Impacting Silicates Using a Fast Indentation Device

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Abstract. Material failure is determined by a suite of deformation mechanisms with differing kinetics, operating together to present 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, in which failure processes are controlled by a designed impulse and are at a suitable scale, offers the possibility of separating operating mechanisms. A highly synchronised, loading test frame has been developed by Diamond and Manchester. It has already been fielded at the ESRF and shown excellent results using ultra-high speed, single bunch imaging 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 and emphasise the need for further fast radiography to elucidate failure mechanisms in solids.

INTRODUCTION

The process defining the end of elastic behaviour in silicates, typically glasses, is fracture. On exceeding the strength of the material, inelastic processes start when fracture is initiated at a flaw on the loaded surface. A crack then propagates with the tip travelling at a speed determined by the stress level. This then accelerates to the Rayleigh wave speed in the material in the limiting case, since the shear wave speed increases with pressure [1].

A regime exists ahead of a driven indenter where a material can support different states (unfailed and failed) for a time dependent upon the speed at which failure processes operate. The indenter morphology determines the deformation zone extent, and the degree of compaction of fragmented material within that absorbs the applied strain. Further, we can control indenter material and geometry to control the nature of the compact. This time is a few tens of microseconds in the case of glass and these processes were captured with radiography using single bunch imaging at the European Synchrotron Radiation Facility (ESRF) for the first time. Fast imaging at MHz allows us to observe the penetration of an indenter, and the compaction of the fragmented material, to capture these processes in this experiment. Glass shows the evolution of unsteady stress states through localisation, failure and compaction as the material accommodates strain and transits to a steady but often metastable state. In application, delayed failure is a feature of such materials, and this plays a critical role in the operation of armour and protection for key components and vital structures [2, 3].

Whilst it has been possible to measure stress states and wave speeds for these states using photography, we have never previously been able to track density change and identify fracture morphology in real time. Thus we have fielded X-ray and optical imaging to quantitatively define these states, observe fracture occurring in the glasses and then to extend to opaque brittle materials. In this work we present radiographic framing sequences with reconstructed streak of these radiographs down an impact axis. We have shown that capability exists to interrogate these phenomena, and we now present quantitative 4D results using X-ray imaging in silicates. This couples understanding of materials’ physics with characterisation of the geometry and kinetics of failure across regimes. We present an overview of the operating mechanisms and suggest analytical and numerical models that may be improved using this data for this class of brittle solids under load [4].
EXPERIMENTAL

Fast indenter

A precise and portable indentation device was designed to investigate failure in range of materials and material classes. The means of operation was a commercially available electrical solenoid (Ledex® Low Profile 6SFM) which delivers a force of approximately 900 N to a projecting indenter and allows it to reach velocities up to 10 m s\(^{-1}\). The solenoid shaft was assembled with the freely moving indenter tip inside the assembly shown in Figure 1. The sample stage of indenter device allows the positioning of samples of interest with rectangular cross sections with a range of thickness varies from 0.5 mm – 15 mm within a space that allows other initial conditions to be varied in a controlled manner. Two open sides allow the X-ray beam to pass uninterrupted through the target to collect transmission images with an adequate field of view. There is additional space to allow optical high speed photography of the target under load.

![Schematic set-up of the portable indenter.](image)

**FIGURE 1.** Schematic set-up of the portable indenter.

Single Bunch X-ray Imaging

The experiments were carried out at ESRF ID19 beam line while the ESRF ring was operated in a dedicated 4 bunch machine operation. Under this operating mode four electron bunches were circulated in the storage ring with a current of ca. 10 mA per bunch. The measured bunch length was 140 ps (FWHM) [5, 6]. The revolution frequency of a single bunch is determined by the storage-ring circumference and is calculated to be ca. 2.8 µs for these experiments. For such single-bunch imaging experiments at ID19, the beam line was operating two U32 undulators in series with ca. 11 mm gap [7].

The usable polychromatic X-ray energy spectrum was largely confined to the energy range between 20 to 50 keV with a mean energy of approximately 30 keV. Imaging was adjusted so that phase contrast allowed cracks and their propagation to be captured and the imaging detector was therefore placed ca. 10 m distance from the experiment. The imaging detector configuration consisted of a 250 µm thick, LuAG scintillator coupled to either Shimadzu HPV-X2 cameras with 1000 x 1000 pixels or a pco.dimax camera. The Shimadzu HPV-X2 camera was used only for single bunch imaging while the pco.dimax was used to capture imaging at the slower speeds, capturing longer time windows. In the latter case, photons corresponding to more than one X-ray bunch were integrated to produce a single exposure. The Shimadzu HPV-X2 camera captured 128, 10 bit greyscale images, allowing fast and continuous image capture without any data transfer problems. The pco.dimax camera was used to capture images at 14.5 kHz frame rate with 400 x 250 pixels per image. The coupling optics and magnification allowed effective spatial resolutions of 11 µm and 8 µm for the pco.dimax and the Shimadzu cameras respectively.

One of the critical aspects of in situ single bunch imaging configuration was to capture indentation and crack propagation to take place with the camera’s recording window. This required the camera aperture to be temporally synchronized with the X-ray flashes from the electron bunches after their travel through the two U32 undulators. Thus the initial triggering signal was passed to the indenter and a proximity sensor detected the position (accuracy ±20 µm) of the shaft sending a 5 V DC pulse to a digital delay generator to trigger image capture in synchronization with the X-ray flashes. In this mode, images were acquired every 1.404 µs and camera aperture was controlled to have a single frame for each X-ray bunch.
MATERIALS

Experiments were conducted on two filled glasses and on z-cut quartz. Shock and materials’ data for the three materials are given in Table 1.

**TABLE 1.** Materials data for the three silicates.

<table>
<thead>
<tr>
<th>Material</th>
<th>Borosilicate (BS)</th>
<th>Quartz</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$ (±0.05 g cm$^{-3}$)</td>
<td>2.23</td>
<td>2.65</td>
</tr>
<tr>
<td>$E$ (GPa)</td>
<td>73.1</td>
<td>97.2</td>
</tr>
<tr>
<td>$\mu$ (GPa)</td>
<td>30.4</td>
<td>31.1</td>
</tr>
<tr>
<td>Poisson’s ratio, $\nu$</td>
<td>0.20</td>
<td>0.15</td>
</tr>
<tr>
<td>$c_L$ (±0.01 mm µs$^{-1}$)</td>
<td>5.56</td>
<td>3.49</td>
</tr>
<tr>
<td>$c_S$ (±0.01 mm µs$^{-1}$)</td>
<td>3.45</td>
<td>2.02</td>
</tr>
<tr>
<td>$K_{IC}$ (MPa m$^{1/2}$)</td>
<td>0.8</td>
<td>0.66</td>
</tr>
<tr>
<td>HEL (±0.5 GPa)</td>
<td>4.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

In all cases the impactor chosen was made from a hardened martensitic steel (440A). Even after a number of impacts, the impactor tip did not show recognisable wear. However, to ensure repeatability and point loading the tip was routinely replaced after every 6-8 impacts.

RESULTS

In this experimental campaign, we observed defect activation and fracture occurring *in situ* using X-ray phase contrast imaging. For fast imaging experiments on these brittle materials (for properties see Table 1), different tip morphologies were used to build up a picture of the micromechanics controlling failure in differing classes of material. Brittle glass targets were loaded from the flat lower face (in the sequences presented below) to drive a divergent failure front out into the sample. This illustrated contrasting behaviours in the amorphous glasses (soda lime and borosilicate) and within crystalline quartz.

**FIGURE 2.** Impact onto borosilicate (L) and quartz (R) targets with flat-faced indenters. A streak image through a vertical axis through the impact point is shown beneath each sequence. Interframe time of *ca.* 68 µs.
Figure 2 shows the impact of a flat impactor onto borosilicate and quartz targets. The image show frames taken from the sequence of radiographs, recorded with phase contrast filtering to highlight fracture within the materials. Each is numbered left to right; top to bottom with an interframe time of ca. 68 µs. Below each sequence is a streak image for each impact, with the dark regions showing the impactor and the image to the right in grey showing damage accruing down the central axis of the target. The flat impactor used in this test fails the borosilicate tile uniformly and penetrates the fragmented material. In the case of the quartz, there are isolated cracks from a flaw on the surface. The magnetic field of the impactor’s coils can be controlled to allow further impacts to occur and in this case the impactor tip returned after the first impact and accrues a second level of damage (see image to the right).

Figure 3 shows a sequence for the impact of a triangular prismatic indenter. In the first impact onto quartz, the impactor propagates four symmetrical cracks with 36˚ between segments along different principal shear planes. The rebound and second impact of the impactor rebounds for the quartz sample inducing limited isolated cracks, while the second impact opens these fragments further, isolating fragments whilst absorbing impact energy. In the case of the borosilicate, uniform damage is activated from the impact point and a cylindrical front can be seen propagating from here. The associated streak image indicates penetration of the fragments by the decelerating indenter which is then brought stationary within the compacted fragments. During the experimental campaign, experiments were repeated with same materials, indenter types and other parameters. Comparison of those image sequences has shown clear, repeatable results found over single and multiple impacts.

**FIGURE 3.** Impact of a triangular prismatic indenter. BS sequence to right and quartz to left. Conditions as in Figure 2. Interframe time of ca. 68 µs.

Representative experimental sequences for the spherical shape indenters are shown in Figure 4. In this case, the borosilicate fails in expanding fronts that are clearly visible and arrest the impactor again. However this geometry allowed further penetration of the indenter and it was slowed rather than halted as seen in the case of the prismatic tip. As previously, there is less damage in the quartz case, whilst with borosilicate there is a greater region of damaged and more limited arrest of the indenter. Systematic analysis is currently underway with the rich data set collected and with a large number of image sequences in this experimental campaign. However is clear that failure is accelerated in the weaker materials and there is ongoing work to quantitatively identify the strength and defect properties of the silicates that gives rise to these behaviours.

Combining fast *in situ* imaging data with *ex situ* X-ray tomographic reconstructions will allow us to extract microstructural changes relative to the pre-shocked state. These results will identify (or allow development of) the key defect, failure and damage models appropriate to the formation of these microstructures. This represents a key step forward in assessment of the controlling mechanisms that drive planetary damage and informs debates on the formation of planetary bodies through accretion. Further, we shall develop key protocols for future dynamic work at GHz imaging rates at ID19 and collaborative work at DLS and DCS at APS (where smaller sample volumes will be
used for simultaneous imaging and diffraction studies). Combining work packages in this way means we may be able to assess the full effect of scaling on materials response. This provides us with the first critical results to allow us to constrain microstructural evolution in response to stress. 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.

**FIGURE 4.** Impact of a spherical indenter. BS radiographic sequence to right and that for quartz to left. Interframe time of ca. 68 µs.

## CONCLUSIONS

A novel impact device has been designed and built to conduct *in situ* fast X-ray radiographic experiments. From the preliminary results, the device built has proven to give excellent results. X-ray imaging was improved using phase contrast and this technique allowed us to observe crack propagation and identify the evolution of fracture morphology in real time. Full data sets are currently being analysed in detail and further experiments are planned to consolidate knowledge of assumed mechanisms and advance analytical and numerical models for this class of brittle solids under load [8]. 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 has been a *tour de force* coupling dynamic failure with quantitative X-ray imaging to shine light upon a key unanswered problem in these fields.

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## REFERENCES