

CONF-941195--1

Fracture Testing and Analysis of Adhesively Bonded Joints for Automotive Applications

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Prepared for the proceedings of the
10th Annual ASM/ESD Advanced Composites Conference

November 7-12, 1994
Dearborn, Michigan

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Abstract

In 1992, the Oak Ridge National Laboratory (ORNL) began a cooperative effort with the Automotive Composites Consortium (ACC) to conduct research and development that would overcome technological hurdles to the adhesive bonding of current and future automotive materials. This effort is part of a larger Department of Energy (DOE) program to promote the use of lighter weight materials in automotive structures for the purpose of increasing fuel efficiency and reducing environmental pollutant emissions. In accomplishing this mission, the bonding of similar and dissimilar materials was identified as being of primary importance to the automotive industry since this enabling technology would give designers the freedom to choose from an expanded menu of low mass materials for component weight reduction.

Early in the project's conception, five key areas were identified as being of importance to the automotive industry. (1) The development of appropriate methods for determining the properties of the adherends and adhesives independent of one another. (2) The determination of accurate, highly standardized fracture test methods for quantifying, not just qualifying, an adhesive/adherend system's resistance to crack growth. (3) Modeling of joints so that designers would be able to examine the effects of minor design changes without entering into an expanded testing program. (4) Non-destructive inspection of production bonds either during the bond formation, after adhesive curing or after component completion. (5) Mechanisms for increasing the manufacturability and reducing the production costs of bonded composites.

This program is in its second year. The tasks under this program are being performed by industry, university and government researchers and are being managed in a joint effort between the ACC Joining Group and ORNL staff members. Plans for expansion of this research project to meet future

research needs are also being considered.

This paper concentrates on the details of developing accurate fracture test methods for adhesively bonded joints in the automotive industry. The test methods being developed are highly standardized and automated so that industry suppliers will be able to pass on reliable data to automotive designers in a timely manner. Mode I fracture tests have been developed that are user friendly and automated for easy data acquisition, data analysis, test control and test repeatability. The development of this test is discussed. In addition, materials and manufacturing issues are addressed which are of particular importance when designing adhesive and composite material systems.

IN THE FUTURE, automobiles will be forced to travel further between refuelings while discharging lower levels of pollutants [1]. Currently automobiles account for just under two-thirds of the nation's gasoline usage and about one-third of the total United States energy consumption. By improving automotive fuel efficiency, the United States can lessen the impact that foreign oil prices have on our economy and lives. In addition, decreased emissions from reduced fuel consumption will provide a cleaner environment for future generations. At current usage rates, a 25% weight reduction in current United States vehicles would save 750,000 barrels of oil each day, reduce the yearly domestic fuel consumption by 13% and prevent 101 million tons of CO₂ from being emitted into our atmosphere each year. [2]

A significant reduction in fuel consumption can only be achieved by one of three means: (1) improving engine and drivetrain efficiency; (2) reducing automotive component mass and thus vehicle weight; or (3) reducing the size and thus weight of an automobile. Engine efficiency improvements are being studied by a wide variety of industry

and government organizations, and great strides are being made in this area. Vehicle down-sizing has been undertaken since the early '70s and is still occurring, however, consumers are reluctant to purchase smaller and smaller vehicles because their transportation requirements dictate the necessity for a family-size car. Reducing component weight and thus vehicle weight, while not sacrificing vehicle size, reducing safety or increasing vehicle cost, can only be accomplished by the use of alternate, lighter weight materials. The goal of this project is to provide one enabling technology, adhesive bonding, which will allow for the use of alternate materials, particularly reinforced polymer composites.

The commercial application of composites has an extensive history in the marine, aerospace and construction industries, but has evolved relatively slowly in the automotive industry during the past 20 years [3,4]. Composite use has traditionally been limited to secondary structures like appearance panels and dash boards, but as the evolution of the automobile continues, fiber-reinforced polymers are being considered for weight reduction in future automotive structures and load-bearing components [5]. A critical aspect of using these materials is the manner in which they are joined. Adhesive bonding is potentially an economical and structurally sound means of joining reinforced polymers and other alternative automotive materials and may overcome a major obstacle to the incorporation of lighter weight materials into automobiles.

As with composites, the major problems limiting the utility of aluminum in automotive structures have been related to joining technologies [6]. Reliable joining methods for aluminum alloys are needed to make the lightweight metal more attractive for structural applications [7].

While much work has been conducted in adhesive bonding for the aerospace, construction, and some consumer goods industries, the automotive industry does not currently have a complete set of processes and methods for evaluating candidate adhesives for use in bonding structural automotive components. The charter of this project is to develop those processes and methods. Emphasis is placed on deriving designer usable test data and models from industry-ready standardized test methods. Since this work is concerned with developing processes and not simply evaluating specific materials for specific applications, only a few materials have been selected and will be subjected to the entire method and process development. After completion of this step, the processes, methods and standards developed will then be verified using other materials. The materials used for the initial phase of this program are: one urethane based adhesive; one epoxy based adhesive; one structural reaction injection molded (SRIM), glass-fiber reinforced urethane composite; a standard E-coated steel; and a standard aluminum alloy. The adhesives are experimental and are being developed and refined by two industry suppliers. The SRIM composite is made with an experimental resin developed by a supplier and the steel and aluminum are standard industry stock.

Experimental Needs

Polymer based composites have historically found their greatest usage in the aerospace and military markets. These industries have expended tremendous resources in developing test methods and test standards for material evaluation and selection. Due to the high performance environments that structural composites were subjected to in these applications and the low factors of safety that were allowable, the test methods were highly involved (and thus expensive) and highly specific to the end use application of the material under evaluation. As a result, when one surveys current aerospace and military industry standards, it becomes apparent that there are so many individual standards for arriving at a specific material property that it is fair to say that there are few standardized standards.

An example of this that the one of the authors recently noted was experimental data being derived by more than 10 members of a consortium involved in basic composite materials research. Each company had its own set of standards for measuring certain material properties. The member of the consortium who was responsible for consolidating the data had a nearly impossible task in drawing conclusions due to the differences in the test methods that were employed.

In the early part of this century the metals industries were forced to adopt a single set of standards. This was due primarily to the limited number of steel producers and the size of their industry. When those producers decided to use a set of standards (ASTM standards) for reporting data, the rest of the industry and other related industries had to follow suit. The structural composites industry has not developed with the same limited number of mega-producers.

High production rate consumer goods industries, such as the automotive industry, cannot bear testing costs in the same manner as the aerospace and military equipment industries. They cannot afford the time required for full-scale, multi-year prototype testing of each material before making material selection and moving into production. High production rate consumer goods also have a greater variability in material properties from one batch of material to another, or from one location in a component to another due to the increased rate of productivity and the need to use less expensive composites. Additionally, consumer goods tend to be made from random chopped or swirled fibers where the aerospace industry relies more heavily on laminates and uni-directional fiber placement. All of these factors point to the need for testing standards that cater to the needs of these industries and can be performed at a cost and schedule that is within acceptable limits.

After consultation with members of the automotive industry, it was determined that standardized and automated test methods need to be developed for the evaluation of composites joined by adhesive bonding. The single-lap shear strength values that are currently employed yield a qualitative

comparison between adhesively bonded joints, but do not produce specific material property values that an engineer can use in designing structural components of an automobile.

The first issue to be tackled in this effort is the development of standards and methods for accurately and precisely predicting the fracture behavior of adhesively bonded joints. The standards must yield designer-usable fracture toughness numbers. Three fracture modes are being considered: opening mode, in-plane shear and mixed opening and shear. The opening or Mode I test method is nearly finalized, and the other two test methods have received preliminary consideration. Before development of the test procedures, certain material use and specimen fabrication issues had to be resolved.

Materials Issues

When the first composite samples were bonded using the epoxy based adhesive, the composite blistered and the adhesive "blew" out of the joint during the adhesive curing cycle, yielding warped samples and joints with little adhesive on the interior. After some quick analysis, the source of the problem became readily apparent.

The composite resin is a polyisocyanurate which has a large affinity for absorbing atmospheric moisture. Upon heating at 150°C (the adhesive cure temperature), the absorbed moisture was constrained from escaping into the atmosphere due to microscopic, localized, thermal constriction of the voids and capillaries in the composite. This allowed sufficient pressure to build inside the composite to produce blistering. Similarly, the thixotropic adhesive was constraining the surface and subsurface moisture from escaping due to its high viscosity. As heating progressed, the adhesive's viscosity decreased, and the steam pressure increased until the adhesive was literally blown out of the joint by the escaping gas. This resulted in a large percentage of disbands in the joint.

The obvious solution to these problems was to eliminate the water before bonding. After an extensive series of tests, it was determined that a 48 hour, 101°C pre-drying treatment would remove more than 95% of the absorbed moisture. Twelve inch square material plaques were then bonded using this composite pre-drying treatment prior to application of the adhesive. After the 45 minute, 150°C adhesive curing cycle, no material problems were noted. In other efforts to reduce the drying time by boosting the drying temperature, it was also determined that 125°C was the highest temperature that the composite could be subjected to for extended periods of time (> 4 hours) without suffering degradation.

The evaluation of the effect of drying time, drying temperature and moisture content on the mechanical strength of adhesive joints was the final step in this evaluation. Single lap shear samples were used to obtain an idea of the relative quality of adhesive joints prepared by pre-drying at two different temperatures for different lengths of times. Samples were prepared by pre-drying one batch of samples at 101°C and

a second batch of samples at 125°C. Drying times for each batch of material were 1, 2, 3, 4, 8, 16, 24, 36 and 48 hours. After drying, single lap shear plates were bonded using the epoxy adhesive (30 mil bondline thickness) and cured for 45 minutes at 150°C. For comparison, a third set of samples were prepared that had undergone no pre-drying treatment. Next, the plates were sectioned into one inch wide lap shear samples which were tested in a conventional Instron using a crosshead speed of 1.27 mm/min. (0.05 in./min.) All samples failed by composite fiber pullout and fiber tear.

Figures 1, 2 and 3 show typical load displacement curves for samples dried for 3, 16 and 48 hours, respectively. From these curves it is apparent that the slope of the "elastic" (polymers are not truly elastic) curve is approximately the same regardless of the drying treatment. Drying the composite at 101°C tends to produce a slight decrease in the apparent "yield strength" of the joints when compared to samples not dried. Increasing the temperature further to 125°C produces and even greater decrease in the apparent "yield strength".

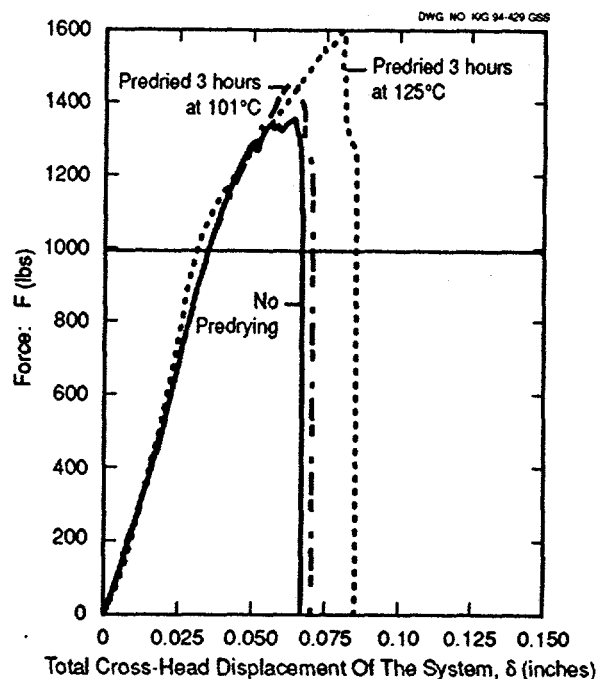


Figure 1. Load vs. Displacement for Composite/Adhesive Single Lap Shear Samples Pre-Dried for 3 Hours.

While decreases in apparent "yield strength" are noted with increasing drying temperatures, the opposite effect is seen on the "ultimate strength" of the samples. Drying the samples at 101°C produces an increase in "ultimate tensile strength" of the joint, and boosting the drying temperature to

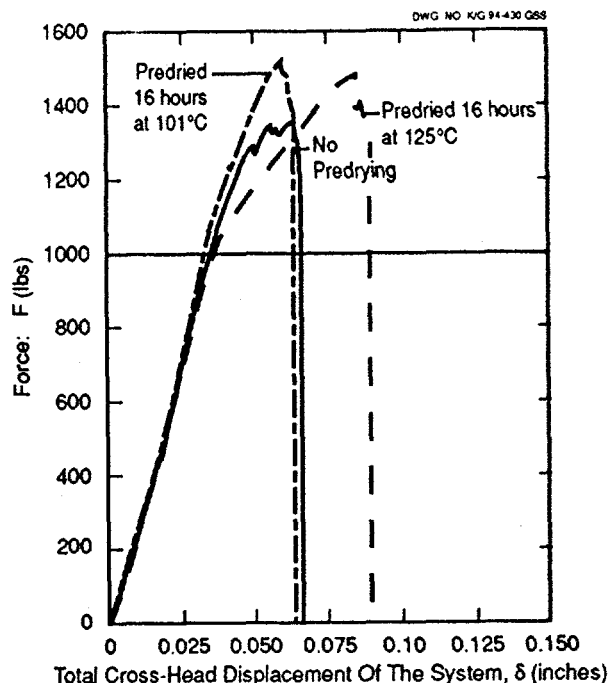


Figure 2. Load vs. Displacement for Composite/Adhesive Single Lap Shear Samples Pre-Dried for 16 Hours.

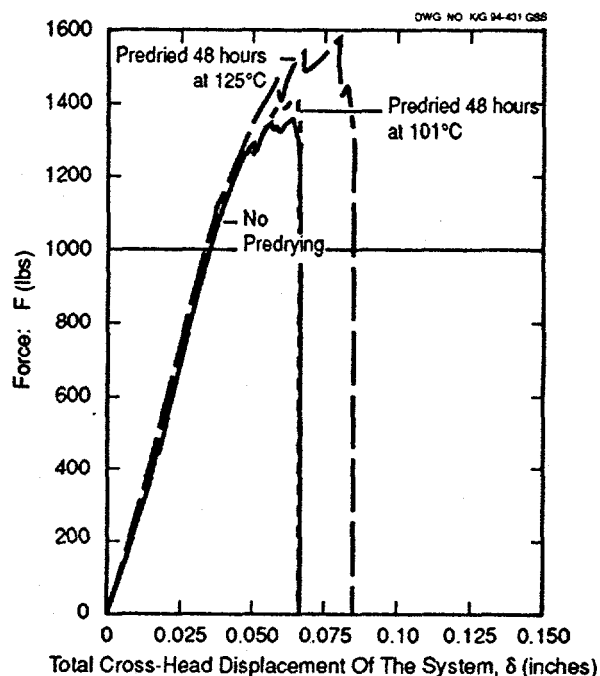


Figure 3. Load vs. Displacement for Composite/Adhesive Single Lap Shear Samples Pre-Dried for 48 Hours.

125°C further increases this system property. The total crosshead displacement, and thus system deformation of the joint, was approximately the same between samples dried at 101°C and those not dried. Samples dried at 125°C had a significantly increased plastic zone which indicates that the composite may have been annealed by the drying treatment.

In conclusion, subjecting the composite to a 101°C drying treatment reduces the apparent "yield strength" but increases the "ultimate strength". Increasing the temperature to 125°C further exaggerates these changes. Using the higher temperature drying treatment also increases the energy absorbing ability of the joint, but at the expense of lowering the apparent "yield strength". Regardless of whether or not a drying treatment is used, satisfactory bonds can be formed with this material combination. By satisfactory it is meant that the strengths of the adhesive and the adhesive/substrate interface exceed the strength of the composite in the near interface region.

Mode I Test Development

Mode I fracture toughness is a mechanical property that defines a material's resistance to crack propagation for a crack acted upon by tensile forces directed normal to the crack surface. The typical test specimen for adhesively bonded joints, the uniform double cantilever beam (UDCB or DCB), is the subject of ASTM Standard Practice D3433.[8] The standard was developed for testing adhesive joints with metallic adherends, but has gained broader acceptance including the determination of the fracture toughness for laminated composites. It has been demonstrated to work quite well for aerospace-grade composites.

Of interest here however, are bonded joints in which the adherends are an automotive-grade composite. Specifically, the composite is made SRIM panel made with a polyisocyanurate resin and randomly oriented continuous glass strand mats. It is a low-cost and rapid process that results in a composite having a higher void content and a lower fiber volume fraction than the typical aerospace-grade composites (Figure 4). Furthermore, due to the randomness of the fiber placement, the uniformity is significantly less than "high-tech" composites resulting in random zones of high-fiber content and resin-rich pockets.

The applicability of DCB testing practices, as typically found in the literature, was investigated with specimens made by bonding two 3 mm (0.125 in.) thick SRIM panels with an epoxy to form a 0.75 mm (30 mil) bondline. Specimens 25.4 mm by 241 mm (1 in. by 9.5 in.) were machined from the bonded panels after the adhesive was cured for 1 hour at 150° C. Hinges for load introduction were bonded to the sample with Hysol® XEA 9359.3 structural adhesive. The specimens were loaded in a 5 kN (1000 lbs.) electromechanical testing machine with a cross-head speed of 5 mm/min.(0.2 in./min.).

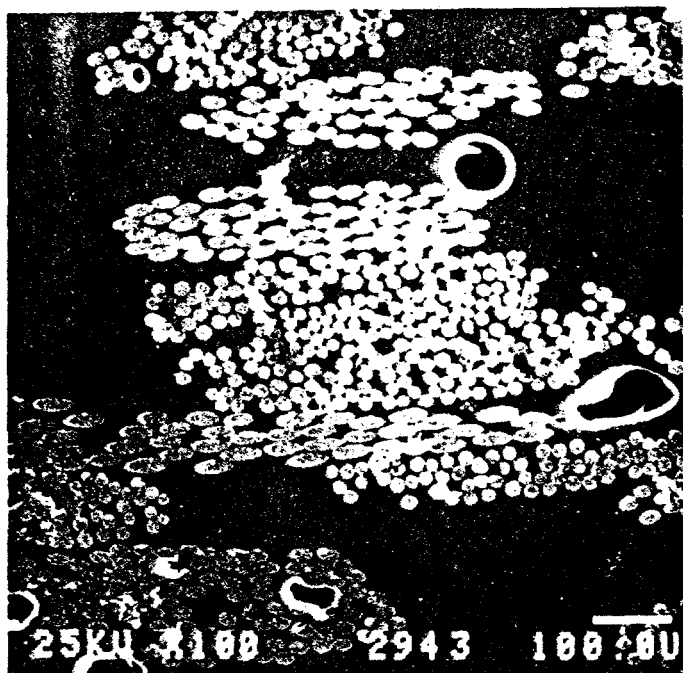


Figure 4. Micrograph illustrating the variability of the SRIM composite. Fibers are concentrated in bundles which are randomly dispersed with resin-rich pockets. These panels exhibit a high degree of porosity in the matrix as well as in the fiber bundles. (magnification=100x)

Crack extension in the adhesive was preempted by damage accumulation in the composite adherends resulting in one of the specimen's arms failing prematurely due to bending as shown in Figure 5. As a result, fracture toughness measurements were not possible, and it was determined that the "standard" DCB geometry was not appropriate for these materials.

In order to conduct a successful fracture toughness test for bonded joints with these SRIM composite adherends, a modified specimen is required. Since the adherends fail prematurely due to excessive bending, it is concluded that stiffening the adherends by bonding on "backing-beams" would be beneficial. Although an earlier paper by the authors [9] espoused the backing-beam concept for reasons discussed below, it appears to be necessary to prevent damage for these materials. Others have used this approach also. Byun, Gillespie, and Chou [10] reported the use of aluminum backing-beams for three-dimensional fabric composite DCB specimens. River and colleagues [11,12] used aluminum and wood backing-beams to test wood DCB specimens. Whitney and Short [13] used steel backing-beams for similar reasons to test the interlaminar shear strength of graphite/epoxy composites using a modified Short Beam Shear (SBS) specimen.

Backing-Beam Concept. The motivation for this work is to develop test procedures that would help resolve theoretical and experimental issues dealing with specimen

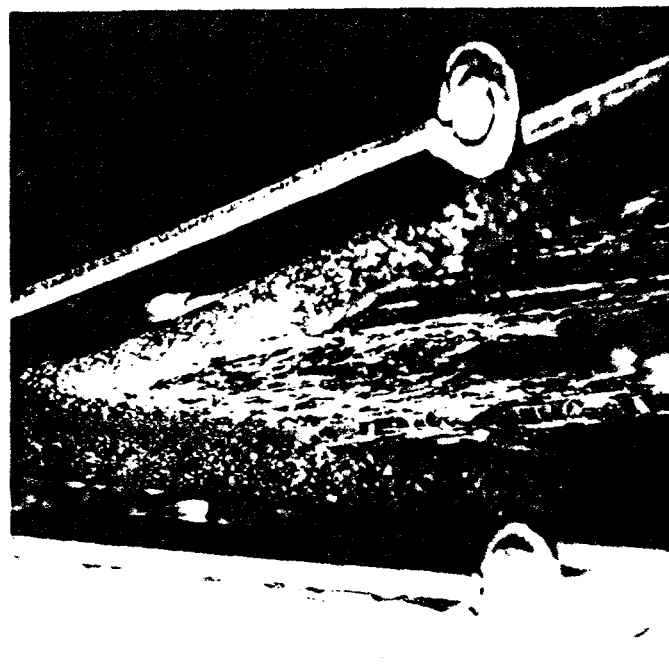


Figure 5. SRIM composite adherends failed prematurely due to excessive bending during traditional DCB test.

design and data reduction and be valid for a wide range of both adherend and adhesive properties using standardized geometries, sizes, fixtures and procedures. To that end, a contoured shape as developed by Mostovoy and colleagues [14-16] was employed for the backing-beams as shown in Figure 6. The Mostovoy specimen, the height-tapered double cantilever beam (HTDCB) is also the subject of ASTM D3433. Employing backing-beams with the Mostovoy contour has advantages for the following reasons:

Small Displacements. In many applications of the DCB, large displacements of the cantilever ends are encountered. This introduces two primary error sources that must be accounted for in the analysis of the results. Firstly, large deflections cause an effective shortening of the cantilever. Secondly, if end blocks (rather than hinges) are used to introduce the load and if deflection is measured at the load-line then end block rotation reduces the deflection. Correction factors can be applied to account for these effects. As a practical testing matter, the correction factors are troublesome, but correction factors can be circumvented by incorporating the backing-beam concept. With this concept the deflections are governed by the stiffer backing beam thereby limiting the deflection to acceptably-small values. In addition, since the backing beams provide the majority of the overall stiffness, the deflections from tests with a wide range of adherend stiffnesses will exhibit a much narrower range avoiding the need to change the test setup for the variety of different adherends of interest to the automotive industry.

Crack Length Measurement. The HTDCB test is designed such that the determination of the strain energy

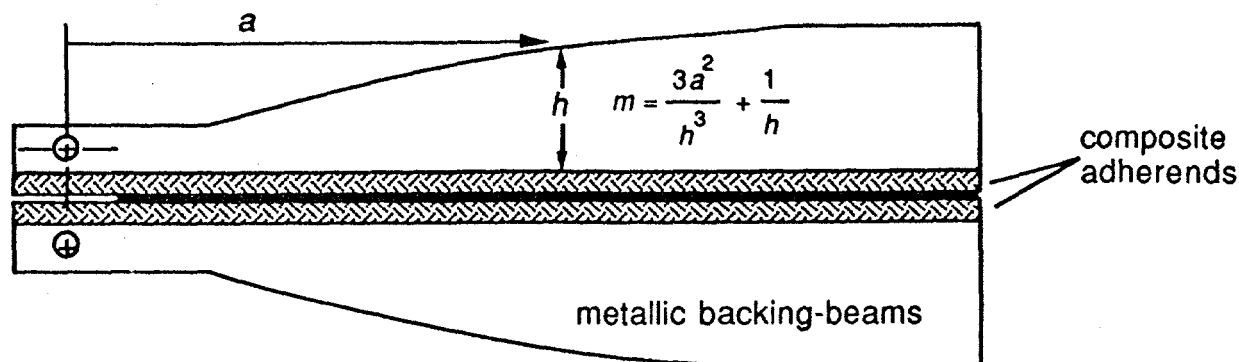


Figure 6. Backing Beam Concept using the Mostovoy Contour.

release rate is independent of the crack length — it is only necessary to measure the load required to drive the crack. In the present case the stiffness of the backing beam is modified by the adherend, and thus crack length independence is lost. If however, the stiffness of the backing beam dominates the overall stiffness, then the toughness should become only weakly dependent on the crack length. Thus the sensitivity of the experimental results to errors in the measurement of the crack length has been minimized. This is a particularly desirable feature when the crack length varies through the width of the specimen.

Anticlastic Curvatures. It has been reported [17] that thin (perpendicular to the crack surface) adherends develop anticlastic curvatures. As a result, strong width-variations of the strain energy release rate develop. By bonding the backing beams to the specimen, it is expected that the curvature and the subsequent variation in the strain energy can be significantly diminished. It is further believed that this would result in crack lengths that are more uniform through the thickness.

Experiments. Backing-beams, 12.7 mm (0.5 in.) wide by 254 mm (10 in.) long with a contour parameter $m = 3.543$ 1/mm (90 1/in.), were machined from 17-4PH stainless steel. SRIM panels (approximately 3 mm thick) were bonded with an experimental epoxy to form a 0.75 mm (30 mil) bondline with an inserted Teflon film to serve as a crack initiator. Composite-epoxy-composite specimens, 12.7 mm (0.5 in.) by 241 mm (9.5 in.), were machined from the panel after the adhesive was cured at 150°C for approximately 1 hour. The backing beams were then bonded to the joint with 3M AF-163-2 film adhesive. When cured, the specimens were loaded in an electromechanical testing machine under displacement control with a cross-head speed of 2.54 mm/min (0.1 in./min). Data acquisition equipment was used to collect and process the data in real-time.

Under these conditions this adhesive exhibited the "run-arrest" response indicative of rate-sensitive adhesives as shown in Figure 7. Neglecting the stiffness of the composite, the initiation and arrest fracture toughnesses, G_{Ic} and G_{Ia} , are

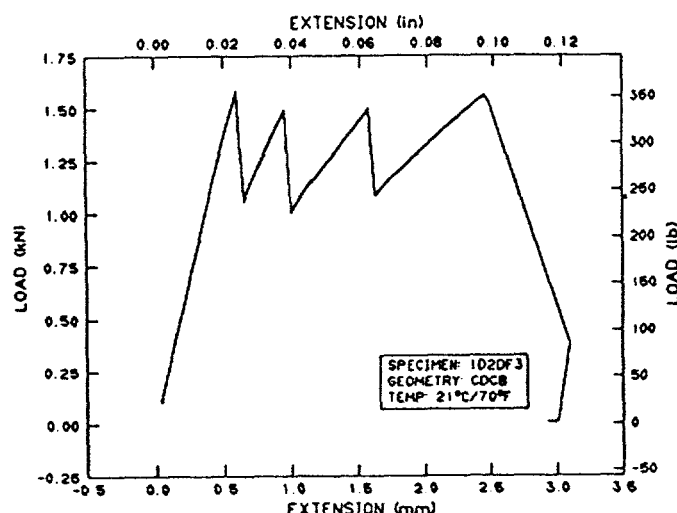


Figure 7. Load-deflection curve for composite/adhesive/composite specimen tested using backing-beams. The sawtooth nature of the curve indicates a run-arrest response.

calculated by

$$G_{Ic} = \frac{4L^2(\max) m}{EB^2}$$

$$G_{Ia} = \frac{4L^2(\min) m}{EB^2}$$

where $L(\max)$ is the load at crack initiation, $L(\min)$ is the load at crack arrest, E is the modulus of the backing-beam, and B is the width of the specimen and backing-beam. For the data plotted in Figure 8, the average initiation toughness was 993



Figure 8. The failure is constrained to the near interface region when employing the backing-beams.

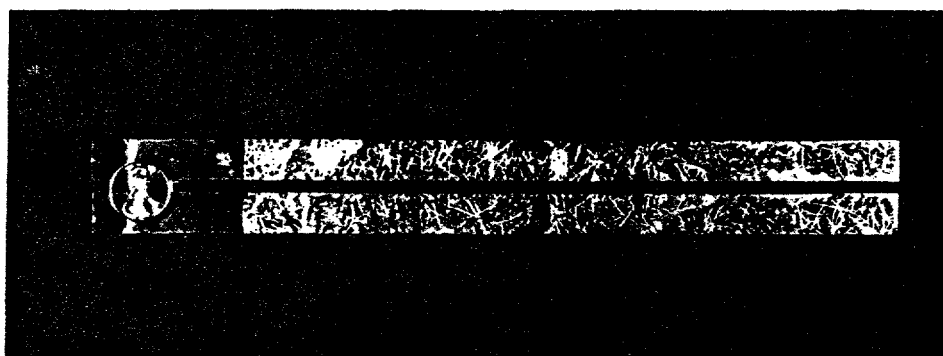


Figure 9. The failure exposes bare fibers indicating that the fiber/matrix interface may be the dominate factor in the failure process.

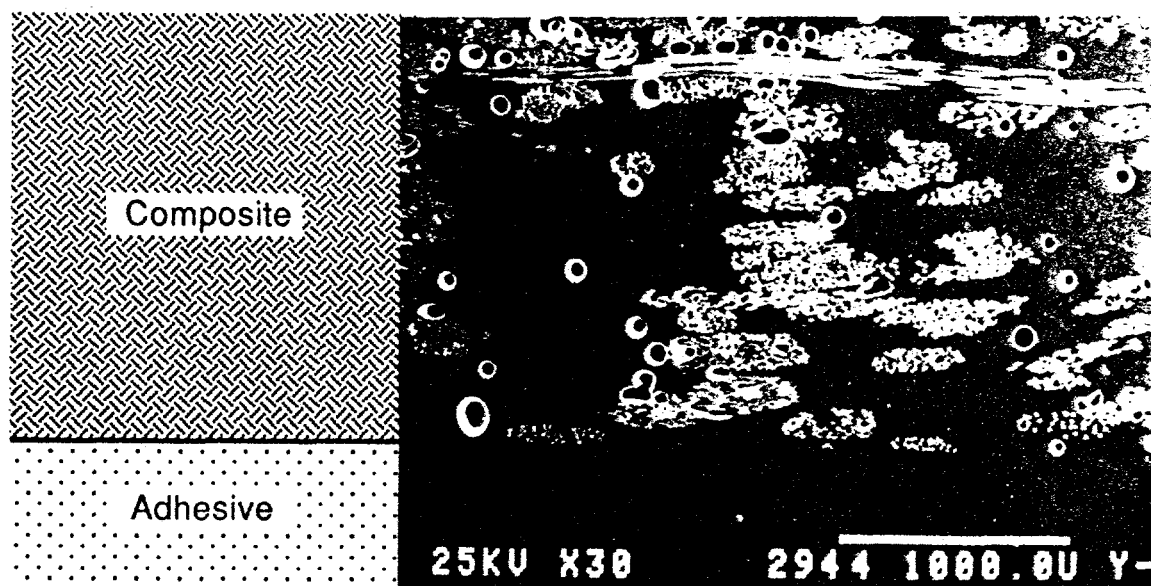


Figure 10. Micrograph indicating a very good composite/adhesive interface. Note the porosity, fiber-bundle concentrations and the resin-rich pockets. (magnification=30x)

J/m² (5.67 in-lb/in²), whereas the average arrest toughness was 471 J/m² (2.69 in-lb/in²). A side view of the failed specimen is shown in Figure 8, and the failure surface is shown in Figure 9. Note that the adhesion to the adherend is excellent (Figure 10), and that the failure generally takes place in the composite. This often exposes the glass fiber in areas of high fiber content near the adhesive/composite interface indicating that the fiber/matrix interface may dominate the response. It has been observed that in some specimens the "crack" location changes from the near interface in one adherend to the near interface in the other. It is hypothesized that this is because the crack follows a path that takes it to the "weakest" interface, that is the interface with the highest local concentration of fibers near the interface. Figure 5 certainly illustrates the variability of the fiber content near the interface and thus leads credence to the hypothesis. It is also quite probable that the distributions of voids, particularly near the high fiber content zones, affects the path of the "crack."

Future Work. In the experiments described in the previous section, the contribution of the composite adherends to the specimen compliance was neglected requiring only the load to be known to calculate the toughnesses G_{Ic} and G_{IIc} . This is an approximation. In future work the crack length and compliance as a function of the crack length will be measured and used to determine G_{Ic} and G_{IIc} . Tests will also be conducted on specimens where the adherends are an E-coated steel. The entire test method will be automated including the crack length measurement. Then the complete process will be repeated for Mode II (shear mode) and Mixed-Mode (opening and shear) test development. Throughout the process, analytical and numerical studies will be conducted to assess the advantages of the backing-beam concept and to define optimal configurations.

Conclusions

Test methods for determining the Mode I fracture toughness of adhesive joints containing automotive-grade SRIM composite adherends were developed. Standard double cantilever beam techniques were found to be inadequate because the adherends failed prematurely due to excessive bending. A backing-beam concept was successfully employed to prevent the adherend failures. Very good adhesion between the epoxy adhesive and the polyurethane, resin-based composite adherends was achieved. The failure surface was observed to expose the glass fibers in the composite near the adhesive/adherend interface indicating a weak fiber matrix interface as a leading factor in the failure. The failure was observed to randomly jump from one interface region to the other, and it was hypothesized that the "crack" followed the path toward the highest local concentration of fibers near the interface.

Acknowledgements

The authors wish to thank the following individuals for their diligent effort in assisting with this project: Felix Paulauskas, Fahmy Haggag, and Ronny Lomax of Oak Ridge National Laboratory; Thomas Meek, University of Tennessee; Dallas Smith, Tennessee Technological University; Carl Weber, BF Goodrich Adhesives and the entire crew of the ACC's Joining Group.

This project is sponsored by the U. S. Department of Energy, Office of Transportation Materials, Lightweight Materials Project. Oak Ridge National Laboratory is operated by Martin Marietta Energy Systems Inc. under contract DE-AC05-84OR21400.

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