such as XRD, in a soon future. In order to pave the way for this extension, we have submitted a long term project (HC-4528) on the ID09 beamline, which aims at developing a laser-driven dynamic compression platform to perform ps time resolved XRD pump-probe experiments at high pressure. Indeed, we have already demonstrated that this time-resolved beamline is particularly well suited to study the kinetics of phase transitions [7,8], but we were limited in pressure due to the use of an existing low power drive laser system. The laser-driven shock compression platform will link a new custom high power EKSPLA laser system with high energy X-ray pulses from the ESRF to perform in situ, time-resolved XRD measurements in materials subjected to wellcharacterized, moderate pressure ( $\leq 1$  Mbar) and short duration shock waves.

In this talk, we will describe the new shock compression platform, and present first experimental results from shock compressed iron and tin samples that demonstrate the facility's capability for acquiring high quality XRD data.

- [1] J. S. Wark, M. I. McMahon, and J. H. Eggert, J. Appl. Phys. 132, 080902 (2022).
- [2] S. Pascarelli et al., Nat. Rev. Methods Primers 3, 82 (2023).
- [3] P. Wen, G. Tao, D. E. Spearot, S. R. Phillpot, J. Appl. Phys. 131, 051101 (2022).
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#### XPCI Observations of Dynamic Compressive Fracture of Fiber Reinforced Polymer Composites and Granite Rocks

M. Hokka, A. Rubio Ruiz, N. Pournoori, M. Kanerva, M. Isakov

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Understanding of fracture and fragmentation behavior of materials at high rates of loading is of great importance for developing new materials and predicting their behavior in various challenging environments and conditions. For example, development of rock drilling technologies requires good fundamental understanding of rock fracture at different conditions. Most rock drilling technologies are based on applying fast mechanical loading (impact) on the rock mass which leads to complex stress states and transient high strain rate phenomena within the rock mass and the drill tools. Rocks contain several different minerals, microcracks, pores, absorbed liquids and gases, and interfaces which leads to complex dynamic mechanical response, fracture, and fragmentation behavior. Similarly, Carbon Fiber Reinforced Polymer (CFRP) composites are used extensively in lightweight applications across various industries including demanding impact-resistant structural designs. The inherent anisotropy of FRP composites, arising from the orientation of reinforcing fibres in a laminate, significantly affects the levels of strength, stiffness, and resistance to specific failure modes. Therefore, predicting how and where CFRP laminates will fail under impact loads is challenging. The XPCI enables the visualisation of damage initiation, propagation and fragmentation of complex materials under dynamic loading. This presentation describes successful cases of the application of ultrafast XPCI as a tool for characterising the failure and fragmentation processes of granite and CFRP. The XPC imaging was carried out at ID-19 of the European Synchrotron Radiation Facility, where a Split Hopkinson Pressure bar device was used to apply dynamic compressive load on the specimen, while imaging deformation of the specimen during loading. This work is complemented by experiments carried out at Tampere University, where also high speed infrared imaging (IRT) and high speed imaging (DIC) were used to characterize the full thermomechanical phenomena related to the dynamic fracture. The compressed angle-ply laminates failed due to combined normal and shear stresses and disintegrated into pieces by intralaminar crack growth [1]. The IR measurements revealed highly localized heating of the specimens. The XPCI images also indicated delamination and fracture at the interface of the  $0^{\circ}$  and  $\pm 45^{\circ}$  angled layers inside the laminate. XPCI offers a helpful tool for observing the formation of internal damage in different materials under dynamic loading conditions [2]. These observations serve as a qualitative reference for calibration and validation of numerical models to simulate damage evolution in granite and CFRP. Some examples of how the XPCI were used to validate finite element models used to simulate the fragmentation process of granite and CFRP are shown in this presentation.

<sup>[1]</sup> Nazanin Pournoori, Guilherme Corrêa Soares, Bratislav Lukić, Matti Isakov, Maria Clara Lessa Belone, Mikko Hokka, Mikko Kanerva, In situ damage characterization of CFRP under compression using high-speed optical, infrared and synchrotron X-ray phase-contrast imaging, Composites Part A: Applied Science and Manufacturing, Volume 175, 2023,107766,

<sup>[2]</sup> A. Rubio, N. Pournoori, M. Isakov, T. Saksala, R. Bjørge, A. Kane, M. Hokka, Progressive weakening of granite by piezoelectric excitation of quartz with alternating current, under review at Journal of Rock Mechanics and Rock Engineering (2023).

#### PORES COLLAPSE AND SPALL FRACTURE: A DIRECT OBSERVATION USING FAST ULTRA-HIGH SPEED X-RAY PHASE CONTRAST IMAGING

Thomas Virazels\*, José A. Rodríguez-Martínez\*,+, Javier Garcia+, Federico Sket+, Bratislav Lukić\*\*, Alexander Rack\*\*, David Pedroche\*, Sergio Puerta\*

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**ABSTRACT** Plate-impact experiments have been performed at the ID19 beamline of the European Synchrotron Radiation Facility on porous plates printed using SLM technique out of AlSi10Mg and Ti6Al4V. The samples contain a large population of voids with different sizes varying from 5 to 120 µm. The tests have been conducted with a meso-scale gas-launcher at different impact velocities ranging from 150 to 650 m/s. The targets have been analyzed by X-ray tomography before and after testing, to obtain a 3D reconstruction of the porous microstructure of the samples. In-situ radiography has allowed to obtain time-resolved measurements of the shape and size evolution of voids. The tests have been monitored with a PDV system which was synchronized with the radiographies to correlate the velocity of the free surface of the target with the initial collapse of the pores and the later spall fracture. Finite element simulations of the experiments, with explicit representation of the porous microstructure have been performed. The analysis of experiments and calculations has provided new insights on the effect of porosity on the void collapse and spall fracture of porous metallic materials fabricated using additive manufacturing.

## Dynamic compaction under shock loading experiments using ultra-high speed X-ray radiography

J. Tartière<sup>1</sup>, <u>M. Arrigoni<sup>1,a</sup></u>, B. Lukic<sup>2</sup>, A. Rack<sup>2</sup>, D. Chapman<sup>3</sup>, P. Pradel<sup>4</sup>, T. de Rességuier<sup>5</sup>, P. Forquin<sup>6</sup>, D. Eakins<sup>3</sup>

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Autoclaved Aerated Concrete (AAC) is a structural material under widespread use due to its attractive low carbon footprint. Through its ability to be compacted, it is able to mitigate shock waves and their mechanical effects. In addition, it is at the same time nonflammable, which is of interest for most structures that must resist both impacts and fire. AAC is therefore a good material for the elaboration of sacrificial coatings in protective design.

However, its mechanical behaviour under impacts or shock loading, especially during the compaction phase, has not been extensively studied, which limits its use in dynamic conditions.

With the aim of a better understanding of its compaction process in the dynamic regime, an experimental campaign was carried out at the European Synchrotron Radiation Facility (ESRF), where two variants of AAC, Siporex<sup>©</sup> (density of 550 kg/m<sup>3</sup>) and Multipor<sup>©</sup> (density of 115 kg/m<sup>3</sup>) were subjected to plate impact loading. Using the single-stage gasgun at the ESRF, three impact velocities ranging from 250 to 400 m/s were achieved and repeated for each material. The dynamic compaction process of AAC could be observed *in-situ* through ultra-fast X-ray phase-contrast radioscopy.

Through the analysis of the acquired X-ray radiographs, a compaction front in the AAC was identified. Its velocity as a function of time, as well as those of the projectile and the buffer, were extracted. Based on shock physics principles, the tracking of the compaction front and the initial velocity of the projectile provide experimental data for the establishment of an analytical model able to reproduce the compaction front dynamics, considering mass-spring systems.

#### Probing the evolution of solid microjets from grooved Sn samples and their impact on Asay foil and piezoelectric mass diagnostics using X-ray radiography

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When a shock wave emerges at a metal free surface presenting geometrical defects such as pits, scratches, or grooves, "ejected matter" (ejecta) can be emitted from these defects in the form of thin jets expanding ahead of the main surface and breaking up into small particles. This process, which is referred to as microjetting, is a major safety issue in ICF and shock-related work where it can disrupt measurements [1], damage nearby equipment [2] or even present safety hazards [3]. It is therefore particularly important to determine the characteristics of the cloud of ejecta. Whereas it is quite straightforward to determine the velocity of the ejecta through laser Doppler velocimetry, the measurement of the ejected mass is more tricky. The latter is generally measured using momentum-based techniques such as piezoelectric probes [4-6] or Asay foils [1,7,8], the analysis of which is based on several unproven assumptions [9,10].

The objective of the current study was to test these assumtions, emphasizing the effect of initial surface geometry on jet mass, but also on jet morphology and velocity. To accomplish this, synchrotron-based radiography was used to image the evolution of solid microjets from grooved Sn samples and their impact on piezoelectric and Asay foils mass diagnostics. Several groove geometries (chevron, fly-cut and square profiles) have been used in order to tune the ejected mass and the impact conditions on the mass diagnostics. The preliminary results obtained in the frame of this shock BAG experiment, open very interesting opportunities for characterizing the ejection process under high pressure shock loading.

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### High Power Nanosecond Laser for Dynamic Shock Compression and roadmap to kJ level at high repetition rate

S. Branly, F. Mollica, F. Falcoz, A. Golinelli, P. Leroy, O. Zabiolle, P-M. Paul

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Key words: Pulse shaped nanosecond laser, Dynamic Shock Compression, High Average Power, High Peak Power

We report here the latest achievements in the development of high energy high repetition rate nanosecond lasers along with our road map aimed at ramping up the average power which is increasingly requested by the users.

Amplitude has developed few years ago a high-power nanosecond amplifier based on Pseudo Active Mirror Disk Amplifier Module (PAMDAM) technology. This technology allows to increase significantly the repetition rate and as a consequence the average power of high energy nanosecond laser. This technology has been developed in both ND:YAG (Premiumlite-YAG) and ND:Glass (Premiumlite-Glass) versions. The Premiumlite-Glass laser is capable of delivering up to 250J at 0.1Hz at 1053nm in a single beam and a single pulse. Thanks to a specific front-end, the pulsewidth of this laser can vary from a few ns to 15ns with a high resolution (125 ps) programmable pulse shape that is suitable for Laser Driven Dynamic Shock Compression. In this report we will present some of the lasers that have already been developed, manufactured, and delivered (European Synchrotron Radiation Facility, Los Alamos National laboratory) and the perspective of scaling the above-mentioned technology to kJ-class laser through THRILL project [1] using PAMDAM technology with higher diameter disks.

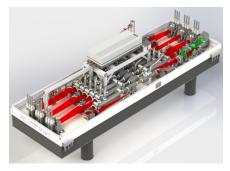


Fig. 1: 3D view of the Premiumlite

#### References

[1] https://www.thrill-project.eu/

#### X-ray absorption spectroscopy of dynamically compressed matter

<u>A Krygier</u><sup>1</sup>, H Sio<sup>1</sup>, S Ali1, A Coleman<sup>1</sup>, F Coppari<sup>1</sup>, J Eggert<sup>1</sup>, J-A Hernandez<sup>2</sup>, P Hesselbach<sup>3</sup>, C McGuire<sup>1</sup>, R Rudd, N Sévelin-Radiguet<sup>2</sup>, R Torchio<sup>2</sup>, C Vennari<sup>1</sup>, R Torcchio<sup>2</sup>, Y Ping<sup>1</sup>

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Dynamic compression is now a widespread technique for investigating material properties at extraordinary pressure, density, and temperature. However, there is a nearly complete lack of temperature measurements across the full scope of this field, leaving thermal effects as a large source of uncertainty. Extended X-ray Absorption Fine Structure (EXAFS) is a powerful diagnostic for characterizing material properties. EXAFS is particularly sensitive to density, temperature, and crystal structure in the range 100s-10000 K, where most materials form a solid at high pressure. Here we present results of experiments at the European Synchrotron Radiation Facility, the National Ignition Facility (NIF), and the Dynamic Compression Sector at the Advanced Photon Source that measured EXAFS from both copper and tantalum compressed to multi-Mbar pressures along both shock and ramp compression paths. We discuss these results in the context of predicted thermal states, thermal diffusion on nanosecond timescale, detailed strength models, and design of future experiments.

#### Ultrafast X-ray Absorption Spectroscopy on Fe and Cu up to shock melting collected at HPLF (ESRF)

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Laser-driven shock compression coupled to brilliant X-rays probes opens new research opportunities in the field of matter at extreme conditions allowing to answer questions relevant for fundamental and planetary science. At beamline ID24 at ESRF (Grenoble, France) a High-Power laser has been coupled to time-resolved X-ray Absorption Spectroscopy (XAS)[1]. XAS allows to reveal the local ionic and electronic structure even in complex states such as the Warm Dense Matter (WDM). I will present here the XAS data on laser-driven shock compressed iron and copper up to shock melting along the Hugoniot curve (around 300 GPa and 6000K). Analysis and modelling of our data allow to address open questions in the phase diagram of these metals up to such extreme conditions, in particular providing Hugoniot temperatures, phase stability under dynamic conditions, and electronic structure up to the dense liquid state.

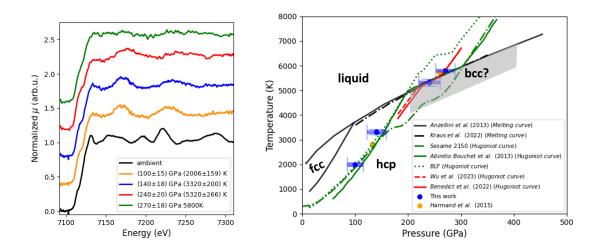


Figure 1: Shocked Fe K-edge XAS spectra as a function of pressure and temperature (left). Iron phase diagram including our data (right).

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#### Stability of the fcc phase in shocked nickel up to 325 GPa

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We have investigated elemental bulk nickel at high shock pressures and temperatures using in situ x-ray diffraction and the velocimetry technique, VISAR, with the goal of bracketing the onset of melt under shock conditions. We observe a solid compressed fcc-Ni phase up to pressures as high as 325 GPa. In the range of approximately 300–375 GPa, we see evidence for the onset of melt, with full melt achieved above 500 GPa. The experiments were conducted at the Matter in Extreme Conditions endstation at the SLAC National Accelerator Laboratory using a flat-top laser drive. Experimental results were supported by 1D hydrodynamic simulations, providing particle velocity, transit time, pressure, and density data that agree well with experiment.

Our work presents the first in situ x-ray diffraction data on shock-compressed nickel up to  $\sim$ 500 GPa and is particularly relevant to computational and experimental researchers studying nickel and other dense metals. The pressures we reached are some of the highest ever reported for shocked nickel, and our identification of a solid compressed phase up to 325 GPa is significantly higher than expected by the majority of melt lines that have been proposed for nickel in the literature.

#### Femtosecond structural probing of warm dense matter with Betatron laser-based X-ray source

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a

Exploring and understanding ultrafast processes at the atomic level is a scientific challenge. Femtosecond X-ray Absorption Near-Edge Spectroscopy (XANES) arises as an essential experimental probing method, as it can simultaneously reveal both electronic and atomic structures, and thus potentially unravel their non-equilibrium dynamic interplay which is at the origin of most of the ultrafast mechanisms. The key point of this investigation is the achievement of a femtosecond X-ray source suitable for routine experiments. The presentation will show the progressive development and improvement of such laser-plasma-based X-ray sources, starting from the picosecond [1,2] down to the femtosecond scale [3]. Time-resolved XANES measurements have been achieved and interpreted using *ab initio* quantum molecular dynamic simulations [4]. This diagnostic was used to shed new light on the non-equilibrium physics involved in various materials, such as local disordering dynamics in warm dense aluminum [5], electronic structure modification in warm dense molybdenum [6] and electron-ion thermal equilibration dynamics in warm dense copper [7]. The current work is focused on the investigation of the phase transition dynamics in non-equilibrium situations [8] and the ultrafast electronic transport in warm dense copper [9].

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### A pulsed power facility for studying the warm dense matter regime.

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Over the past decades, notable efforts have been devoted to study the thermodynamic and transport properties of metals in the warm dense matter regime. Isochore measurements were performed on the *Enceinte à Plasma Isochore* facility (EPI) located at CEA DAM Île-de-France, giving access to electrical conductivity and internal energy of homogeneous plasmas up to 1-2 GPa for density ratios such as  $1/30 \le \rho/\rho_0 \le 1/10$ , where  $\rho_0$  is the standard density [1-4]. Experimental data collected with the EPI facility enabled the validation of theoretical properties of various metals calculated with quantum molecular dynamics simulations [5,6].

To explore further the properties of plasmas at higher pressures  $(2 \le P \le 5 \text{ GPa})$  and density states  $(1/3 \le \rho/\rho_0 \le \rho_0)$ , we have developed a new facility called *Enceinte à Plasma Pulsé* (EPP). The EPP is based on the pulse Joule heating technique [7,8], where micrometer thin metallic foils confined between two sapphire plates are heated by a microsecond electrical discharge. *In situ* measurements provide access to electrical conductivity and thermodynamics properties such as dissipated internal energy variation, pressure and density. In this talk we will present the experimental setup of the EPP facility, the implemented diagnostics, and preliminary experiments conducted on aluminum samples.

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#### Exploring the deep interior of ice giant planets with density functional theory and shock-compression experiments

#### Mandy Bethkenhagen

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About two thirds of known exoplanets are classified as super-Earths and mini-Neptunes due to their similar size compared to their Solar System cousins. To date, it remains unclear if similar-sized exoplanets share a complementary composition of their interiors. Insights from atomic scale simulations are therefore key in moving forward. This presentation provides an overview of the recent advances made in the study of ice-rich giant planets such as Uranus and Neptune by investigating high-pressure phases of water, ammonia, and carbon hydrates. Accurate equation of state data, phase diagrams, and transport properties such as electrical conductivity are obtained by combining density functional theory, machine learning potentials and insights from high-pressure experiments, which can provide useful insights for planetary modeling.

### Combining ultrafast X-ray spectroscopy and diffraction to investigate deep planetary interior conditions.

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The advent of high-brilliance X-ray sources has transformed the field of dynamic compression, allowing to deploy a variety of *in situ* characterization techniques and to gain a detailed understanding of matter's behaviour when subjected to extreme conditions and high strain-rates. Here, we present results from recent experiments that used the Linac Coherent Light Source XFEL to perform a simultaneous structural (X-ray Diffraction, XRD) and spectroscopic (X-ray Emission Spectroscopy, XES) characterization of shock-compressed silicates up to and beyond melting at multi-Mbar pressures [1-2]. Such detailed studies of liquid silicates are necessary to understand our planet's formation, as well as to model planetary evolution. Indeed, the early stages of the Earth took place in the liquid state (the so-called "magma ocean" stage), and they have set the chemical composition of the Ferich core and the silicates mantle.

At the LCLS, the Matter in Extreme Conditions long-pulse laser allows us to compress and heat the samples (*e.g.*, Grossular-Andradite, glassy MgSiO<sub>3</sub>) to extreme pressure and temperature conditions, which are particularly challenging to reach using conventional, static-compression techniques [3]. Furthermore, the use of ultrafast, dynamic compression ensures that the molten samples maintain their initial composition, as it prevents chemical migration, which generally affects Laser-Heating Diamond Anvil Cell experiments. A single XFEL pulse allows us to collect high-quality XRD and XES data *in situ*, monitoring simultaneously the evolution of the structure at the atomic level and the spin and oxidation state of the Fe present in the samples. With this unique setup, we can study the correlation between the atomic and electronic structure of silicates as they undergo compression and melting.

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#### Time-resolved X-ray absorption spectroscopy of shock-loaded magnesiosiderite: possible implications for the origin of magnetite in Martian meteorite ALH84001

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Carbonates are widespread in the Earth's geological processes. They play a crucial role in our planet's carbon cycle, serving as significant reservoirs of carbon, and are also present in a variety of environments on the surface of the Earth. In recent decades, carbonates have also been identified in various extra-terrestrial locations, including Mars [1] and the Ryugu asteroid [2], which are targets of impacts. Carbonates were also identified in the Martian meteorite ALH84001, where the shock impact-driven decomposition of iron carbonate (siderite) was proposed to be behind the origin of magnetite (Fe<sub>3</sub>O<sub>4</sub>) nanocrystals [3].

Two previous studies on natural siderite have demonstrated the shock-induced decomposition of siderite into iron oxides and carbon-bearing species [4,5]. Magnetite or magnetite-like phases in these studies were detected in the post-shock recovery samples using Raman spectroscopy and transmission electron microscopy (TEM). However, these measurements don't give clues about the timing or mechanisms of this transformation. To determine *in-situ* the mechanism and shock conditions of this transformation, we have performed time-resolved X-ray absorption spectroscopy (XAS) during laser shock compression on natural (magnesio)-siderite samples.

In this presentation, we will look at the X-ray absorption spectroscopy and velocity interferometry results of natural magnesio-siderite crystals shocked up to 200 GPa. Under shock compression conditions, with an increase in pressure, the magnesio-siderite crystals go from a high-spin state to a low-spin state, before supposedly reaching a partially molten state at the highest pressure (and temperature) conditions, consistent with the observations in static high-pressure experiments [6]. Under shock release conditions, i.e., at times when the sample is probed after the shock breakout, the sample recrystallizes back to the starting magnesio-siderite, with a slight decomposition to iron oxide. This presentation will discuss the different possible pathways for this transformation, in the context of the longstanding debate about the origin of magnetite in the Martian meteorite ALH84001.

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#### Phase transitions and electronic properties of Fe2O3 under laser compression by

ultrafast in-situ X-ray absorption spectroscopy

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Understanding the structural changes of hematite ( $\alpha$ -Fe2O3) under extreme conditions of pressure and temperature is crucial for gaining insights into the physical properties of planetary interiors such as Earth and super-Earths (2 to 10 times more massive). At ambient conditions, hematite is a rhombohedral structured antiferromagnetic insulator [1,2]. Its high-pressure behaviour has been largely studied over the past decades using static compression [1,3,7]. Several phase transitions were reported but particular attention was given to the structural, electronic and magnetic changes occurring ~50 GPa: a 10% volume cell drop accompanied by a change of crystal symmetry corresponding to a distorted perovskite ( $\zeta$ -Fe2O3) [3], a Mott insulator-metal transition and the collapse of the iron magnetic moments corresponding to the high-spin (HS) to low-spin (LS) electronic phase transition [3-7]. However the exact nature of the phase transition in this pressure area remains controversial [4,6,7]. The question of which transition drives the other one, electronic or structural, is still under debate.

Here, we report ultrafast time-resolved X-ray Absorption Near Edge Spectroscopy (XANES) measurements obtained on laser-compressed Fe<sub>2</sub>O<sub>3</sub> at the High Power Laser Facility (HPLF) of ESRF-ID24 beamline [8]. Our XANES data provide information on time-resolved structural transformations by showing changes in the pre-edge, the white line and the 1<sub>st</sub> Extended X-Ray Absorption Fine Structure (EXAFS) oscillation within hundreds of ps after the shock breakout from the samples. More severe spectral changes are observed at longer delays between the X-ray probe and the shock, during its thermodynamic release. We will present a detailed time-resolved study of the XANES changes as a function of pressure and temperature, along the Fe<sub>2</sub>O<sub>3</sub> Hugoniot thermodynamic path and release. For further understanding of the XANES features, preliminary FDMNES [9] and Quantum Espresso [10] ab-initio calculations will also be presented.

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#### The HED instrument at European XFEL: Unique capabilities to study material properties of laser-compressed matter

#### Erik Brambrink for the HED-HIBEF instrument

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The HED instrument at the European XFEL offers unique capabilities to study extreme states of matter, delivering X-ray bursts with up to 2 mJ of pulse energy, fs pulse duration and photon energies up to 25 keV. This allows in-situ studies of highly transient states using various x-ray diagnostics such as diffraction, scattering and imaging [1].

Amongst other drivers, HED provides a 400 TW/25 fs short pulse laser RELAX [2] and a 100J/ns laser DIPOLE-100X, both capable running at 10 Hz enabling high repetition rate experiments for experiments requiring signal integration due to low scattering cross sections.

We will present the status of the instrument with a particular focus on the commissioning and first user experiments with the DIPOLE-100X laser.

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#### First results obtained using the DiPOLE laser at the European XFEL

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In May 2023, after a long delay because of the COVID pandemic, the first laser compression science was conducted on the HED beamline at the European XFEL using the DiPOLE high rep-rate laser. After the commissioning of DiPOLE, which took place only two weeks before the main experiment, a large international user consortium of over 80 researchers collected the first diffraction data from a number of different dynamically compressed materials. The science focussed on iron and iron alloys at planetary core conditions, carbon polymorphs at extreme P-T and the transition to diamond, and studies of how materials "flow" and transform under dynamic loading. There was also a strong technical development aspect in the research, focussing on optimising the laser drive and VISAR, target development, and 10 Hz operation. The beamtime was a great success, perhaps more successful than the consortium members had dared to hope, and demonstrated that a community proposal was the <u>ideal</u> way to conduct the first experiment using DiPOLE. In this talk I will give some background to DiPOLE and the planning for the experiment, as well as show some of the first results from the different materials studied.

#### Towards the direct measurement of bulk temperature in shockcompressed matter using inelastic X-ray scattering at an XFEL

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Direct and accurate measurements of thermodynamic and transport properties are essential for understanding the behavior of matter at extreme pressures and temperatures. While X-ray diffraction measurements have allowed in situ measurement of structure and density [1], the direct measurement of bulk temperature remains a challenge. In shock compression experiments, it is often estimated from hydrodynamic simulations or inferred using streaked optical pyrometry, which requires a priori knowledge of the material properties at extreme conditions. On the time scale of nanosecond shock compression, for temperatures less than 4000 K, the intensity recorded in SOP experiments decreases and the accuracy of the technique degrades. This limitation is particularly hindering for the investigation of high-pressure, moderate temperature states of matter such as those generated using double shock or quasi-isentropic compression. Furthermore, due to the small penetration depth of optical photons in solid density materials, this technique only gives access to the surface temperature unknown.

Here, I will describe experiments conducted at the High Energy Density instrument at the European XFEL and the Matter in Extreme Conditions at the LCLS using high-resolution inelastic X-ray scattering with milli-electronvolt to measure temperature [2]. With the installation of high repetition rate drive lasers at hard X-ray free electron lasers [3], this technique has the potential to become a powerful to investigate temperature as well as transport of shock-compressed matter.

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#### Single pulse extended x-ray absorption fine structure capability at the Dynamic Compression Sector

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Determining real-time changes in the local atomic order of dynamically compressed materials is an important need in condensed matter and materials science. However, x-ray measurements capable of probing this length scale – such as extended x-ray absorption fine structure (EXAFS) – are difficult due to the single event and short duration (~ 10 ns) of these experiments. Here we describe our single pulse, transmission geometry EXAFS capability at the Dynamic Compression Sector (DCS), located at the Advanced Photon Source (APS) for laser-driven compression experiments [1]. We use a flat plate of highly oriented pyrolytic graphite (HOPG) as the spectrometer element to energy disperse x-rays transmitted through the sample. This approach allows us to perform EXAFS measurements between 9 and 13 keV with EXAFS spectra covering 100s of eV and an energy resolution of ~ 10 eV (at 10 keV). Excellent pulse-to-pulse reproducibility and a good signal-to-noise ratio have been demonstrated. Recent EXAFS measurements to determine temperatures on shock-compressed Pt between 72 and 325 GPa will also be presented [2].

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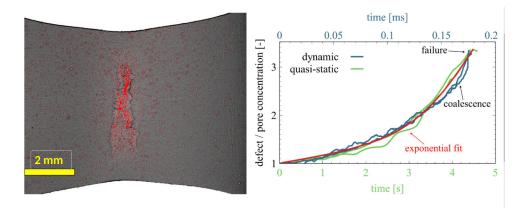
#### Ductile failure in metals: How does void growth rate depend on loading rate?

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Damage and failure in ductile metals is governed by the growth of internal voids under tension, ultimately leading to the coalescence of individual voids to form a macroscopic crack. Resolving this complex mechanism in-situ, i.e., during a dynamic loading event, is a challenge. Observing how microvoids grow and coalesce is inherently elusive, as the process occurs beyond the surface of non-transparent materials. However, understanding of this process holds paramount significance to accurately model metal behaviour under extreme loading conditions.

This talk reports on early work performed at ID19 [1]. In this research, both slow and fast tensile loading methods were employed to strain and fracture specimens additively manufactured from a Scandium-Aluminium alloy. Fast experiments were performed using a Split Hopkinson Tension Bar [2] at a strain rate of 1000 /s. Imaging was performed using Shimadzu HPV-X2 cameras with a spatially overlapping optical relay. Experiments at a strain rate of  $10^{-2}$  /s were performed using a screw-driven universal testing machine and a Photron SAZ camera. In both slow and fast cases, it was possible to identify bright defects which are likely voids using a threshold/clustering algorithm. The total void volume grows exponentially as a function of the accumulated plastic strain. This suggests that it is possible to determine the relation between growth rate as and strain rate and measure a hitherto inaccessible parameter required for predictive modelling of ductile failure.



<u>Figure 1</u>: Results obtained at ID19: left image shows one fast phase-contrast radiograph, analysed using a specific classification algorithm. Right image shows the total void volume at low and high velocity loading.

#### References

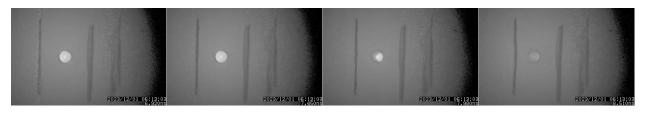
 P. Jakkula, A. Cohen, B. Lukić, D. Levi-Hevroni, A. Rack, G. Ganzenmüller, and S. Hiermaier. In *Proceedings of the 26th DYMAT Technical Meeting*, pages 87–92. doi:10.6094/UNIFR/228460.
 P. Jakkula, A. Cohen, B. Lukić, D. Levi-Hevroni, A. Rack, G. Ganzenmüller, and S. Hiermaier. *Instruments*, 6(3):38, 2022. doi:10.3390/instruments6030038.

#### Dynamic Shear Localization and Vortical Flow during Shock Wave Induced Pore Collapse in Sucrose

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Pore collapse under shock wave loading is one of the primary mechanisms of hotspot formation, which leads to ignition and shock to detonation transition (SDT) in energetic materials. In this study, X-Ray phase contrast imaging (XPCI) is employed to observe cylindrical pore collapse in sucrose (an energetic material simulant) when subjected to shock loading. Experiments were conducted at the European Synchrotron Radiation Facility (ESRF), France at the ID19 beamline. An imaging system consisting of three high speed cameras was used to record the subsurface time evolution of several phenomena, including cylindrical pore collapse, jetting, vortex generation and crack formation. A single-stage gas gun was used to generate shock pressures between 0.5 GPa and 6 GPa in the sucrose samples. Shock hugoniots of sucrose was extracted from digital image correlation (DIC) analysis of the X-ray phase contrast images. In the case of vortex structures, simulations indicate that the post-collapse flow is influenced by shear strength, even though the collapse itself is predominantly hydrodynamic. This allows strength to be inferred at high pressures. The key to the analysis technique is that the velocity gradient decays with both time and radial distance, meaning that strength dominated volumes will exist even for high-pressure shots. The result is something analogous to a tamped Richtmyer-Meshkov experiment, but without the undesirable convolution of tamper and sample strength. Since these phenomena are controlled by the dynamic strength of the material, they offer a promising approach to indirectly infer it. Secondary shocks were also observed due to the jet impingement of the upstream pore wall on the downstream surface as observed in the time-series below:



<u>Figure 1:</u> 28mm x 28mm sucrose block with a 1 mm diameter cylindrical hole is impacted at a velocity of 570 m/s from the left. This resulted in hole collapse with jet impingement on the downstream surface of the hole. The three vertical lines are gold thin films (10 um thickness) which are used to track the particle velocity of the sucrose after impact. One can observe cylindrical shock in the 4<sup>th</sup> image (on the extreme right) with its center on the downstream pore wall. This experimental shot was conducted at ID19 in ESRF, France.

#### A new approach for shockwave experiments in arbitrary geometries: pulsed power driven exploding wires and foils

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Recent advances in high energy density science and machine learning optimization have led to increased interest in the design of shock compression experiments in increasingly complex geometries. Examples include developing optimized geometries to suppress growth of the Richtmyer-Meshkov instability (RMI) [1], enhance jetting to produce fast material outflows [2], and increase pressures and temperatures for inertial confinement fusion [3]. These designs can be produced by training a neural network on a set of hydrodynamics simulations to optimize a selected fitness parameter, but the resulting geometries are often unintuitive. Simultaneously, the results may be strongly dependent on material models (strength, failure, and equation-of-state) and numerical implementation of the code. As such, it is crucial to develop quantitative experimental capabilities to verify the physics of these complex designs.

The pulsed power driven electrical explosion of wires and foils is a novel method to perform shock-driven hydrodynamic experiments in arbitrary geometries at facilities such as synchrotrons and XFELs [4]. It offers multiple advantages: the shockwave (Mach ~ 1-3) can be shaped to the requirements of the experiment; the pressure (P > 300 MPa) generated by the explosion enables the use of liquid and solid targets in hydrodynamic regime; the use of multi-MHz synchrotron radiography enables highly resolved data acquisition within a single experiment, eliminating uncertainties regarding repeatability of initial conditions; and it is possible to directly estimate compression ratios from X-ray attenuation.

In this talk, I will present the pulsed-power hydrodynamics platform utilised in a series of campaigns at the ID19 Microtomography beamline of the European Synchrotron. I will discuss several of its applications including measurements of convergent RMI, verification of optimized designs for the suppression of planar RMI via impedance defects, and other experiments that highlight the capabilities of this approach. The platform will shortly be available as part of the Shock Beamtime Allocation Group for external users.

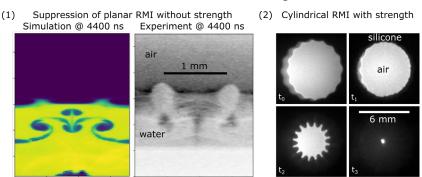


Figure 1: Two example experiments performed by the pulsed-power platform at the European Synchrotron. References

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### Measuring material strength in laser compressed Ta via *in situ* X-ray diffraction at the Dynamic Compression Sector

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We present X-ray diffraction measurements of shock and ramp compressed tantalum performed at the Dynamic Compression Sector (DCS). By using a 'tilted target' geometry, we can find the sample shear strain by observing deviations in the Debye-Scherrer line position as a function of azimuthal angle [1]. This can be related to strength via the shear modulus, allowing for comparison with other high strain rate-strength experiments, such as Rayleigh-Taylor instability [2] and planar ramp release [2]. Additionally, we study the effects of alloying, by measuring how the strength in Ta-W alloy changes with increasing W concentration.

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#### Phase transitions in amorphous materials under shock compression

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Amorphous or glassy materials are kinetically trapped metastable structures, essentially "froz en liquids" with no long-range order. Their structural evolution at high pressures (P) and temperatures (T) through crystallization, polyamorphic transitions, and melting is currently a topic of significant fundamental interest and practical importance. For example, amorphou s carbon (a-C) is being explored as a promising ablator fuel capsule for inertial fusion energy application, which promises to overcome the limitations of diamond - currently used ablator material - by significantly lowering the fusion fuel's adiabat.

By combining the computational power of exascale supercomputers and quantum-accurate de scription of billions atoms with machine-learning interatomic potentials we uncover novel stabl e and metastable states generated through dynamic loading of amorphous precursors. By obs erving the pressure-induced evolution of atomic order we gain fundamental understanding of kinetics, metastability, phase transitions, and liquid structure at extreme conditions generated by shock compression.

Our cutting-edge molecular dynamics simulations guide DiPOLE shock compression experiments at European X-Ray Free Electron Laser (EuXFEL) facility to observe predicted phenomena and as sist in atomic-scale analysis of the results.

#### Macroscopic and microscopic study of Glassy GeO<sub>2</sub>

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Germanium dioxide is an important material for technology and material science as well as for geophysics and planetary science. It can exist in three polymorphic phases at ambient conditions: quartz, rutile and glass. Generally, GeO<sub>2</sub> is regarded as a chemical and structural analog of SiO<sub>2</sub>, in particular, the structure of glassy GeO<sub>2</sub> is a good model for amorphous or molten SiO<sub>2</sub> [1]. All GeO<sub>2</sub> phases have a larger sensitivity to pressure, undergoing pressureinduced changes at much lower pressures than their SiO<sub>2</sub> analogues. However, its phase diagram is poorly studied, especially above Mbar range. Here, we will present an exhaustive macroscopic and microscopic study of laser compressed glassy GeO<sub>2</sub> in the range of pressures up to TPa range. Experiments have been performed at LULI laboratory where equation of state (see figure 1), optical properties and phase transitions using X-ray diffraction [2] along the Hugoniot have been investigated. More recently an experiment on HPLF at ESRF allowed us to complete investigations adding the electronic structural changes and local ionic order measurements using XANES diagnostic.

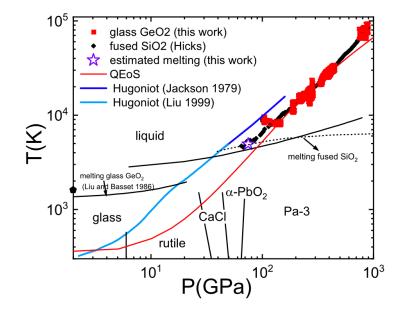


Figure 1: Phase diagram of glass GeO<sub>2</sub> from this work and previous literature (static and dynamic data).

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### Dynamic behaviour of zirconium-based metallic glasses under laser shock compression

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The continuous increase of small sizes space debris motivates the study of innovative materials behaviour under shock compression to reinforce the actual space structure shields. Previous studies have highlighted the potential of Zirconium-based metallic glasses as shielding components with hypervelocity impact experiments on a Whipple shield configuration (ISS configuration).

This work is focused on the dynamic behaviour of ZrCuAl metallic glasses (or amorphous metallic alloys). Instead of experiments on gas-guns, high-power laser pulse have been chosen as shock generator in order to reach strain rates levels representative of hypervelocity impacts of space debris.

Experimental campaigns conducted on high power laser facilities at the Laboratoire pour l'Utilisation des lasers Intenses and CEA DAM DIF enabled us to: complete the Hugoniot curves for bulk metallic glasses and ribbons metallic glasses from 5 to 100 GPa; highlight an evolution of the spall strength with the strain rate reaching 13.6 GPa i.e. almost 7 times the quasi-static value [1]; observe crystallisation of Zr50Cu40Al10 alloy by the mean of XRD measurements under shock compression in the 50-80 GPa pressure range.

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#### Studies of Dynamically Compressed Methane-Hydrogen with Raman Spectroscopy

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Hydrogen (H<sub>2</sub>) and methane (CH<sub>4</sub>), as the simplest molecule and the simplest hydrocarbon, respectively, hold paramount significance in the domain of condensed simple-molecular matter. Their mixtures play significant roles in planetary physics and they are the amongst the most prevalent molecules in giant planets. Above 10 GPa, H<sub>2</sub> and CH<sub>4</sub> molecules form several different Van Der Waals Compounds: CH4(H2)2, (CH4)2H2, CH4H2, CH4(H2)4, (CH4)3(H2)25 [1,2]. It is known that the phase diagram of methane is strongly controlled by kinetic effects, as if is continuously compressed it stays at a ahombohedral phase A until 240 GPa or cubic phase by going through extremely sluggish transition from phase A at around 10 GPa [3,4].

Furthermore, the formation of methane hydrates (MH) and the structural evolution are also shown compression rate dependent [5]. However, though the importance of kinetics in determining CH<sub>4</sub> phase diagram is known well, the role of compression rate in the formation of CH<sub>4</sub>-H<sub>2</sub> compounds has not explored in the previous works.

We have conducted dynamic compression experiments on mixtures of methane (CH4) and hydrogen (H2) at different concentrations in gas membrane-driven dynamic Diamond Anvil Cells (dDAC) with compression rates various from 0.1 GPa/s to 500 GPa/s[7], coupled with Time Resolved Raman Spectroscopy (TTRS). Our research reveals that the formation of methane-hydrogen Van Der Waals compounds is compression rate dependent. Moreover, their crystal growth and morphologies could be systematically controlled by the compression rate.

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