Quantifying Adhesive Bond Strength for Advanced Instrumentation Research

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Abstract - *The goal of instrumentation is to install a sensor on a structure in order to capture any alterations (temperature, strain, etc.). However, during large scale research projects, the process for selecting the appropriate adhesive can be subjective due to the lack of quantitative metrics associated with evaluating bond strength. Common practice within the structures division of the Air Force Research Laboratory's Aerospace Systems Directorate (AFRL/RQV) is to pinpoint a small pool of adhesives that will work based on project requirements and begin installation based on the adhesive that is the most readily available. A wedge-peel test method has been identified as a method to evaluate the fracture behavior of adhesively bonded joints when subjected to a mechanical load. The data, along with the adhesive's material properties, will be used to down-select adhesives from the pool normally constructed at the start of a project. In addition, this reference tool will be used to reduce man hours and guide the investigation to better understand how the adhesives interact with the material to which they're attached. All information will be compiled into a database containing material property data from various substrates to determine any existing relationships.*

Keywords: Instrumentation, high temperature, composites, adhesives, bond strength, wedge

1 Introduction

A critical aspect of sensor installation is the bond strength between the adhesive and the substrate. There are many different tests, both qualitative and quantitative in nature, that can provide insight into the molecular interaction between materials[1]. However, it is important to note that caution must be taken in selecting the test and how the resultant data is interpreted. For example, ASTM-D-3808 (the "Spot Adhesion Test")[2] is a qualitative test, employed frequently by RQV, to quickly eliminate unlikely candidates for an upcoming project. Using a sample specimen, small quantities of selected adhesives are placed on the substrate using manufacturer specifications. Because it has been well established that not all adhesive-substrate pairs are equally compatible[1,3,3], this test can used to guide the process of selecting the proper adhesive. But, is the selected adhesive the **best** adhesive for the desired application? In order to answer this, the characteristics of both the adhesive and the substrate must be considered.

To determine the strength of a joint, one must resort to testing which is an important aspect of adhesive science and technology. As previously stated, there are many accepted test standards quantifying bond strength, but each differ depending on the goals of the researcher. For this effort, a simple peel test (referencing ASTM-D-3762[5]) was designed due to the ease of specimen manufacturability and comparative cost of required test equipment. The goal was to evolve RQV's adhesive selection process into a system based on more than subjectivity to determine the best adhesive for the selected test material. In addition, the data from this test, along with others, has been entered into a database that will be used as a reference tool for future projects in an effort to reduce the required amount of man-hours for a single project.

2 Methodology

2.1 Rationale

In 2009, instrumentation researchers within the Structural Validation branch (RQVV) designed an adhesive "button" to protect a thermocouple (TC) junction at high temperatures. Previous methods produced either data irregularities or were limited to specific materials. Qualitative results have shown an improvement in both life-cycle duration and data accuracy for TC's encapsulated using this method[6](see Figure 1). However, quantitative results are required to validate this method.

Figure 1. The practice of covering the TC junction and exposed wires was altered to a two part silicone injected into a "button" shaped acrylic mold to be used on test specimens.

The ASTM standard was modified to focus on the bond line formed between the "button" and substrate by driving a manufactured wedge through the bond line until failure. While this test produces numerical data that can provide comparisons between specimens, it has been noted that peel strength is not an inherent fundamental property of an adhesive nor is this a representative failure mode of in-flight thermomechanical loads. The value of the force required to initiate or sustain a peel is not only function of the adhesive type but also depends on the particular test method, rate of loading, nature, thickness of the adherend, and other factors[7]. However, studies have shown that the data from a peel test can be used to measure the "work of adhesion" which can be translated into adhesive fracture energy[8,9].

It cannot be overstated that data interpretation can vary based on the experimental setup and test specimens. For adhesives, average stresses at failure used in predictive analyses have been shown to produce false projections due to the complexity of the stress state within the bond region[10]. But technological advances have been able to prove that by accounting for the energy alterations caused by the applied load and the creation of the new surface area, the stress state and displacement can be accurately predicted. A fracture mechanic approach uses these stresses combined with the displacements and strains, along with conservation of energy principles, to predict failure conditions[11]. Delamination, the separation of fibers at stress free edges, is a commonly observed failure mode that can be model by the three basic fracture modes that occur:

- Mode I - Crack opening mode.
- Mode II - Sliding shear mode.
- Mode III - Scissoring shear mode.

With proper experimental testing, the corresponding strain energy release rates for each mode can used to predict delamination onset and growth[12]. For a wedge test, Mode III fractures are not observed and the strain energy release rate for the remaining modes ($G_{I,II}$) can be calculated using the following equations based on the nodal displacements shown in Figure 2:

$$G_I = \frac{Y_i \triangle v_m + Y_j \triangle v_l}{2\triangle a} \qquad (1)$$

$$G_{II} = \frac{X_i \triangle u_m + X_j \triangle u_l}{2\triangle a} \qquad (2)$$

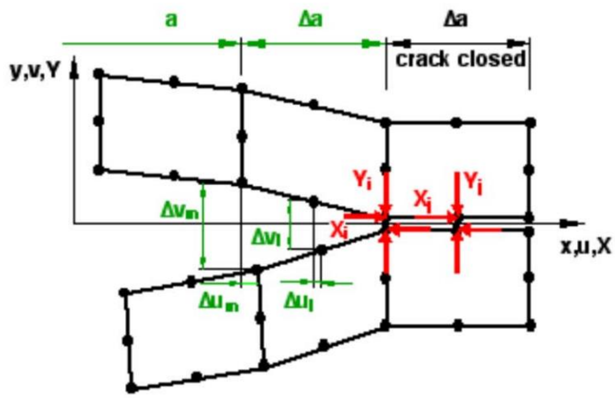


Figure 2. Vectors in green denote displacements due to Mode I fracture and vectors in red denote Mode II displacements.

The total strain energy release rate (sum of the observed components) and nodal displacements can be used in conjunction with finite element analysis (FEA) software to predict delamination. Techniques similar to these, as discussed in Section 4, will be employed to increase the knowledge provided by the adhesive database.

2.2 Test Article Description

The first iteration test specimens were cut to the approximate size of 6" x 2" x .25" with an adhesive button installed on the surface. Dimensions were selected based on ease of manufacturability in an effort to develop a future standard, however, this caused issues during initial testing. As shown in Figure 3, using the screw grips in the MTS Q-Tester provided a secure test fixture, but the specimen length combined with the load caused the specimen to bend.

Figure 3. The length of the test specimen allowed a moment arm to be created once the force of the wedge was applied.

In an effort to resolve these issues, two solutions have been devised (see Figure 4). The first is a repeatability guide to ensure that the wedges strike the adhesive at the bond line during each test. The wedge travels down the upper slot while the specimen is secured within the lower slot. Assuming the specimens match the dimensions of the guide, the offset vectors the wedge into the bond line creating the desired failure mode. Additionally, the screw grips were discarded due to lack of grip pressure for both the specimen and the wedge. The wedge grips are known to be much more secure and can fit both the specimen and the guide within the clamps to also aid in replication between tests.

Figure 4. Proposed solutions to bending issues observed during first tests.

2.3 Procedures

The first two tests focused on base materials and adhesives most familiar to the research team: carbon-carbon substrates and graphite adhesives. To date, it has shown to provide the strongest bond for thermocouple attachment during the instrumentation process. Two different graphite adhesives were selected for the baseline tests. The first was Aremco's Graphi-Bond 551-RN. This is a one part solution that has been selected in the past because of its ease of use, but has a high curing temperature. The second, GrafTech's UCAR, is a two part mixture that has been utilized on various past projects. Both have had favorable results, but again, this process was established to determine which has the better bond strength. The specimens were loaded into the guide shown in Figure 5. A vice was used to keep the test article secure during testing.

Figure 5. Example specimen clamped into MTS fixture prior to loading.

The load rate was steadily decreased to .062 in/s after a series of practice runs in order to ensure that the rate would not affect the results. After each test was complete, the sites were photographed underneath a microscope for installation comparisons and the numerical results compared. Each sample was tested five (5) times for statistical analysis. Because the C-C/graphite adhesive pairing has been so widely used historically within RQVV, it was determined that the values obtained through these tests would serve as the 'goodness' value for subsequent tests. This is based on the subjective assumption that this pairing (C-C and graphite) cannot be surpassed. So all other substrate-adhesive pairs should be within 25% or exceed the results obtained.

3 Results

Table 1 shows the preliminary results from the baseline tests.

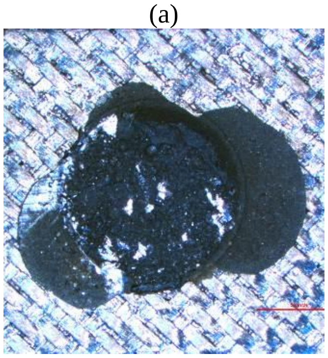
Table 1 - Preliminary Results (Peak Load in Newtons)

	Group 1	Group 2
Test 1	2.0933	2.4432
Test 2	2.5144	2.3798
Test 3	2.4800	2.2198
Test 4	2.1176	2.5976
Test 5	2.5342	2.4879

Group 1, using the 551-RN adhesive, has an average peak load 2.35 N while Group 2 has an average of 2.43 N. These are promising results as both graphite adhesives performed similarly. As stated in Section 2.3, these values will be referenced to compare the quality of other bond strenghts.

Qualitatively the failure modes differed slightly as shown below.

(a)



(b)

Figure 6. Part (a) shows a failure of the 551-RN adhesive and part (b) shows a UCAR failure.

Figure 6a displays a 551-RN failure, specifically from Test #4 which was significantly lower than the previous run. Note the voids and large area of separation to the right of the installation. It has been hypothesized that this caused the decrease in bond strength. Conversely Figure 6b, which corresponds to UCAR Test #1, shows an installation with a lesser number of void pockets corresponding to a higher a bond strength value in Table 1.

As it can be seen, the curing process of the mixtures can make a difference in how the adhesive interacts with the substrate. This will be documented and compared future tests.

4 Future Work

While the results provide an insight into the adhesive-substrate interaction, they do not provide a definitive answer to the question of what adhesive is most suitable for any given program. As previously stated, there are a number of different experiments that can determine bond strength based on varying factors. In order to create a more robust tool, this test will be the first in a series of experiments to compare the strength of adhesive under different conditions.

It is the goal of RQVV to determine which adhesive has the highest strength, but can also withstand the projected in-flight thermomechanical loads for any given program. Current efforts will look to evaluate established adhesives that have been used on previous programs. Future efforts will focus on new high temperature adhesives capable of reaching temperatures in excess of 3000°F[4]. These tests will follow a two-stage process:

1. Experiments will be tested under room temperature conditions to ensure that proper fixture specifications are being met.
2. After room temperature tests are complete, the fixture will be re-designed to incorporate a thermal load and the results will be compared.

Thermal testing, a well established capability within RQVV at Wright-Patterson Air Force Base, will aid in validating the performance of an adhesive at various temperatures. MTS frames, such as the one depicted in Figure 7, will be utilized to provide the thermal load while the MTS hydraulics provide the mechanical forces.

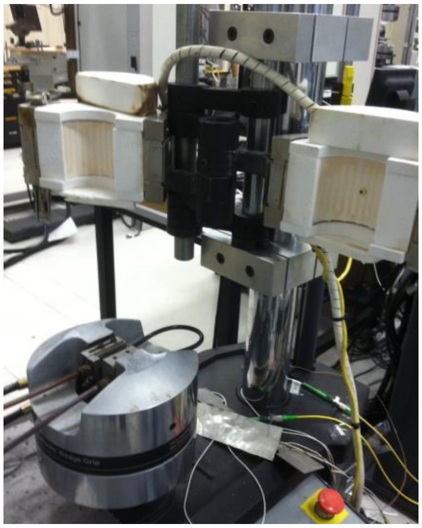


Figure 7. MTS frame setup that provides both thermal and mechanical loads.

Finally, future parallel efforts will seek to model these interactions based on the provided material properties acquired for both the adhesive and the substrate. As discussed in Section 2.1, fracture mechanics and other modeling tools are viable options for simulating the observed experimental failure mode. The modeling objectives will be to first correlate experimental data to simulation data and then use the model as a predictive tool for future tests.

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