In situ damage assessment using synchrotron X-rays in materials loaded by a Hopkinson bar

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Split Hopkinson or Kolsky bars are common high-rate characterization tools for dynamic mechanical behaviour of materials. Stress–strain responses averaged over specimen volume are obtained as a function of strain rate. Specimen deformation histories can be monitored by high-speed imaging on the surface. It has not been possible to track the damage initiation and evolution during the dynamic deformation inside specimens except for a few transparent materials. In this study, we integrated Hopkinson compression/tension bars with high-speed X-ray imaging capabilities. The damage history in a dynamically deforming specimen was monitored in situ using synchrotron radiation via X-ray phase contrast imaging. The effectiveness of the novel union between these two powerful techniques, which opens a new angle for data acquisition in dynamic experiments, is demonstrated by a series of dynamic experiments on a variety of material systems, including particle interaction in granular materials, glass impact cracking, single crystal silicon tensile failure and ligament–bone junction damage.


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1. Introduction

After John Hopkinson [1] explored tensile wave propagation in an iron wire in 1872, Bertram Hopkinson in 1914 invented a pressure bar to measure the pressure produced by high explosives or high-speed bullet impact [2]. Kolsky in 1949 used the principle of wave propagation in elastic bars and developed an experimental method for the characterization of dynamic material properties, known as split Hopkinson pressure bar or Kolsky bar [3]. Since its introduction, this method has been continuously improved and widely used in characterizing various types of engineering materials. For example, Krafft et al. used a gas gun to generate a stress wave instead of the explosive used by Kolsky [4]. Hauser et al. [5] used a velocity generator to successfully produce a stress wave with constant amplitude. Lindholm incorporated most of the previous improvements and presented an updated version of the Kolsky bar for dynamic characterization, which became the most widely used version for decades [6–9]. This technique was also expanded to tension [10–12], torsion [13,14], tri-axial compression [15–17] and tension/compression–shear [18–20] versions. To subject a specimen to desired testing conditions and to recover the specimen after a well-defined loading history, pulse-shaping and momentum-trapping techniques were introduced [21–23]. These modified and extended versions of the Kolsky bar are very effective to obtain stress–strain response of various materials averaged over the specimen volume at various strain rates up to $10^4$ per second.

Recent developments in multi-scale modelling of material behaviour have led to a desire for more detailed deformation information in the specimen rather than just the average strain over the specimen length. Digital image correlation has been successfully integrated with Kolsky bar experiments to record the dynamic evolution of the full-field strain on the surface of the specimen [24]. For transparent materials, the damage history inside the specimen can be recorded by a high-speed camera [25,26]. However, when the specimen is optically opaque, or the transparency is destroyed by damage, the detailed deformation distribution or damage evolution inside the specimen is no longer visible. To record and explore the internal damage histories in such specimens, X-rays are a natural choice to image through the specimen thickness.

X-rays have been widely used in medicine and security applications to view objects inside covers. However, to introduce X-rays into Kolsky bar experiments for real-time in situ measurements with sufficient spatial and temporal resolutions, there are two specific requirements that must be addressed owing to the high-rate deformation and low-density contrast within one material. The high-rate deformation demands high intensity from the X-rays. In most conventional applications such as medical radiographs or airport security screening, an X-ray image process can take seconds of time. As the duration in Kolsky bar experiments is typically only a fraction of a millisecond, the intensity of the X-rays to be used in Kolsky bar experiments will need to be orders of magnitude higher. To reach this level of intensity, one may use a flash X-ray for very short exposures or a very bright continuous X-ray source. Flash X-ray can capture only an X-ray image at one or a few specific instants. In order to track damage development processes inside specimens, we used continuous X-rays generated by synchrotron radiation at the Advanced Photon Source (APS) at Argonne National Laboratory in the USA. In conventional radiographing, the density difference between the object to be observed and the background is typically very large, e.g. bones under soft tissues and metal objects under clothes. These significant density differences facilitate radiographs to have high resolution and contrast. In a specimen of an engineering material under Kolsky bar loading, such drastic density difference does not exist, even in multi-phase composite materials. Proper experimental techniques must be introduced to enhance the contrast in the resultant images. In this study, we used X-ray phase contrast imaging (PCI) to enhance the interfaces between material phases or features inside the specimen on the imaging plane [27].

We integrated the dynamic loading capability from Kolsky bars with the high-speed, high-intensity X-ray imaging capability from synchrotron X-ray PCI to reveal dynamic damage evolution inside a specimen deforming at high rates, which was briefly reported earlier [28]. The experimental technique has further evolved. This paper introduces the experimental
techniques in detail and presents the application of the new experimental method in the dynamic characterization of various material systems. The schematic of the experimental set-up under discussion is shown in figure 1, with a representative sample in the testing section. Kolsky tension and compression bars were specifically designed and constructed to fit in the X-ray hutch of APS beamline 32-ID-B where the high-speed X-ray imaging capability resides. Owing to the space limitation inside the X-ray hutch, the transmission bars had to be replaced by load cells. So, we actually used the original Hopkinson bars, rather than the split version, in the experiments reported in this paper. The bars were operated remotely from a control room to apply a controlled dynamic loading on the specimens. The high-speed images of the damage evolution inside the specimens were obtained by passing the high-intensity X-ray beam through the specimens onto a scintillator, which converts X-rays to visible light, and then recorded using a high-speed camera after magnification.

In the following sections, we first introduce the synchrotron X-rays, PCI, and their unique features that are required in Hopkinson bar experiments. Then, we describe the Hopkinson bars designed and used for the integration of this high-rate loading method with high-speed, high-energy X-rays. The integration of these two powerful experimental methods, including the synchronization among the set-ups and the instrumentations, is then presented. The resultant
new experimental technique was used to conduct dynamic experiments on brittle particles, single crystal silicon, glass and a biological tissue. The experiments and results for each material, together with the unique capabilities of the integrated Hopkinson bar/X-ray PCI technique, are presented and discussed.

2. Synchrotron X-rays at the Advanced Photon Source

To continuously track the dynamic deformation history and damage evolution in the specimens, we used the synchrotron radiation source available at APS in Argonne National Laboratory. APS provides X-ray beams with sufficiently high intensity levels that are necessary to image high-speed events created by Hopkinson bar loading. The third-generation X-ray sources have high photon energy, high photon fluxes, high coherency and high pulse repetition rates. Ultrafast, multi-frame, single-pulse imaging with high temporal (less than 100 ps) and spatial (approx. 2 \( \mu \)m) resolutions can be generated for capturing the dynamic events produced by impact loading from Hopkinson bars or shockwaves. Owing to the highly transient nature of shockwave experiments, even with the intensity level available at APS, it can still be quite challenging to obtain a series of X-ray images which track microstructural evolution during shock loading [27]. However, Hopkinson bar experiments have durations one to two orders of magnitude higher than shock experiments. Thus, this high-rate X-ray diagnostic method is much more adept at tracking microstructural or damage evolutions inside the specimens loaded in the strain rate regime produced by Hopkinson bar loading.

In the experiments reported in this paper, X-ray beam measurements were performed at the APS beamline 32ID-B. The X-ray beam used an APS undulator A with a period of 3.3 cm and length of 2.4 m and is capable of providing white or monochromatic X-rays. To maximize the number of photons in the X-ray pulses, we selected the white beam option. The undulator source size is roughly 0.260 mm wide and 0.013 mm high. The specimen is located approximately 40 m away from the undulator light source. The beam cross section has an elongated two-dimensional Gaussian shape. The beam spot size at the half maximum beam size at 10 keV is 1.3 mm wide and 0.6 mm high [29]. The actual beam spot size on the specimen can be controlled using adjustable slits in both horizontal and vertical directions. The window size used in the experiments reported in this paper was 1 mm wide and 0.7 mm high.

The spectrum characteristics of the X-ray beam are varied via altering the undulator gap [27], which was typically between 20 and 30 mm for the Hopkinson bar experiments. When the gap is increased from 20 to 30 mm, the fundamental (first harmonic) shifts from 9 to 13 keV, with peak broadening [27], which is accompanied by a decrease in photon number by a factor of three [27]. The fundamental of the X-rays used in our experiments was centred at roughly 9 keV, corresponding to an undulator gap of about 20 mm. The photon flux through a 1 \( \times \) 1 mm pinhole located 35 m away from the source owing to this fundamental is roughly $1.9 \times 10^{16}$ ph s\(^{-1}\). As a reference, the photon flux for the full energy range (5–100 keV) is $2.4 \times 10^{16}$ ph s\(^{-1}\).

The time structure of X-ray pulses or the corresponding electron bunches depends on the operation modes of the APS electron storage ring. As schematically shown in figure 1, the pulse train is circular. It takes 3.68 \( \mu \)s for the electrons to travel one revolution around the circular ring. The number of photons in an X-ray pulse, i.e. the intensity of the X-rays, scales linearly with the bunch current [27] (http://www.aps.anl.gov/accelerator_systems_division/accelerator_operations.physics/srparameters/node5.html). The bunch separation can be varied from 2.84 ns to the microsecond time scale. The nature of dynamic loading and duration of the experimental event directly determine the necessary recording frame rate and also the total number of required frames to properly investigate the phenomenon of interest. This then sets the necessary X-ray flux and timing structure of the synchrotron pulse along with placing restrictions on the imaging system, such as frame exposure time and frame rate. Owing to the duration of the Hopkinson bar experiments, which is of the order of 100 \( \mu \)s, a temporal resolution of 100 ns to 10 \( \mu \)s is acceptable, which refers to the X-ray integration time for a single frame, thus allowing for the aforementioned white beam X-ray selection.
In the design of experiments probing a dynamic phenomenon, it is important to consider the current of the bunch/superbunch (number of photons), the bunch width (temporal resolution) and bunch separation (frame rate ceiling). The dynamic event must be synchronized with these X-ray characteristics along with detectors, in order to properly capture the attributes possessed by the phenomenon of interest. As previously described, for the Hopkinson bar experiments, of the various fill modes offered by APS, the hybrid fill mode was deemed most appropriate. In this run mode, a single electron bunch containing 16 mA is isolated from the remaining bunches with a 1.594 μs gap. The remaining eight groups of seven consecutive bunches (septuplets), which have a time gap of 51 ns, possess a maximum of 11 mA per group and a periodicity of 68 ns [30]. This mode is denoted as ‘1 + 8 × 7’, where ‘1’ stands for the singlet and ‘8 × 7’ stands for the eight groups of septuplets, thereby being referred to as a superbunch. Hopkinson bar experiments can be performed using either the singlet or the superbunch mode, with the former possessing a less than 100 ps pulse width, 3.68 μs bunch spacing and bunch current of 16 mA, whereas the latter possesses values of 500 ns, 3.68 μs and 88 mA, respectively. This is opposed to the standard 24-bunch mode, which possesses values of less than 100 ps, 153 ns and 4.25 mA, respectively. It was decided to use the superbunch from the hybrid fill mode for the experiments reported in this paper, which inherently decreases the temporal resolution. However, owing to the long experimental duration (approx. 100 μs), this was deemed appropriate owing to utilization of the superbunch drastically increasing X-ray brightness.

Owing to the small spot size of the X-ray beam, it is necessary to magnify the X-ray images. A single crystal scintillator was used to convert the X-ray signal to visible light, which can then be magnified and recorded by a high-speed optical camera. The scintillator was Lu₃Al₅O₁₂:Ce or LuAG:Ce (Crytur Ltd, Czech Republic) with dimensions of 10 × 10 × 0.1 mm, a decay time of 45–55 ns, and an emission spectrum peak of 530 nm [27,31]. This decay time, along with the short duration of exposure to the superbunch (500 ns), was much shorter than the frame separation (n × 3.68 μs). For the experiments described in this work, n ranged from 1 to 5. Thus, the ghosting effect [27] owing to exposure on the preceding frame was deemed negligible in the current scheme. A Photron Fastcam SA1.1 with a 12-bit CMOS sensor was used to capture the high-speed images through a 45° mirror, 10 × microscope objective and a tube lens; this allowed variation of frame rate and exposure time with relative ease. The full frame size of this sensor was 1024 × 1024 pixels, with each pixel being 20 μm in size. The frame rate could be varied from 5400 to 675 000 frames per second (fps), with reduced image size at increasing frame rates owing to information transfer limitations, e.g. 1024 × 1024 pixels at 5400 fps, 320 × 128 pixels at 100 000 fps, 128 × 64 pixels at 300 000 fps and 64 × 16 pixels at 675 000 fps. Images were stored in on-board memory allowing for a total recording time of at least 1 s. The camera could be phase-locked or synchronized to an external source and also triggered by an external transistor–transistor logic (TTL) signal.

3. X-ray phase contrast imaging

To enhance the contrast among various phases inside the specimen material on the X-ray image, we used the method of PCI, which uses the differences in the change of X-ray phases as they pass through the sample to enhance contrast in the resultant images. A material exhibits a complex index of refraction for X-rays, \( n = (1 - \delta) + i\beta \), with \( \delta \) and \( \beta \) representing phase shift and absorption, respectively. For contact image radiography or absorption imaging, the sample–detector distance (\( z \)) equals zero, and the contrast on the resultant image comes from the absorption of X-rays by different phases of the specimen material. For propagation-based PCI, \( z \) is a finite number within the near-field Fresnel region, with the Fresnel number being greater than or equal to 1. If the phase object is heterogeneous and \( \beta \) is small, then X-ray phase spatial variations are induced, \( \gamma = \gamma(x, y, z) \), along with an induced local curvature to the transmitted wavefront. Furthermore, during the propagation from sample to detector, the wavefront undergoes interference and overlap which thereby adjusts the intensity [32,33]. This variation in wavefront intensity owing to the propagation effects is proportional to the Laplacian of \( \gamma = \gamma(x, y, z = 0) \), thus generating edge enhancement [32,34]. This is the main advantage of
using the PCI technique to resolve structural alterations and inhomogeneities in low atomic number materials, which are inherently difficult to analyse with contact image radiography. Furthermore, X-ray PCI possesses the ability to effectively penetrate through low atomic number materials with high levels of spatial resolution, thereby allowing one to look through a material or structure rather than solely analysing the surface of a specimen, which is commonly the case for imaging analysis during dynamic events of non-light transparent materials. X-ray PCI also avoids the high scattering of visible light encountered in conventional imaging methods.

In principle, PCI requires spatially and spectrally coherent X-rays. However, the spectral coherence requirement can be relaxed thus enabling the use of white X-ray beams [35], thereby allowing the use of multiple harmonic X-rays. Thus, dynamic PCI is possible in the presence of highly coherent, and high flux white beam X-rays which are produced with a great degree of repetition from synchrotron undulator sources. Our experiments are carried out in the intermediate (Fresnel) regime (short imaging distances), where the contrast is mainly due to absorption and ‘edge enhancement’ effects. In this regime, the edge enhancement contrast (to first order) is proportional to the Laplacian of the projected electron density of the sample, and is independent of the X-ray wavelength [35]. Quantitative phase extraction can, indeed, be performed with a polychromatic beam under some assumptions, in the case of homogeneous and weakly absorbing objects. However, images in the edge-enhancement regime are straightforward to understand and analyse (secondary fringes not visible, especially with polychromatic beams). Quantitative phase retrieval is not necessary in this study, especially in that we do not do tomographic (three-dimensional) reconstructions. The processes described in this paper are too fast for computed tomography scans.

4. Hopkins tension and compression bar for X-ray hutch

The X-ray hutch at APS is not accessible when the X-rays are on. In addition, the physical space of the hutch is limited. We designed and constructed a set of modified Hopkinson bars to perform a variety of experiments in both tension and compression. The Hopkinson bars are oriented perpendicularly to the X-rays, as illustrated in figure 1. The distance from the X-ray beam to the hutch door is approximately 3 m. In the opposite direction, the distance from the X-ray beam to the hutch wall opposite the door is about 0.8 m. Owing to the space limitation, these Hopkinson bars were designed to use a load cell in place of transmission bars. However, the space limitation does not necessarily compromise the experimental results. This design was pursued under the premise that because the X-ray window is roughly 1 mm square as described earlier, the specimens are small in dimensions, ensuring a very large impedance mismatch between bar and specimen. Under this condition, the amplitude of the transmitted pulse would be very low. Using a load cell is an effective approach to record the dynamic loading history in the specimen.

Both the tension and compression bars operate from a unified remote-control system. However, each bar has an incident bar with a set of semiconductor strain gauges on the surface, load cell as the transmission bar, striker and gas-gun barrel. Gas pressure regulation comes in the form of an operating panel (with pushbuttons), to allow both attended and unattended operation of the gas system, which can be connected to only a single Hopkinson bar at a time. Data acquisition is handled in a similar manner, where a fast acquisition oscilloscope can be armed, monitored and triggered from a remote location. Once the experiment has finished, data can be saved and reviewed without entering the X-ray hutch.

The two Hopkinson bars shared the same gas tank, firing mechanism and baseplate assembly. The firing system consisted of a zero-pass solid-state relay, which was triggered by a 3 V input DC, thereby allowing a 120 V AC to flow into a fast-response solenoid valve. This valve was located close to the inlet nozzle of each barrel assembly, allowing for the minimal ramp-up pressure needed to ensure consistent delay timing from solenoid trigger to striker-incident bar impact. Timing differences between solenoid valve opening and strain gauge activation were obtained
as a function of air pressure in preparation experiments before the X-ray beam was turned on, thereby allowing for the necessary accurate determination of the timing sequence in dynamic measurements.

Selection of load cell capacity was carefully chosen for each experimental scheme in order to provide appreciable excitation during the loading history, while simultaneously ensuring prevention of response overload. The output from the strain gauge/Wheatstone bridge assembly was amplified by a differential preamplifier possessing a 100 kHz bandwidth. Both load cell and strain gauge voltage signals were concurrently collected with an oscilloscope. These signals were further synchronized with the PCI images in efforts to correlate visual material deformation history with the recorded loading and bar velocity response. It is also important to note that the strain gauge signal was further used to both trigger a PCI camera and determine the bar-end velocity history undergone during the impact event.

The 12.7 mm diameter tension bar (figure 2), constructed from 7075-T651 aluminium alloy, is approximately 2.15 m long, providing sufficiently long loading time during a single loading to perform both tension and fibre-pullout type of experiments. As with any Hopkinson bar, alignment is critical. While alignment of the bar is straightforward, the relative size of the small specimens, such as a single-crystal silicon specimen (4 µm thick), may result in undesired sample

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**Figure 2.** Hopkinson tension and compression bar set-up perspective drawing (a) and a photo taken inside the X-ray hutch (b). (Online version in colour.)
motion, should misalignment with the bar axis be present. Hence, the load cell was affixed to a series of stages that provide four degrees of freedom for alignment: \(X, Y\) and \(Z\) translation as well as rotation about the \(Z\)-axis. The ability to adjust the position of the sample is useful not only for alignment of small samples, but also for alignment of samples with irregular and inconsistent geometries, such as natural materials or biological tissues, providing a means to accept a wide range of sample lengths and grip geometries.

The striker, a brass tube, being interchangeable with different lengths and thicknesses, allows a multitude of stress-wave intensities and durations to be achieved. For Hopkinson bars with large impedance mismatch, strain-rate constancy is a simple matter. Very little pulse shaping is required, and as such, a thin piece of foam-like material will provide a quick ramp up to a long, flat plateau in the loading signal.

Contrary to the tension bar, the compression bar, also 12.7 mm in diameter, is designed for much stronger materials, and as such, is constructed from a high-strength steel alloy. While the same principle regarding impedance mismatch holds true for this design, alignment is not as critical as sample geometries are more easily controlled. In addition, with keeping in mind that high-strength materials such as ceramics and glass require large forces to induce failure, a solid backstop is needed for this application to ensure minimal load cell deflection instigated from the firing mechanism.

In comparison with the tension bar, the compression bar is only 1.7 m long, because all of the materials so far explored require only small strains to cause failure. However, to achieve large stresses, the compression bar striker must travel faster than is necessary for the tension bar. In this case, the barrel is 1 m long and fires a 0.3 m striker up to 12 m s\(^{-1}\). Because the load cell occupies the typical location of the transmission bar, isolating the stress wave in a trap bar becomes a non-trivial matter. In this design, the trap bar is in the form of a tube near the striking face. Similar to the tension bar, the compression bar has a flange, but works in reverse. After the striker impacts the incident bar, the stress wave runs its course and returns to the striking end. At this point, the striking face experiences the second pass of the stress wave. In the preparation of the experiment, a predetermined gap was set between the flange and the tube. After the passage of the first compressive pulse, the flange will come into contact with the trap bar, resulting in a majority of the stress amplitude in the second and further reflected pulses being removed from the incident bar [23].

When the compression bar is used to characterize softer materials, such as foam and rubber, achieving constant strain rate is trivial due to the large impedance mismatch. However, for brittle materials, a ramp pulse is often desired, but is easily generated with any moderately thick pulse shaper, the behaviour of which can be quantitatively modelled [22] in the design phase of the experiments.

Within the X-ray hutch, the beam operates at a fixed height (depending on mode of operation). While this provides stability for the imaging system, the Hopkinson bars must be moved to bring the sample into the X-ray viewport. By mounting the tension and compression bars on a single table, experiments can be conducted on either set-up by switching the gas and data acquisition systems to the desired Hopkinson bar. Even though the sample location relative to the X-ray beam changes with the type of experiment, grip geometry and sample geometry, the point of interest can always be moved into the X-ray viewport. This is achieved using a 200 mm horizontal actuator, powered by a high-resolution stepper motor. Despite the ability of the equipment to move with high precision, the spatial measuring systems provide information in increments of 10 \(\mu\)m, which in itself still provides a suitable level of accuracy for positioning the sample during experiments inside the X-ray hutch.

In conjunction with the horizontal actuator, a set of three vertical actuators provides vertical motion. By working together, the entire system can be positioned with the same level of accuracy as the horizontal actuator, but in addition, also provides a means to level the surface on which the Hopkinson bars are mounted. Once the bars are at the same height as the X-ray beam, most of the experiments require only the use of a single actuator (located underneath the sample section) to reposition the sample by up to a few hundred micrometres in either direction. In
situations where the sample must move vertically by a larger distance, all of the actuators must be used in order to avoid damages caused by side-loading induced by the system being tilted. Thus, the entire Hopkinson bar assembly is effectively used as a three-axis stage, thereby allowing for proper location of the sample within the X-ray beam.

5. Integration of Hopkinson bar loading and X-ray diagnostics

A schematic of the synchrotron/imaging and Hopkinson bar assemblies is shown in figure 1. The X-ray source, slits, shutters, Hopkinson bar, scintillator and detector system are all represented in efforts to depict the entire assemblage in a simplistic manner. Owing to the brightness of the source, an upstream shutter system is used in order to minimize damage to the sample and optical equipment. As can be seen in figure 1, the Hopkinson bar apparatus is placed at a 90° orientation with respect to the beam pathline in a location that allows for the sample to be exposed to the X-ray beam. The sample is placed in the X-ray beam path, between the bar end and a load cell (force transducer). As previously described, the entire bar system is movable on a micrometre-scale ‘stage’, thereby allowing for proper bar location within the beamline path. During the dynamic loading process undergone by the sample owing to the impinging Hopkinson bar end movement, X-rays passing through the two-dimensional slits are allowed to travel down the beamline path via opening of the slow shutters. The X-rays are then able to penetrate through the dynamically loaded sample and are then converted to visible light via the downstream scintillator, being located 18 and 23 cm away from the axes of the tension bar and compression bar, respectively. The visible light is then reflected by a 45° mirror through either a 5× or 10× microscope lens, and then ultimately passes into the aperture of the high-speed camera. Depending on the selection of the magnification lens within the optical assembly, the spatial resolution is 2 or 4 μm [27]. The scintillator and optical components are located in a solid aluminium casing, which can be moved remotely in order to achieve high levels of resolution during beam flow, whereas the entire imaging assemblage is placed on a moving optical table, thereby allowing for proper location of the system within the beamline path.

Intrinsically, the most critical aspect of amalgamating both the Hopkinson bar apparatus with the X-ray beamline and detection system is the intricate synchronization of both systems. The open/close time of the X-ray window, firing of the bar apparatus, the actual dynamic event and corresponding image records must all be properly located in time in order to capture the phenomenon of interest (figure 3). Owing to the nature of using the extremely bright superbunch, the frame separation is inherently $n \times 3.68 \mu s$. Thus, voltage pulses ($P_0$) supplied by the master clock (which are also separated by 3.68 μs) from the synchrotron are used to synchronize the high-speed camera with the superbunch pulses via co-centring the camera shutter window with the time span of the superbunch. In order to increase the frame separation from the electron superbunch periodicity ($3.68 \mu s$), a digital delay/pulse generator (DG535) was used in order to skip a certain number of $P_0$ pulses, thereby acting as a frequency divider, and this voltage output is sent to the camera as an external phase-locking signal. Upon being triggered by an external signal derived from the strain gauges attached to the Hopkinson incident bar, the camera began to record the dynamic event.

The timing sequence, which can be seen in figure 3, is detailed as follows. At time $t_{-3}$, a single shot voltage signal is sent from the DG535 to fire the striker of the Hopkinson bar. A delay trigger from the Hopkinson bar firing signal is then sent at $t_{-2}$ in order to open the slow shutter, followed by another delayed signal sent at $t_1$ which is used to close the slow shutter. Within this timing window, being the active time window for the dynamic process to occur, a voltage signal derived from the strain gauges on the incident bar is used to trigger a fast-response data recording oscilloscope at time $t_0$, which then rapidly (50–80 ns) sends an output voltage signal to trigger the high-speed camera. The shutters are necessary owing to the excessive exposure to the high flux, white beam X-rays which are capable of damaging the sample and optics downstream. Owing to the longevity of the recording capability of the camera system (more than 1 s), no delay time is needed between the incident loading waveform and ultimate sample loading. Values for
Figure 3. Timing/synchronization schemes for the Hopkinson bar PCI experiments. (Online version in colour.)

the opening \( t_{-2} \) time of the slow shutter are determined by the Hopkinson striker travel time, which was calibrated as a function of specific tank pressures before the X-rays were active, thereby allowing for the achievement of necessary predetermined bar end velocities. Opening and closing times of the slow shutter are also varied in order to correct for the firing system jitter (less than 10 ms) and experiment longevity, e.g. if multiple loading pulses through the sample are desired.

6. Demonstrative experiments

Here, we briefly present four experiments, two in compression and two in tension, to demonstrate the unique capabilities established by the integration of Hopkinson bars and high-speed X-ray PCI. The two representative compression experiments are sand particle collision and edge cutting on glass. The two representative tension experiments are the dynamic failure of a single crystal silicon specimen and the dynamic damage process in a tendon–bone joint.

(a) Sand particle collision

Constitutive relations and deformation characteristics of granular materials depend significantly on failure of individual grains. Under quasi-static loading, failure mechanisms were found to include: single abrasion fracture, multiple abrasion fractures, major splitting of a grain into two or more particles, breakage of subparticles and pulverization of grains into many small pieces [36]. Under dynamic loading, grain failure in sand was also observed in post-mortem analysis [37,38]. However, the dynamic failure process was never observed. To investigate the \textit{in situ} failure of grains of sand under dynamic compression loading, we placed two Ottawa sand particles of nominal diameter 0.6 mm in a custom made aluminium sample holder and loaded in compression using a 0.6 mm diameter steel gauge pin.

\textbf{Figure 4} shows the image sequences of one of the experiments. The X-ray window is positioned to view the damage development near the contacting point of the two sand particles. The X-ray PCI technique clearly shows the cracks developed inside the sand particles at high speeds, which have been impossible to image. In all the experiments, one of the sand grains was observed
to pulverize when the first compressive stress wave arrived at the bar end. The other sand grain pulverized on the arrival of subsequent reflected stress waves. Both the incident-bar strain signal and the analysis of the high-speed images show that the bar end moves at the velocity of 4.9 m s$^{-1}$. The load data show that the load at the pulverization of the first grain was on average 206 N with a large standard deviation of 43 N. The grain fails when the local loading exceeds its capacity. However, local loading conditions depend on both the applied load and the boundary/contact conditions. The irregular sand grains lead to a large scatter in failure load. In our experiments, the order of pulverization for the grains was observed to be random. The failure mode of only pulverization indicates that the load path in the sand medium has to be re-established either through bypassing the pulverized particle or after the vacancy is physically closed when neighbouring particles contact again, thus seriously limiting the dynamic load propagation speed through the sand. This limitation effectively isolates incoming shock waves and other intensive dynamic loading.

(b) Edge cutting on glass

Although glass is an optically transparent material, its transparency is lost as soon as damage/cracks develop in the glass material owing to light reflections from the new surfaces created by the mechanical damage. In the experiments reported here, we used a knife blade that was driven by the end of the compression Hopkinson bar and cut into the edge of a glass specimen as schematically shown in figure 5. Also shown in figure 5 are the high-speed X-ray PCI images that record the damage/cracking process in a borosilicate glass as the ceramic-coated, tool-steel knife cut into it. The images show that, in response to the advancing edge-cutting blade, the borosilicate glass develops angular cracks at the initiation of damage. The angular cracks grow, and more angular cracks form as the blade is driven further into the glass plate. This is an important damage mechanism that spreads the concentrated load from the knife edge to a wider area as the damage grows deep into the glass material. A conventional high-speed optical technique would not be able to reveal the damage process owing to the reflection of light from the crack surfaces.
Figure 5. Dynamic edge-cutting experiment on a borosilicate glass. The schematic at the top is the experimental set-up at the test section. The three serial images in the lower row are the high-speed X-ray PCI images of the glass damage process at a frame rate of 45 260 fps. The dominating failure mode in this class is inclined crack propagation as indicated by the arrows. (Online version in colour.)

(c) Single crystal silicon tensile failure

Structures have been built at microscales with unique failure mechanisms that are not yet understood, in particular, under high-rate loading conditions. Consequently, microelectromechanical systems devices can suffer from inconsistent performance and insufficient reliability. To understand the failure mechanisms in microscaled specimens deforming at high rates, we subjected single-crystal silicon microspecimens that were 4 $\mu$m thick to dynamic tensile loading at a strain rate of 90 s$^{-1}$ using the Hopkinson tension bar in the APS X-ray hutch. The uniaxial tensile loading was applied along the $<110>$ crystallographic direction of the silicon single crystal. Figure 6 shows that failure mode is extensive fragmentation of the silicon microspecimen into many pieces when the tensile loading reaches a peak around 1.25 GPa. Optical high-speed imaging is capable of recording the failure process also. However, owing to the small dimensions of the fragments (some are close to the wavelengths of visible light), the resolution is poor. X-rays have wavelengths orders of magnitude shorter, thus having further potential for the realization of high-speed images of small objects at much higher resolution.
(d) Dynamic tensile damage of a tendon–bone joint

To understand the damage/injury at a tissue interface, a series of dynamic tension experiments were conducted on rat medial collateral ligaments (MCLs) with the region of interest being the interface between the MCL and femur. All tissue samples were harvested from animals euthanized under the conditions of the Purdue Animal Care and Use Committee, from both fresh and recently frozen animals. In these experiments, the MCL was secured via two grips to the tension Hopkinson bar and a load cell. The femur grip was fixed to the load cell, allowing the femur to remain motionless and within the X-ray field of view. The failure modes of the adult tissue samples were primarily rupture, whereas the failure modes for the juvenile tissue samples were primarily avulsion. The high-speed X-ray PCI images shown in figure 7 indicate that striations become visible just prior to failure. Unlike typical Hopkinson bar experiments, failure does not happen during the first stress wave. Instead, loading continues with successive passes of the stress wave. Even after failure has initiated, the MCL still retains a capacity for load. The MCL finally fails after a majority of the tissue has been pulled from the adjacent bone. It is noted that, in a conventional X-ray radiograph, the bone will have a sharp image, whereas soft tissues show up as a blurred light cloud. Using the X-ray PCI technique, detailed damage features inside the ligament are clearly imaged.

7. Conclusion

We integrated Hopkinson tension and compression bars with high-speed X-ray PCI technique which was used to visually analyse the dynamic damage and failure processes of a variety of materials. The combination of these two powerful experimental methods allows the continuous
in situ assessment of the damage evolution inside specimens undergoing dynamic deformation, thus opening up an additional angle of observation in high-rate experiments. The effectiveness of this new dynamic experimental method is demonstrated by a series of dynamic experiments on particle interaction under dynamic compression, high-rate edge cutting into glass, tensile failure of single crystal silicon and ligament–bone junction damage under high-rate tension.

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