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Author(s):

George T. Gray III, William R. Blumenthal,
Carl P. Trujillo, Robert W. Carpenter II

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INFLUENCE OF TEMPERATURE AND STRAIN RATE ON THE MECHANICAL BEHAVIOR OF ADIPRENE L-100

George T. Gray III, William R. Blumenthal, Carl P. Trujillo, and Robert W. Carpenter II
Materials Research and Processing Science
Los Alamos National Laboratory,
Los Alamos, NM 87545, U.S.A.

Abstract: The effect of sample thickness, strain rate, and temperature on the mechanical response of Adiprene-L100 is presented. The compressive stress-strain response of Adiprene-L100 was found to depend on both the applied strain rate; $0.001 \leq \dot{\epsilon} \leq 7000 \text{ s}^{-1}$, and the test temperature at high-rate; $77 \leq T \leq 298 \text{ K}$. Due to the slow, dispersive wave propagation in Adiprene-L100, thinner sample thicknesses are needed to assure uniform, uniaxial stress conditions within Hopkinson Bar samples; the optimal sample thickness being dependent on test temperature. Decreasing temperature from 298 to 77K at 3000 s^{-1} was found to increase the maximum flow stress in Adiprene-L100 from 10 to ~210 MPa.

Résumé: Nous présentons l'influence de la vitesse de déformation, $\dot{\epsilon}$, de la température d'essai, T , et de l'épaisseur de l'échantillon sur le comportement mécanique sous grandes vitesses de sollicitation de l'Adiprene-L100 : sa réponse en compression dépend à la fois de $\dot{\epsilon}$ et de T . À cause de la nature dispersive de la propagation des ondes dans l'Adiprene-L100, les épaisseurs des échantillons doivent être faibles pour assurer un état de contrainte uniforme dans l'éprouvette; l'épaisseur optimale dépend de la température d'essai. À 3000 s^{-1} , une décroissance de T , de 298 à 77K, entraîne une augmentation de la limite d'écoulement maximale de 10 à ~210 MPa.

1. INTRODUCTION

The high-strain-rate stress-strain response of polymeric materials has received increased interest in recent years related to: 1) the need for predictive constitutive model descriptions for use in large-scale finite-element simulations of automotive crash-worthiness and aerospace impacts, including foreign-object damage such as during bird ingestion in jet engines utilizing polymeric composite components, and 2) focused emphasis on understanding the polymeric binder-constituents in explosive and propellant composites. The establishment of more physically-based constitutive models to describe complex loading processes and polymeric composites requires a detailed knowledge of the separate and synergistic effects of temperature and strain rate on the mechanical response of polymers.

A significant number of previous studies have probed the high-rate constitutive response of a wide variety of polymers starting with Kolsky in 1949 through the present [1-5], including several in-depth reviews by Walley summarizing a broad spectrum of polymers [6, 7]. Beginning with the high-rate pressure-bar studies of Kolsky [1] on polythene (polymerized ethylene) and rubber it was found that: a) the effective elastic modulus of polymeric materials is strongly influenced by strain rate, b) polymeric materials during high-rate loading continue straining after the maximum flow stress is achieved in more ductile polymers, c) some polymers exhibit no permanent plastic flow after substantial high-rate straining; the recovery effect being time dependent, and d) stress wave propagation through some polymers, such as polythene, is so dispersive that over a given sample length no transmitted Hopkinson bar signal is measured. Studies since the original work of Kolsky have documented the strong influence of strain rate, temperature, moisture content, and imposed hydrostatic pressure on the mechanical response of a wide range of polymeric materials [6, 7]. Experimental results for a large number of polymers exhibited a linear relationship between the compressive yield stress of the polymer and log

(strain rate)[7, 8]. This observation supports the concept that a single deformation rate theory appears adequate to describe the plastic response of polymers over a wide range of imposed strain rates[7].

While numerous studies have investigated the influence of strain rate on the constitutive response of a range of polymers, the influence of temperature at high-strain rate on polymer mechanical behavior has received more limited attention[8, 9]. Testing at temperatures below 298K for a range of polymers has shown that both the loading modulus and peak flow stresses measured increase with decreasing temperature[8, 9]. Based upon these results it is apparent that the construction of robust material models for high-rate system simulations describing polymeric constitutive behavior requires systematic knowledge of the independent effects of temperature and strain rate.

The early work of Kolsky [1] on plastics and rubber also identified the importance of utilizing thin sample thickness when studying the high-rate constitutive response of plastics and rubber. This constraint is driven by the need to assure accurate and reproducible constitutive data in the Hopkinson Bar[2, 10] on samples in a uniform stress state. Given the documented influence of temperature on the elastic and plastic properties of ductile polymers it is important to evaluate sample geometry effects over a range of temperatures. The objective of this paper is to present results illustrating the effect of systematic variations of sample thickness, strain rate, and temperature at high-strain rate on the constitutive response of Adiprene-L100. The experimental apparatus utilized for conducting temperature controlled Hopkinson Bar testing is also described in detail.

2. EXPERIMENTAL TECHNIQUES

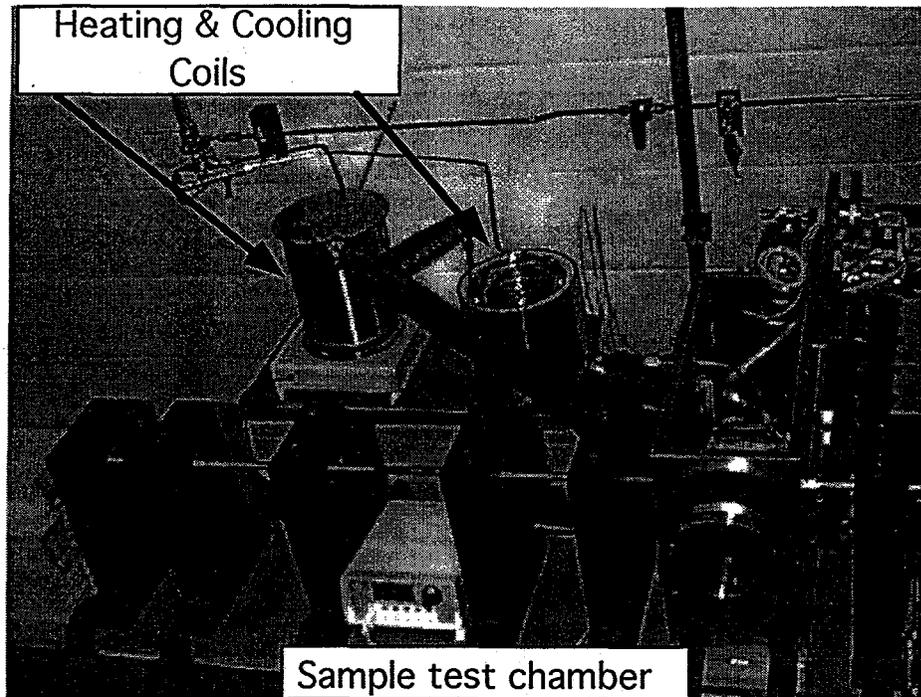
This investigation was performed on Adiprene-L100 plate material. Vulcanizates of Adiprene L-100, a polyether urethane-based rubber, when cured utilizing polyol exhibit outstanding quasi-static properties at low temperatures. They do not become brittle at 200K, although stiffening occurs as the temperature is reduced. Adiprene is also attractive in the industrial environment because it is 5 to 10 times more resistant to degradation by moisture than are polyester-based urethanes. The broad range of temperatures over which Adiprene-L100 remains flexible make it a good choice for an experimental study focused on investigating the influence of temperature at high-strain rate on polymer behavior.

Cylindrical compression samples 6.35-mm in diameter with three different lengths of 3.2, 5, and 6.35-mm (yielding sample length-to-diameter ratios of 0.5, 0.8, and 1 to 1, respectively) were machined normal to a 12.5-mm thick Adiprene-L100 plate. Quasi-static compression tests were conducted at strain rates of 0.001 and 0.1s^{-1} at 298K in laboratory air exhibiting a relative humidity of ~15%. Dynamic tests were conducted as a function of strain rate, 1500-7000 s^{-1} , and temperature, 77 and 298K, utilizing a split-Hopkinson pressure bar[11]. The split-Hopkinson bar used for this study was equipped with 9.4-mm diameter Ti-6Al-4V bars that improve the signal-to-noise level needed to test extremely low strength materials as compared to the maraging steel bars traditionally utilized for Hopkinson-Bar studies on metallic materials where the highest possible bar strength is necessary to maintain elastic conditions in the bars.

The inherent oscillations in the dynamic stress-strain curves and the lack of stress equilibrium in the specimens at low strains make the determination of yield strength inaccurate at high strain rates. Temperature variations between 200 and 350K on a split-Hopkinson bar have been achieved utilizing a specially-designed gas manifold system developed at the Los Alamos National Laboratory (LANL) where samples were cooled and heated using Helium gas within a 304-stainless steel containment chamber held at a partial vacuum, as shown in Figure 1. Helium gas, due to its high inherent thermal conductivity, was selected as the heat-transfer medium to heat/cool a range of materials including polymers, energetic and propellant materials. The He gas is cooled below ambient temperature by passing the He through a copper coil positioned within a liquid nitrogen dewier, while elevated temperatures are achieved by heating the He in a similar coil within a glycerin-filled beaker warmed to ~398K by a heating plate. Samples were lubricated using either a thin layer of molybdenum disulfide grease or molybdenum disulfide spray lubricant.

The sample temperature is monitored using a thermocouple positioned directly adjacent to the sample. Variations in He gas flow rate to the manifold surrounding the sample were found to allow fine temperature control. The heat transfer time required to heat or cool a 1:1 aspect ratio sample of Adiprene-L100 was determined using a thermocouple inserted into the middle of a dummy sample. A minimum time interval of 5 minutes was determined to be necessary to equilibrate the sample

temperature after attaining a stable helium gas temperature within the manifold surrounding the sample. A similar procedure is being utilized at LANL for evaluating the temperature dependency of the high-strain-rate response of energetic materials.



Figures 1: Photograph of specialized split-Hopkinson pressure bar set-up using Ti-6Al-4V bars and a He-gas manifold heating / cooling system to allow controlled temperature testing at high strain rates.

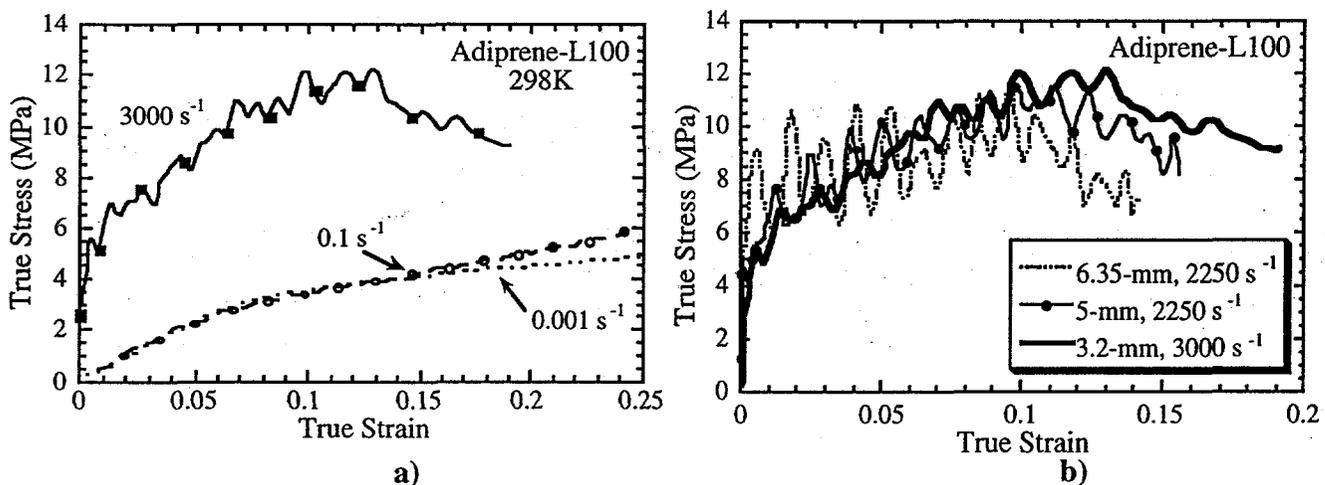
3. RESULTS AND DISCUSSION

The compressive true-stress versus true-strain response of Adiprene L-100 was found to depend on the applied strain rate, varied between 0.001 and 7000 s^{-1} , and the test temperature, varied between 77 and 298K at a strain rate of 3000 s^{-1} . The yield strength of Adiprene L-100 at 298K is shown in Figure 2a to increase from 2 MPa at 0.001 s^{-1} to $\sim 6 \text{ MPa}$ at a strain rate of 3000 s^{-1} accompanied by an ~ 8 -fold increase in apparent loading modulus. The yield strength at 0.001 s^{-1} at 77K was measured to be 250 MPa . These data on Adiprene-L100 are consistent with the pronounced influence of strain rate and temperature on the mechanical behavior of ductile polymers[6,7]. Due to the documented dispersive nature of wave propagation in ductile polymers and the influence of sample size on attaining a uniform stress state, the high-rate constitutive response of Adiprene-L100 was measured using three sample thicknesses at a constant diameter. Similar to the early findings of Kolsky[1] and more recent studies of Diah et. al.[2], the high-rate stress-strain response (using a 1-wave analysis[12]) of Adiprene-L100 at ambient temperature was found to depend on sample thickness as shown in Figure 2b. While the maximum flow stress attained for each sample thickness is nominally the same for a strain of 10% , the details of the ringing-up of the sample as well as the fall off in flow stress after the maximum stress level was reached is significantly different. The $1:1$ aspect ratio sample exhibits the most dispersive ring-up as well as the most rapid fall off in flow stress of the three sample thicknesses, while the 3.2-mm thick sample shows a clear yield strength and the lowest ringing amplitude.

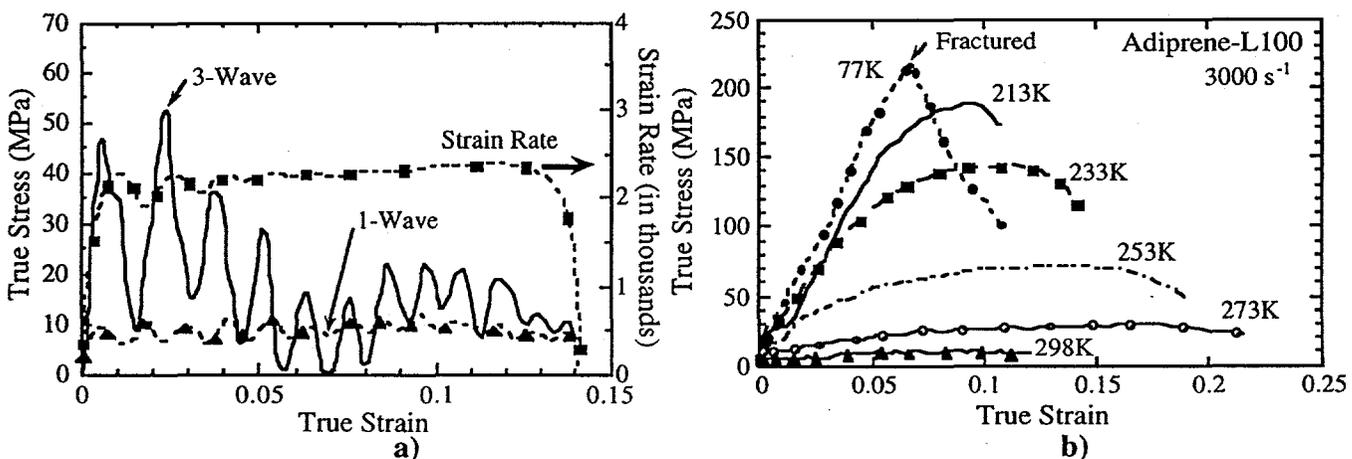
To understand the different responses as a function of sample thickness, it is instructive to examine the different analyses used to calculate sample stress from the Hopkinson bar strain as shown in Figure 3a. In the 1-wave analysis the sample stress is directly proportional to the bar strain measured from the transmitted bar. This waveform characteristically exhibits low oscillation amplitude because the deforming sample effectively damps much of the ringing inherent in the incident pulse as it propagates through the sample. More importantly, the 1-wave stress analysis reflects the conditions at the sample-transmitted bar interface and is often referred to as the sample "back stress". Alternatively in a 2-wave analysis, the sum of the synchronized incident and reflected bar waveforms (which are opposite in sign) is proportional to the sample "front stress" and reflects the conditions at the incident/reflected bar-sample interface. Unfortunately, both the incident and reflected waveforms contain substantial inherent

oscillations which, compared to the transmitted waveform, cause uncertainty in the interpretation of stress, especially near the yield point. In addition, these harmonic oscillations are subject to "dispersion" due to the wave speed dependence of different frequencies that causes asynchronization of the overlapped waveforms and, therefore, inaccuracy in the calculation of the "front stress". A dispersion correction analysis has been developed [12] to account for these changes in phase angle of the dominant harmonic oscillation of all three strain signals. This analysis results in more accurate and smoother stress-strain curves, especially near the yield point. Finally a third stress-calculation variation that considers the complete set of three measured bar waveforms, the 3-wave analysis, is simply the average of the 2-wave "front" and the 1-wave "back" stress.

A valid, uniaxial Hopkinson bar test requires that the stress state throughout the sample achieve equilibrium during the test and this condition can be checked readily by comparing the 1-wave and 3-wave (or 2-wave) stress-strain response. When the stress state is uniform throughout the sample, then the 3-wave stress oscillates about the 1-wave stress. When the stress state is not uniform throughout the sample, then the 3-wave stress diverges and exceeds the 1-wave stress values. Previous Hopkinson bar studies of ceramic materials using this 1-wave versus 3-wave comparison have shown quite dramatically that a sample is not in stress equilibrium when divergence is observed [13]. In ceramic and cermet materials this divergence correlates very well with the onset of non-uniform plastic flow and/or fracture events. The pronounced difference in the initial 1-wave and 3-wave signals for the 6.35-mm thick Adiprene L-100 sample in Figure 3a can accordingly be viewed as an indication of a sluggish ring-up to stress-state equilibrium and a marginally valid Hopkinson bar test at strains $> 5\%$, even though a stable strain rate is indicated throughout the entire test. The ambient temperature data in Figure 2b therefore asserts the need to utilize thinner sample aspect ratios when studying the complete high strain rate constitutive response of low sound speed, dispersive materials. Based upon the data in Figure 2b, all subsequent testing in this study utilizes the 3.2-mm thick samples with an aspect ratio of 0.5:1.



Figures 2: Stress-Strain response of Adiprene-L100, a) as a function of strain rate at 298K, and b) as a function of sample thickness at high-strain rate.



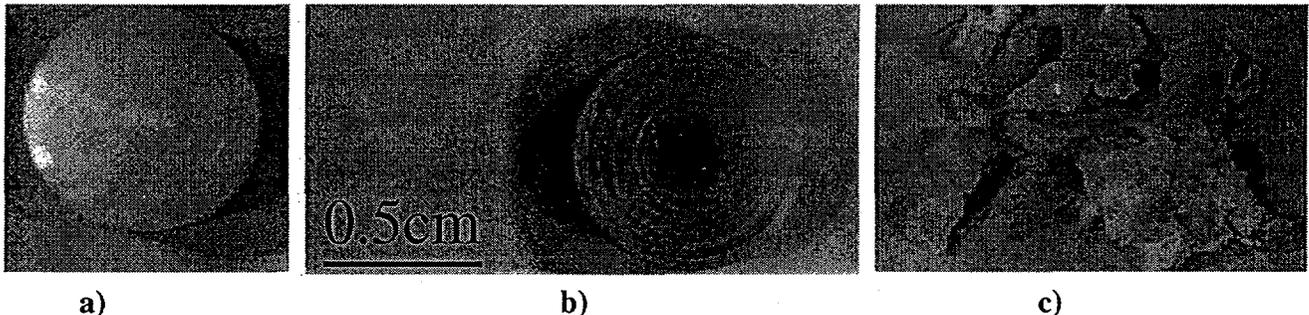
Figures 3: Stress-strain response of Adiprene-L100, a) for a 6.35-mm thick sample showing 1- and 3-wave stress curves in addition to the strain rate; and b) as a function of temperature at high strain rate using 3.2-mm thick samples.

Another interesting observation from the data of Figure 2b is the difference in the stress fall-off as a function of sample thickness at strains over 10%. Taking into account that the lubrication conditions were the same for all samples, these results suggest the dynamic stress relaxation of Adiprene-L100 is a function of sample size. Further experiments are needed to validate these observations.

At high strain rate, the yield strength of Adiprene L-100 was found to be strongly dependent on temperature as seen in Figure 3b, increasing from 10 MPa at 298K to 50 MPa at 253K and finally to a value of ~215 MPa at 77K. Coincident with this flow stress increase is an ~15-fold increase in the loading modulus with decreasing temperature at high strain rate. The structural response of Adiprene L-100 was found to exhibit minimal permanent plastic set at temperatures > 200K. At ambient temperature and a loading rate of 3000 s⁻¹, Adiprene L-100 samples were found to essentially fully recover their initial sample length via time-dependent relaxation processes (i.e., creep following stress removal) as seen in Figure 4a.

Attempts to achieve higher strain rates (> 5000 s⁻¹) at 298K proved difficult-to-unsuccessful. For example, at a strain rate of 7000 s⁻¹ the 1-wave and 3-wave signals diverged for the entire test (invalidating the stress analysis as discussed previously) and the samples were found to tear on axis leaving a small central hole in the sample as seen in Figure 4b. The sample aspect ratio needed to achieve stress state equilibrium was found to be dependent on the test temperature. While a 3.2-mm thick sample was needed at 298K to obtain well-posed Hopkinson Bar data on Adiprene-L100, samples with higher aspect ratios proved suitable at temperatures below ~240K. These observations clearly demonstrate that stress equilibrium within polymeric samples is influenced by material elastic properties.

With decreasing temperature, the Adiprene-L100 samples exhibited increasing levels of permanent deformation. However, at 77K and a strain rate of 3000 s⁻¹, Adiprene L-100 loaded essentially elastically to the fracture point denoted in Figure 3b and then suffered catastrophic brittle fracture into a number of fragments (Figure 4c),



Figures 4: Macro-photographs of Adiprene-L100 following Hopkinson bar testing at: a) 298K and a strain rate of 3000 s⁻¹; b) 298K and a strain rate of 7000 s⁻¹ which caused tearing around the central axis; and c) at 77K and a strain rate of 3000 s⁻¹ which caused brittle fracture.

The predominantly visco-elastic nature of Adiprene-100 response suggests that this material may be an excellent candidate for a matrix material with superior impact and shock mitigation properties. The high strain rate wave propagation behavior also indicates that kinetic effects are as important as in other previously documented polymeric materials. In addition, the temperature dependence of the high strain rate stress-strain response of Adiprene-L100 demonstrates that thermal effects play an important role in the ability of polymer chains to extend and rotate to accommodate imposed strains. The pronounced dependence of the apparent loading modulus on temperature for a fixed high-strain rate, is consistent with considering Adiprene-L100's temperature response as a viscosity term reflecting how the local polymer chains realign. Advanced material constitutive models for polymers will need to incorporate both strain rate and temperature effects on mechanical behavior.

4. SUMMARY AND CONCLUSIONS

Based upon this study of sample thickness, strain rate, and temperature at high strain rate on the constitutive response of Adiprene-L100, the following conclusions can be drawn: 1) the compressive stress-strain response of Adiprene-L100 was found to depend on both the applied strain rate; $0.001 \leq \dot{\epsilon} \leq 7000 \text{ s}^{-1}$, and the test temperature at high-rate; $77 \leq T \leq 298 \text{ K}$, 2) due to slow, dispersive wave propagation in Adiprene-L100, low aspect ratio samples are needed to assure uniform stress conditions within Hopkinson bar samples; the optimal sample thickness being dependent on test

temperature, and 3) decreasing temperature from 298 to 77K at 3000 s⁻¹ was found to increase the maximum flow stress in Adiprene-L100 from 10 to ~210 MPa.

Acknowledgments

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