High-speed Imaging of a Bulk Metallic Glass During Uniaxial Compression

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High–speed imaging of a bulk metallic glass during uniaxial compression

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High–speed imaging directly correlates the propagation of a particular shear band with mechanical measurements during uniaxial compression of a bulk metallic glass. Imaging shows shear occurs simultaneously over the entire shear plane, and load data, synchronized and time–stamped to the same clock as the camera, reveal that shear sliding is coincident with the load drop of each serration. Digital image correlation agrees with these results. These data demonstrate that shear band sliding occurs with velocities on the order of millimeters per second. Fracture occurs much more rapidly than the shear banding events, thereby readily leading to melting on fracture surfaces.

The microscopic manifestation of plastic deformation in metallic glasses is the shear band. The localization of shear bands in space and time is the root cause of the macroscopic brittleness of amorphous metals. Numerous experimental efforts have been undertaken to characterize shear band propagation in metallic glasses1–27 utilizing techniques such as nanoindentation,18 thermal imaging,19 and acoustic emission measurements.14,21–24 Direct optical imaging of shear bands has been performed by Neuhäuser,5 Hampel et al.,6 and Song et al.13 One method gaining recent popularity is the use of a piezoelectric load cell10,14–17 and strain gages applied to the specimen.10–13 In this work, we directly correlate optical images of shear band propagation with mechanical measurements made using a piezoelectric load cell.

The nature of the propagation of shear in metallic glasses has been a question of interest.9,10,13,16,25,28 One possibility is that shear proceeds in a simultaneous manner across the entire shear plane. In this case, the shear offsets for the same shear band on opposing sides of a compression specimen grow at the same rate. The digital image correlation results from Song et al.13 strongly indicate that this is the case. Another possibility, however, is that the shear propagates in a progressive fashion, much like a dislocation in a crystalline metal. A further possibility is that the shear occurs as some combination of these two, e.g., the shear proceeds first progressively as the shear band initiates and then simultaneously as shear continues.28 Our imaging of shear band propagation builds on the work of Song et al.13 but the significantly improved temporal and spatial resolution presented here allows the viewer to directly observe the simultaneous nature of the shear, and because the images are synchronized and time–stamped to the same clock as the camera, the load drop is definitively associated with the shear event for each serration.

Earlier work has consistently shown that for bulk samples, the shear band velocity is too slow (whether using either the simultaneous or progressive shear velocity) to result in a significant temperature rise.8–10,14–16 The energy released during the final failure event, however, is much larger than the energy released during serrated flow due to the combination of the large load drop and, as demonstrated in this work, the much shorter timescale for the final failure event. The large energy dissipation and high temperatures lead to the well–known vein pattern morphology on the fracture surfaces of metallic glass specimens.29

The only experimental observations that indicate that significant shear band heating occurs prior to failure were made by Lewandowski and Greer.26 In their experiments, fusible tin coatings deposited on double–notched four–point bend–test specimens were observed to melt at shear bands. Lewandowski and Greer used their coupled thermal and mechanical measurements to estimate peak temperatures in shear bands and reported temperature increases greater than 1000 K; however, we note that the specimens for which melting of the tin was observed in fact fractured before inspection with scanning electron microscopy. We have speculated that the shear bands on which the tin melted formed during the fracture event.10 This notion is supported by the work of Bengus et al. who tested metallic glass specimens in tension. They found that shear bands formed near the specimen ends during the fracture event due to reflections at the specimen grips of the elastic waves emanating from the fracture plane.27 In tension, the crack was observed to propagate at a velocity approaching the transverse wave speed, and we speculate that shear bands that form during fracture, but away from the fracture surface, may propagate at similarly high speeds, thereby leading to significant heating.

Three–millimeter–diameter rods of amorphous Zr45Hf12Nb5Cu15.4Ni12.6Al10 and Zr52.5Cu17.9Ni14.6Al10Ti5 were prepared using arc melting and suction casting in water–cooled copper molds. The methods used to prepare specimens for testing have been described elsewhere.10 The specimen dimensions were 6 mm in the direction of the loading axis with a cross–sectional area of 2 mm × 1.5 mm. Since Zr45Hf12Nb5Cu15.4Ni12.6Al10 is relatively ductile compared to Zr52.5Cu17.9Ni14.6Al10Ti5 and therefore sustains more serrations prior to failure, Zr45Hf12Nb5Cu15.4Ni12.6Al10 was used for studying shear band propagation prior to failure and...
Zr$_{52.5}$Cu$_{17.9}$Ni$_{14.6}$Al$_{10}$Ti$_5$ was used to study the fracture event. Seven samples of Zr$_{45}$Hf$_{12}$Nb$_5$Cu$_{15.4}$Ni$_{12.6}$Al$_{10}$ and six samples of Zr$_{52.5}$Cu$_{17.9}$Ni$_{14.6}$Al$_{10}$Ti$_5$ were tested.

A screw–driven Instron 5584 mechanical test system was used to perform quasistatic uniaxial compression tests at a nominal strain rate of $10^{-4}$ s$^{-1}$. A 60 kN Kistler piezoelectric load cell with a 180 kHz low–pass filter was used to acquire the load data. Tungsten carbide platens that were constrained by a steel sleeve with windows to facilitate imaging were used to compress the samples. For a schematic diagram of a similar load train, see Ref. 10. Strain gages applied to opposing sides of a specimen demonstrate that specimens machined to the tolerances given in Ref. 10 and tested in this load train show uniaxial loading with essentially no bending. An extensometer was used to acquire the displacement data to generate stress–strain curves; however, a typical extensometer does not reliably distinguish individual serrations.

Shear bands were imaged using a Vision Research Phantom v310, one–megapixel digital high–speed camera. Images were acquired at a rate of 12.5 kHz during serrated flow with an exposure time of 10 µs and a rate of 80 kHz during fracture with an exposure time of 2 µs. During serrated flow, the field of view was 224 pixels × 624 pixels, and during fracture, the field of view was 64 pixels × 200 pixels. The load data from the piezoelectric load cell were synchronized and time-stamped to the same clock as the camera using an analog signal acquisition module from Vision Research (SAM–3–PCI–DT3010). The acquisition rate for the synchronized load data was approximately 1.175 MHz.

Figure 1 is a true stress–strain curve for a specimen of Zr$_{45}$Hf$_{12}$Nb$_5$Cu$_{15.4}$Ni$_{12.6}$Al$_{10}$ that failed in compression. The yield strength is 1.75 GPa. The serrated flow that typifies the plastic deformation of metallic glasses is visible. Serrated flow is characterized by repeated cycles of a sudden stress drop followed by elastic reloading. Twenty–two serrations were observed during this test and produced a plastic strain of approximately 0.4%.

Figure 2 is a single image that highlights a shear band in the same specimen as shown in Figure 1. In contrast to previous work, our imaging rate is sufficiently fast such that the nature of the shear can be ascertained by direct visual inspection. The linked video clearly shows that the shear proceeds in a simultaneous fashion. The sequence of images was acquired at a rate of 12.5 kHz for a total length of 560 µs and was slowed in the video to display at a rate of approximately 8 frames per second. The video first shows the shear occurring in forward motion. Then the sequence of images was reversed using software, and the forward and reverse frames are looped to enhance the viewer’s understanding of the nature of the shear. (It is recommended that the video be viewed in full screen mode and played continuously to allow the viewer to see the simultaneous nature of the shear.) This particular serration occurred at approximately 2.54% total true strain. It is the serration immediately preceding the failure event and precedes the failure event by 0.74 s. The shear band formed previously and was reactivated at this point. Failure later occurred on this same shear plane. The images from compression testing of multiple specimens show that the same shear band can be activated multiple times throughout a test, but that deformation does not exclusively proceed on one particular shear band once it is activated.

Figure 3 is a plot of the load as a function of elapsed time as acquired by the Vision Research module during the same serration as shown in Figure 2. The load data were acquired at a rate of approximately 1.175 MHz, much faster than the 12.5 kHz imaging rate. A load drop of 150 N is observed. The data points corresponding to the images that comprise the video are indicated; thus, Figure 3 demonstrates that the simultaneous shear that occurs is coincident with the load drop of the serration. This point is critical since some thermal modeling relies on the notion that the shear step forms at a high rate during an internal stress redistribution surrounding the shear band prior to the observed load drop.
We note that our imaging does not preclude the possibility that progressive shear occurs first as a shear band initially propagates followed by simultaneous shear across the entire shear plane of the band, but even if this is the case, a measurable load drop does not occur during the progressive shear.

To the best of our ability to discern shear band propagation from the images, only one shear band occurs per serration. Although we only show the results for one specimen, the relevant behavior is similar for all seven specimens of Zr\(_{45}\)Hf\(_{12}\)Nb\(_{5}\)Cu\(_{15.4}\)Ni\(_{12.6}\)Al\(_{10}\) with a different number of serrations for each test.

Digital image correlation was performed on the images associated with Figure 2 to determine the displacement adjacent to the shear band as a function of time and position across the width of the sample. Figure 4(a) is a plot of the displacement parallel to the shear band at various locations as a function of image number during the load drop for the same serration as shown in Figures 2 and 3. The open symbols represent locations above the shear band (\(\pm y\)) while the filled symbols represent locations below the shear band (\(-y\)). The locations above the shear band are moving in the direction of the \(+x\) axis, while the locations below the shear band are moving in the direction of the \(-x\) axis. Figure 4(b) shows the approximate locations of each point in Figure 4(a) and the orientation of the axes.

For the shear band shown in Figure 4 with a final offset size of 4.6 \(\mu\)m and an elapsed time of 560 \(\mu\)s, an average shear band velocity (or plastic displacement rate \(\dot{u}_{\text{plastic}}\)) of 8 mm/s is calculated. This velocity is expected to produce a maximum temperature increase in the shear band \(\Delta T\) on the order of approximately thirty degrees when the shear is simultaneous according to

\[
\Delta T = \frac{\tau \dot{u}_{\text{plastic}}}{K} \sqrt{\frac{\kappa \Delta t}{\pi}},
\]

where \(\tau\) is the shear stress acting on the shear band, \(K\) is the thermal conductivity, \(\kappa\) is the thermal diffusivity, and \(\Delta t\) is the elapsed time during the serration.

Figure 5 shows images and the corresponding load data for a Zr\(_{52.5}\)Cu\(_{17.9}\)Ni\(_{14.6}\)Al\(_{10}\)Ti\(_{5}\) specimen failed in compression acquired at a rate of 80 kHz. The fracture occurred on a pre-existing shear band. Images I and II show that the sample was still intact. There appear to be no signs of fracture at these moments. Image III shows that the fracture event had already occurred because the specimen was no longer making contact with the bottom platen when Image III was taken. If the sample was not making contact with the platen, then it was no longer bearing load and was at least effectively, if not completely, fractured. Note that this particular specimen was cut using a diamond saw and did not adhere to the precision tolerances specified earlier. Of the six samples that were machined to the precision tolerances, the images of fracture for six specimens showed that the fracture occurred in 20 \(\mu\)s or less using an imaging rate of 50 kHz, an exposure time of...
inclined at approximately 45°. The fracture occurred on a pre-existing shear band. Images I and II show that the sample was still intact. Image III shows that the fracture event had already occurred because the specimen was no longer making contact with the bottom platen when Image III was taken.

3 µs, and a field of view of 120 pixels × 320 pixels. All images at the moment of fracture show what appear to be molten droplets being ejected from the fracture surface as also observed by Gilbert et al.34

The load cell is unable to accurately track the fracture event due to the short timescale over which it occurs. As shown in Figure 5, the sample broke into two pieces before the load registered zero. This is an artifact of the low-pass filter in the electronics of the load cell. Figure 5 demonstrates that the failure is much more rapid than the load cell indicates. The upper bound for the elapsed time during fracture is estimated as the time between images II and III, or 12.5 µs. A low-pass filter with a cut-off frequency of 180 kHz, such as that used in the load cell, attenuates signals with timescales on the order of 1/(180 kHz) = 5.6 µs. Since the fracture event occurs on a timescale less than 12.5 µs, it should be expected that the load data for the fracture event do not accurately reproduce the temporal features of the data. This, however, is not a concern for the shear banding events that occur prior to fracture because the timescale associated with shear banding events is two orders of magnitude longer than the timescale associated with the cut-off frequency of the filter.

A lower bound velocity for the propagation of fracture is approximated as the ratio of the length of the fracture plane to the elapsed time during fracture. For a shear band inclined at approximately 45° to the loading axis across a specimen width of 1.492 mm, this gives an average velocity of 170 m/s. This value is many orders of magnitude faster than the shear band velocities prior to fracture during serrated flow, but also more than an order of magnitude slower than the shear wave speed of approximately 2200 m/s (we note again that 170 m/s is an approximate lower bound value). These results do not give any indication of why fracture initiates; the results do, however, suggest that fracture is not the progression of a shear banding event because the timescale of the fracture event is more than an order of magnitude faster than shear band propagation. Future work will focus on this question.

The authors gratefully acknowledge contributions to the design of the load train from M. M. LeBlanc at the Lawrence Livermore National Laboratory (LLNL) and a useful discussion with T. C. Hufnagel at Johns Hopkins University and also J. N. Florando at LLNL. We thank E. Schwarz and J. Psilopoulos, applications engineers at Trilion Quality Systems for assistance with the image correlation results. We also thank B. Derr for video editing assistance and D. Johnson for machining of parts, both from Bucknell University. This work was supported by the National Science Foundation under Grant DMR–1042734 with E. M. Taleff as the program director.

FIG. 5. Images (acquired at a rate of 80 kHz) and the corresponding load data for a Zr52.5Cu17.9Ni14.6Al10Ti5 specimen failed in compression. The sample broke into two pieces before the load cell registered zero. This is an artifact of the low-pass filter in the electronics of the load cell. Figure 5 demonstrates that the failure is much more rapid than the load cell indicates. The upper bound for the elapsed time during fracture is estimated as the time between images II and III, or 12.5 µs. A low-pass filter with a cut-off frequency of 180 kHz, such as that used in the load cell, attenuates signals with timescales on the order of 1/(180 kHz) = 5.6 µs. Since the fracture event occurs on a timescale less than 12.5 µs, it should be expected that the load data for the fracture event do not accurately reproduce the temporal features of the data. This, however, is not a concern for the shear banding events that occur prior to fracture because the timescale associated with shear banding events is two orders of magnitude longer than the timescale associated with the cut-off frequency of the filter.

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