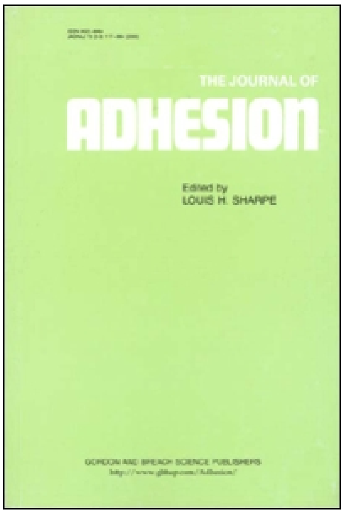


**The Journal of Adhesion**
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

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**To cite this Article** Spigel, Barry and Roy, Samit(1994) 'Comparison of the Adhesive Shear Modulus in Bulk and Bonded States', The Journal of Adhesion, 47: 1, 151 — 163
**To link to this Article: DOI:** 10.1080/00218469408027096
**URL:** http://dx.doi.org/10.1080/00218469408027096

*J. Adhesion*, 1994, Vol. 47, pp. 151–163
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Printed in the United States of America

# Comparison of the Adhesive Shear Modulus in Bulk and Bonded States*

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*(Received November 20, 1992; in final form September 29, 1993)*



A method is proposed for determining the *in situ* shear modulus of a structural adhesive from a sandwich beam loaded in 3-point bending in which the adhesive is contained as a thin layer. Expressions for calculating the elastic shear modulus of the adhesive layer from compliance data on the beam are derived, and experimental tests to validate the theory are conducted. To verify the test results, tensile tests are also conducted, and the shear modulus for bulk adhesive is determined using the constitutive equation for an isotropic material relating tensile modulus and Poisson's ratio to shear modulus.

However, the bulk shear modulus as traditionally determined from a tensile test was up to an order of magnitude greater than the *in situ* shear modulus obtained from the 3-point bend test. A finite element simulation and sensitivity study replicated the experimental results of the 3-point bend tests, and showed that using the shear modulus obtained from the tensile tests would result in significant errors in predicting material and joint behavior. In addition, torsion tests were conducted on bonded cylinders to measure directly the shear modulus. The shear modulus from the torsion test was in agreement with the *in situ* modulus obtained from the 3-point bend test. This combined experimental-computational approach validated the 3-point bend test as a means to determine the *in situ* adhesive shear modulus. Finally, micrographs of the interface of the 3-point bend specimen indicated that adhesion occurred by the extension of adhesive pillars to the surface of the adherends. This pillaring phenomenon may have resulted in a lack of bonding along significant portions of the interface, and may explain the compliance of the 3-point bend specimens and, subsequently, the lower shear modulus. The repeatability of the experiments and the substantiation of the results of the experiments by finite element analysis suggest that this pillaring phenomenon may be a mechanism of adhesion.

KEY WORDS Adhesive; shear modulus; *in situ*; bulk; bending; scanning electron microscopy; mechanical properties; adhesion; finite element simulation; theory; experiment.

## INTRODUCTION

Adhesive bonding is used increasingly to join structural components in the aerospace and automotive industries. Optimal joint design requires knowledge of all adhesive properties beyond just the shear strength. A method is proposed that determines the *in situ* shear modulus of structural adhesives from test data on a sandwich beam loaded in 3-point bending in which the adhesive is contained as a thin layer. This test method

*Presented at the International Symposium on "The Interphase" at the Sixteenth Annual Meeting of The Adhesion Society, Inc., Williamsburg, Virginia, U.S.A., February 21–26, 1993.

has the advantages of being simple to conduct and, because the adhesive is used as a bonding agent, representative of shear behavior in a joint.

Based upon earlier work by Moussiax *et al.*,[1] in which a method for calculating the shear modulus of an adhesive using an end-loaded cantilever beam was described, expressions for calculating the elastic shear modulus of the adhesive layer from compliance data on a beam in 3-point bending were derived. Experimental tests using Lord's Fusor 320/322 paste adhesive (Lord Corp., Erie, PA, USA) and 3M's AF-563 film adhesive (3M Co., St. Paul, MN, USA) were then conducted to validate the theory. The shear modulus obtained from the 3-point bend tests was compared with the shear modulus obtained from tensile tests of bulk adhesive and with the shear modulus obtained from torsion tests.

## THEORY

The solution for determining the deflections in an adhesively-bonded cantilever beam subjected to an end load was determined.[1] As shown in Figure 1, the maximum deflection for an adhesively-bonded cantilever beam is given by

$$\delta = \beta \frac{Pl^3}{2Eb(h+t)^3} \tag{1}$$

where

$$\beta = \left(1 + \frac{t}{h}\right)^3 \left[4\left(1 - \frac{1}{\gamma^2}\right) + \frac{3E}{2G}\left(\frac{h}{l}\right)^2 + \frac{12}{\gamma^2}\left(\frac{1}{\bar{\alpha}^2} - \frac{1}{\bar{\alpha}^3}\tanh\bar{\alpha}\right)\right] \tag{2}$$

and

$$\bar{\alpha} = \alpha\gamma$$

$$\alpha^2 = 3\frac{G_a}{E}\left(\frac{l}{h}\right)^2 \frac{(1+2t/h)^2}{t/h} \qquad \gamma^2 = 1 + \frac{1}{3(1+2t/h)^2}$$

where

$l$ : length of the cantilever
$h$ : thickness of adherend
$t$ : half the thickness of the adhesive layer
$E$ : Young's modulus of adherends
$G$ : shear modulus of adherends
$G_a$ : shear modulus of adhesive

The deflection term consists of the expression for an isotropic cantilever beam and a coefficient of adhesion, $\beta$, which accounts for the adhesive layer. A rigorous derivation for $\beta$ can be found in Reference 1. $\beta$ consists of three separate terms. The first term is related to the bending of the adherends and is only a function of geometry. The second term is due to shear deflection and is also a function of adherend geometry. In general, this term is a small percentage of the bending term. The last term contains the adhesive properties through the parameter $\bar{\alpha}$. For very stiff adhesives, this term approaches zero, but for very deformable adhesives it can increase to four times the bending term.[1]

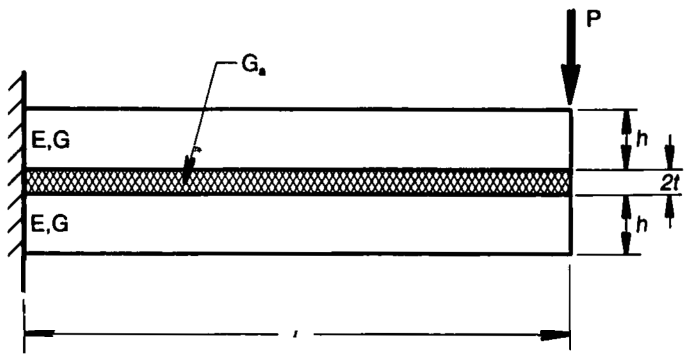


FIGURE 1 Geometry of a bonded cantilever beam.

Using the mechanics of materials, Equation (1) can be readily extended to a bonded beam loaded in 3-point bending. It can be shown that for a simply supported beam of length, $l$, centrally loaded by a force, $P$, as shown in Figure 2, Equation (1) becomes:

$$\delta = \beta \frac{Pl^3}{32Eb(h+t)^3} \tag{3}$$

The defintion of $\beta$ remains the same as given in Equation (2).

By defining the adhesive deformability as the ratio of the Young's modulus of the adherend to the shear modulus of the adhesive, $E/G_a$, the dependency of the adhesion coefficient $\beta$ on the adhesive properties and the adhesive and adherend geometry can be seen (Figure 3). This relationship is shown for a constant normalized adhesive thickness $(t/h)$ while varying the adherend length $(l/h)$. It is imperative that the geometry of the specimen be tailored to maximize the sensitivity of $\beta$ to $E/G_a$. The high sensitivity zone where the curves are steadily increasing can be shifted by changing the geometry of the beam. From Figure 3, this region is seen to be approximately three decades wide. Care

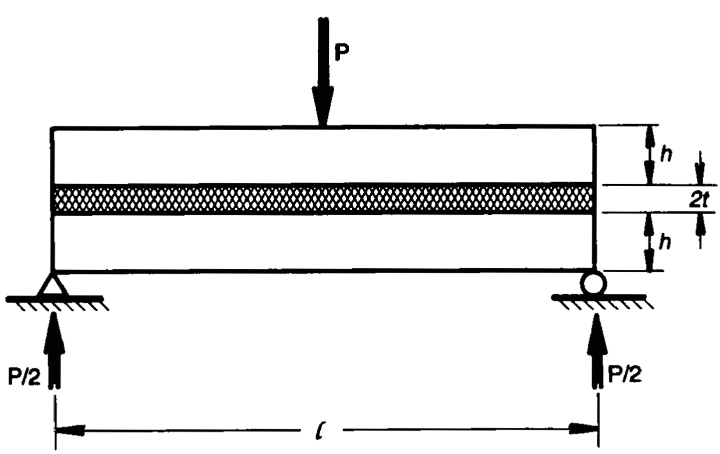


FIGURE 2 Geometry of a bonded beam in 3-point bending.

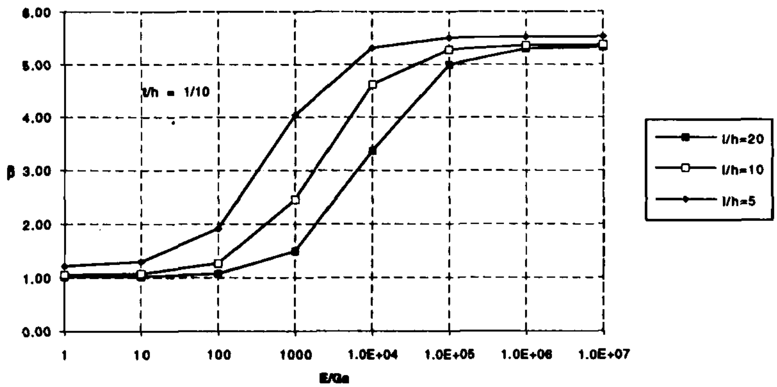


FIGURE 3 Dependence of the adhesion coefficient of a 3-point bend specimen on the adhesive deformability for various $l/h$.

must be taken to avoid the region where $\beta$ approaches 1, whereby the adhesive is very stiff and behaves like an isotropic beam, or where $\beta$ reaches an asymptote, whereby the adhesive is very deformable and the one beam behaves like two unbonded beams of thickness $h$.



## EXPERIMENTAL DETAILS

Five bending specimens were fabricated using 1018 steel adherends and Lord Fusor® 320/322 paste adhesive. An additional five specimens were fabricated using 1018 steel adherends and 3M's AF-563 film adhesive. The adherend's gage section was five inches (12.7 cm) long, one inch (2.54 cm) wide, and one-quarter inch (0.64 cm) thick. The adhesive thickness was 0.05 inch (0.13 cm) ($t = 0.025$ inch (0.064 cm)). The steel adherends were grit blasted and wiped with acetone.

To determine the adhesive shear modulus from the 3-point bend specimen, the following procedure is followed:

1. The deflection at the midpoint of the specimen is monitored continuously and the load/deflection curve is determined.
2. From Equation (3), the experimental adhesion coefficient, $\beta_{exp}$, is determined for a given load and measured deflection.
3. From Figure 3, the intersection of $\beta_{exp}$ and the appropriate curve for the geometry of the specimen is found. (For the specimens tested in this program, the curve representing $t/h = 1/10$, $l/h = 20$ was used). The adhesive deformability ratio can then be determined.
4. With E known, the adhesive shear modulus, $G_a$, can then be determined.

Finally, tensile specimens of both the Lord and 3M adhesives were fabricated following the guidelines for Type 1, ASTM D 638, Standard Test Method for Tensile Properties of Plastics. The specimen thickness was 0.05 inch (0.13 cm), and the specimens were strain-gaged to determine the tensile modulus and Poisson's ratio. The shear modulus was then derived from the constitutive relationship for isotropic materials relating Young's modulus, E, and the Poisson's ratio, $\nu$, with the shear modulus:

$$G = E/2(1 + \nu) \tag{4}$$

This provided a comparison with the *in situ* shear modulus determined from the 3-point bend specimens. Torsion tests on the adhesive were also conducted to provide a more direct measure of the shear modulus. The decision to conduct these tests was made after evaluating the results of the 3-point bend and tensile tests as explained in the following section.

## RESULTS AND DISCUSSION

The experimental data for the two adhesives is shown on Table I. Note that the bulk shear modulus value obtained from the Lord 320 tensile specimens is an order of magnitude higher than the average *in situ* shear modulus obtained from the 3-point bend specimens, and that the average bulk shear modulus for the 3M 563 adhesive is nearly an order of magnitude higher than the 3-point bend average *in situ* modulus. Typical 3-point bend test load-deflection curves for the Lord 320 adhesive are shown on Figure 4, and tensile stress-strain curves are provided on Figure 5. The 3-point bend curves are linear up to the maximum measured deflection, and the tensile curves are linear to approximately 0.23% strain. The calculations for both the bulk and *in situ* shear modulus were derived from the linear portions of the curves. Similar results were obtained from the tests conducted with the 3M 563 adhesive.

A review of the test data and machine calibrations was undertaken, and no discrepancies were reported. As can be seen from Figures 4 and 5, the 3-point bend load-deflection curves and the tensile stress-strain curves were repeatable. Durometer hardness tests, using Shore durometer hardness Type D-2, were also conducted on the post-test 3-point bend and tensile specimens. For both specimens, the hardness number was the same, indicating that there was no difference in the cure of the adhesives.

To determine the cause of the discrepancy in the adhesive shear moduli, additional 3-point bend tests using the Lord 320 adhesive were conducted. Torsion tests using the same adhesive were also conducted. Two precision-machined, 1018 steel adherends were bonded together to form an annular ring of adhesive. They were hexagonal and transitioned to a ring with a 0.812-inch (2.06 cm) outer diameter and 0.500-inch (1.27 cm) inner diameter. Tolerances as low as 0.0005 inch (0.0013 cm) were required to obtain proper alignment in the test fixture. The adhesive was contained as a 0.05-inch (0.13 cm) layer, the same as used for the 3-point bend specimens. The steel adherends

TABLE I
Shear moduli of lord 320 and 3M 563 adhesives

| Adhesive | Tensile Modulus (ksi) | Poisson's Ratio | Shear Modulus (ksi) (average $\pm$ standard deviation) | |
|---|---|---|---|---|
| | | | Tensile Test | 3-pt Bend Test |
| Lord 320 | 505 | 0.38 | $183 \pm 41$ | $12.1 \pm 1.6$ |
| 3M 563 | 243 | 0.30 | $93 \pm 5$ | $11.3 \pm 0.2$ |

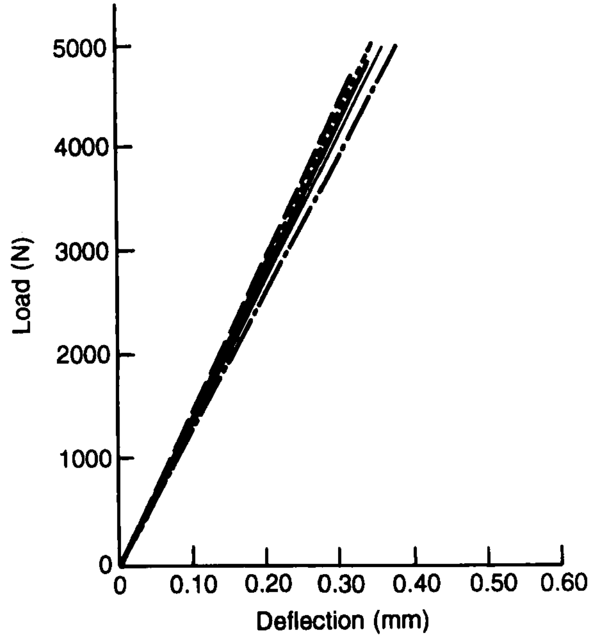


FIGURE 4 3-point load deflection curves for Lord 320 adhesive.

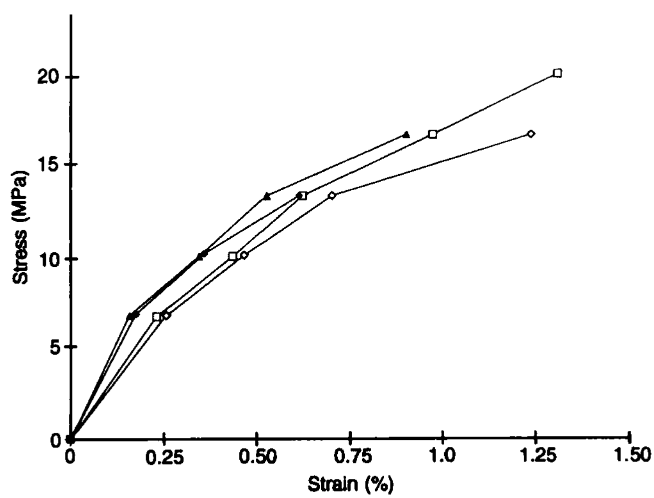


FIGURE 5 Tensile stress-strain curves for Lord 320 adhesive.

were grit blasted and wiped with acetone before bonding. The torsional shear strain was obtained from the angle of twist measured on the outer diameter.

The results of the additional 3-point bend tests and the torsion tests are shown on Table II. Torque stress-strain curves are given in Figure 6. The *in situ* shear modulus

TABLE II
Additional shear modulus values for Lord 320 adhesive

| | Shear Modulus (ksi) (average ± standard deviation) |
|---|---|
| 3-pt Bend Test (second set) | 8.7 ± 0.2 |
| Torsion Test | 17.5 ± 4.0 |

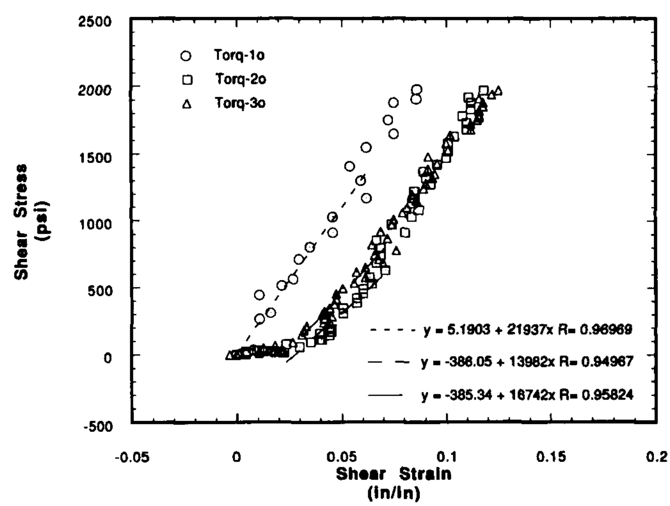


FIGURE 6 Torque stress-strain curves for Lord 320 adhesive.



derived from the 3-point bend tests is 30% lower than the previous information given in Table I, though the standard deviation was much lower. The Lord 320 adhesive is known to have different properties due to its mixing and cure cycle,[2] which could explain the differences between the two sets of data. However, this does not explain the order of magnitude difference between the bulk and *in situ* shear modulus. The torsion test results showed good agreement with the results of the 3-point bend shear moduli. The results of the torsion tests provide additional support to the hypothesis that the 3-point bend test can be used to determine the (*in situ*) shear modulus of an adhesive.

Simultaneous to the additional testing, a finite element simulation of the 3-point bend tests was carried out using the finite element code NOVA-3D that had been specially developed for the analysis of adhesive bonds.[3] The role of the finite element analysis was to (a) replicate and verify the experimental observations, and (b) study the sensitivity of the 3-point bend specimen to changes in material properties using the theory of elasticity.

Invoking symmetry, only one-half of the beam width was modeled. Twenty noded brick elements were used for the analyses. Linearly elastic finite element simulations of the 3-point bend tests were performed for the Lord Fusor® 320/322 and the 3M AF-563 adhesive systems. Steel adherends were used in both cases. The results from the simulations replicated the experimental data almost exactly, thus ruling out error in measurement of deflection as a possible source of discrepancy in the evaluation of the adhesive shear modulus. It also revealed that, for either adhesive system, the use of an adhesive shear modulus derived from tensile test data using isotropic relations (Equation (4)) resulted in approximately 50% lower mid-span deflection as compared with the shear modulus obtained from the 3-point bend test. Figure 7 shows the effect of adhesive shear modulus on the deflection of the 3-point bend specimen for the Lord 320 adhesive, while holding the tensile modulus and Poisson's ratio constant. Additional analyses were carried out to investigate the influence of adhesive tensile modulus and Poisson's ratio on the mid-span deflection of the 3-point bend specimen for the Lord 320 adhesive. This procedure, although in apparent contradiction with Equation (4), was nevertheless performed to study the exclusive parametric dependence of the beam deflection on the adhesive shear modulus. As can be seen from Figures 8 and 9, adhesive tensile modulus and Poisson's ratio, respectively, have negligible effect on the deflection of the specimen. The finite element results vindicate the accuracy of Equation (2). Similar results were obtained for the 3M 563 adhesive.

It is interesting to note that Morman, *et al.*,[4] used a doubly-clamped sandwich beam to determine the *dynamic* shear modulus of the adhesive layer. Morman followed a derivation for determining the dynamic shear modulus similar to the procedure employed by Mouissaux and modified by this effort to obtain the static adhesive shear modulus, though he used doubly-clamped boundary conditions at both ends of the specimen. In contrast to the static adhesive shear modulus results reported herein, Morman found that the calculated values for the *in situ* dynamic shear modulus were in agreement with the corresponding values from specimens made of bulk adhesives.

Dolev and Ishai[5] noted a discrepancy between bulk and *in situ* stress-strain curves in shear. The *in situ* results obtained from a napkin ring test did display more variability



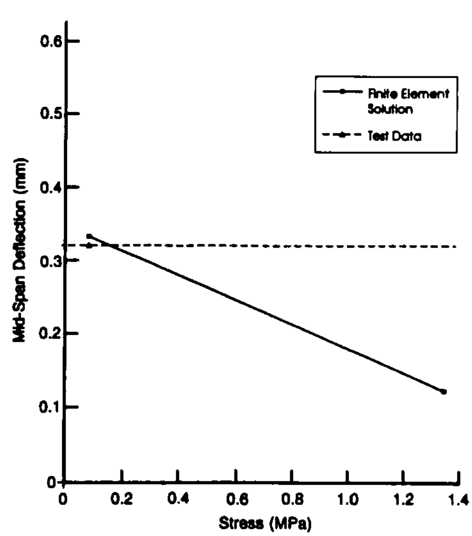


FIGURE 7 Effect of adhesive shear modulus on midspan deflection of a 3-point bend specimen.

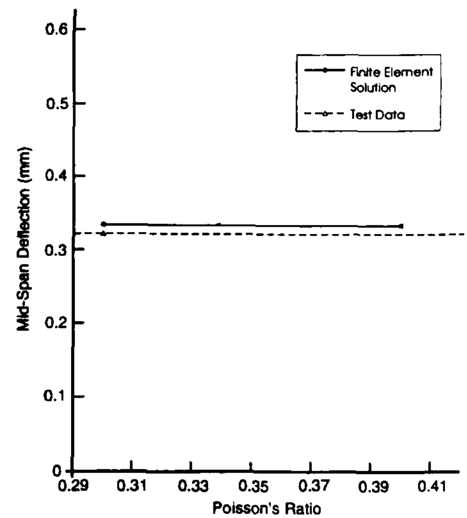


FIGURE 8 Effect of adhesive tensile modulus on midspan deflection of a 3-point bend specimen.



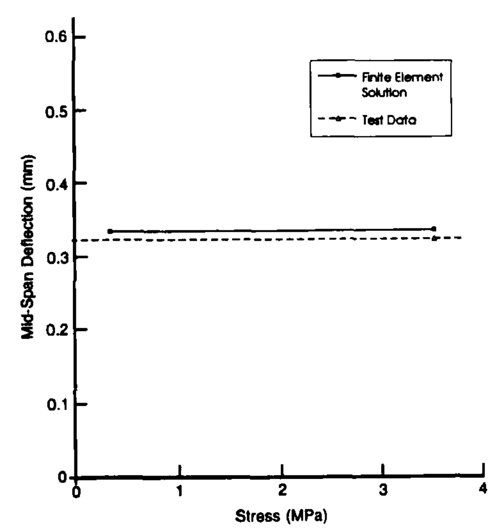


FIGURE 9 Effect of adhesive Poisson's ratio on midspan deflection of a 3-point bend specimen.

than the bulk shear test results from a torsion tube. The authors attributed the variability to the nonuniform and complicated state of stress within the adhesive layer, particularly close to the ends of the bonded joint. Still, in comparing their experimentally obtained shear moduli with computed results from Equation (4), the authors found fairly good agreement. Similarly, Sancaktar and Brinson[6] compared elastic shear moduli obtained from symmetric rail shear (bulk) tests, symmetric lap shear (bonded) tests, and tensile tests. They also found good agreement in the shear moduli.

The contrast in agreement of bulk and shear moduli between past results and this study prompted an examination of the adhesive interface of a Lord Fusor® 320/322 sandwich beam specimen using a field emission scanning electron microscope (FESEM). The specimen was sectioned along its width and mounted in a thermosetting resin and automatically polished. Then a 10 Å gold coating was applied. Figure 10 shows a typical interfacial region at 5000x magnification. The adhesive is at the top of the micrograph; the steel is the gray homogeneous region at the bottom. There are significant areas of separation between the adhesive and steel, as evidenced by the cracks along the interface. The white, particulate matter is a polishing artifact; it is adhesive that has been drawn across the steel during the polishing process. The interfacial region in the area of the large rectangle in the center of the photograph was examined more closely since it appeared to be an undisturbed region (the rectangle is an electron burn from the microscope that occured when observations were made at higher magnifications. It provides a convenient marker). Figure 11 shows the region of interest at 35,000x magnification. The interface has two distinguishing features: first, there is no contact between the adhesive and the steel along significant portions of the interface. Second, where adhesive bonding has occurred, pillars or ligands of adhesive appear to extend from the surface of the adhesive to the surface of the steel. These pillars are much in evidence along the right side of the photograph. A closer examination of the interface near the dark artifact in the steel is shown on Figure 12. Note that the scale

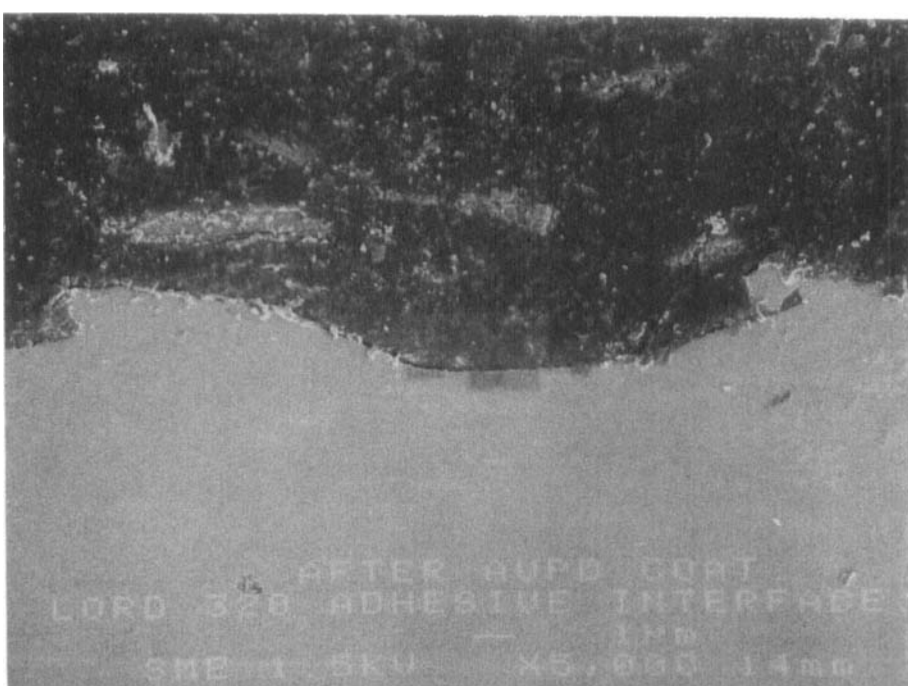


FIGURE 10 Adhesive bondline at 5000X.

in the 75,000x magnification micrograph is now 100 nanometers, and that the picture unfortunately blurs as the limits of the FESEM's resolution with this sample are approached. The adhesive pillars are clearly identifiable and appear to be somewhat random in width and distribution. The pillars are approximately 40 nanometers in length, with thicknesses varying from roughly 25–125 nanometers.

It is noted that these pillar structures were observed at both the top and bottom interfaces along the one-inch width of the sandwich specimen, and do not appear to be a consequence of the fabrication process. Indeed, the sandwich beam specimens were fabricated by placing the top adherend over the adhesive, which was spread evenly over the bottom adherend. Shims were layered adjacent to the specimens to control the thickness of the bondline. A plate was then set on the shims, which served to squeeze out any excess adhesive. Once this was accomplished, the adhesive and the adherends bonded under little or no pressure.

The question also arose as to whether the fracture surface was caused by the polishing process during sample preparation. While this possibility cannot be completely discounted, it is highly unlikely due to the presence of other polishing artifacts on the surface and the lack of such artifacts inside the cracks along the interface. The consistency of the mechanical test results, both with the sandwich beam and torsion specimens, led the authors to believe that the adhesion process (influenced by differential thermal expansion, moisture and surface preparation) was the more likely cause of the fracture topography.

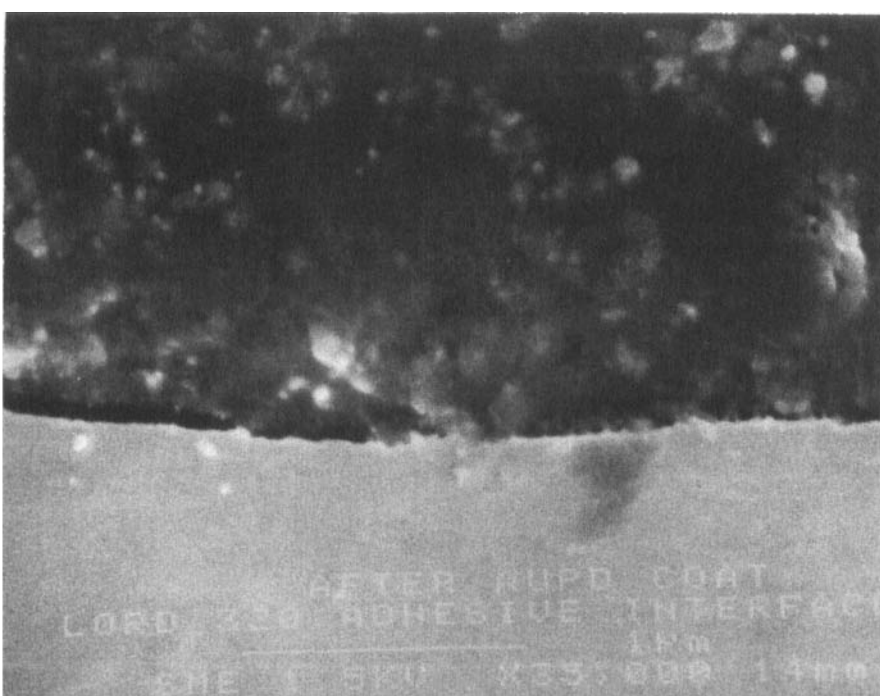

FIGURE 11 Adhesive bondline at 35000X.

These micrographs appear to indicate that adhesion at the interface of the Lord 320/322 paste adhesive and 1018 steel occurs by the extension of adhesive pillars to the surface of the adherend. These pillars, and the lack of bonding along significant portions of the interface, may explain the order-of-magnitude reduction of the *in situ* adhesive shear modulus as compared with the bulk shear modulus. Due to program limitations which precluded specific investigation, it can only be assumed that a similar process occurred between the 3M AF-563 adhesive and the 1018 steel adherends. The occurrence of the pillars in other adhesive/adherend systems, and the mechanisms by which they are formed, are a subject for further research.

## CONCLUSIONS

A sandwich beam containing a thin adhesive layer and loaded in 3-point bending has been shown to be a simple, accurate test method for determining the *in situ* shear modulus of the adhesive. Expressions for calculating the elastic shear modulus of the adhesive layer from compliance data on the beam were derived and experimental tests were conducted that validated the theory. The experimental shear modulus was in agreement with the *in situ* shear modulus directly measured from a torsion test of a bonded cylinder. However, the bulk adhesive shear modulus as traditionally determined from a tensile test was an order of magnitude greater than the *in situ* shear



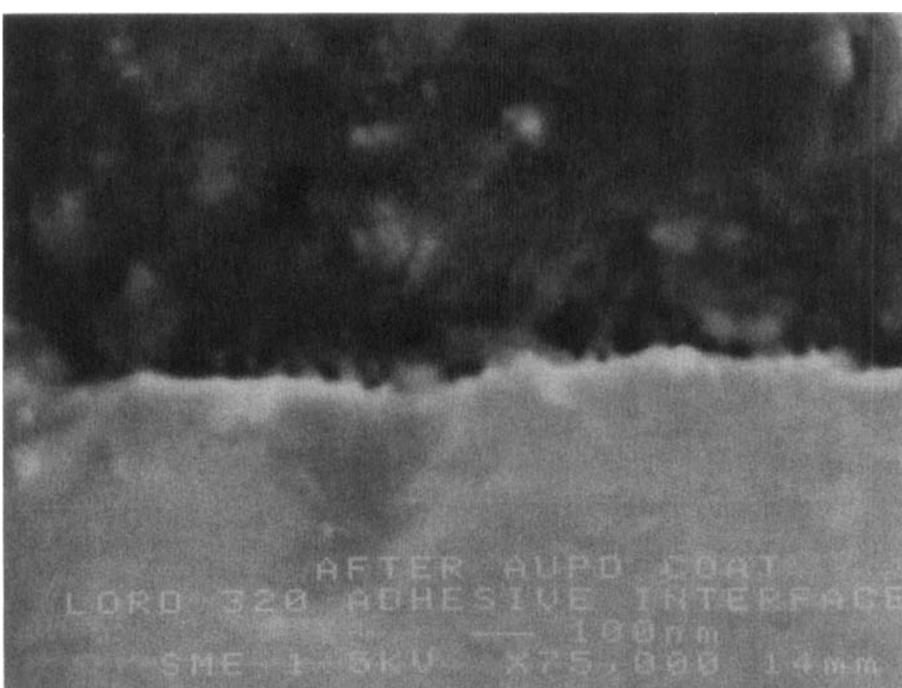


FIGURE 12 Adhesive bondline at 75000X. (Note scale change.)

modulus obtained from both the 3-point bend and torsion tests. A finite element simulation and sensitivity study replicated the experimental results of the 3-point bend tests, and showed that using the shear modulus obtained from the tensile tests would result in significant errors in predicting material and joint behavior. In addition, micrographs of the interface of the 3-point bend specimen indicated that adhesion occurred by the extension of adhesive pillars to the surface of the adherends. The lack of bonding along significant portions of the interface may explain the enhanced compliance of the 3-point bend specimens and, subsequently, the lower shear modulus. As such, there are two summary findings resulting from this study:

1. The repeatability of the experiments and the substantiation of the results of the experiments by finite element analysis suggest that this pillaring phenomenon may be mechanism of adhesion.
2. This experimental-computational approach has clearly demonstrated that the traditional practice of applying a shear modulus derived from tensile tests of bulk adhesive to model an adhesive bond could cause up to 50% error in predicted values. A more accurate test method, such as the 3-point bend test, should be used to measure the *in situ* adhesive shear modulus.



## Acknowledgements

This research was supported by the National Center for Manufacturing Sciences under Contract Number 14-06-0916. Mr. Kerry Barnett was the technical monitor.

## References

1. E. Moussiaux, H. F. Brinson, and A. H. Cardon, "Bending of a Bonded Beam as a Test Method for Adhesive Properties," Virginia Polytechnic Institute and State University, Report VPI-E-87-9 and CAS/ESM 87-2, 1987.
2. C. Li, R. Dickie, and K. Morman, "Dynamic Mechanical Response of Adhesively Bonded Beams: Effect of Environmental Exposure and Interfacial Zone Properties," *Polymer Engineering Science* **30**, (1990).
3. S. Roy and J. N. Reddy, "A Finite Element Analysis of Adhesively Bonded Composite Joints With Moisture Diffusion and Delayed Failure," *Computers & Structures* **29**, 1011–1031 (1988).
4. K. Morman, C. Li, F. Zhang, and R. Dickie, "Determination of the Complex Shear Modulus of Structural Adhesives Using a Doubly Clamped Sandwich Beam," *Experimental Mechanics* 124–131 (1992).
5. G. Dolev, and O. Ishai, "Mechanical Characterization of Adhesive Layer *in situ* and as Bulk Material," *J. Adhesion* **12**, 283–294 (1981).
6. E. Sancaktar, and H. Brinson, "The Viscoelastic Shear Behavior of a Structural Adhesive," in *Adhesion and Adsorption of Polymers*, Polymer Science and Technology Series, **Vol. 12A**, L. -H. Lee, Ed. (Plenum Press, New York, 1980), pp. 279–299.