High-density polyethylene damage at extreme tensile conditions

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Abstract. In-situ and postmortem observations of the dynamic tensile failure and damage evolution of high-density polyethylene (HDPE) are made during Dynamic-Tensile-Extrusion (Dyn-Ten-Ext) loading. The Dyn-Ten-Ext technique probes the tensile response of materials at large strains (>1) and high strain-rates (>10^5 s^-1) by firing projectiles through a conical die. Postmortem sectioning elucidates a mechanism of internal damage inception and progression. X-ray computed tomography corroborates shear damage with cracks nearly aligned with the extrusion axis but separated by unfailed internal bridges of material. In-situ measurements of damage are made with the impact system for ultrafast synchrotron experiments (IMPULSE) using the advanced imaging X-ray methods available at the Advanced Photon Source. Multiple frame phase-contrast imaging (PCI) elucidates the evolution of damage features in HDPE during Dyn-Ten-Ext loading that is observed in postmortem sectioning and X-ray tomography.

1. Introduction

Dynamic-Tensile-Extrusion (Dyn-Ten-Ext) is presented as a technique for investigating extreme tensile deformation and damage in polymers in conjunction with postmortem and in-situ characterization. The Dyn-Ten-Ext technique was developed at Los Alamos National Laboratory to examine extreme tensile conditions in metals [1,2] and has been extended to polymers including polytetrafluoroethylene [3–6], polychlorotrifluoroethylene [3–6], polyurea [6,7], polycarbonate [8], and high-density polyethylene (HDPE) [6,9,10] with postmortem characterization. It is especially attractive for probing tensile deformation and damage in polymers, which are ductile, highly strain-rate sensitive and exhibit significant sensitivity to hydrostatic tension. The apparatus consists of a conical extrusion die that is fixed to the end of the gun barrel, forcing the specimen to extrude through it at a high velocity (figure 1 inset). The leading edge of the specimen is relatively unaffected by the extrusion process, but the aft portion rapidly decelerates inside the die, pulling the extruded ligament between the two ends in high strain-rate tension, typically to large strains and ultimately to failure. Finite element simulation of high-density polyethylene (HDPE) [6] undergoing Dyn-Ten-Ext predicts that the ligament achieves a true strain approaching 2 at a strain rates exceeding 10^5 s^-1 in the critical tensile section during extrusion (figure 1). Simulations using ALE3D of Dyn-Ten-Ext loading of polycarbonate have yielded equivalent extreme states of stress, strain and strain-rate [8].
To this point, diagnosis of Dyn-Ten-Ext has been limited to high-speed photography and velocity measurements during the test followed by postmortem characterization. Recent experimental efforts at the Advanced Photon Source (Argonne, IL) have demonstrated the ability to use PCI and Laue diffraction to examine materials shock compressed using a gas-gun system [11–16]. The current work investigates industrial grade HDPE extruded sheet (Cope Plastics, Godfrey, IL) under Dyn-Ten-Ext loading with in-situ and postmortem observations. This material is the same pedigree as previously investigated by Brown et al. [6,9,10,17–18]. The density is $969.8 \pm 1.4$ kg m$^{-3}$ as measured by He pycnometry, with a 134°C melt temperature and 80.9% crystallinity based on differential scanning calorimetry. High-speed photography and postmortem characterization including optical microscopy on sectioned samples and X-ray tomography on recovered samples were performed at LANL, while in-situ PCI of dynamic phenomena at ns to μs timescales were performed at APS. Recovered samples have been observed to have a nominal residual temperature above ambient but there is no indication of melting. During continuum deformation any increase in temperature will offset the effect of increasing strain-rate on the flow stress under temperature–strain-rate equivalency [17,19]. This effect could be amplified during localization associated with damage and failure processes.

Figure 1. Simulation of Dyn-Ten-Ext for a 7.62 mm diameter HDPE sphere at 447 m s$^{-1}$ and exit diameter of 2.8 mm shows the ability to achieve loadings above a strain of 2 and strain-rate of $10^7$ s$^{-1}$. See Furmanski et al. [6] for details.

Figure 2. Sequences of images of Dyn-Ten-Ext with a 7.62 mm diameter HDPE sphere at (a) low, (b) intermediate, and (c) high velocity. Damage is (a) stable and limited, (b) stable and terminal, and (c) unstable and catastrophic. Red boxes illustrate the nominal size and location of the X-ray window at APS.

2. Dyn-Ten-Ext at LANL Taylor gun facility
Dynamic-Tensile-Extrusion tests typically yield two categories of observable behavior: stable continuum deformation of an extruded jet and stochastic localization including damage, failure, or fragmentation. The stable bulk deformation is studied to verify continuum descriptions of material
behavior and develop continuum failure criteria, while the latter can be used to investigate dynamic instabilities and the final stages of damage progression. At lower driving velocities HDPE, extrudes a long stable extrusion (figure 2). Though some variation in cross-section can be observed along the length of the extrusion suggesting initiation of localization or necking, the extrusion arrests due to retained strength and stays intact. With increased velocity the behavior transitions from stable deformation to failure, first with a single fracture surface and at higher velocities diffuse catastrophic fragmentation.

The progression of damage to failure is evident upon cross-section of the recovered specimens (figure 3), even in samples at lower velocity that appear intact from inspection of the outer surface. Damage progresses along a pseudo-axial path that links an internal flaw to the exterior edge of the specimen (figure 3a). Closer examination of the intact specimen (figure 3b) reveals an internal failure at the specimen axis in the form of a macroscopic chevron (shear-dominated) crack, which is followed by a shear-mode pullout of the core behind the flaw in a strain concentrating process. X-ray computed tomography reveals the damage mechanism to be a shear-cracking process (figure 3c). Both the localized shear-failure and diffuse crazing are processes strongly exacerbated by hydrostatic tension (compression would close cracks and prevent growth). However, postmortem characterization is not able to distinguish the temporal evolution of damage or isolate the deformation during the test from the posttest viscoelastic-viscoplastic relaxation of the polymer.

Figure 3. Postmortem images of arrested damage in 7.62 mm diameter HDPE Dyn-Ten-Ext sample at 450 m s\(^{-1}\). (a) Optical image of recovered specimen cross-section (note the specimen was cut in the plane normal to loading to remove it from the die). (b) Optical micrograph showing chevron tensile failure and shear damage. (c) X-ray computed tomography showing shear-mode cracks in damage region ahead of and behind chevron failure. Red boxes illustrate the nominal size and location of the X-ray window at APS. Extrusion direction is left to right in all images.

3. Dyn-Ten-Ext at APS IMPULSE

The impact system for ultrafast synchrotron experiments, or IMPULSE, is a 12.6 mm bore light-gas gun designed specifically for performing dynamic compression experiments using the advanced imaging and X-ray diffraction methods available at synchrotron sources as introduced by Jensen et al. [11]. The multiple frame PCI capability, used here, has been demonstrated in previous investigations for acquiring dynamic data on the nanosecond time scale of the impact event with incident 80 ps X-ray pulses [13–15]. The following experiments were performed similarly at the APS Sector 32 beamline using the standard mode with the undulator gap and the sample-to-scintillator distance set to 30 mm.
and 200 mm respectively. During experiments the X-rays transit through a evacuated target chamber, interact with the Dyn-Ten-Ext sample located at the muzzle of the gun, and impinge upon the detection system (scintillator and a detector) configured for phase contrast imaging that enhances imaging of edges or surfaces such as cracks, as described in detail elsewhere [16]. Unlike Dyn-Ten-Ext tests performed at the LANL Taylor gun facility where the samples are size matched to the bore of the gun, a sabot and stripper design was incorporated with the die to allow Dyn-Ten-Ext tests to be performed on IMPULSE, as shown in figure 4. The sample is a 7.62 mm diameter hemisphere and the die has a 9° taper and 3.607 mm exit diameter. An optical beam interrupt at the die exit triggered diagnostics. The red square in figure 4 illustrates the nominal size and location of the X-ray PCI and nominally corresponds with the red boxes in figures 2 and 3 from data acquired with the LANL Taylor gun facility. In each case, the top of the 2.1 by 1.4 mm X-ray window was positioned approximately 300 μm above the inner surface of the die exit to capture 60% of the extrusion radius (i.e., excluding the center-line region). Velocity of the sample through the barrel and extrusion process was measured by photonic Doppler velocimetry (PDV), as shown in figure 5, and replicated velocity profiles from HDPE Dyn-Ten-Ext tests performed at the LANL Taylor gun facility.

**Figure 4.** Sabot and die assembly used to image internal tensile damage and failure processes in-situ with PCI at the APS. An optical beam interrupt at the exit of the die triggered the diagnostics. PDV monitored travel of the HDPE hemisphere down the barrel and throughout the extrusion process. The red square illustrates the nominal size and location of the X-ray PCI.

**Figure 5.** Representative PDV data recorded from the HDPE hemisphere during the Dyn-Ten-Ext experiment at APS on IMPULSE. PDV was used to monitor the hemisphere’s travel through the barrel and extrusion process. The data points correspond to image times in figure 6.

Figure 6 shows multiple-frame PCI from three separate experiments fired at approximately 550 m s⁻¹, nominally corresponding to the high-speed photography in figure 2c. Average inter-frame velocities for the extrusion trip were determined from the PDV measured velocities in figure 5 and the PCI images offset in figure 6 by the distance calculated from the average inter-frame velocities and inter-frame times. In the case of a simple rigid body translation these offsets would allow reconstruction into a single long image. Dyn-Ten-Ext is much more complicated, where not only is the sample moving through the reference frame but is also deforming and damaging, as evident in figure 2. Therefore, the offset based on the extrusion tip velocity is at best a maximum offset. Figure 6a–d (taken at the die exit, evident on the left edge of the image, 1.03 ± 0.153 μs after extrusion and at three 3.366 μs intervals thereafter) illustrates the breakdown of this shift. In all four PCI images
the dominant feature is the sloping sample edge near the top of image, which is self-similar suggesting a near steady-state extrusion process consistent with the high-speed optical photography in figure 2. Within the sample volume a number of lines are observed in the PCI images aligned with the extrusion direction. This apparent evolving meso-structure may be indicative of the diffuse crazing observed in figure 3. Since there are no particular features in either the sample edge or meso-structure that correlate from image to image, both the bulk deformation and damage seem to be dominated by the proximity of the die boundary condition. Conversely, figure 6e–h taken approximate three die diameters from die exit illustrates the utility of this shift (taken 10.16 mm from the die exit 21.233 ± 0.153 µs after extrusion and at three 5.049 µs intervals thereafter). The sample edges in all four PCI images lineup up in a continuous undulation suggesting a nearly rigid body bulk deformation, again consistent with the high-speed optical photography in figure 2. Short white lines are added between the images to aid in guiding the eye. Within the sample volume, the meso-structure lines are clearer and more complex in the PCI than near the die. This is particularly true in the bottom portion of the PCI images nearer to the centerline of the extrusion where nucleation and growth of macro-scale fracture was observed in figure 3. Finally, figure 6i–l (taken 10.16 mm from the die exit 33.638 ± 0.153 µs after extrusion and at three 5.049 µs intervals thereafter) shows no indication of the sample edge but does feature fragmentation consistent in size and shape with figure 2, suggesting the sample has failed and the images are primarily of free space between the fore part of the sample that is in free flight and the aft part of the sample contained in the die. The stochastic nature of damage and potential for this gap during failure are consistent with high-speed photography in other experiments.

**Figure 6.** Multiple-frame PCI from three separate experiments fired at 550 m s\(^{-1}\). In (a–d) PCI was taken at the die exit. In (e–f) and (i–l) PCI was taken 10.16 mm from the die exit in two separate experiments.

**4. Conclusions**

The Dynamic-Tensile-Extrusion technique is a relatively simple and effective experimental approach for studying the high strain-rate and large-strain constitutive behavior of materials, with a particular potential for ductile polymers that exhibit both high strain-to-failure and strong sensitivity to strain-rate. Both deformation and failure phenomena under extreme mechanical conditions can be directly observed in Dyn-Ten-Ext, which in turn enable inference of the phenomena driving damage and
failure in the material. As an integrated test with complicated stress, strain, and strain-rate gradients, with simple well-defined boundary conditions, Dyn-Ten-Ext is well suited for the refinement and validation of material models under extreme conditions. Combining the novel loading and in-situ observation from PCI with IMPULSE at APS has great potential to extend the spatial and temporal understanding of damage nucleation and growth in the extreme tensile loading regime.

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