

### FLUID DYNAMICS MEETS SYNCHROTRON X-RAY HIGH-SPEED IMAGING

ESRF, Grenoble, France

# 21 - 22 MARCH 2024

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### Fluid dynamics meets synchrotron X-ray high-speed imaging

ESRF Auditorium - Grenoble - France



21 - 22 March 2024



| THURSDAY 21 MARCH   |   |   |  |  |  |
|---|---|---|--|--|--|
| 08:15 - 09:00   | Registration and Welcome coffee   | ESRF Central Building Entrance Hall                                       |  |  |  |
| SESSION 1   |   |   |  |  |  |
| 09:00-09:30   | Welcome and Introduction to EBS ESRF  | Veijo Honkimaki<br>ESRF   |  |  |  |
| 09:30-10:20   | Invited Talk - To see what cannot be seen   | Michel Versluis<br>University of Twente                                   |  |  |  |
| COFFEE BREAK  |   |   |  |  |  |
| 10:35 – 11:25   | Invited Talk: Application of x-ray imaging to the characterization of high-speed cavitating flows                       | <b>Olivier Coutier-Delgosha</b><br>Virginia Tech                          |  |  |  |
| 11:25 – 11:45   | Poster pitch 1  |   |  |  |  |
| LUNCH BREAK   |   |   |  |  |  |
| SESSION 2   |   |   |  |  |  |
| 13:15 – 14:05   | Invited Talk: Concentration Measurements in Dense Particle Clouds Induced by<br>Jet Impingement on a Granular Substrate | <b>Laura Villafañe Roca</b><br>University of Illinois at Urbana-Champaign |  |  |  |
|   | Contributed talks on Cavitation and Shock dynamics<br>Chair: Outi Supponen  |   |  |  |  |
| 14:10 - 14:25   | Pulsed power driven exploding wires: a new approach for shockwave experiments in arbitrary geometries                   | Jergus Strucka<br>Imperial College London                                 |  |  |  |
| 14:25 - 14:40   | Underwater shock waves interacting with antibubbles serving as liquid payload carriers                                  | Guillaume Bokman<br>ETH Zurich  |  |  |  |
| 14:40 - 14:55   | X-ray visualization of a shock-wave-induced cavitation cloud inside liquid perfluorohexane droplets                     | Samuele Fiorini<br>ETH Zurich   |  |  |  |
| 14:55 – 15:10   | Unravelling bubbles formation and bubbles dynamics in pulsed laser ablation in liquids                                  | <b>Vito Coviello</b><br>Università di Padova                              |  |  |  |
| 14:10 – 15:25   | Flow instabilities in cavitating microchannels  | <b>Matevz Dular</b><br>University of Ljubljana                            |  |  |  |
| COFFEE BREAK  |   |   |  |  |  |
| SESSION 3: Contributed talks on Breakup, Coalescence and Jetting processes<br>Chair: Nathanaël Machicoane |   |   |  |  |  |
| 15:40 - 15:55   | Shape mode-induced repeated jetting of ultrasound-driven microbubbles   | Marco Cattaneo<br>ETH Zurich  |  |  |  |
| 15:55 – 16:10   | Synchrotron x-ray phase-contrast imaging of ultrasonic drop atomization   | Anunay Prasanna<br>ETH Zurich   |  |  |  |

| 16:10 – 16:25   | High-speed synchrotron x-ray analysis of non-contact jetting process  | Jesper Sallander<br>Mycronic AB           |  |  |  |
|-----------------|---|---|--|--|--|
| 16:25 – 16:40   | High-speed spray formation probed by Synchrotron X-ray  | Santanu Kumar Sahoo<br>CNRS               |  |  |  |
| 16:40 – 16:55   | Poster pitch 2  |   |  |  |  |
| 16:55 — 18:55   | ESRF tours and Beamline visit at ID19 (2 X 50 min)  |   |  |  |  |
| 19:00-21:00     | Poster session (poster prize) + cheese and wine evening event   | ESRF Central Building Entrance Hall       |  |  |  |
| FRIDAY 22 MARCH |   |   |  |  |  |
| 08:45-09:00     | Casual coffee and discussion on ESRF access   |   |  |  |  |
| 09:00 - 09:50   | Invited Talk: Multiphase flows from swarming bubbles to schooling fish: from the advancement of optic measurements to data assimilation | <b>Rui Ni</b><br>Johns Hopkins University |  |  |  |
|                 | SESSION 3: Contributed talks on Instrumentation and Techniques<br>Chair: Alexander Rack   |   |  |  |  |
| 09:50 - 10:05   | Multiphase Flow Studies at the APS 7 BM Reamline  | Alan Kastengren                           |  |  |  |

| 10:05 – 10:20  | Advances X-Ray Techniques for Fluid Dynamics at Lab and Synchrotron Scale   | Simo Mäkiharju<br>University of California Berkley                          |  |  |  |
|--|---|---|--|--|--|
| 10:20 - 10:35  | X-ray multi-projection imaging: enabling fast volumetric information for fluid dynamics                                       | Pablo Villanueva-Perez<br>Lund University                                   |  |  |  |
| COFFEE BREAK   |   |   |  |  |  |
| SESSION 4: Contributed talks on Flow in porous media and Granular system<br>Chair: Marie-Jean Thoraval |   |   |  |  |  |
| 10:50 – 11:05  | 4D imaging of Haines jumps with sub-millisecond temporal resolution   | <b>Dag Werner Breiby</b><br>Norwegian University of Science &<br>Technology |  |  |  |
| 11:05 - 11:20  | 4D X-ray velocimetry of multiphase flows in porous media  | Tom Bultreys<br>Ghent University  |  |  |  |
| 11:20 – 11:35  | Dynamics of Binary Granular Materials: Unraveling the Phenomena of Rising Granular Bubbles<br>and Splitting Granular Droplets | Jens Metzger<br>ETH Zurich  |  |  |  |
| 11:35 – 11:50  | X-ray analysis of air bubble rise in granular suspension: Application to concrete de-airing                                   | <b>Bastian Strybny</b><br>Institute of Building Materials Science           |  |  |  |
| 11:50 – 12:00  | Closing remarks take-home message   |   |  |  |  |
| LUNCH BREAK  |   |   |  |  |  |





#### Invited Talk: To see what cannot be seen

#### Michel Versluis<sup>a</sup>

We heavily rely on our vision for routine daily tasks, but at the forefront of science we need to resort to instruments, to see far beyond what can be seen. Our target objects are either too small, or too far, or too fast to be captured by conventional means. For that purpose, we have developed confocal and superresolution microscopes, extremely large aperture telescopes with adaptive optics and ultra-high-speed imaging camera systems equipped with ultrashort illumination. Fluid flows typically suffer from a lack of optical contrast, and we must rely on fluorescence, tracer particles or contrast agents to aid localization and tracking, e.g. for time-resolved 3D image velocimetry. Still, many phenomena of interest remain hidden with the inability of these methods to penetrate deep into complex media. For example, the human body is largely opaque to visible light, and we rely instead on magnetic resonance imaging, ultrasound and x-ray to retrieve and display anatomical, functional, and molecular information. Similarly, while imaging within microfabricated micro-electromechanical systems (MEMS) devices of the nanotechnology industry we face the same problems: microscale flow phenomena that are hindered by opacity, limited imaging depth and resolution, lack of contrast, and optical distortion. High energy x-rays have the ability to penetrate deep into dense objects, allowing the distortion-free examination of structures, reactions, and flows. Here we make use of ultrafast x-ray phase-contrast imaging to study the mechanisms underlying a bubble entrainment process within a drop-on-demand inkjet printhead that suffers from strong optical distortion<sup>1</sup>. Piezoacoustic actuation leads to oscillatory flows that can lead to strong deformations of the air-liquid interface at the nozzle exit. These deformations may lead to an inward directed air jet with bubble pinch-off and to the subsequent entrainment of an air bubble, which then destabilizes the operation of the inkjet printhead. We demonstrate good agreement between experiments and direct numerical simulations based on the volume-of-fluid method. We show the different classes of bubble pinch-off phenomena obtained in experiments, as well as those captured numerically. These results provide a fundamental understanding of the oscillating meniscus and the resulting flows inside an inkjet printhead.



**Figure:** Ultrafast x-ray phase-contrast imaging and direct numerical simulations based on the volume-of-fluid method to study the mechanisms underlying the bubble entrainment in a piezoacoustic printhead. Meniscus position during different time instants of the bubble entrainment process as observed in experiment (grayscale images (a)-(c)) and the corresponding numerical simulations (red curves (d)-(f)).

<sup>&</sup>lt;sup>a</sup> Physics of Fluids group, Faculty of Science and Technology, University of Twente, The Netherlands

<sup>&</sup>lt;sup>1</sup> Maaike Rump, Youssef Saade et al., Phys. Rev. Fluids 7, 104004 (2022).





## Invited Talk: Application of x-ray imaging to the characterization of high-speed cavitating flows

#### Olivier Coutier-Delgosha<sup>1</sup>

Ultra-fast x-ray imaging has been used in the last 20 years to characterize the structure and the dynamics of unsteady cavitating flows. The first application, in the early 2000s, was focused on the measurement of the volume fraction of gas with x-ray sources available in labs, using phase contrast imaging based on the difference of absorption between liquid and vapor. The volume fraction was obtained first globally and later locally at frequencies of a few thousands of Hertz, which enabled to resolve the large-scale flow instability called cloud cavitation. Seeding the flow with small radio-opaque particles and using the high beam intensity available in Synchrotrons has enabled a few years later to also access the velocity field: the large space resolution combined with a high contrast provide a clear visualization of all interfaces, in addition to the variations of grey levels inside the bubbles and the particles. By post-processing the images containing both the vapor bubbles and the seeding particles, the two populations could be separated. PIV algorithms applied to both populations have provided the instantaneous velocity fields of the liquid and vapor phases, which has enabled to identify a significant slip effect between the two phases. The data were obtained at a frequency of 12,000 Hz, using high speed shutters and complex synchronization between the x-ray flashes, the shutter, and the camera frames, which enabled to discuss not only timeaveraged fields, but also the instantaneous dynamic effects. The Reynolds stresses could also be obtained, which has given a significant insight into the turbulence inside attached cavities and rediscuss some approaches in physical modelling. Evidence of different mechanisms responsible for the cloud cavitation instability, such as the re-entrant jet, the condensation shock, the cloud collapse-induced pressure wave, and a Kelvin-Helmholtz instability at the cavity interface, has been observed. More recent developments have taken advantage of the higher speed cameras available on the market, which currently enable to record the data at about 300,000 Hz without sacrificing the space resolution. These capabilities enable to resolve in time the motion of the vapor bubbles and particles, opening the door to a better characterization of local phenomena such as bubble collapses and bubble / bubble interactions. The mechanisms of cavitation inception are also further investigated, by studying local effects such as the entrapment of nuclei in low momentum areas in the boundary layer, or the effects of surface munhace



**Figure:** Example of visualization of cavitation with x-ray imaging. Cavitation develops at the throat of a venturi type section; the flow goes from left to right.

<sup>&</sup>lt;sup>1</sup> Kevin T. Crofton Department of Aerospace and Ocean Engineering, Virginia Tech, USA





#### Invited Talk: Concentration Measurements in Dense Particle Clouds Induced by Jet Impingement on a Granular Substrate

#### L. Villafañe<sup>a</sup>, N. Rasmont<sup>a</sup>, G. Elliott<sup>a</sup> and J. Rovey<sup>a</sup>

The impingement of a high-speed jet on a granular surface causes dense clouds of ejecta particles that evolve as the surface near impingement gets eroded into a crater. Plume surface interactions (PSI) have farreaching implications for the safety of future manned missions to the Moon and Mars such as sandblasting of nearby infrastructure and clogging devices by lifted regolith, and compromising the lander stability on the altered surface topography. Experimental characterization of PSI phenomenology is necessary for understanding of the key erosion regimes as a function of exhaust plume, atmosphere, and surface regolith properties, and to improve PSI predictive models. The large amount of lifted particles challenge surface, flow, and ejecta measurements as ejecta clouds are optically opaque at conditions representative of planetary landings.

To circumvent the problem of excessive attenuation of waves in the visible by dense particle-fluid mixtures we use a novel millimeter-wave interferometric technique. A frequency modulated continuous-wave (FMCW) radar and a passive reflector are used to measure path-integrated particle concentrations from the phase shift the millimeter-waves experience as they propagate. A system of one single radar and multiple passive reflectors allows resolving local concentrations with the aid of tomographic reconstruction techniques. This technique can measure particle concentrations orders of magnitude larger than those at reach with conventional laser-based techniques, with high temporal resolution (10 kHz with the of-the-shelf radar used), and without the need of highly specialized or costly equipment. Results are presented of the ejecta cloud concentration evolution caused by a Mach 5 jet impinging on a bed of glass microspheres of 109 mean diameter, for different jet expansion ratios and nozzle to surface distances. Concentration measurements are complemented with particle trajectory models to study the role



**Figure:** Concentration of ejecta as a function of radial distance to the nozzle axis, r, and time for a measurement plane height of 3 (a) and 6 (b) nozzle diameters, for a moderately underexpanded jet with nozzle exit plane 10 nozzle diameters above surface, jet mass flow 8.6 g/s and ambient pressure 800 Pa. (c) shows instantaneous snapshot of ejecta cloud from lateral camera at 100 ms after jet operation.

<sup>&</sup>lt;sup>a</sup> Aerospace Engineering, University of Illinois at Urbana-Champaign, USA





#### Pulsed power driven exploding wires: a new approach for shockwave experiments in arbitrary geometries

J. Strucka<sup>1</sup>, K. Mughal<sup>1</sup>, B. Lukic<sup>2</sup>, Y. Yao<sup>1</sup>, D. Maler<sup>3</sup>, S. Efimov<sup>3</sup>, A. Rack<sup>2</sup>, Ya. Krasik<sup>3</sup>, J. P. Chittenden<sup>1</sup>, S.N. Bland<sup>1</sup>

<sup>1</sup>Plasma Physics Group, Imperial College London, UK, <sup>2</sup>European Synchrotron, France, <sup>3</sup>Technion – Israel Institute of Technology, Israel

Recent advances in high energy density science and inertial confinement fusion have led to increased interest in the design of shock compression experiments in increasingly complex geometries. Examples include suppression of the Richtmyer-Meshkov instability (RMI) via impedance defects [1], enhancement of jetting to produce fast material outflows [2,3] and increase in pressures and temperatures for inertial confinement fusion [4]. Designs that obtain such effects can be produced by training a neural network on a set of hydrodynamics simulations to optimize a selected fitness parameter, but the resulting geometry is 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 effect of these complex designs and their underlying dynamics.

The pulsed power driven electrical explosion of wires and foils is a novel method that can be used to perform shock-driven hydrodynamic experiments in arbitrary geometries at facilities such as synchrotrons and XFELs [5]. Compared to other commonly used dynamic compression techniques, 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 regimes where material strength is not important; 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 shortly discuss some of its applications including measurements of convergent RMI, suppression of planar RMI, cumulative hydrodynamic effects, and other experiments that show the capabilities of this approach. The platform will shortly be available as part of the Shock Beamtime Allocation Group for external users.



(a) planar RMI instability

(b) cylindrical RMI instability

**Figure:** (a) A series of radiographs showing the growth of planar Richtmyer-Meshkov instability on an aerogel-water interface. The multi-MHz radiography shows periodic cavitation behind the interface. (b) Example of Richtmyer-Meshkov instability growth in cylindrical geometry to investigate convergent effects.

[1] D. M. Sterbentz et al., *Physics of Fluids* 34, 082109 (2022), doi: 10.1063/5.0100100

- [2] D. M. Sterbentz et al., J. Appl. Phys. 134, 045102 (2023), doi: 10.1063/5.0156373
- [3] D. Maler et al., J. Appl. Phys. 135, 045901 (2024), doi: 10.1063/5.0186659
- [4] D. A. Yager-Elorriaga et al., Phys. Plasmas 29, 052114 (2022), doi: 10.1063/5.0087215
- [5] J. Strucka et al., Physics of Fluids 35, 044108 (2023), doi: 10.1063/5.0144839





### Underwater shock waves interacting with antibubbles serving as liquid payload carriers

<u>Guillaume T. Bokman</u><sup>a</sup>, Luc Biasiori-Poulanges<sup>a</sup>, Bratislav Lukić<sup>b</sup>, Kevin Schmidmayer<sup>c</sup>, Claire Bourquard<sup>d</sup>, Enea Baumann<sup>a</sup>, Alexander Rack<sup>b</sup>, Britton J. Olson<sup>e</sup> and Outi Supponen<sup>a</sup>

Antibubbles, the inverse of bubbles, encase a droplet in a gas layer within a liquid and can be relevant in fluid transport and mixing methods. More recently, they have been promoted in biomedical applications such as targeted drug delivery<sup>1</sup>, where the dynamical response and payload release of drug-loaded microbubbles is induced by low-amplitude sound waves, such as ultrasound or therapeutic shock waves. This study experimentally investigates the interaction of millimetric antibubbles with laser-induced shock waves. Synchrotron X-ray phase contrast images provide a reflection-free, undistorted and temporally resolved visual access to the intricate droplet dynamics within the gas layer upon impact with the liquid surrounding the antibubble. Numerical simulations support the experimental recordings and offer insights into antibubble payload release dynamics. Two new droplet release regimes, supplementing the one first reported by Biasiori-Poulanges et al.<sup>2</sup>, are discovered. The regime type depends on the shape and speed of the antibubble surface upon droplet contact. At high impact velocities, droplets exhibit dynamics akin to those of a droplet impacting a liquid film, generating a sheet-jet whose radius evolution scales with the square root of the droplet's characteristic time. In certain circumstances a liquid jet may form inside the antibubble before contacting the droplet and its angle of deflection upon impact is found to scale with the distance from the droplet center. This work lays the experimental and numerical groundwork to understanding the payload release dynamics of antibubbles in the context of controlled targeted drug delivery.



Figure: Image sequence of the antibubble dynamics following the passage of an underwater laser-induced shock wave. The upper half of the images displays numerical results where the pressure inside of the region with a volume fraction of air greater than 0.5 is displayed in blue, and the droplet payload is highlighted in black. The pressure is normalised to the bubble's initial pressure,  $p_{b,0}$ . half shows The lower corresponding experimental radiographs. The nondimensional time, ρ  $r_{b,0}\sqrt{\frac{1}{p_{b,0}}}$ is indicated and the scale corresponds to 1mm.

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<sup>&</sup>lt;sup>e</sup> Lawrence Livermore National Laboratory, Livermore, CA 94550, USA

<sup>&</sup>lt;sup>1</sup> Kotopoulis et al., Ultrasonics Sonochemistry 85, 105986 (2022).

<sup>&</sup>lt;sup>2</sup> Biasiori-Poulanges et al., Appl. Phys. Lett. 120, 26 (2022).





## X-ray visualization of a shock-wave-induced cavitation cloud inside liquid perfluorohexane droplets

### Samuele Fiorini<sup>a</sup>, Gazendra Shakya<sup>a</sup>, Guillaume Bokman<sup>a</sup>, Stefanos Nikolaou<sup>a</sup>, Anunay Prasanna<sup>a</sup>, Bratislav Lukić<sup>b</sup>, Alexander Rack<sup>b</sup> and Outi Supponen<sup>a</sup>

Acoustic Droplet Vaporization is an ultrasound-based technique that employs acoustically-responsive phase-change nano- or microdroplets as cavitation nuclei in the bloodstream, with the goal to perform targeted drug delivery [1] and non-invasive embolotherapy [2]. Currently, vaporization of heavy perfluorocarbon droplets requires a high ultrasound peak negative pressure [3]. This carries an inherent risk of generating unwanted cavitation activity that can cause damage in the tissues surrounding the target area. Here, we present a proof of concept for fluorocarbon droplet vaporization without the need of any incident negative pressure, by focusing a laser-induced shock wave inside the droplet. Interestingly, a spherical core of liquid fluorocarbon suspended in water acoustically behaves like a "liquid bubble", meaning that no change in the sign of the pressure is expected upon reflection of the shock wave against the internal interface of the droplet. Instead, we believe that a so-called Gouy Phase Shift [4] can occur in the droplet focal point, leading to a partial shock wave sign inversion after crossing it. In other words, tension in the liquid can arise due to the focusing of the compression phase of the shock. In order to confirm our hypothesis, we combined high-speed shadowgraphy with propagation-based phase-contrast x-ray imaging to clearly visualize vapor nuclei formation inside the droplet. The use of x-ray enables visual access to the inside of the droplet, while simultaneous shadowgraphy with a finely collimated light source enabled to capture sharp images of the propagating shock wave. Experimental results reveal pronounced cavitation activity in the distal side of the droplet behind the wave's focal point, as can be seen in Figure 1. Further simulations were carried out in k-Wave [5] and ECOGEN [6], both showing the generation of a tension area after the shock wave crosses the focus, which is in good agreement with the location of the experimentally detected cavitation cloud. Our results provide new insights into the vaporization physics of perfluorocarbon droplets and demonstrate the effectiveness of shock wave focusing in creating very localized negative pressure region inside the droplet core, potentially without the need of any rarefaction wave travelling in the surrounding medium.



Figure 1: X-ray image sequence of the initiation of vaporization in a perfluorohexane droplet. The arrow indicates the direction of propagation of the shock wave.

<sup>4</sup> Lee et al., *ACS Photonics*, 7, 11 (2020).

<sup>&</sup>lt;sup>a</sup> Institute of Fluid Dynamics, D-MAVT, ETH Zürich, Switzerland

<sup>&</sup>lt;sup>b</sup> ESRF – The European Synchrotron, Grenoble, France

<sup>&</sup>lt;sup>1</sup> Chen et al., Journal of Controlled Release, 172, 3 (2013).

<sup>&</sup>lt;sup>2</sup> Harmon et al., Scientific Reports, 9, 11040 (2019).

<sup>&</sup>lt;sup>3</sup> Shakya et al., Advanced Drug Delivery Reviews, 206, 115178 (2024).

<sup>&</sup>lt;sup>5</sup> Treeby et al., Journal of Biomedical Optics, 15, 2 (2010).

<sup>&</sup>lt;sup>6</sup> Schmidmayer et al., Computer Physics Communications, 251, 107093 (2020).





## Unravelling bubbles formation and bubbles dynamics in pulsed laser ablation in liquids

V. Coviello<sup>1</sup>, D. Amans<sup>2</sup>, A. Sollier<sup>3,4</sup>, L. Berthe<sup>5</sup>, V. Amendola<sup>1</sup>, B. Lukic<sup>6</sup> and A. Rack<sup>6</sup>

Laser-induced cavitation bubbles are centrally important in all applications of pulsed laser ablation in liquid (PLAL), such as nanoparticle synthesis<sup>1</sup>, and a thorough understanding of their dynamics is therefore crucial to improving their effectiveness. Despite the use of various optical imaging techniques in the study of laserinduced cavitation bubbles, it is still challenging to capture high temporal resolution images of the initial stage of bubble formation (first 100 ns) and final stage of bubble collapse at its minimum size, because these techniques are all affected by multiple scattering that can obscure the inner details. In order to overcome these limitations, we have conducted experiments on the ID 19 beamline of the European Synchrotron Radiation Facility (ESRF), combining high-resolution multi-frame shadowgraphy imaging to observe the short times, with X-ray phase contrast imaging (XPCI) to observe inside the bubbles over longer times. We found that whatever the liquid used (water, ethanol, solution of H<sub>2</sub>O<sub>2</sub>, PDMS), the bubble formation is triggered by the propagation of the shock wave. This suggests that the bubble formation is not induced by heat transfer from the plasma to the liquid, but by a standard cavitation phenomenon, i.e. a negative peak pressure following the shock propagation. It also suggests that the plasma is isolated from the liquid phase after a few tens of nanoseconds, but the characteristics time of vapor formation appears a bit longer than the one predicted by molecular dynamics<sup>2</sup>. The pressure reached at the shock front range from a few GPa to tens of GPa for the 100 mJ laser pulses used in our work<sup>3</sup>. We also observed thanks to the XPCI images that the surrounding liquid can enter into the bubble from the triple line, when the maximum size of the bubble is reached, as shown in the Figure below. This behavior is unexpected since we usually assume adiabaticity of the vapor during the first oscillation of the bubble.



**Figure:** X-ray phase contrast image showing the entrance of some surrounding liquid into the bubble from its triple line at the bottom right of the image. The image is taken during laser ablation of a titanium surface in water, with a magnification of x4 corresponding to a field of view of  $3.2 \times 2$  mm (H×V). The Nd:YAG laser parameters are 5 ns pulse duration, 100 mJ per pulse, and 532 nm wavelength.

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<sup>&</sup>lt;sup>6</sup> ESRF-The European Synchrotron, CS40220, F-38043 Grenoble, France

<sup>&</sup>lt;sup>1</sup> Kanitz et al., Plasma Sources Sci. Technol. 28, 103001 (2019).

<sup>&</sup>lt;sup>2</sup> Shih et al., Nanoscale 10, 6900 (2018).

<sup>&</sup>lt;sup>3</sup> Chemin et al., Appl. Surf. Sci. 574, 151592 (2022).



#### Flow instabilities in cavitating microchannels

Ž. Boček<sup>a</sup>, M. Petkovšek<sup>a</sup>, S.J. Clark<sup>b</sup>, K. Fezzaa<sup>b</sup>, <u>M. Dular<sup>a</sup></u>

We discuss cavitation inside a micro-Venturi channel. In addition to the conventional imaging we used high speed X-Ray measurements, which can reveal further details of the process. These were conducted at the Argonne National Laboratory – Advanced photon source (Sector 32-ID-B), which provides a highly intense X-Ray beam with a broad energy spectrum from 7 to 40 keV. Images were captured by a Phantom TMX 6410 high-speed camera with a 10x magnification through a LuAG:Ce scintillator screen.

While the initial aim of the study was to establish supercavitating conditions inside a micro-Venturi, yet we found that this regime is suppressed due to the formation of a Kelvin-Helmholtz instability, which triggers a semi periodical attached cavity collapse<sup>1</sup>. In depth observations revealed that this is, besides the re-entrant jet and the shock wave, a third mechanism leading to the shedding of cloud cavitation.

In a follow up study we introduced a stream of oil into the flow and observed the emulsification process<sup>2</sup>. It was determined the formation of the Kelvin-Helmholtz instability at the vapor oil interface interacts with the oil stream and acts in a similar way as the Rayleigh-Taylor instability in the ultrasonically driven emulsification<sup>3</sup>. It can therefore be considered an essential feature to be considered in the optimization of continuous emulsification processes.



**Figure:** The development of the Kelvin Helmholtz instability in a cavitating microchannel (throat crosssection  $450\mu m \times 450\mu m$ ,  $v_{throat} = 31m/s$ ,  $p_{upstream} = 4bar$ ). Visible light (upper image) and X-Ray imaging (lower image).

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<sup>&</sup>lt;sup>b</sup> Advanced Photon Source, Argonne National Laboratory, USA

<sup>&</sup>lt;sup>1</sup> Podbevšek et al. International journal of multiphase flow 142 (2021)

<sup>&</sup>lt;sup>2</sup> Boček et al. *Ultrasonics Sonochemistry* **101** (2023)

<sup>&</sup>lt;sup>3</sup> Boček et al. *In preparation* (2024)





#### Shape mode-induced repeated jetting of ultrasound-driven microbubbles

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Ultrasound-driven microbubbles have emerged as promising agents for targeted drug delivery due to their ability to induce mechanical effects on surrounding cells and tissues, thereby enhancing drug permeability. The mechanical stresses generated by microbubble oscillation in proximity to cells can lead to the puncturing of cell membranes, a phenomenon known as sonoporation. However, the precise physical mechanisms underlying sonoporation at clinically relevant acoustic levels remain elusive. Our recent observations indicate that ultrasound contrast agent microbubbles form cyclical jets capable of impacting a nearby substrate, suggesting a significant role in sonoporation<sup>1</sup>. Unlike previously reported single inertial jets formed at high amplitudes<sup>2</sup>, these jets can repeatedly hit the same spot within a low-amplitude ultrasound burst. This study explores the fundamental mechanism behind the periodic formation of jets by examining larger bubbles driven by ultrasound near a substrate, utilising high-speed and high-magnification phasecontrast X-ray imaging to overcome light refraction challenges observed in visible light shadowgraphy imaging. The experiments unveil that shape modes are instrumental to generate repeated jets against a substrate under low acoustic driving pressures. The primary lobe of the zonal shape mode that is first developed is the most likely to produce a jet, as depicted in Fig. 1(a). Additionally, we identify a clear threshold for the maximum acceleration of this lobe ( $a_{th} = 5 \times 10^5$  m s) for it to produce a jet across a wide range of bubble sizes and shape mode orders, as shown in Fig. 1(b).



**Figure 1:** (a) X-ray images of the jetting dynamics of a bubble driven at 30 kHz and 11 kPa. (b) Measured maximum tip acceleration for bubbles across a range of bubble sizes and shape mode orders. The red solid line represents the apparent tip acceleration threshold for jetting.

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- <sup>1</sup> Cattaneo et al., IEEE International Ultrasonics Symposium (IUS), pp. 1-4 (2023).
- <sup>2</sup> Prentice et al., Nature Physics, 1.2 (2005).

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#### Synchrotron x-ray phase-contrast imaging of ultrasonic drop atomization

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Ultrasonic atomization is employed to generate size-controllable droplets for a variety of applications, including food and drug encapsulation, creation of metal powders and pulmonal drug delivery. Here, we investigate the physics behind the process by studying the atomization of a single drop pending from an ultrasonic horn using spatiotemporally resolved synchrotron x-ray phase-contrast imaging. This technique provides excellent edge contrast due to the spatial beam coherence of the x-rays, ensuring that all ejected droplets are in focus with no overlapping issues and enabling the measurement of millions of ejected droplets, which is not possible with conventional imaging techniques. The high energy flux provided by the synchrotron source additionally allows to image the process at high frame rates (80000 fps). Furthermore, the ability to capture and resolve interfaces with high accuracy allows us to clearly distinguish entrained air and cavitation activity within the deformed drop. The drop's interface initially forms Faraday waves, which undergo several transitions before ejecting micrometric droplets as seen in Fig. 1(a). The size distribution is found to be controlled by the fluid properties and the driving frequency. The results also show that the ejection dynamics in viscoelastic drops is dictated by extended ligament formation, entrainment of air, and ejection of drop-encapsulated bubbles as depicted in Fig. 1(b). Finally, we elucidate the differences between capillary wave-based and cavitation-based atomization using two different configurations - (i) confined water drops and (ii) trapped air bubbles in pendant drops. The occurrence of cavitation-based events within the drop (see Fig. 1(c)) are identified and found to quicken the onset of daughter droplet ejection while impeding their size control.



Figure 1: X-ray phase-contrast images showing the different mechanisms of ultrasonic drop atomization driven with a horn frequency of  $f_d = 20$  kHz. The snapshots are labeled with their non-dimensional times,  $t/T_d$ , where  $T_d =$  $1/f_d$  is the time period of the driving ultrasonic excitation. (a) Formation of Faraday waves and ejection of droplets from a pendant water drop (b) Extended ligament formation in a viscoelastic drop with the boxes highlighting entrainment of air and formation of drop-encapsulated bubbles (c) Trapped bubbles in a viscoelastic drop highlight cavitation-based ejection that is significantly different from the wave-based ejection as seen in (a).

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#### High-speed synchrotron x-ray analysis of non-contact jetting process

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The ability to dispense minute volumes of functional fluids accurately on substrates is of importance in a large number of applications such as the pharmaceutics, electronics, and printing industries. Non-contact methods of application, such as inkjet printing and jet printing of fluids, where a material is accelerated through a nozzle via pneumatic or piezo-based actuation, are growing in importance due to their flexibility of use, increased electronic design space, lower manufacturing cycle times, and decreased cost.

Experimental monitoring of the process is complicated by the small patial dimensions and short temporal durations. The process of material deposition is a complex system of hydrodynamic resistance in the device in combination with break-off and impact dynamics of droplets, phenomena affected by the material properties of the complex fluid that is being used. The use of high-energy photon-based methods with high spatial and temporal resolution offers the possibility to probe the inner workings of the ejection process and use this information to guide the development of future jet printing heads together with numerical simulations.

In this work, a photon-based imaging using a synchrotron light source together with a high-speed image acquisition device to record the development of the front of the fluid ejection process in order to ascertain the effect of front shape on deposition accuracy and repeatability, specifically at the start of a series (strip) of deposits, see Figure 1. The imaging was performed at 150 kHz and with a pixel size of 1 um. It is known that the filling of the nozzle affects the volume and quality of the resulting deposit, but information about the actual position of the meniscus inside the device during consecutive deposits is not currently available. The experimental data was compared with numerical simulations of the jetting system using the IPS IBOFlow package<sup>1</sup>. The comparisons show good agreement and also accentuate the importance of the position of the meniscus for predictive ability of the simulations.



Figure 1: Synchrotron image of jetting head during ejection process.

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<sup>&</sup>lt;sup>1</sup> G.E. Mårtensson, et al., SSMT. 33, 266 (2021).





#### High-speed spray formation probed by Synchrotron X-ray

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In coaxial two-fluid atomization, a liquid jet is destabilized and broken up by a fast gas jet, forming a spray. Such turbulent two-phase flows play a large role in many engineering applications, and in the case of rocket propulsion or metal powder manufacturing the gas flow presents angular momentum (referred to as swirl) and Mach numbers neighboring 1. Such spray formation processes are optically dense and involve extremely small space and time scales, which we tackle using Synchrotron X-ray high-speed imaging. Qualitative observations of the time-resolved gasliquid interfaces show both the destabilized liquid jet and inner details, such as entrapped air bubbles and gas recirculations (Fig. 1), and lead to the establishment of regimes for the morphology of the liquid jet, whose transition can be explained by kinetic energy balance between each phase. When this balance strongly favors the gas flow, a strong gas recirculation displaces the liquid jet, exiting through a smaller section that is no longer centered on the nozzle axis. This break of symmetry is accompanied by the emergence of long-time dynamics. This is explored by recovering equivalent path length maps using ANKAPhase [1] to compute the temporal evolution of the center of mass of the liquid jet. In addition to studying the spray formation processes, the resulting spray droplet size and spatiotemporal distributions and the consequences of the change in regimes of the liquid jet are explored using laser interferometry.



**Figure 1:** Morphologies of the liquid jet for increasing gas Weber number  $We_g$  at a liquid Reynolds number of 800. The green arrows schematically indicate the suspected geometry of the surrounding gas flow.

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<sup>[1]</sup> T. Weitkamp et al., J. Synchrotron Radiat. (2011). 18 (4), 617-629





#### Invited Talk: Multiphase flows from swarming bubbles to schooling fish: from the advancement of optic measurements to data assimilation

#### Rui Ni, Johns Hopkins University

In this presentation, I will explore various complex multiphase flows that stand to benefit from synchrotron X-ray techniques. These flows inherently involve more than one phase, typically a carrier phase and a dispersed phase, which could manifest as solid particles, deformable bubbles, or even dynamic entities like live schooling fish. While the success of 3D optical reconstruction, employing multiple high-speed cameras from various angles, has provided valuable insights into these problems, it has encountered limitations.

The dispersed phase, whether solid particles, deformable bubbles, or living organisms, poses challenges as it can either block or reflect light. As the concentration of the dispersed phase increases, traditional optic diagnostics in the visible light spectrum face difficulties. On the contrary, X-ray methods excel in scenarios where the medium is optically opaque. However, the synchrotron source used in X-ray methods has its own limitations, such as the inability to create orthogonal beams intersecting for high-speed tomography.

This second half of the presentation aims to address the question of whether a synergistic approach can be developed by combining both optical and X-ray methods, allowing for a more comprehensive understanding of the physics involved in these complex multiphase flows. I will present an illustrative example highlighting the potential of data assimilation techniques that leverage multiple diagnostics. This approach enables us to uncover hidden physics within a problem that may remain elusive when relying on a single diagnostic method alone.





#### Multiphase Flow Studies at the APS 7-BM Beamline

#### A. L. Kastengren<sup>a</sup>

Due to the impact of multiple scattering, dense multiphase flows are some of the most difficult flows to study with visible light diagnostics. X-ray diagnostics offer distinct advantages in these flows due to their relative insensitivity to phase boundaries. High-speed x-ray phase contrast imaging has provided clear visualizations of previously unseen phenomena in a number of multiphase flowfields. However, imaging alone provides limited information in multiphase flowfields. Over the past 25 years, the Advanced Photon Source has used a wealth of diagnostics, including phase-contrast imaging, to study multiphase flows. The combination of phase-contrast imaging with other x-ray diagnostics can provide insights unavailable by imaging alone. This presentation will describe several diagnostics that have been applied at the APS. Timeresolved focused-beam radiography has been used to determine quantitative liquid mass distributions in spray flowfields at high spatial and temporal resolution<sup>b</sup>, which directly complements the qualitative morphology and quantitative velocity information available from high-speed imaging. X-ray fluorescence microprobe measurements have quantitatively probed mixing in liquid-liquid<sup>c</sup>, liquid-gas<sup>d</sup>, and gas-only flowfields, including for combusting flowfieldse. The role of hard x-ray microtomography in complementing high-speed imaging will be detailed, as it provides both boundary conditions for computational modeling and a better understanding of the cause of flowfield asymmetries<sup>f</sup>. Finally, x-ray scattering-based measures of micron-scale droplet size<sup>g</sup> and liquid temperature<sup>h</sup> will also be briefly described, which provide insights generally invisible in high-speed phase-contrast imaging. Several example measurements from the literature will be shown to illustrate the capabilities and limitations of these techniques, as well as how their combination with imaging can provide insights unavailable from any one diagnostic alone.



**Figure:** a) Three sequential frames from high-speed phase-contrast imaging (20 kHz) of an impinging jet atomizer with one stream doped with contrast agent (50% KI by mass in water vs. pure water)<sup>i</sup>, b) Demonstration of transmittive mixing in a similar impinging jet atomizer by x-ray fluorescence<sup>c</sup>

- <sup>e</sup> Meng, et al., Combustion and Flame, 251, 112686 (2023)
- <sup>f</sup> Moon, et al., AIAA Paper 2024-1996 (2024)
- <sup>g</sup> Matusik, et al., Atomization and Sprays, **29**, 199 (2019)

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<sup>&</sup>lt;sup>b</sup> Bothell, et al., International Journal of Multiphase Flow, 125, 103219 (2020)

<sup>&</sup>lt;sup>c</sup> Halls, et al., Optics Express, 22, 1730 (2015)

<sup>&</sup>lt;sup>d</sup> Peltier, et al., *Experiments in Fluids*, 58, 111 (2017)

<sup>&</sup>lt;sup>h</sup> Rahman, et al., *Applied Optics*, **60**, 2967 (2021)

<sup>&</sup>lt;sup>i</sup> Halls, et al., Optics Express, 25, 1605 (2017)





#### Advances X-Ray Techniques for Fluid Dynamics at Lab and Synchrotron Scale

#### S. A. Mäkiharju<sup>a</sup>

X-ray techniques have become increasingly accessible and useful for the fluid dynamics community as more synchrotron beamlines have become accustomed to accommodating fluid dynamics experiments, and in the laboratory scale the sources and detectors have evolved to enable the undertaking of evermore ambitions experiments - from time-resolved to those requiring extreme dynamic range. The latter has been enabled by the increasing capabilities of photon counting detectors, which provide us with nominally infinite dynamic range by enabling unlimited integration times with nominally no added noise even when utilizing minutes or hours per exposure. In this brief talk we will examine a few fluid dynamics experiments which can benefit both from synchrotron as well as laboratory scale X-ray experiments. First, we consider an Xray particle velocimetry (XPV) application requiring both a small yet bight focal spot (or parallel beams). While such measurements have been conducted at synchrotron facilities for over a decade in a relatively small number of pioneering studies<sup>1,2</sup>, even more accessible XPV in-lab has recently become progressively more capable (figure 1a&b). Second, we consider a slowly evolving flow in an optically opaque metal confinement - one that is unavoidable due to extreme thermal gradients and pressures around 300 MPa, with spikes on the order of GPa being possible. Figure 1c shows the contents of an isochoric vitrification chamber in which low contract materials can be imaged in previously unobtainable detail owing to photon counting detectors. Beyond the steady equilibrium points, such as the one shown, quantification of flow field during the transients is of significant scientific value for the cryopreservation community and hence part of ongoing research.



**Figure: a)** in-lab 1 kHz PPV imaging setup and **b)** radial flow profile with measurement compared to the theoretical Poiseuille flow profile shape (from Parker et al.<sup>7</sup>). **c)** ice shown in teal and vitrified solution in grey within the 10 mm inner diameter bore of an isochoric metal chamber (from Ali et al.<sup>8</sup>).

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<sup>&</sup>lt;sup>1</sup>Lee et al., J. of Applied Physics (2005)

<sup>&</sup>lt;sup>2</sup> Dular et al., Experiments in Fluids (2012)

<sup>&</sup>lt;sup>3</sup> Makiharju et al., Experiments in Fluids (2022)

<sup>&</sup>lt;sup>4</sup> Parker et al., Measurement Science and Technology (2022)

<sup>&</sup>lt;sup>5</sup> Parker et al., Experiments in Fluids (2023)

<sup>&</sup>lt;sup>5</sup> Parker et al., Flow Measurement and Instrumentation (2024)

<sup>&</sup>lt;sup>7</sup> Parker et al., Cryobiology, (2024)

<sup>&</sup>lt;sup>8</sup> Ali et al., to be submitted, (2024).





### X-ray multi-projection imaging: enabling fast volumetric information for fluid dynamics

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Key words: X-ray imaging; Fast dynamics; X-ray methods; Fluid dynamics

X-ray multi-projection imaging (XMPI) [1] is an X-ray imaging technique capable of probing natural processes with a micrometer to nanometer resolution and resolving millisecond to microsecond dynamics. This technique splits a single X-ray pulse or flash into several angularly resolved beams, which illuminate the sample simultaneously. This approach, therefore, avoids scanning the sample as required by current 3D X-ray techniques, such as state-of-the-art timeresolved tomography. Thus, it prevents the introduction of forces due to the scanning process that may hinder the studied dynamics, which is especially relevant for non-rigid systems like the ones studied by fluid dynamics.

In this oral contribution, we intend to introduce the concept of XMPI and report our proof-ofconcept experiments with micrometer resolution at ESRF-EBS [2] and the European XFEL [3]. In such experiments, we demonstrated the possibility of acquiring volumetric information without rotating the sample at kHz and MHz acquisition rates at ESRF-EBS and the European XFEL, respectively. As an example of the applications of this enabling technique, we will present our study of MHz binary droplet collisions at 1.1 MHz acquisition rate with micrometer resolution [3]. We will also discuss the limits of XMPI, among them the challenge of reconstructing 3D movies from sparse projections. To address this, we will introduce ONIX [4], a novel reconstruction approach that combines several experiments on similar events to retrieve 3D movies from as few projections as three. We envision that XMPI will become an enabling tool to image fast fluid dynamics in 3D, opening the opportunity to study complex processes not yet observed.

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#### 4D imaging of Haines jumps with sub-millisecond temporal resolution

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Multiphase flow in porous media is a topic of high societal and scientific interest.<sup>14</sup> Advances in X-ray sources and detectors have enabled time-resolved studies of flow in three-dimensional porous media through computed tomography (CT). Drainage, which denotes the replacement of a wetting fluid by a non-wetting fluid in a porous medium, is associated with a wide range of pore-scale processes.<sup>1,3,4</sup> The most spectacular of these is arguably *Haines jumps*, i.e., the sudden fluid arrangement whereby a new pore volume is filled with the invading non-wetting fluid.<sup>2-4</sup> By designing an experimental setup with a repeatable drainage dynamics across a static glass shard structure, combined with an innovative hydraulic pump – X-ray probe method, and a compressed-sensing inspired reconstruction algorithmn<sup>5</sup>, we demonstrate the visualization of Haines jumps in 4D (3D + *time*) with sub-millisecond temporal resolution.<sup>1</sup> For the first time, we were able to follow the detailed dynamics of the fluid rearrangements, including Haines jumps, surpassing the previously achieved temporal resolution by several orders of magnitude. Thus, we are able to appreciate the fine details of the liquid menisci, including their shape and propagation velocity, rather than just the "frozen" still images before and after the rapid dynamics as previously reported. The experimental scheme presented here opens for studying a wide range of dynamical processes in soft materials with time-resolved 3D microscopic X-ray imaging.

#### Acknowledgements

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#### 4D X-ray velocimetry of multiphase flows in porous media

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Many natural and industrial processes depend on fluids displacing each other in porous materials. However, multiphase flow dynamics in porous media are still poorly understood due to the complex interplay between capillary, viscous and inertial forces. Research on this topic has been hampered by the lack of methods to measure flow fields in optically opaque, microscopic 3D geometries : while X-ray micro-computed tomography (micro-CT) has enabled the visualization of fluid distributions and menisci in the pores, measurements of the underlying flow dynamics (i.e. velocity fields) have so far remained impossible. In this work, we introduce a novel 4D micro-velocimetry method based on synchrotron micro-CT with fast imaging rates (up to 4 Hz at 2.75 µm voxel size) at the TOMCAT beamline of the Swiss Light Source. This was used to perform Lagrangian particle tracking of µm-scale tracer particles in the flow through the pores of natural rock and filter samples. The measurements resulted in time-resolved and fully three-dimensional (3-component) flow fields during unsteady-state drainage, where oil displaces water from the pores at slow, constant injection rates. The data enabled to calculate how fluid displacements convert interfacial energy into kinetic energy, corresponding to velocity perturbations in the pore-scale flow field. Our analysis suggests that these perturbations are long-ranged, impacting the pore-scale viscous-capillary force balance and the representative volume needed for averaging. Furthermore, the perturbations enhance solute and colloid transport in unsaturated porous media. Overall, we show that 4D X-ray velocimetry opens new pathways to investigate flow in porous materials, relevant to e.g. groundwater pollution remediation and subsurface storage of CO<sub>2</sub> and hydrogen.



**Figure:** 4D particle tracking of micrometer-scale flow tracers in the non-wetting phase (oil) during a drainage experiment on a porous filter sample. The two stills are from tomograms 1.25 s apart ; each with an acquisition time of 0.25 seconds and a voxel size of 2.75  $\mu$ m. The grey transparent surface shows the oil meniscus in the pores. Oil invades pores in sudden movements called Haines jumps, triggering acceleration in the 3D flow field related to oil redistribution in the surrounding pores. This flow field perturbation extends throughout nearly the entire sample and results in remarkably tortuous flow paths.

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#### Dynamics of Binary Granular Materials: Unraveling the Phenomena of Rising Granular Bubbles and Splitting Granular Droplets

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Granular materials are ubiquitous in nature and an integral part of numerous industrial processes. However, due to their complex behavior on the cross-roads of classical gases, liquids, and solids, we are far from having a complete understanding of their dynamics<sup>1</sup>. Furthermore, the inherent opacity of granular materials makes it difficult to study their behavior in three dimensional systems without perturbing probes.

Recently, the existence of gravitational instabilities has been demonstrated for binary granular materials in a quasi-two-dimensional bed that is subjected to vibro-gas-fluidization<sup>2</sup>. Here, a cluster of particles that is immersed in a bulk of particles with smaller particle size and larger density can form a coherently rising structure resembling a gas bubble rising in a liquid. However, a sinking and splitting cluster reminiscent of a liquid drop falling in a miscible fluid is observed if the surrounding bulk particles are larger and lighter<sup>3</sup>. The existence of such liquid-like phenomena is astounding considering the absence of surface tension in dry granular media.

This talk elucidates the underlying mechanisms behind the rising and sinking of granular clusters in two dimensional systems exploiting high speed camera imaging and numerical simulations. An analytical buoyancy limit is derived delineating the border between rising and sinking structures and is used to compile a dimensionless regime map<sup>4</sup>. Furthermore, the use of magnetic resonance imaging is demonstrated to overcome the limitation to quasi-two-dimensional beds.



Figure: a) A granular cluster (white) forms a rising "granular bubble" in a pool of bulk particles (black) when being exposed to vibro-gas-fluidization (simulation). b) Instantaneous snapshot of a rising granular bubble (experiment). c) Time-averaged solid flow field in the bubble frame of reference (experiment). An internal circulation pattern with striking similarity to bubbles in classical fluid mechanics is found.

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### X-ray analysis of air bubble rise in granular suspension: Application to concrete de-airing

#### Bastian Strybny<sup>1</sup>, Julian Link<sup>1</sup>, Marcus Zuber<sup>2</sup>, Valérie Vidal<sup>3</sup> and Michael Haist<sup>1</sup>

The mechanical properties of concrete are strongly influenced by its porosity. A high air content can be caused through the processing of concrete on the construction site (mixing, transportation and placing), when air bubbles cannot escape due to improper rheological properties of the fresh concrete. Whereas the air-bubble rise has been extensively studied in both Newtonian as well as low to medium yield stress Bingham or Hershel-Bulkley fluids, the de-airing mechanisms in concrete – i.e. a reactive, polydisperse, thixotropic, multi-phase suspension with solid volume fractions as high as 90 vol.-% remains an open scientific question.

The proposed contribution deals with investigations to quantify the influences of the rheological and granulometrical properties of the fresh concrete on the rising behavior of air bubbles. For this purpose, a novel test setup, which enables injecting defined air bubbles into the sample was developed. As concrete is a really opaque system, the air bubble rising dynamics was quantified by means of X-ray techniques. The image analysis was carried out using MATLAB algorithms for image segmentation. The focus of the experimental investigation was on the correlation between the rheology of the cement suspension and the rising velocity, bubble shape and the trajectory of the uprising air bubbles.

A key point of interest in addition was to study the effect of larger aggregates – both as monodisperse as well as polydisperse distributions in different sizes and solid volume fractions.

The results hereby showed, that a rising air bubble in cement suspension creates an uprising channel for subsequent bubbles that follow the same trajectory. The rising velocity of the secondary bubble is increased, caused by locally lower viscosities due to shear induced particle migration in the uprising channel. Furthermore, the rheological properties in combination with the granulometry of the aggregates play an important role in governing concrete de-airing. Significant changes in the bubble rising behavior arise through the bubble volume, the viscosity of the carrier fluid as well as the size and packing density of the larger aggregates.

The findings of this project help to improve the understanding of the basic mechanisms of concrete deairing but can also be applied to other non-Newtonian granular systems, such as in the process technology or bio sector.



**Figure:** X-ray measurements of rising air bubbles after image segmentation in cement paste without aggregates  $\Phi$ paste =0.36 (a), 0.38 (b), 0.40 (c) and with 60 vol.-% of monodispersed aggregates with a size of 8 mm (d).

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#### Shock induced cavitation in water

#### M. Arrigoni<sup>1</sup>, E. Tanné<sup>1</sup>, A. Sollier<sup>2</sup>, M. Monloubou<sup>1</sup>, S. Kerampran<sup>1</sup>, B. Lukić<sup>3</sup>, A. Rack<sup>3</sup>,

Cavitation of a liquid can be obtained after a compressive wave transmission into a liquid container. The process of a formation, the growth and collapse of a bubble in fluid is thus an active phenomenon that dissipate energy and mitigate strain-rates. The time scale of cavitation (~hundreds of  $\mu$ s) is longer than that of the shock transit time (~ $\mu$ s) created by the impact of a projectile and a fraction of this energy is stored in the bubble and released during the bubble existence, until it collapses. Having in mind that water is available in large quantity, it is transparent and the cavitation process consecutive to an impact on water compartmentalized targets could offer the possibility actuating a part. However, cavitation process is triggered above a certain shock intensity and duration and open literature does not exhibits clear data and models that can predict it.

The presented work shows a collection of experimental results of shock induced cavitation process for a range of impulse and pressure produced by a variety of dynamic experiments such as laser shock, gas gun and Hopkinson bar. The target edifice is a transparent tank made of PMMA filled with water and capped by a metal plate. Pressure sensors are located at the inner back wall of the tank to measure the shock pressure into the water. The cavitation bubble is captured by a high-speed camera and a series of laser shock experiments was performed at ESRF with ultra-fast X-ray in order to avoid the shadow effect of the light in the growth of the bubble. At the impact, a layer of bubbles nucleates at the target/water interface, then merges to a flat bubble, expands until reaching a maximum radius, then starts to collapse from the contact radius with the plate. The collapse produces a second shock in the water and then an annular vortex moving forward is formed. Series of experiments obtained shows some interesting features that could enhance energy dissipation and mitigate consecutive strain-rates during an impact event.



**Figure:** On the left, bubble produced by a steel ball (diam. 14 mm) launched by a gas gun at 50 m/s on a sandwich of stainless-steel plate (2 mm), water (3 cm) and PMMA (3 cm). On the right, cavitation due to laser shock on aluminium 0.8 mm plate confined with water, the impact is coming from the bottom.

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#### Numerical methodology for validation of small-scale blast test design of steel plates subjected to an impact from underwater blast wave generator

D. Čekerevac<sup>1</sup>, C. Rigueiro<sup>2</sup>, A. Santiago<sup>1</sup>, E. Pereira<sup>3</sup> and J. Gois<sup>4</sup>

This study is focused on a numerical methodology that supports an experimental evaluation of the performance of a plate or a sandwich panel subjected to an underwater blast generator (WBWG) which is formed by an explosive charge detonated inside a water container. WBWGs are characterized by a high conversion rate of explosive detonation energy into the kinetic energy of water confinement, making them more suitable for these experimental studies compared to air blasts. An optimization of parameters of IWL equation of state (EoS) for definition of expanded detonation products of selected explosives charges used in the preliminary blast tests and the assessment of the charge sizes was performed through combination of in-house thermochemical computer code THOR calculations and Ansys Autodyn numerical modelling. Calibration of JWL parameters can also be done experimentally based on cylinder test results<sup>1</sup>, however the computational predictions of thermochemical codes such as THOR provide a cost-effective alternative<sup>2</sup>. The performance of the discussed methodology for the case of PBX explosive charge was evaluated in comparison to a deformed shape of the steel plate subjected to WBWG impact in a small-scale blast test. Test configuration consist of a steel place placed over a 50 mm thick low-density polyurethane back plate serving as a shock absorber. The plate is subjected to a blast impact resulting from a detonation of PBX charge within a small water container. The experiments were performed with charges of 50 g inserted in a plastic film. The study starts with a definition of the physical properties of explosive charge (such as composition, density and energy of formation) which are needed for THOR calculations of CJ detonation product properties, and adiabatic and isentropic expansion. Theoretical expansion curves obtained from THOR are then used for optimization of JWL parameters by minimizing the difference between values of JWL predictions and those of theoretical curves. Hydrocode Autodyn was used to model the expansion of detonation products, propagation of the shock into the steel plate and the plate's deformation. It was found that the size of the water container and the explosive charge have appropriate dimensions to be used in the small-scale tests. The model provides a way to assess the shock transmission and attenuation in water domain and to compare the deformed shape of the plate validating the effect of the small scale WBWG. The JWL expansion curve, the numerical model and experimental configuration are shown in the Figure.



**Figure:** Comparison of JWL expansion curve with adiabatic and isentropic theoretical expansion curves for PBX charge composition (a), Autodyn model (b) and experimental configuration (c)

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#### Bubble clouds generated by single and multiplunging jets

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The impact of a plunging jet with a liquid surface induces air entrainment beneath the surface, resulting in the formation of a bubble cloud. This ubiquitous phenomenon is widely encountered in nature, like breaking waves in water bodies, and in industrial techniques like reducing foam formation in chemical processes. The depth (*H*) of this bubble cloud is an important parameter for modeling in such applications. Using laboratory scale experiments and novel optical probes to carefully measure the local bubble void fraction ( $\phi$ ), we demonstrate that a simple momentum balance including only liquid inertia and buoyancy force due to the bubble cloud volume provides a very good estimate for the depth *H*, when  $\phi$  is known. Furthermore, we show that bubble clouds can be classified as inertial or buoyancy-dominated based on a Froude number given by a characteristic bubble terminal speed, cloud depth and the cloud's net void fraction. Thereby, our findings help unify a large body of data in the literature corresponding to a wide range of injector diameters (250 µm - 20 cm) and cloud depths from few centimetres to a few metres. Finally, we use a simple set-up of closely packed multi-injectors as model to investigate air entrainment by large scale plunging jets. Our preliminary results confirm that inertia imparted by the plunging liquid jet and the bubble cloud volume are sufficient to determine the cloud depth even in such complex cases, provided the bubble void fraction is given.





#### Reactive transport instability during flow in porous rock cores.

#### M. Dzikowski<sup>1</sup>

Highly localized flow paths or channels, termed wormholes, arise during the dissolution of fractured and porous media under certain flow conditions<sup>23</sup>. Their growth is governed by positive feedback between the flow field and chemical reactions at pore surfaces. Those newly created channels become preferential flow paths, in which both the flow and reactant transport become spontaneously localized. Since dissolution fingers dramatically increase the permeability of the rock, wormholing is important both for industrial applications and in hydrogeological studies.

This early study presents several core flooding experiments showcasing the occurrence of reactiveinfiltration instabilities during both single (HCl) and two-phase (HCl and Nitrogen) flow. Leveraging the capabilities of the Fast-X laboratory at NCBJ in Świerk (Poland) and the ID19 beamline at ESRF in Grenoble (France), these in-situ experiments sought to capture high-resolution images of developing wormholes to understand their interaction with the surrounding porous matrix.

During the two-phase experiments, the acid solution was injected in discrete pulses, alternating between 30 minutes of continuous flow and 30 minutes without flow. This approach mirrored the CO2 experiments conducted by Ott<sup>4</sup>. In contrast, nitrogen was supplied continuously at a constant pressure exceeding the capillary threshold, ensuring a relatively stable nitrogen flow. Though preliminary analysis did not reveal significant interaction between the growing wormhole and nitrogen, observations suggest presence of different types of two-phase regimes inside and around the growing structure, ranging from bubbly flow within the wormhole itself, to fully locked area and branches. For comparison, single phase experiments were also performed in the same conditions, where acid was pumped in turns, but no gas was present in the system.



Figure: A. Wormhole developed in highly heterogeneous limestone during combined acid/nitrogen flow. Trapped nitrogen bubbles in blue.

- **B.** Wormhole developed in homogenous gypsum cast during single phase flow
- **C.** Structure developed in the same conditions and in a similar core as B, but in dry core pre-saturated with nitrogen; Presence of capillary effect rendered the structure more compact.
- **D**. Cross section of A at 2 different times, dark black zone is a gas bubble traveling the chanell. Last picture is an overlap the boundary of the bubble shape is still visible in later times indicating a partially dissolved zone.

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## Analysis of powder dynamics in laser powder bed fusion (LPBF) by 3D segmentation of projected particle trajectories

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Spatter in laser powder bed fusion (LPBF) refers to the ejection of particulate matter as a result of vaporisation phenomena in the laser-matter interaction zone. Not only does the removal of powder from the bed reduce productivity, the molten spatter can also redeposit on previously processed layers, creating defects that degrade mechanical properties. For this reason there is interest in tracking spatter characteristics in situ. There exists a multitude of spatter tracking techniques, but most suffer from poor illumination sources and insufficient spatial and/or temporal resolution to follow fast moving particles, thus uncertainties are large. Here, we present a new technique for accurately tracking spatter during in-situ LPBF with ultra high speed synchrotron X-ray radiography. At 40k frames per second and 4 µm pixel size, we have sufficient flux and X-ray image contrast at the ID19 beamline at ESRF to identify and track small and fast-moving particles. Treating the image sequence as a 3D volume, with time as the third dimension, we segment particle trajectories as fibre-like objects. The proposed technique enables measurement of spatter volume, states of spatter particles, and projected velocity and exit angle with a high degree of accuracy.



Figure: Time series radiography data imported into 3D slicer software as Z-series. Each coloured fiber is a segmented particle trajectory.





#### Liquid fraction for the flow of alkanes through an anodic alumina membrane

T. Loimer<sup>1</sup>, D. I. Petukhov<sup>2</sup>

Anodic alumina membranes are produced by anodic oxidation of metallic aluminum<sup>1</sup>. Depending on the process parameters, the final membranes have very evenly distributed, straight, round pores with pore sizes between about 20 nm and more than 200 nm, see Fig. 1. The thickness of the membranes is usually abount 0,1 mm. Due to its topology, the flow through this material can realistically be described as a flow through parallel, round capillaries.

For the case of a vapor near saturation flowing through an anodic alumina membrane, due to the small pore size a large pressure difference and, hence, a temperature difference due to the Joule-Thomson effect results. The downstream side of the membrane is colder than the upstream side. Therefore, for a flow where the fluid is in a state of a vapor near saturation upstream of the membrane, the fluid may condense, liquid flows through a part of the membrane, the fluid evaporates and a vapor farther away from saturation than at the upstream side leaves the membrane at the downstream side. This process has been theoretically described<sup>2</sup>, the mass flow has been measured<sup>2</sup> and in one experiment the fraction of liquid within the membrane has been determined using confocal Raman spectroscopy<sup>3</sup>. However, an accurate measurement of the length of the liquid flow region would provide an additional corroboration of the description of the flow.



Figure 1: Scanning electron microscopy images of (a) the bottom side and (b) a cross section of an anodic alumina membrane (Fig. 4 from Ref. 1).



Figure 2: Flow with condensation and evaporation within a pore.

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#### Vapor condensation in fractured porous media revealed by multimodal tomographies and numerical modeling

A. Nemati a, B. Lukić b, A. Tengattini c, M. Briffaut d, P. Séchet a

The study of phase change in processes involving two-phase flow in porous media remains relatively underexplored due to the intricate nature arising from the strong coupling between heat and mass transfer and the heterogeneity of the medium. However, condensation in porous media plays a crucial role in various applications, including steam-based gas recovery, underground contamination removal, the integrity of geothermal, CO2 storage reservoirs, durability of concrete structure, and porous fabric and insulation condensation. The objective of this study is to provide a deeper understanding of the subject by conducting rapid neutron tomographies, and X-ray imaging during vapor injection experiments and introducing a novel numerical approach to model the process.

The identification and quantification of water is revealed using 3D rapid in-situ neutron imaging, acquired at 30-second intervals per tomography. Such temporal resolution is possible thanks to the high neutron flux of the Institute Laue Langevin Grenoble (ILL) using the imaging instrument NeXT (Neutron and X-ray Tomograph). The experiments were preceded by a calibration and correction campaign where the quantification of water content was fitted to empirical correlation and the spurious deviations arising from the scattering of neutrons are accounted for using black body (BB) grid method. The in-situ experiment consists of the injection of a predefined mixture of air and water vapor at a constant flow rate into cylindrical samples of Fontainebleau sandstone with a splitting crack along their height. Successive rapid neutron tomographies are acquired during the injection of vapor to investigate the evolution of water content and condensation process inside the sample. Furthermore, the sample is scanned by synchrotron microtomography with 6.5 micrometers pixel size at ID19 beamline of the European Synchrotron Radiation Facility (ESRF). This allows for extracting the microstructure and morphology of the crack and porous matrix, and its impact on the spatio-temporal accumulation of liquid water, and understanding its migration within the crack and matrix. The results [3,4] show that water initially emerges near the inlet and spreads toward distant areas. Condensed water generally has the tendency to occupy tighter spaces within the sample. The condensed water diffuses into the porous matrix due to capillary effects and pressure buildup in the crack.

Preliminary results of an original numerical model that has been developed in OpenFoam also provides additional insights. The model solves heat transfer and two phase flow equations with phase change mass transfer term. It is capable of modeling water condensation and temperature fields within a domain of heterogeneous porous media contributing additional insights to the phenomena.



**Figure:** (a) schematic of the experimental setup during the in-situ imaging, (b) relationship between the pore size distribution (revealed by synchrotron microtomography) and water accumulation (neutron intensity, revealed by neutron imaging)

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#### X-ray high-speed imaging of hydrodynamic cavitation and biomass particle passing through Venturi tube

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Cavitation is a harmful phenomenon for hydraulic machineries, as severe impact is produced at cavitation bubble collapse. However, it can be utilized for mechanical surface treatment of metallic materials<sup>1</sup> and water treatment. Regarding previous studies, hydrodynamic cavitation through Venturi tube was applicable for pretreatment of biomass<sup>2</sup> and the intensity of sonoluminescence of the hydrodynamic cavitation was closely related to number of vortex cavitation<sup>3</sup>. It was also found that the vortex cavitation was associated with angulated bubbles rather than spherical bubbles<sup>4</sup>.

In the present paper, in order to make clear the mechanism of pretreatment of biomass by hydrodynamic cavitation, the hydrodynamic cavitation and biomass passing through Venturi tube was observed by X-ray high-speed imaging at beamline 28B2 of SPring-8. Figure 1 shows aspect of hydrodynamic cavitation and biomass particles passing through Venturi tube. The throat diameter of Venturi tube was 2.0 mm, the upstream pressure of the throat was 0.078 MPa and the downstream pressure of the throat was 0.027 MPa, respectively. Note that the pressures were described in absolute pressure. The recording speed was 69,600 frame/sec. As shown in Fig. 1, the vortex cavitation, which consisted from angulated bubbles, was observed and the biomass particles were passing through near the cavitation.

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Figure 1: Aspect of vortex cavitation and biaomass particles observed by X-ray high-speed imaging

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## X-ray radiography 4D particle tracking of heavy spheres suspended in a turbulent jet

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The suspension of a heavy sphere by an upward jet is a classical fluid mechanics experiment to demonstrate the fluid forces acting on an object. In the range of the parameter space where the sphere can be suspended, the dynamics can either be regular, i.e., with oscillations around an equilibrium position, or chaotic, with extreme events leading to large deviations from that equilibrium region. The existence and characteristics of suspension regimes of several heavy spheres in such flow configurations remain open questions. Spheres compete for the equilibrium position and come very close to each other, resulting in large local particle concentrations that prevent direct imaging. Relatively high speed X-ray radiography along with the radioSphere analysis technique is leveraged here to study the time-resolved 3D trajectory of each individual sphere in a vertical jet. radioSphere is an X-ray analysis method that retrieves the 3D information out of a single 2D radiography using a priori knowledge of the imaging geometry (E. Andò et al., 2021), which due to the imaging modality imposes no limitations on the optical properties of the water. The 3D + time kinematics yield the evolution of the statistics of the position and velocity of the spheres as a function of the number of spheres and for two jet Reynolds numbers. Drastic changes in behavior occur when many spheres are present, leaving a clear signature on the temporal dynamics and on the exploration of the flow volume, where spheres can remain on the bottom of the vessel for long periods of time, resulting in only partial suspension. In addition to the suspension capacity, the interactions between spheres are explored with statistics of pair separation distances, which, together, allow for quantitative arguments to introduce suspension regimes of a collection of spheres in an upward vertical jet.





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### X-ray imaging and numerical modeling of shear band formation during solidification

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High-pressure die casting (HPDC) is an advanced manufacturing process for producing intricate components of lightweight aluminium alloys at high production rates. However, HPDC is prone to potential defects such as porosity and shear bands. The formation of shear bands depends on the semi-solid flow (SSF) patterns, solid fraction gradient normal to the bulk flow, die shape, and the applied pressure. A parametric study quantifying these effects is however not available. In this study, we captured the effect of solid fraction and SSF patterns in custom alumina die-pistons mounted on a bespoke thermomechanical rig using fast synchrotron X-ray imaging. The effect of solid fraction on the semi-solid interface velocity, eutectic band width and porosity was quantified. Further, sphericity and volume of porosity defects were quantified using ex-situ CT scans and correlated with the in-situ radiographs. Additionally, the effect of SSF on the microstructural inhomogeneity was quantified. Based on these observations, a mechanism is provided for formation of a novel porosity that is related to the presence of Externally Solidified Crystals. A macroscopic model is also developed to capture the SSF with band of porosity and deformation during the flow. A permeability variation across the flow was assumed to depict the solid fraction gradient, which captures the shear band formation during the SSF. Understanding the rheological behaviour of SSF will be crucial in developing the numerical models for shear band prediction. Therefore, synchrotron X-ray imaging is necessary to capture shear band formation and the underlying physics in the defect occurrence during the SSF.



**Figure:** Synchrotron radiograph sequence during semi-solid flow due to deformation, displaying nucleation & growth of pores during deformation and solidification Velocity profiles for different permeability variations in axisymmetric cylinder (K=permeability variation shown in figure)



#### Multiphase flows in deep-sea sedimentary layers

V. Vidal<sup>a</sup>, A. Gay<sup>b</sup> and G. Varas<sup>c</sup>

Sedimentary basins are large areas in the older and deeper oceanic regions where particles accumulate due to the sedimentation of organic and mineral matter. These kilometer-deep layers host many fluids, gas or liquid, which migrate due to physico-chemical or tectonic processes generating a local pressure increase leading to fluid escape<sup>1,2</sup>. Although located far from land, the fluid migration structures, also called "pipes", represent strong geohazards for human activities such as transoceanic telecom fibers or offshore resources. However, even the most recent seismic techniques cannot capture their dynamics.

Laboratory experiments provide the opportunity to model and characterize fluid migration in watersaturated sands<sup>3</sup>. A first series of experiment consists of a cylindrical tank filled with particles up to a height  $h_g$ , analogue to the sediment layer. The grains are then immersed in water (height  $h_l$  above the granular free surface, Figure a). Gas is injected at constant flow rate at the cell bottom center, through an injection nozzle (black arrow, Figure a). As the particles are opaque, the fluid migration patterns through the granular layer cannot be observed. A camera on the top of the experiment can locate the gas emission at the surface and characterize its spatial and temporal evolution.



**Figure:** (a) Gas invasion in a water-saturated sediment layer.  $h_g$  and  $h_w$  indicate the initial granular layer height and the water layer height above the grains free surface, respectively. In this 3D experiment, the grains are opaque and only the gas emission at the surface can be quantified. (b,c) Snapshots of gas pathways in a 2D experiment (Hele-Shaw cell) with (a) a single or (b) multiple gas injection points. The black arrows indicate the injection nozzles.

To get an insight on the gas dynamics inside the granular medium, experiments have also been performed in a quasi-2D configuration. The experimental cell consists of two glass plates separated by a thin gap (typically 2 mm), also known as a Hele-Shaw cell. A homogeneous light panel located behind the cell makes possible to visualize the gas pathways (Figure b,c). The case of a single injection point (Figure b) or multiple injection points (Figure c) have been investigated. In particular, we have characterized the formation of a fluidized zone around the main vertical gas flow (light gray region, Figure b), and the interaction between fluidized zones and gas pathways for multiple injection sources (Figure c). Geophysical implications can then be discussed, with comparison to field data from live or fossil structures such as the Beauvoisin pockmark (France) or Cape Turnagain pipe field (New Zealand).

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#### Modelling the Liquid Droplet Impact Behaviour of Polymer Materials

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The ambitious goal of achieving net-zero and the current energy crisis have generated a strong demand for lightweight polymer materials, now extensively utilised in the transport and energy sectors. However, these materials are vulnerable to various extreme conditions, including dynamic impact loading and environmental ageing. Particularly, high-velocity liquid droplet impact can cause significant erosion damage on the leading edge of wind turbine blades (Figure 1a). It is imperative to develop a comprehensive understanding and computational models to predict the degradation of polymer materials under such extreme conditions.

This study introduces a multi-physics phase field model designed to forecast material degradation during liquid droplet impact. The model utilises the Arbitrary Lagrangian-Eulerian (ALE) technique in combination with a predefined multiphysics interface for Two-Phase Flow and Phase Field [1]. The ALE method captures the dynamics of the deforming geometry and evolving boundaries through a moving grid. The boundary between water and air is tracked using the phase-field method. Simultaneously, the deformable solid is represented using various constitutive models, including linear elastic, Von Mises plasticity, and continuum damage mechanics [2]. The computational model is then validated against experimental results [3], with a comparison of displacement, strain distribution, and strain wave history between modelling and experimentation (Figure 1b). Our model exhibits strong agreement with the experimental results. The plasticity model and fracture model also offer insights into the mechanisms of failure. This initial work establishes a novel approach to modelling the degradation of polymer materials under extreme conditions.



Figure 1: (a) Leading-edge erosion (b) Snapshots of X and Y displacement contours

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