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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

	Experiment title: **Impacting brittle materials using a designed fast indentation device**	**Experiment number:** **MA-2981**
Beamline: ID19	**Date of experiment:** from: 12 Sep 2016 to: 14 Sep 2016	**Date of report:** 25 Feb 2017 *Received at ESRF:*
Shifts: 6	**Local contact(s):** Dr. Alexander Rack and Dr. Margie Olbinado	

Names and affiliations of applicants (* indicates experimentalists):

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[1]University of Manchester, [2]Diamond Light Source.

Report:

Impacting brittle materials using a designed fast indentation device

Neil Bourne[1], Wajira Mirihanage[1], Christoph Rau[2]
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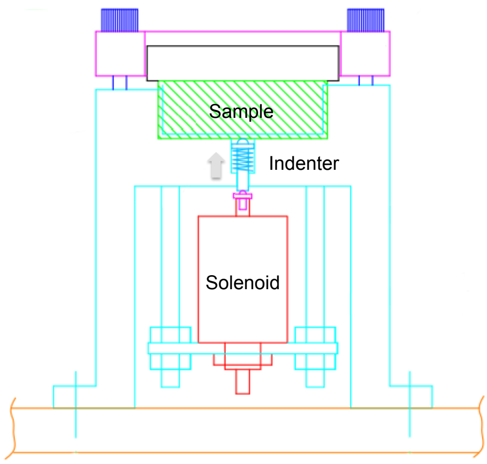

Fig. 1 *DMC impact rig.*

1. Abstract Material failure is determined by a suite of deformation mechanisms with differing kinetics operating together and presented an integrated response to an observer. To elucidate processes requires separating one from another in order to construct physically-based descriptions of behaviour[1]. Observing a material where failure processes are controlled by a designed impulse and are at a suitable scale offers the possibility of separating operating

mechanisms. A highly reproducible synchronised loading test frame has been developed by Diamond and Manchester. It was fielded at the ESRF in MA-2981 and shown to produce useful results on simple test problems (see Fig. 1). The device was designed to investigate failure in a range of materials and a vairety of classes of material were fielded in the experiments conducted. These included experiments to study the fracture of glass and consolidation in graphite with instrumented testing and high-speed radiography. Further we were able to contrast observed behaviours with experiments on single crystal silicon wafers as well as to look at quartz plates and their fracture in comparison with soda lime glass. The fielding of the experiment and the performance of the device, have shown that the excellent data gathered on failure may be extended in future beamtime with fundamental studies on shock consolidation. We wish to compress silicate particles in order to assess the possibility that addition of quantities of ice and salts might allow particulate material to attain a solid structure after impact. This would enable us to demonstrate consolidation of dust in the early solar system into *planetesimals* as hypothesised in several works[2,3].

2. Scientific context: The process defining the end of elastic behaviour in an amorphous glass is fracture. On exceeding the strength of the material, inelastic processes start when a crack is initiated at a flaw on the loaded surface, the tip travels at a speed determined by the stress level that accelerates to the Rayleigh wave speed in the material in the limiting case with the shear wave speed increasing with pressure.[4]

A regime exists in front of a driven indenter where a material can support different states (unfailed and failed) for a time dependent upon the speed at which the failure processes operate. An example is shown from a radiographic image taken at ESRF (Fig. 2). The indenter morphology determines the deformation zone extent and the degree of compaction within that absorbs the applied strain. Further we can control indenter material and geometry. This time is a few microseconds in the case of glass and was captured using single bunch imaging at ESRF for the first time. Glass shows the evolution of unsteady stress states through localisation, failure and compaction as the material accommodates strain and transits to the steady state. In application, delayed failure is a feature of such materials, and this plays a critical role in the operation of armour and protection in key components and vital structures[5].

Whilst it has been possible to measure stress states and wave speeds for these states using photography, it has never been previously possible to track density change and identify fracture morphology in real time. Thus we hope to field X ray and optical imaging to quantitatively define these states, observe fracture occurring in the glasses now that timing and impulse loading have been optimised, and then to extend to opaque brittle materials. We have shown capability exists to interrogate these phenomena, and we believe we now have the means to demonstrate for the first time quantitative 4D results using X ray imaging in brittle solids. Further experiments will allow us to consolidate knowledge of assumed mechanisms and advance analytical and numerical models for this class of brittle solids under load[6]. Further we wish to use the impactor to compress silicate grains with and without ice coatings and under vacuum to attempt to test these hypotheses using high-speed radiography and post-loading XRT to interrogate the compacted material.

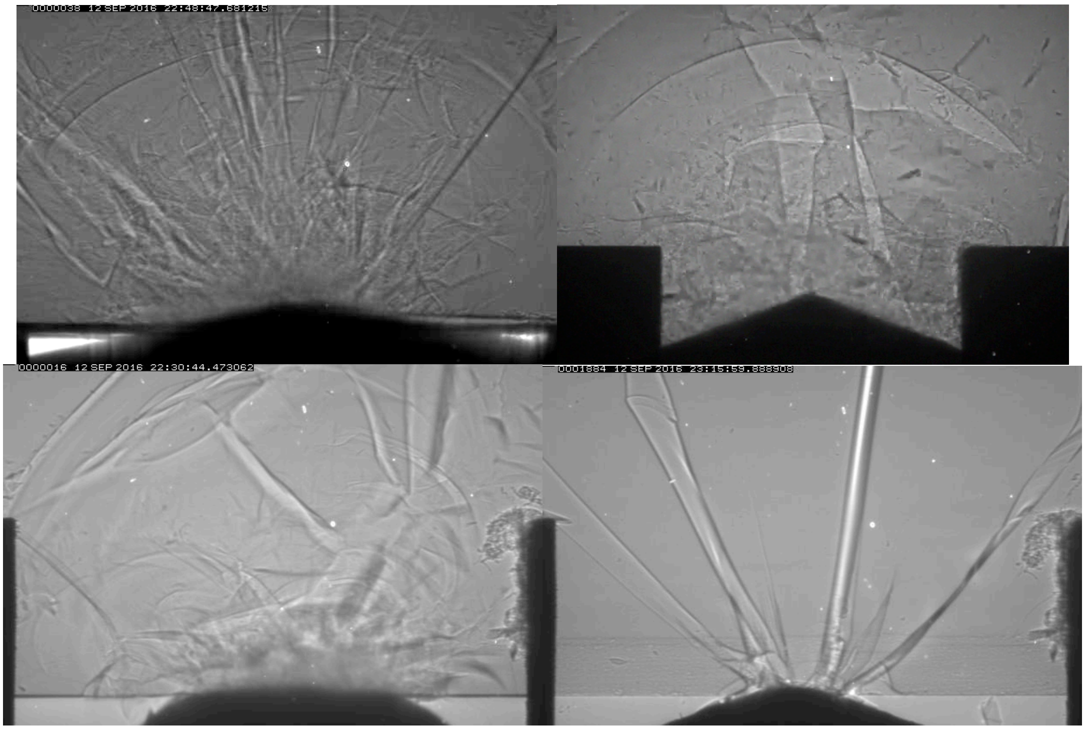

Fig. 2 *Impact and fracture in quartz and glass with different indentors and impact speeds.*

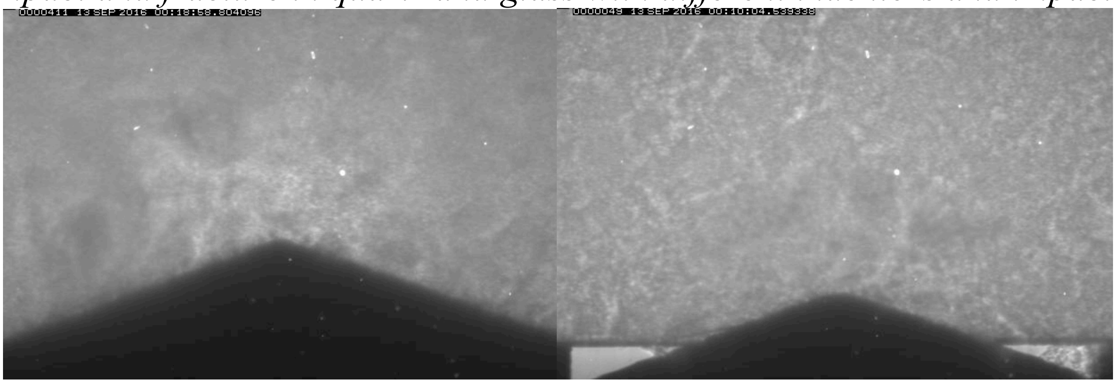

Fig. 3 *Impact and fracture in nuclear graphite with edge indentor.*

3. Experimental results We have investigated a brittle and a porous material indented using different tip morphologies to build up a picture of generalized failure in these material classes. Glass plates were loaded from a linear face to drive a divergent failure front out into the target. Frames from such sequences captured during the experiments on silica materials is shown in Fig. 2 above. The impulse was accurately matched to the shutter controlling transmission of X rays through the sample and to trigger imaging cameras and assembled stress field diagnostics. In other experiments nuclear carbon and graphite tiles were impacted and mixed mode fracture and compression was observed (Fig. 3). We hope to come back to ESRF with an additional impact device that will be constructed with a more powerful driving force to increase velocity and thus force new modes of failure within the sample. After the shot microstructure was characterised off line and this process is ongoing to field experiments in a future call.

Beamline(s) and beam time: The beam conditions on ID19 proved ideal for studying the low-density glasses and silicates investigated in this experiment. Without this information the linkage between loading conditions and the failed and retrieved resulting fragments is lost. Samples were mounted on beam line rotation stages and imaged with phase contrast transmission imaging using standard beam line protocols. Tomography and imaging with *ca.*

three micron resolution was conducted over the 6 shifts allocated but we shall look to apply for longer time allocations in future work now the technique has been proven.

The monochromatic XMT allowed us to extract microstructural changes relative to the pre-shocked state and shock conditions. These results identified (or allow development of) the key defect, failure and damage models appropriate to the materials tested. We have identified the needed for faster indentation (greater stress developed) to investigate and initiate these new failure modes. This will be the subject of our follow on proposal for future experiments. We will develop key protocols for future dynamic work at GHz imaging rates at ID19. The 3D mapping of phase and damage that will result will allow model construction and then stringent validation. The role of these processes remains a key area of debate in the field, and the project will yield high impact publications. We intend to use the data to support a suite of comprehensive studies for which further funding proposals are under development. With the fast imaging already conducted at ESRF, this experiment will be a *tour de force* coupling dynamic failure with quantitative X ray imaging to shine light upon a key unanswered problem in these fields.

References

1. Bourne, N. K., *Materials in Mechanical Extremes*, Cambridge: Cambridge University Press, (2013).
2. Goldreich, P., Ward, W. R., Astrophysical Journal, **183**: 1051, (1973).
3. Youdin, A.N., Physics and Astrophysics of Planetary Systems **41**, 187-207, (2010).
4. Millett, J. C. F., Bourne N. K., and Rosenberg, Z., J. Appl. Phys., **87**, 8457-8460, (2000).
5. Bourne, N. K., Millett, J. C. F., and Rosenberg, Z., Proc. R. Soc. Lond. A., **452**, pp. 1945-1951, (1996).
6. Bourne, N. K., Rosenberg Z., and Field J. E., Proc. R. Soc. Lond. A., **455**, 1267-1274, (1999).
7. Resnyansky, A. D., Romensky, F. I., and Bourne, N. K., J. Appl. Phys., **93**, 1537-1545, (2003).

Papers submitted

American Physical Society Shock Compression of Condensed Matter 2017 submitted

Impacting brittle materials using a designed fast indentation device

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Material failure is determined by a suite of deformation mechanisms with differing kinetics operating together and presented an integrated response to an observer. To elucidate processes requires separating one from another in order to construct physically-based descriptions of behaviour. Observing a material where failure processes are controlled by a designed impulse and are at a suitable scale offers the possibility of separating operating mechanisms. A highly reproducible synchronised loading test frame has been developed by Diamond and Manchester. It has already been fielded at the ESRF and shown useful results using ultra-high speed single bunch image mode on simple test problems. Now that the device has been proven, we show studies on the compression and fracture of glass and quartz. The results indicate several modes of failure within the targets and emphasise the need for further fast radiography to elucidate failure mechanisms in solids.

* work carried out at ESRF beamline ID19.