High Strain-Rate Compressive Behavior of Bulk Structural Adhesives: Epoxy and Methacrylate Adhesives*

Takashi YOKOYAMA**, Kenji NAKAI** and Norfazrina Hayati MOHD YATIM***

**Department of Mechanical Engineering

Okayama University of Science, Okayama 700-0005, Japan

E-mail: yokoyama@mech.ous.ac.jp

***Graduate School of Engineering

Okayama University of Science, Okayama 700-0005, Japan

Abstract

The present paper describes the determination of high strain-rate compressive stress-strain loops for bulk specimens of two different epoxy and methacrylate structural adhesives on the standard split Hopkinson pressure bar with a tapered striker bar. The full compressive stress-strain data including unloading process are obtained over a wide range of strain rates from 10^{-3} to 10^3 /s at room temperature. The effects of strain rate on the initial (secant) modulus, flow stress, dissipation energy and hysteresis loss ratio are studied. The experimental results show that both bulk structural adhesives exhibit highly strain-rate dependent viscoelastic behavior like polymeric materials.

Key words: Bulk Structural Adhesive, Compressive Stress-Strain Loop, Dynamic Viscoelasticity, Elastic After-Effect, Hopkinson Bar, Strain Rate, Strain Recovery

1. Introduction

Structural adhesive bonding has extensively been used in automotive, aircraft and railway industries; wide classes of components are assembled using adhesive joints. In order to ensure their structural integrity in service, we need to know the dynamic behavior of the adhesive joints, which is greatly affected by the mechanical response of both adherends and adhesives to dynamic loading. So far, the impact compressive ⁽¹⁾, tensile ⁽²⁾, shear ⁽³⁻⁷⁾ and torsional ⁽⁸⁾ responses of the adhesive joints have often been determined on the drop-weight impact test apparatus or modified split Hopkinson pressure bar (SHPB) ⁽⁹⁾. Numerical simulations of SHPB tests on double-lap adhesive joints were performed using the finite element technique to examine the effects of material and geometrical properties of adhesives and adherends, and striker-bar impact conditions on the accuracy of the SHPB method ⁽¹⁰⁾. In an effort to isolate the effects of adherends and joint geometry on the dynamic response of the adhesive joints, the dynamic stress-strain properties of bulk structural adhesives were accurately evaluated in both tension and compression ⁽¹¹⁾. Recently, constitutive equations for bulk materials of structural adhesives have been proposed based on elasto-viscoplastic rheological models ^(12, 13). However, the dynamic stress-strain loops ⁽¹⁴⁾ and associated energy dissipation in the bulk structural adhesives have not been fully understood yet.

The objective of the present work is to determine the high strain-rate compressive stress-strain behavior for bulk specimens of two different structural adhesives on the SHPB. Bulk structural adhesive sheets with a uniform thickness were fabricated by a special

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solidification procedure. Cylindrical specimens machined from the bulk adhesive sheets were tested at room temperature. The low and intermediate strain-rate compressive stress-strain data were obtained using an Instron testing machine. The influences of strain rate on the initial (secant) modulus, flow stress, dissipation energy and hysteresis loss ratio were discussed in detail. It is shown that both bulk structural adhesives evidently exhibit intrinsic strain-rate dependent viscoelastic behavior and an elastic after-effect ⁽¹⁵⁾ following complete unloading like polymers.

2. Experimental Details

2.1 Test Adhesives and Tensile Properties

Commercially available two-part epoxy structural adhesive (thermoset), Scotch-Weld[®] DP-460 (Sumitomo 3M Limited, Japan) and methacrylate structural adhesive (thermoplastic), Plexus[®] MA560-1 (ITW PP&F Japan Co., Ltd.) were chosen. The former is suitable for use with metallic adherends, and the latter is suitable for use with non-metallic (or composite and polymer) adherends. Both bulk adhesive sheets with almost no porosity were fabricated by a special solidification technique⁽¹⁶⁾ and cured for 24 hr at room temperature, but not post-cured at higher temperatures. This is because the adherends to be joined do not tolerate high temperatures in industrial applications. Figure 1 gives a picture of two dumbbell-shaped specimens (JIS K7162) ⁽¹⁷⁾ with a gage length of 50 mm machined from the bulk adhesive sheets with a thickness of 3 mm. Tension tests were carried out in the Instron testing machine (Model 5500R) at a crosshead speed of 1mm/min (or strain rate of 3.3×10^{-4} /s). Typical tensile properties of the two bulk adhesives including glass transition temperatures are listed in Table 1.

Fig.1 Picture of static sheet tension specimens of bulk structural adhesives: DP-460 (top) and MA560-1 (bottom)

Table 1 Nominal tensile and physical properties of two different bulk adhesives

Adhesive	Initial modulus* E [GPa]	Tensile strength σ_{UTS} [MPa]	Elongation ϵ_f [%]	Glass Transition Temperature T_g [K]
DP-460	2.1	44.1	8.9	331
MA560-1	0.7	12.6	N/A**	332

* defined as the secant modulus at 0.2% strain, ** not available ($\epsilon_f > 100\%$)

2.2 Design of Compression Specimens

Figure 2 shows cylindrical specimens machined out of the bulk adhesive sheets with a thickness of 5 mm as depicted in Fig. 3. Following the designation of ASTM E9-89a ⁽¹⁸⁾, the slenderness ratio l/d (= length/diameter) of the static specimen was taken as large as 1.875 by stacking three thin identical specimens to prevent barreling of the specimen due to end friction during compression loading. The slenderness ratio of the impact specimen was taken as 0.625, falling in an appropriate slenderness ratio range between 0.5 and 1.0, suggested by Gray⁽¹⁹⁾ in the conventional SHPB tests (see, Table 2).

Fig.2 Picture of static and impact compression specimens: DP-460 (top); MA560-1 (bottom)

Fig.3 Picture of bulk adhesive sheets and locations of cylindrical specimen extraction: DP-460 (left) and MA560-1 (right)

Table 2 Nominal dimensions of static and impact compression specimens

Strain rate	Length l [mm]	Diameter d [mm]	Slenderness ratio l/d
Static/Intermediate	15	8	1.875
Impact	5	8	0.625

2.3 Low / Intermediate Strain-Rate Compression Testing

The low and intermediate strain-rate compression tests were conducted on the specimens with $l/d = 1.875$ using the Instron testing machine at a crosshead speed of 1 mm/min and 100 mm/min, respectively. Lubricant (or petroleum jelly) was applied to anvil/specimen interfaces to reduce the frictional effects. The specimens were loaded up to given strains and unloaded at the same two crosshead speeds. At least two repeatable tests were carried out at each crosshead velocity.

2.4 High Strain-Rate Compression Testing

A schematic of the SHPB apparatus ⁽²⁰⁾ is given in Fig. 4. The apparatus consists of two 2024-T4 Al alloy bars of 2000 mm in length and 10.1 mm in diameter. Instead of using a uniform striker bar, a tapered striker bar with a PEEK guide collar was used to generate constant strain-rate stress-strain data. A picture of the tapered striker bar is shown in Fig. 5.

Fig.4 Schematic of conventional SHPB apparatus (associated recording system not shown)

Fig.5 Shape and dimensions of tapered striker bar

Table 3 Mechanical properties of striker, input and output bars used in SHPB system

	Striker bar	Input bar	Output bar
Material	AA2017-T4	AA2024-T4	
Young's modulus E (GPa)	73	73	
Longitudinal wave velocity c_0 (m/s)	5120	5130	
Yield strength σ_Y (MPa)	405	450

Fig.6 Lagrangian $x-t$ diagram for SHPB test on bulk adhesives

The mechanical properties of the striker, input and output bars are listed in Table 3. Figure 6 depicts a Lagrangian $x-t$ diagram illustrating the details of the elastic wave propagation in the input and output bars. The Al alloy bars with low impedance were used to reduce a drastic impedance mismatch between the bulk adhesive specimen and the conventional steel bars, which resulted in a transmitted strain signal with a very low signal-to-noise ratio. The specimen was held in place between the input and output bars by applying a very small pre-compression load with turning of the head of a support block. As in the static tests, lubricant (or petroleum jelly) was applied to the bar/specimen interfaces to reduce the frictional effects. A pulse shaping technique⁽²¹⁾ was applied to generate well-defined incident strain pulses without higher frequency components in the input bar. Namely, a 0.2 mm thick 1050 Al disk of nearly 10 mm in diameter was attached onto the impact (left) end of the input bar using a thin layer of petroleum jelly. When the input bar was impacted with the tapered striker bar launched through the gun barrel, a compressive strain pulse (ϵ_i) was generated in the input bar and travels towards the specimen. At the bar/specimen interface, because of the impedance mismatch, part of the strain pulse was reflected back into the

input bar (ε_r) and the remaining part was transmitted through the specimen into the output bar (ε_t). The incident, reflect and transmitted strain pulses were then recorded with two pairs of strain gages mounted on the input and output bars. The output signals from the strain gages were fed through a bridge circuit into a 10-bit digital storage oscilloscope (Iwatsu: DS-9121), where the signals were digitized and stored at a sampling time of $1 \mu\text{s}/\text{word}$. The digitized data were then transferred to a 32-bit personal computer (PC-9821Xb: NEC) for data processing.

From the elementary one-dimensional theory of elastic wave propagation, we can determine the nominal strain $\varepsilon(t)$, strain rate $\dot{\varepsilon}(t)$ and stress $\sigma(t)$ in the specimen from the SHPB test records as ⁽²²⁾

$$\varepsilon(t) = \frac{u_1(t) - u_2(t)}{l} = \frac{2c_0}{l} \int_0^t \{\varepsilon_i(t') - \varepsilon_r(t')\} dt' \quad (1)$$

$$\dot{\varepsilon}(t) = \frac{\dot{u}_1(t) - \dot{u}_2(t)}{l} = \frac{2c_0}{l} \{\varepsilon_i(t) - \varepsilon_r(t)\} \quad (2)$$

$$\sigma(t) = \frac{P_2(t)}{A_s} = \frac{AE}{A_s} \varepsilon_t(t) \quad (3)$$

Here u and P are the displacement and the axial force on both ends of the specimen, respectively, (see the inset in Fig. 4); A and E are the cross-sectional area and Young's modulus of the Hopkinson (2024-T4 Al alloy) bars; A_s is the cross-sectional area of the specimen. Equations (1) to (3) are derived under the assumption of dynamic force equilibrium across the specimen, which can be expressed as

$$P_1(t) = P_2(t) \quad \text{or} \quad \varepsilon_i(t) + \varepsilon_r(t) = \varepsilon_t(t)$$

where

$$P_1(t) = AE[\varepsilon_i(t) + \varepsilon_r(t)], \quad P_2(t) = AE\varepsilon_t(t)$$

In the above derivations, the incident and reflected strain pulses are time-shifted to the specimen-input bar interface, and the transmitted strain pulse is time-shifted to the specimen-output bar interface (see, Fig.6). Eliminating time t through Eqs. (1) to (3) yields the nominal (or engineering) compressive stress-strain and strain rate-strain relations. In this work, the compressive stress and strain are taken as positive.

3. Results and Discussion

3.1 SHPB Tests

A number of the SHPB tests were conducted on the bulk adhesive specimens at room temperature. Figure 7 indicates typical oscilloscope traces of incident and reflected strain pulses (ε_i and ε_r) and transmitted strain pulse (ε_t) from the SHPB test on the epoxy adhesive DP-460. The recorded signal data are neither smoothed nor averaged electronically. Note that the duration ($\approx 400\mu\text{s}$) of the reflected and transmitted strain pulses is much longer than that ($\approx 250\mu\text{s}$) of the incident strain pulse. This is because it takes much time for the specimen stress to decay to zero due to relaxation phenomena (see, Fig. 9), causing often the overlapping between the transmitted strain pulse and its strain pulse reflected from the free (or right) end of the output bar. In the present tests, the overlapping of the two strain pulses is successfully avoided by the use of the long output bar. Figure 8 gives the resulting axial stress histories at the front and back ends of the specimen. The nearly overlapping histories clearly indicate that dynamic stress equilibrium (stress uniformity) is achieved in

the specimen over the entire duration of loading. Figure 9 shows the dynamic compressive stress and strain histories for the epoxy adhesive DP-460. Note that the dynamic strain history lags behind the dynamic stress history during loading and unloading, suggesting typical viscoelastic retardation effects.

Figure 10 presents the resulting dynamic stress-strain and strain rate-strain loops in compression for the epoxy adhesive DP-460. The strain-rate flips its sign from compression (loading) to tension (unloading) at the peak of the loading strain. The strain rates $\dot{\epsilon} = 920/s$ and $\dot{\epsilon} = -300/s$ given, respectively, denote the average strain rates during loading and unloading process. As in the low and intermediate strain rate tests, the dynamic stress-strain loop is not closed, and consequently, a residual strain of about 0.02 is not completely recovered to zero in time.

Figures 11 and 12 show the compressive stress-strain loops for the two different bulk adhesives at three different strain rates. The initial slope (or initial modulus E) and the area within the loop for both adhesives increase greatly with increasing strain rate. Furthermore, initial residual strains after complete unloading are found to decrease with increasing strain rate for the epoxy adhesive DP-460, but to remain almost unvaried for the methacrylate adhesive MA560-1. The strain-rate dependent stress-strain behavior for both bulk adhesives is consistent with that for the bulk epoxy adhesive Hysol 9466⁽¹²⁾, determined using the SHPB.

Fig.8 Front and back stress histories on each face of epoxy adhesive DP-460 specimen

Fig.9 Dynamic stress and strain histories for epoxy adhesive DP-460

Fig.10 Dynamic compressive stress-strain (solid line) and strain rate-strain (dashed line) loops for epoxy adhesive DP-460

Fig.11 Compressive stress-strain loops at three different strain rates for epoxy adhesive DP-460 (solid, double and open circles marked on horizontal axis indicate initial residual strains)

Fig.12 Compressive stress-strain loops at three different strain rates for methacrylate adhesive MA560-1 (see key in Fig. 11)

3.2 Strain-Rate Dependence

In order to evaluate the effects of strain rate on the compressive properties of the two different bulk adhesives, the measured values for the initial modulus (as the secant modulus at 0.002 strain), flow stress at given strains of 0.02 and 0.04, dissipation energy and hysteresis loss ratio up to $\varepsilon_{\max} \doteq 0.076$ and 0.084 are plotted in Figs. 13 to 15 against the average strain rate. As seen from Fig. 10, the initial strain rate in the strain rate-strain loop is much smaller than the average strain rate during loading. Therefore, the average strain rate associated with the initial modulus is estimated from the area under the strain rate-strain relation up to 0.002 strain divided by 0.002. Figure 16 illustrates that the dissipation energy (= hysteresis loss) U_d is defined as the area within the stress-strain loop. The hysteresis loss ratio $H_e^{(23)}$ is then calculated as the ratio of the hysteresis loss U_d to the area W under the stress-strain curve during loading. The initial modulus, flow stress at given strains of 0.02 and 0.04 and dissipation energy increase significantly with increasing strain rate for both adhesives. In contrast, the hysteresis loss ratio for the epoxy adhesive DP-460 significantly decreases with increasing strain rate, while that for the methacrylate adhesive MA560-1 remains almost unchanged. The dissipation energy is mostly converted to heat during high rate deformation, causing the adiabatic temperature rise within the specimen.

Fig.13 Effect of strain rate on initial modulus for two different bulk adhesives

Fig.14 Effect of strain rate on flow stress at given strains of 0.02 and 0.04 for two different bulk adhesives

Fig.15 Effect of strain rate on dissipation energy and hysteresis loss ratio up to maximum strain $\varepsilon_{\max} \cong 0.08$ for two different bulk adhesives

Fig.16 Schematic illustration of dissipation energy and hysteresis loss ratio under stress-strain curve up to maximum strain $\varepsilon_{\max}$

In an attempt to quantitatively evaluate the rate dependence of the flow stress, two different strain-rate sensitivity parameters β and n ⁽²⁴⁾ are introduced. The two parameters estimated for the respective bulk adhesives are summarized in Table 4, where σ_1 and σ_2 are stresses at the average strain rates $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$, respectively, for the fixed two values of $\epsilon = 0.02$ and 0.04 . It is obvious that the methacrylate adhesive MA560-1 exhibits much higher strain-rate sensitivity than the epoxy adhesive DP-460, irrespective of prescribed strains. The two parameters β and n are not constant, and dependent on the strain as well as on the strain rate. However, these parameters are useful as a measure of the strain-rate sensitivity.

Table 4 Strain-rate sensitivity parameters for two different bulk adhesives within a range of strain rates from $\dot{\epsilon}_1 = 1.1 \times 10^{-3} / \text{s}$ to $\dot{\epsilon}_2 \cong 10^3 / \text{s}$

Adhesive	$\beta = \frac{\sigma_2 - \sigma_1}{\log(\dot{\epsilon}_2 / \dot{\epsilon}_1)} \left \begin{matrix} [\text{MPa}] \\ \epsilon = \epsilon_0 \end{matrix} \right $		$n = \frac{\log(\sigma_2 / \sigma_1)}{\log(\dot{\epsilon}_2 / \dot{\epsilon}_1)} \left \begin{matrix} [-] \\ \epsilon = \epsilon_0 \end{matrix} \right $	
	$\dot{\epsilon}_2 > \dot{\epsilon}_1$		$\dot{\epsilon}_2 > \dot{\epsilon}_1$	
	$\epsilon_0 = 0.02$	$\epsilon_0 = 0.04$	$\epsilon_0 = 0.02$	$\epsilon_0 = 0.04$
DP-460	3.56	7.82	0.033	0.055
MA560-1	7.09	9.02	0.107	0.090

3.3 Strain Recovery and Elastic After-Effect

We now consider the time dependence of strain recovery at different unloading strain rates. As seen in Fig.11, the amount of initial residual strains after complete unloading varies, depending on the strain rate when the maximum loading strain is almost the same ($\epsilon_{\text{max}} = 0.076$). The initial residual strain slowly decays to nearly zero in time, whose behavior is known as an elastic after-effect ⁽¹⁶⁾, inherent to polymeric materials. Figures 17 and 18 show the histories of recovery strains during unloading and the initial residual strains at three different unloading strain rates. The horizontal axis indicates the time after the start of unloading on each stress-strain curve (note that for convenience sake, the origin on a logarithmic time scale is set equal to $1 \mu\text{s}$). The strains at a start point on the dashed lines correspond to each initial residual strain just after unloading. Arrows on the dashed lines indicate the phenomenon of “elastic after-effect”. The residual strains at 120 and 300 seconds after complete unloading were precisely determined through measurements of the specimen length with a digital sliding caliper (Mitutoyo Corp., Model CD-15C).

The initial residual strains in the DP-460 get smaller with increasing unloading strain rates, whereas those in the MA560-1 are recovered to nearly the same value of strain, independent of the unloading strain rates. This different strain recovery behavior may arise from differences in the molecular structures between the thermosetting adhesive (DP-460) and the thermoplastic adhesive (MA560-1). Thermosetting adhesives are composed of cross-linked long-chain molecules, unlike the thermoplastic adhesives. As seen from Figs. 13 and 14, the cross-linked adhesive (DP-460) exhibits higher stiffness and strength than the thermoplastic adhesive (MA560-1). Nevertheless, why the thermosetting adhesive (DP-460) shows the unloading strain-rate dependence of initial residual strains is not clear at present. A similar unloading strain-rate dependence of initial residual strains was also reported by Yi, *et al* ⁽²⁵⁾ on a thermosetting polyurea at strain rates ranging from 10^{-3} to $1/\text{s}$. Further study is required to elucidate the effect of strain rate history on the strain recovery process in the bulk adhesives.

Fig.17 Histories of recovery strains during unloading and initial residual strains at three different unloading strain rates within epoxy adhesive DP-460 specimen

Fig.18 Histories of recovery strains during unloading and initial residual strains at three different unloading strain rates within methacrylate adhesive MA560-1 specimen

4. Summary and Conclusions

The influences of strain rate on the compressive stress-strain loops for the two different bulk adhesives DP-460 and MA560-1 for structural bonding have been investigated using the conventional SHPB with the tapered striker bar. The effects of strain rate on the amount of the initial residual strains in the compression specimen were also examined under almost the same maximum loading strain conditions. From this experimental work, we can draw the following conclusions:

- 1) The two different bulk adhesives exhibit intrinsic dynamic viscoelastic behavior and a slight “elastic after-effect” following complete unloading like polymers.
- 2) The initial (secant) modulus, flow stress at fixed strains and dissipation energy for the two different adhesives increase greatly with increasing strain rate.
- 3) The hysteresis loss ratio for the DP-460 becomes lower than that for the MA560-1 at higher strain rates, implying that the former is more excellent than the latter in the hysteresis loss performance.
- 4) The MA560-1 shows even higher strain-rate sensitivity than the DP-460.

- 5) The amount of the initial residual strains for the DP-460 after complete unloading varies, depending on the unloading strain rate when the maximum loading strain is almost the same.
- 6) The use of the tapered striker bar in the SHPB tests is effective for obtaining valid dynamic stress-strain data at nearly constant strain rates.

The use of the two structural adhesives should be selected properly for any purpose of adhesive bonding, considering their dynamic features. Further work is needed to examine the effect of temperature as well as strain rate on the stress-strain behavior of both bulk structural adhesives.

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