The Application of the Iosipescu Shear Test to Structural Adhesives

I. GRABOVAC* and C. E. M. MORRIS

DSTO, Materials Research Laboratory, Melbourne, Victoria 3032, Australia

SYNOPSIS

The Iosipescu shear test method, adapted for adhesives, was used to evaluate shear stress–strain properties of five epoxy-based film adhesives. The shear strength of some adhesives, determined by this technique, was found to differ greatly from that determined by either lap shear and/or thick adherend tests. Accurate measurements of the adhesive bond-line deformation enabled evaluation of the adhesive elastic and plastic properties as well as the calculation of shear modulus. A high-speed video camera was employed to augment observation on the joint deformation.

INTRODUCTION

The Iosipescu shear test method, originally developed for isotropic materials, was first introduced in an English language paper in 1967.[1] Initially, its acceptance in the field of organic materials was slow and basically confined to fiber-reinforced composites. Over the years its popularity has grown, primarily through the work of Walrath and Adams.[2] In a recent survey of nine in-plane shear test methods, Lee and Munro[3] concluded that, overall, the Iosipescu method is the most practical technique currently available for testing composite materials, as it is relatively simple to conduct, employs small, easily fabricated (composite) specimens, and is capable of measuring both shear strength and stiffness. Moreover, viscoelastic analysis, performed on isotropic specimens by Iosipescu[1] and Sullivan et al.,[4] have shown that a quite uniform shear stress distribution is achieved provided the specimen notches and loading are optimized.

It was suggested,[5] however, that with slight specimen modification the technique could be adapted to test adhesives and adhesive bonds, but this suggestion does not appear to have been pursued. Recently, work conducted at this laboratory has focused on adapting the Iosipescu method for shear stress–strain analysis of film adhesives. The method development and description of specially designed auxiliary instrumentation required for this application is given elsewhere.[6] In this paper, the application of that method to a selection of hot-curing, epoxy-based, structural film adhesives is presented. It is assumed that the shear stress is uniformly distributed in the adhesive layer since the specimen and test grips geometry are as given in the original Iosipescu work.[1] The large disparity in strength and elastic modulus between the aluminum adherends and the adhesive that fills the specimen discontinuity at the shear plane should further support this assumption.

EXPERIMENTAL

Sample Preparation

Five commercially available epoxy-based film adhesives were employed in this work (Table I). AF 130 and AF 163-2K were obtained from 3M Co., St Paul, MN; FM 300 and FM 1000, from American Cyanamid, Havre de Grace, MD; and BSL 322 from Ciba-Geigy Ltd., Duxford, UK. All adhesives were stored at −18°C until used.

Modification of the Iosipescu specimen comprised cutting the specimen and introducing an adhesive between the roots of the notches and, thus, by bonding the two adherends, reconstituting the original

* To whom correspondence should be addressed.

Journal of Applied Polymer Science, Vol. 43, 2033–2042 (1991)

 CCC 0021-8995/91/112033-10$04.00

Table I Epoxy Film Adhesives Studied

Adhesive	Commercial Name	Cure Schedule Used	Film Adhesive Type
A	AF 130	1 h at 177°C, 0.34 MPa	Supported
B	BSL 322	1 h at 177°C, 0.31 MPa	Supported, modified
C	FM 300	1 h at 177°C, 0.31 MPa	Supported, modified
D	AF 163-2K	1 h at 121°C, 0.31 MPa	Supported, modified
E	FM 1000	1 h at 177°C, 0.34 MPa	Supported, modified

specimen shape. The geometry and dimensions of the modified Iosipescu specimen are shown in Figure 1. No lateral notches were used on any of the specimens. Adherends were machined to very close tolerances from aluminum alloy 5083, temper H321, obtained from Alcan Australia Ltd.

For comparison, lap shear and thick adherend tests were also conducted. These joints were constructed using Alclad or bare 2024-T3 aluminum, 1.6 or 6.4 mm thick (for lap shear or thick adherend specimens, respectively). For lap shear specimens, joint geometry and preparation methods were in accord with standard procedures.[7] Two thick adherend geometries were employed: a standard (12.7 mm) overlap[8] and a short (5 mm) overlap.[9]

In all four cases, surface preparation consisted of a solvent wipe and vapor degrease with 1,1,1-trichloroethane followed by an FPL etch.[10] No primer was used in any instance.

The lap shear and thick adherend joints were made in a heated platen press, with an average heat-up rate of 3.8°C/min, using the cure cycle recommended for the particular adhesive (Table I). For the Iosipescu specimens, a specially designed bonding jig was used that enabled two test specimens to be prepared at a time. The design is described in detail elsewhere.[6] Assembled specimens were cured in an air-forced oven at the specified adhesive cure temperature. The heat-up rate was approximately 2°C/min, and the cure temperature was kept to within ±2°C. Upon termination of a cure cycle, specimens were left to cool down overnight.

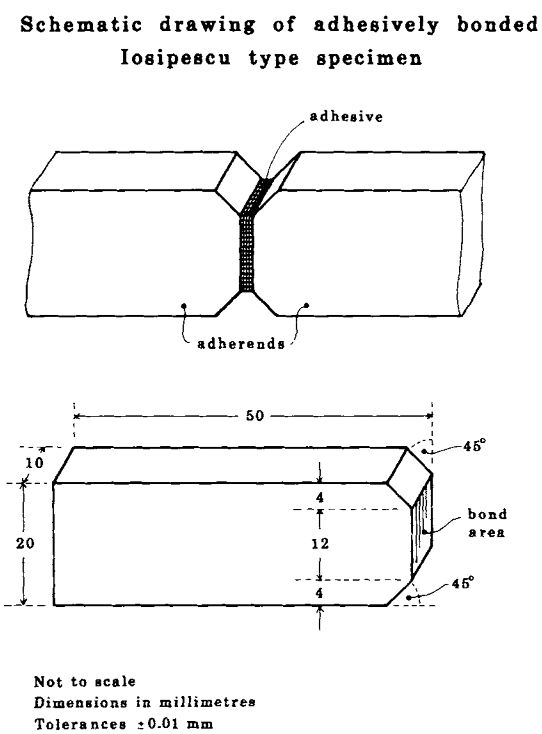

Figure 1 Iosipescu-type specimen for adhesive testing.

Specimen Testing

All joint testing was carried out in a temperature-controlled room (at 23 ± 2°C) using an Instron model 1185 universal testing machine at a constant loading rate of 9 MPa/min.

To obtain meaningful and reliable shear stress–strain data from the Iosipescu tests, the adhesive bond-line thickness, normally around 0.13 mm, must be accurately determined. This was achieved by the use of a modified, commercially available instrument that enables bond-line thickness to be measured with an accuracy of 2.5×10^{-3} mm, provided considerable care is exercised in specimen handling and conditioning.[6]

The small bond-line deformation due to application of the shear force was measured as a relative displacement of adherends with an in-house, purpose-designed and -built shearometer[6] (patent applied for) having a measuring range of about 0.5 mm. Basically, the shearometer consists of four electrical resistance strain gauges adhesively bonded to a two-prong elastic fork and wired in a Wheatstone bridge circuit. Each prong terminates in a small steel block, which enables physical attachment to the adherends.[6] The power supply, signal conditioning, and recording was provided by internal Instron circuitry accessible through a standard extensometer receptacle. There is a linear relationship between the shearometer displacement and the Wheatstone bridge output.[6] The raw data were corrected for the contribution from the elastic deformation of the adherends, determined under identical test conditions using an identically shaped, all-aluminum (nonbonded) specimen. The test grips, used to apply uniform shear force to the specimen between the "V" notches, were the established Iosipescu design.[1]

Progress of the Iosipescu tests was also followed with high-speed video cameras. Two SP2000 cameras, manufactured by Spin Physics, San Diego, CA, were used, one focused on a hand-drawn grid covering the specimen bond line and surrounding area and the other focused on the load recording device of the testing machine. The signals from both cameras were recorded simultaneously in order to relate visual events to stress on the specimen. Recording speed was 2000 frames/s, allowing a maximum recording time of 45 s. The recorded data were downloaded from the high-speed video tape onto a Sony U-matic video cassette for storage and subsequent image processing on the Interactive Image Analysis System manufactured by Kontron, Germany. The specimen image was magnified 6×. The image memory and array processor consisted of 4 × 256 kByte image memory capable of supporting a resolution of 512 × 512 pixels.

RESULTS AND DISCUSSION

Shear Stress Analysis

The shear-force application on all the types of adhesive joints considered in this paper was performed in tension at a constant loading rate. The effect of change in this loading rate on the ultimate joint strength was investigated for Iosipescu-type specimens bonded with FM 300 over the loading range 4.83 to 13.10 MPa/min (Fig. 2). Statistically, the results did not show any significant deviation over the loading range examined for this adhesive. A somewhat higher average shear stress was obtained at a loading rate of 8.97 MPa/min, which was coincident with the value specified by the ASTM method for adhesive shear strength evaluation by tension loading of metal-to-metal joints.[11] To obtain comparative results for all adhesives, studied here by Iosipescu, and lap shear tests, the same loading rate

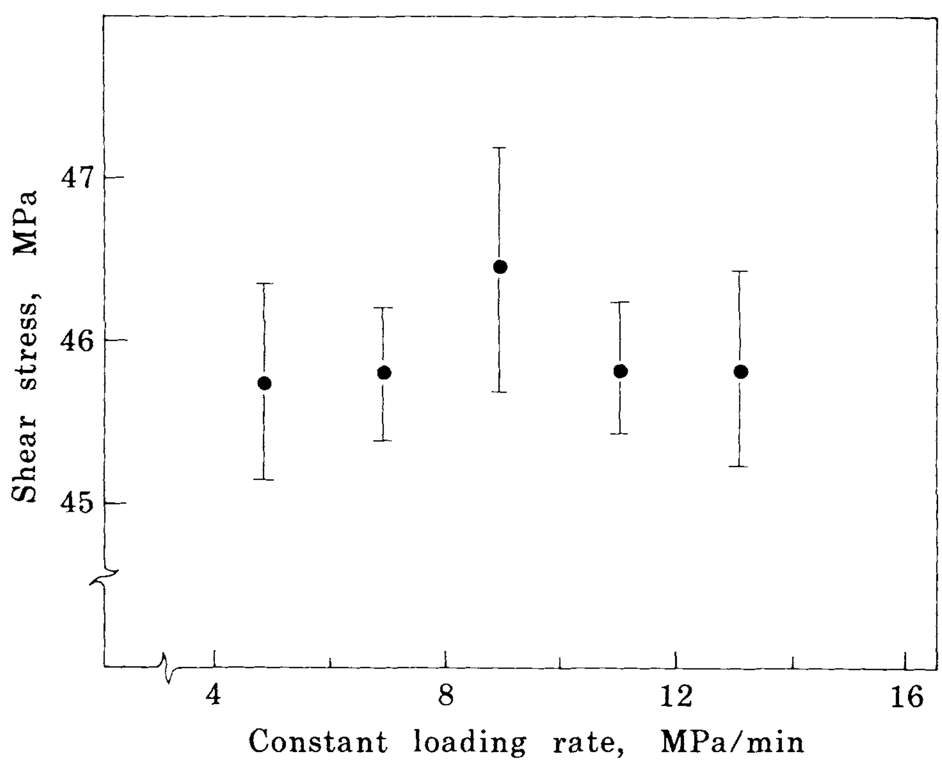

Figure 2 Effect of loading rate on shear stress.

of 8.97 MPa/min was adopted in all further work. With regard to adhesive shear strength, it is noted that the overall value of about 46 MPa (Fig. 2) is approximately 16% higher than that reported later for a batch of FM 300 with a different thermal history. Although the test was found to be insensitive to varied loading rates for FM 300, these results indicate the test's capability to differentiate between batches of different quality.

The shear strength of the five commercial adhesives, determined by lap shear and Iosipescu tests, is illustrated in Figure 3. Clearly, for adhesives AF 130, BSL 322, and FM 1000, values obtained by these two methods differ markedly. For adhesives FM 300 and AF 163-2K, these differences are reduced to within the experimental error. For comparison, Figure 3 also shows the results from the thick adherend test with a standard overlap for AF 130 and FM 1000 and with a short overlap for AF 130.

Basically, the variations in measured shear strength of an adhesive arise from a combination of test geometry and adhesive ductility. Problems stemming from the asymmetric nature of the lap shear test geometry are well known.[12,13] During the application of the load, distortion of the adherends occurs, resulting in a very uneven stress distribution and in tensile forces at the ends of the overlap. Once these forces exceed either the adhesive's cohesive strength or its adhesion to the adherend, rupture takes place. Depending on adhesive ductility, failure may be premature, as in brittle adhesives such as AF 130. Modified adhesives such as FM 300, AF 163-2K, and FM 1000, however, can withstand higher loading by dissipating the peel and cleavage forces at the ends of the overlap, thus producing a higher shear strength that may result in significant inelastic deformation of thin adherends. The thick adherend test configuration significantly reduces, but not entirely eliminates, bending effects. In the case of AF 130 and FM 1000, adherend bending was noticeable at loads over approximately 5 kN, but because the joint strength remained within the adherend's elastic region, no permanent deformation was observed. Reduced adherend bending, compared with the lap shear case, resulted in significantly increased shear strength for AF 130 and marginal changes for FM 1000. A further decrease of adherend bending, by using the short overlap specimen, caused a further rise in shear strength for AF 130. However, due to other effects, such as the increase in the edge effect relative to the small overlap area and some adherend bending, the value is still well below that obtained by the Iosipescu shear test. The uniform shear stress distribution and freedom from adherend

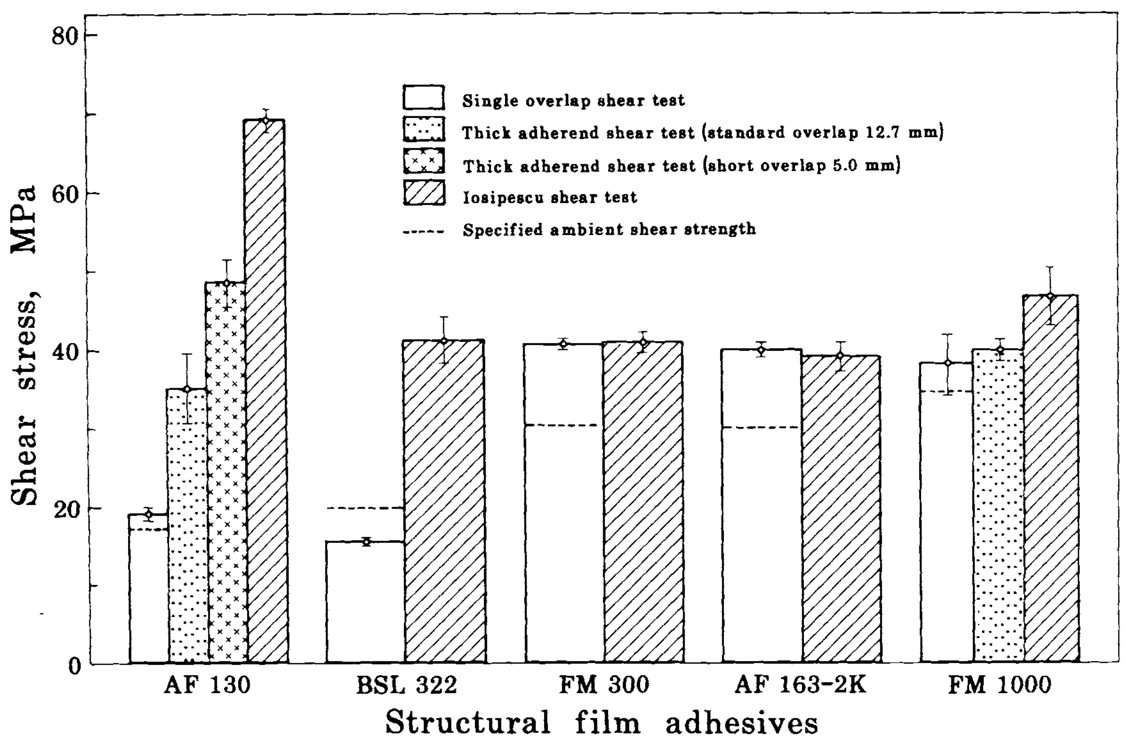

Figure 3 Strength of commercial adhesives determined by different shear tests.

distortions of the Iosipescu configuration permits determination of the true shear strength of the adhesive.

In the case of AF 130, attention must be paid to the orientation of the glass fiber support in the adhesive joint with respect to the axis of the shear force, as anisotropy of shear strength exists in the warp and weft directions. A related effect has been noted previously.[14] In this case, all joints using AF 130 were constructed with fibers aligned in a "stronger joint" configuration with respect to the shear axis. The difference between the two possible extremes is around 18% as measured by the Iosipescu shear test.

Stress–Strain Analysis

The joint geometry related to the shear stress–strain analysis of an adhesively bonded Iosipescu specimen and a model shear stress–strain curve is illustrated in Figure 4(a) and (b), respectively. From this, it is seen that the shear strain, shear strain-to-failure, and the modulus are given by

$$\gamma = \frac{\delta}{t} = \tan \Theta \qquad (1)$$

$$\gamma_f = \gamma_e + \gamma_p \qquad (2)$$

$$G = \frac{\tau}{\gamma} \qquad (3)$$

where γ = shear strain, γ_e = elastic shear strain, δ = displacement, γ_p = plastic shear strain, t = bond line thickness, G = adhesive shear modulus, γ_f = shear strain to failure, and τ = shear stress on adhesive.

The reproducibility and consistency of the test technique is demonstrated by the four replicate shear stress–strain traces for AF 163-2K shown in Figure 5. The traces are typical of all the other modified adhesives investigated, including the observed variation of the strain-to-failure parameter (γ_f). The differences in strain-to-failure for replicates of an adhesive, although small, could be related to the variation of the bond-line thickness; this aspect is the subject of further study. Average shear stress–strain curves for the five adhesives investigated are illustrated in Figure 6, and the mechanical properties derived from these curves are presented in Table II.

The five adhesives examined here are each of a different epoxy formulation, and each of the modified systems contains a different class of modifier. The brittle systems AF 130 and BSL 322 do not appear to possess any significant nonlinear behavior, contrary to Hart-Smith's[15] observation for brittle systems of "significant non-linear behaviour near the upper limit of their operating environments." For AF 130, where the strain-to-failure is extremely small (and thus the accuracy impaired), the shear modulus is significantly higher than that of the other systems examined (Table II). A possible contribution of a glass cloth carrier to its stiffness in this case should not be excluded. The value of the shear modulus for this adhesive, approximately 2.5 GPa, is still only one-tenth that of the aluminum alloy used in this work.[16] BSL 322 also shows typical brit-

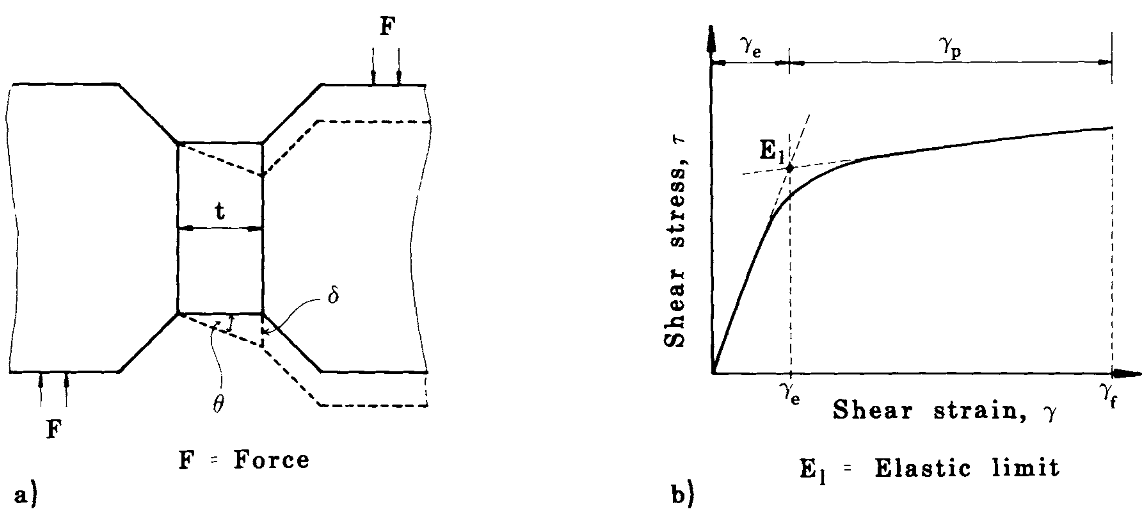

Figure 4 Model diagrams for shear stress–strain analysis: (a) joint geometry; (b) shear stress–strain curve.

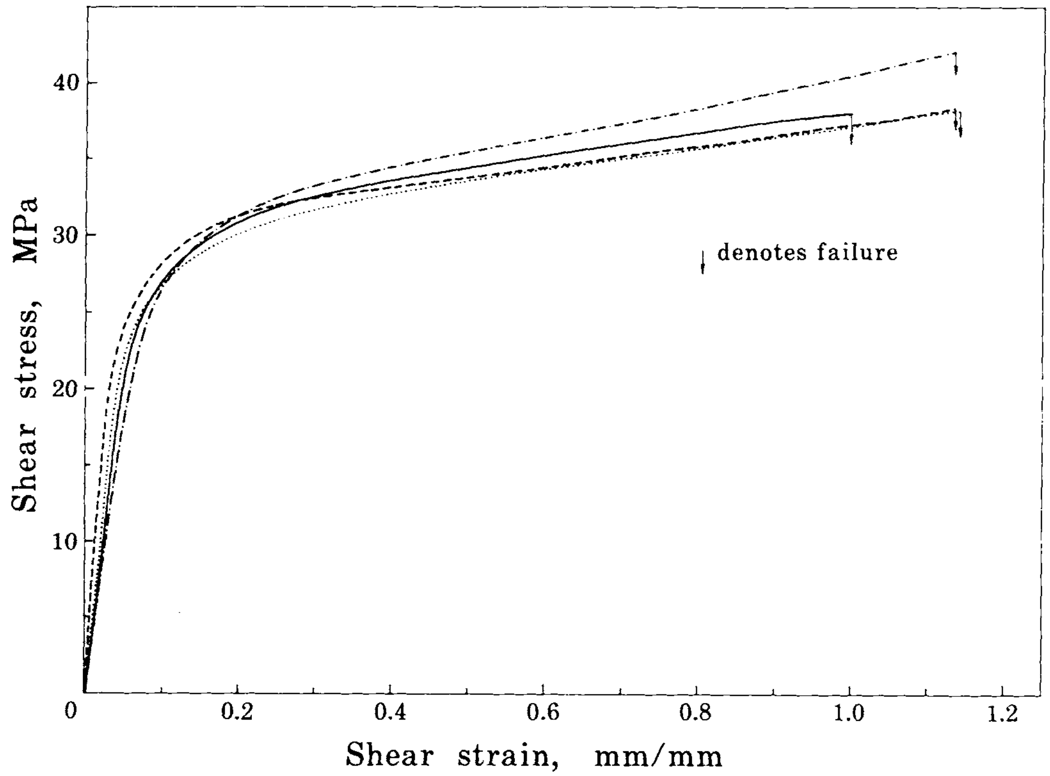

Figure 5 Replicate shear stress–strain curves for AF 163-2K.

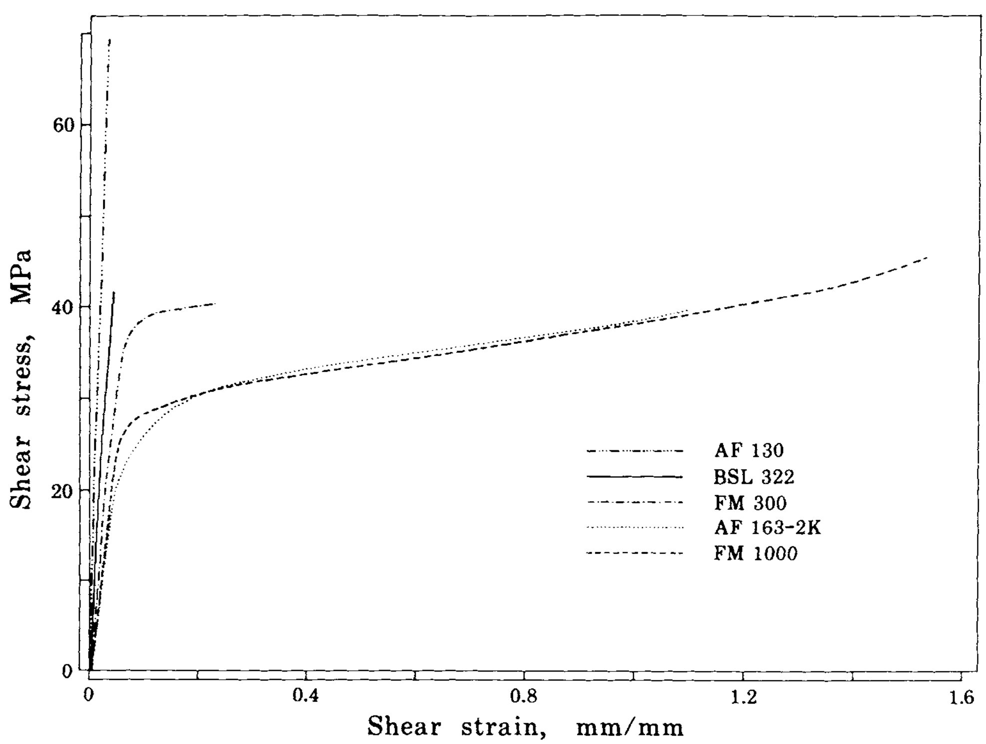

Figure 6 Average shear stress–strain curves of commercial adhesives.

Table II Shear Properties Derived from Stress–Strain Data

	Elastic Limit, E_1		At Failure		
Adhesive	Stress, τ (MPa)	Strain, γ	Stress, τ (MPa)	Strain, γ	Modulus, G (MPa)
AF 130	—	—	68.9 ± 2.1	0.036 ± 0.019	2480 ± 1050
BSL 322	—	—	41.5 ± 2.7	0.046 ± 0.009	1260 ± 150
FM 300	38.5 ± 0.4	0.051 ± 0.011	40.4 ± 1.1	0.239 ± 0.041	790 ± 180
AF 163-2K	29.4 ± 0.4	0.069 ± 0.017	39.4 ± 1.8	1.102 ± 0.070	450 ± 130
FM 1000	28.7 ± 1.1	0.057 ± 0.011	44.9 ± 1.8	1.533 ± 0.157	520 ± 90

Average of three and four specimens for adhesives A and B, and C, D, and E, respectively.

tle system behavior despite the presence of a modifier. In this case, it is not clear what the role of the modifier is or whether the adhesive was affected in any way by its prolonged subzero storage of approximately 9 years. (The lap shear strength was somewhat low.)

The other systems exhibited various degrees of ductility. The high-temperature cured (177°C) adhesives FM 300 and FM 1000 possess significantly different mechanical properties although their ultimate shear strengths differ little. For example, the elastic limit, E_1, of FM 1000 is only about 75% that of FM 300, but its strain-to-failure is about six times larger. AF 163-2K, although cured at a lower temperature (121°C), possesses a shear stress–strain curve that closely matches that of FM 1000, the difference being a lower shear stress and strain-to-failure (Fig. 6). Additionally, as a result of using a tangent method to determine E_1 (Fig. 4b), the estimated value of the elastic limit is almost identical to that of FM 1000, but, as shown in Figure 6, its actual deviation from linearity occurs significantly earlier in the test.

Comparison of the shear data obtained in this work with those of other workers using other methods is difficult as not many such studies have been reported and the adhesive employed is often not identified. Table III shows shear modulus results, in all cases by means of thick adherend tests, employing either the Krieger[18] or Althof et al.[9] version of the shearometer. (In some instances, the modulus value has been calculated from the published shear stress–strain curve.) By comparison, the shear modulus results, reported in this work (Table II), are consistently lower. It is believed that the observed disparity between the two sets of results lies in the asymmetric test configuration associated with the thick adherend lap shear method. Further investigation in this area is continuing. The shear modulus of a number of unnamed modified epoxy adhesive systems, determined by a torsional butt joint technique, were reported by Stringer[21] as being in the range 400–750 MPa. In this case, the problem of undisclosed adhesive identity combined with the nonuniform stress distribution in the adhesive layer make any direct comparison of shear data difficult.

The effect of the bond-line thickness on shear stress and strain to failure, in the present case, cannot be evaluated to the extent studied by Stringer[21] because of the very small bond-line thickness variation (20% maximum compared with 4–5 times for a given adhesive in Stringer's study). However, in the absence of more conclusive evidence, the initial results tend to indicate, at least, that there is a re-

Table III Literature Values of Shear Modulus

Adhesive	Shear Modulus (MPa)	Source of Data	References
BSL 322	1630	Quoted value	17
FM 300	700,720	Calculated from stress/strain diagram	18, 19
AF 163-2K	690	Calculated from stress/strain diagram	20
FM 1000	750	Quoted value	9

lationship between the bond-line thickness and the extent of the bond-line shear distortion for a given adhesive.

Regarding the reproducibility of the data, the variation of shear modulus and strain-to-failure (Table II) was largest for AF 130. This is due mainly to the very small strain involved; by analyzing a greater number of specimens and using digital data acquisition, the scatter of the results should be reduced. Additionally, a redesigned shearometer should minimize the operator variability during the clipping-on procedure, which was found to be a critical factor affecting reproducibility.

The failure process was also examined by the use of a high-speed video camera. It was hoped, by this means, to gain further insights into the relative displacement of the two adherends during the test and the speed of the fracture. (Knowledge of the fracture speed would enable calculation of the minimum kinetic energy involved, which could be used in the design of a new automatic detachment mechanism for the improved shearometer.) This approach was a partial success. Figure 7 shows two successive frames taken at the maximum camera speed of 2000 frames/s, before and after fracture, for the most brittle and ductile adhesives, AF 130 and FM 1000, respectively. In both cases, the fracture occurs within 0.5 ms. Superimposed on the right-hand side of the video image is the strip chart showing the load, recorded simultaneously with another camera. AF 130 developed 6.9 kN in this instance before failing and is a typical case of a "weaker joint" with respect to the glass fiber orientation, discussed earlier, for which the weave construction pattern is just discernible in Figure 8.

Inadequate resolution of the image-processing equipment prevented use of the video images for measurement of the relative displacement of the adherends. This limitation also prevented direct determination of the extent of adherend elastic deformation, evaluated indirectly by the use of a shearometer-equipped dummy Iosipescu specimen.[6] To overcome this restriction, object magnification of about two orders of magnitude would be required

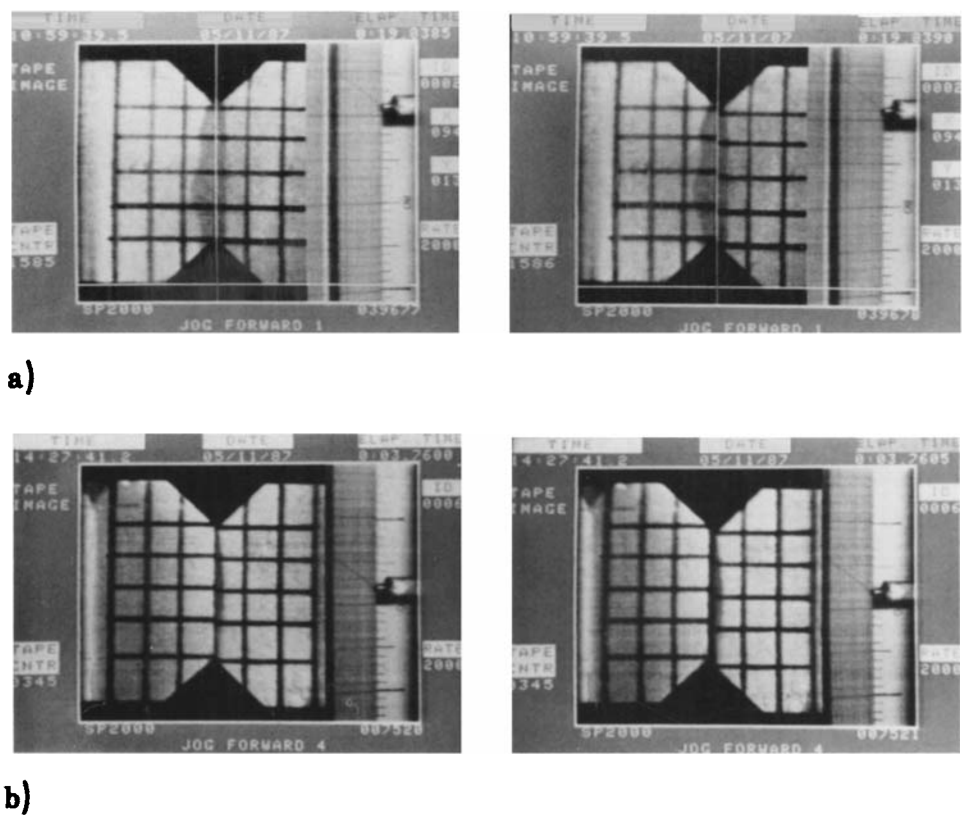

Figure 7 Specimen image before and after break: (a) adhesive AF 130; (b) adhesive FM 1000.

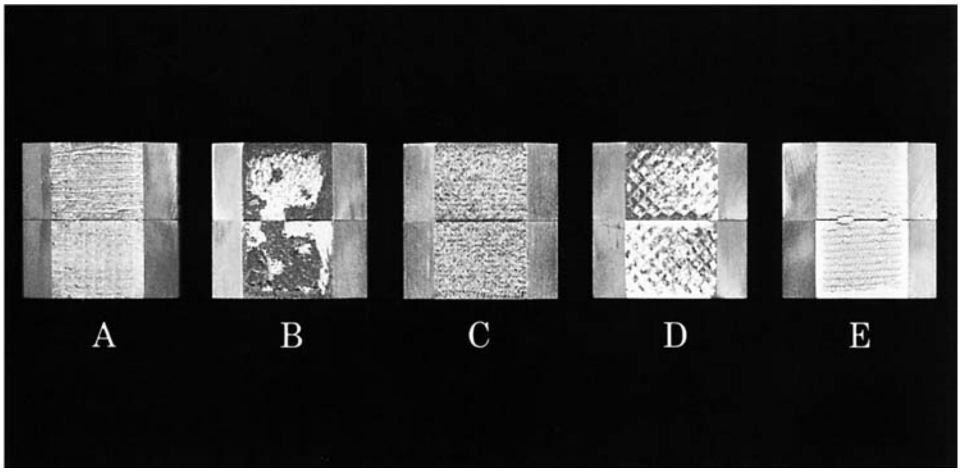

Figure 8 Surface appearance of fractured specimens.

together with a suitable microgrid covering the areas of interest. However, apart from the visual record of the failure event, the video recording was useful in observing the occasional small specimen tilting during the test. Although smaller than the error of one pixel, i.e., $< 1°$ from the original load-free condition, this indication of tilting or specimen rotation emphasizes the importance of proper specimen loading and fixing in the tension grips, as discussed elsewhere.[6]

The mode of failure observed throughout this work was typically cohesive except for BSL 322, which showed some indication of a partial adhesive failure, possibly a result of its age. Figure 8 shows the typical surface appearance of the fractured Iosipescu specimens.

CONCLUSIONS

The Iosipescu shear test, adapted for use with adhesives, has been shown to be a suitable technique for determination of both adhesive shear strength and stiffness in systems ranging from brittle to tough. It also permits determination of the elastic and plastic components of the shear deformation and the total strain-to-failure.

Compared to other tests based on a single overlap configuration, the advantage of this test is especially obvious in evaluation of shear stress–strain properties of strong brittle adhesive systems that are very susceptible to premature failure at even minimal deformation of the adherends. Some potential avenues for further enhancement of the ease of use of the method and reproducibility of results have been identified.

The authors wish to acknowledge the technical assistance of Mr. A. W. Camilleri for performing a part of the experimental work.

REFERENCES

1. N. Iosipescu, *J. Mater.*, **2**(3), 537 (1967).
2. D. E. Walrath and D. F. Adams, *Exp. Mech.*, **23**(1), 105 (1983).
3. S. Lee and M. Munro, *Composites,* **17**(1), 13 (1986).
4. J. L. Sullivan, B. G. Kao, and H. Van Oene, *Exp. Mech.*, **24**(3), 223 (1984).
5. A. D. Jonath, in *Structural Adhesives and Bonding Conference,* El Segundo, CA, March 13–15, 1979, p. 72.
6. G. W. Wycherley, S. A. Mestan, and I. Grabovac, *Journal of Testing and Evaluation, JTEVA,* **18**(3), 203 (1990).
7. Adhesives, Heat Resistant, Airframe Structural, Metal-to-Metal. U. S. Fed. Spec., MMM-A-132A, August 1981.
8. J. Romanko and W. G. Knauss, in *Developments in Adhesives-2,* A. J. Kinloch, Ed., Applied Science, London, 1981, p. 173.
9. W. Althof, G. Klinger, G. Neumann, and J. Schlothauer, *Environmental Effects on the Elastic—Plastic Properties of Adhesives in Bonded Metal Joints,* DFVLR, Braunschweig, Germany, B. Garland, Translator, Royal Aircraft Establishment, Farnborough, Hants, England, Trans. No. 1999, 1979.
10. ASTM D 2651, *ASTM Standards,* Vol. 15.06, Sec. 15, ASTM, Philadelphia, 1988, p. 167.
11. ASTM D 1002, *ASTM Standards,* Vol. 15.06, Sec. 15, ASTM, Philadelphia, 1988, p. 45.

12. U. Yuceoglu and D. P. Updike, *J. Eng. Mech. Div. Am. Soc. Civ. Eng. ASCE,* **107**(EM1), 55 (1981).
13. W. A. Lees, Ed., *Adhesives in Engineering Design,* Springer-Verlag, The Design Council, London, 1984, p. 45.
14. F. C. Tolan, S. A. Mestan, and C. E. M. Morris, *J. Appl. Polym. Sci.,* **29,** 4415 (1984).
15. L. J. Hart-Smith, in *Developments in Adhesives-2,* A. J. Kinloch, Ed., Applied Science, London, 1981, p. 1.
16. *Metals Handbook,* Volume 2—Properties and Selection: Nonferrous Alloys and Pure Metals, 9th Edition, American Society for Metals, Metals Park, OH, 1979, p. 104.
17. J. P. Jeandrau, *Int. J. Adhes. Adhes.,* **6**(4), 229 (1986).
18. R. B. Krieger, in *I. Mech. E. Conf. Publ. 1986,* (*6 Int. Conf. Struct. Adhes. Eng.*), Bristol, U.K., July 2–4, 1986, p. 1.
19. R. A. Jurf and J. R. Vinson, *J. Mater. Sci.,* **20,** 2979 (1985).
20. Scotch-Weld AF 163-2, Structural Adhesive Film, 3M Product Data Sheets, 3M Deutschland GmbH, May 1980.
21. L. G. Stringer, *J. Adhes.,* **18,** 185 (1985).

Received June 6, 1989
Accepted January 23, 1991