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TEMPERATURE EFFECTS ON ADHESIVE BOND STRENGTHS AND MODULUS FOR COMMONLY USED SPACECRAFT STUCTURAL ADHESIVES

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ABSTRACT

A study was performed to observe how changes in temperature and substrate material affected the strength and modulus of an adhesive bondline. Seven different adhesives commonly used in aerospace bonded structures were tested. Aluminum, titanium and Invar adherends were cleaned and primed, then bonded using the manufacturer's recommendations. Following surface preparation, the coupons were bonded with the adhesives. The single lap shear coupons were then pull tested per ASTM D 1002 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint over a temperature range from -150°C up to +150°C. The ultimate strength was calculated and the resulting data were converted into B-basis design allowables. Average and B-basis results were compared. Results obtained using aluminum adherends are reported. The effects of using different adherend materials and temperature were also studied and will be reported in a subsequent paper. Dynamic Mechanical Analysis (DMA) was used to study variations in adhesive modulus with temperature. This work resulted in a highly useful database for comparing adhesive performance over a wide range of temperatures, and has facilitated selection of the appropriate adhesive for spacecraft structure applications.

1. INTRODUCTION

1.1.1.1 Purpose

This research was carried out at the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration.
The purpose of this effort was to study how changes in temperature affected the bond line strength and modulus of structural adhesives that are commonly used in aerospace applications. These materials are used every day in the space community and while much information is known about them at non-extreme temperatures, there is little to no data at the extreme ends of the spectrum. As space exploration continues, there is an increasing need for additional data over a wider range of temperatures. This study explores bond line strength and adhesive properties from -150°C up to +150°C. While this is not intended to be a complete database of B-Basis allowables for these adhesives, it is to be used as a reference during the design process for the Materials Group at the Jet Propulsion Laboratory (JPL) and those in related positions.

1.1.1.2 Scope

This report covers how temperature effects bond line strength and physical properties of seven adhesives. The adhesives are: Scotch-Weld Epoxy Adhesive 2216 B/A Gray (EC 2216), STYCAST 2850 FT Catalyst 9 (2850 Cat 9), STYCAST 2850 FT Catalyst 24 LV (2850 Cat 24), Hysol 9309.3, Hysol 9360, Hysol 9361 and Hysol 9394. These adhesives were chosen based on their frequent use at JPL. There is a great deal of data from JPL and the vendor about these adhesives at room temperature, due to the fact that ASTM D 1002 lap shear testing is used to verify the strength and cure of a bondline that will be used for flight. However, this test is only done at room temperature and little data have been collected at the lower and higher temperatures.

Lap shear coupon testing per ASTM D 1002 is a standard method for checking the ultimate strength of an adhesive. This method was chosen for the study because of its simplicity and ease. We did increase the thickness of the adherends from 1.6 mm to 3.2 mm to reduce problems with adherend bending and peel loads.

The lap shear testing was preformed initially on aluminum substrates. Work was performed using titanium and Invar substrates and will be reported in a subsequent paper. The initial conclusion that was made, was that bond strengths to titanium and Invar adherends were substantially lower than those obtained using aluminum adherends. Additionally, Dynamic Mechanical Analysis (DMA) was used to monitor variations of modulus with temperature.

2. CHARACTERIZATION OF BONDLINE STRENGTH AT DIFFERENT TEMPERATURES

2.1 Tests Performed

To evaluate the seven adhesives, two different types of tests were run. First, ASTM D 1002 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint was performed on the lap shear samples. Next, samples of just the adhesive were made, then, characterized rheologically using the technique of Dynamic Mechanical Analysis (DMA) to obtain modulus information over a wide temperature range (approximately -130 ºC to +150 ºC). Using these two methods, data was collected, providing details about the bondline properties at various temperatures.

2.2 ASTM D 1002

2.2.1.1 General Test Description

ASTM D 1002 used single lap joints that are pulled in an Instron machine to determine the ultimate strength of the adhesive. The samples are made from Aluminum 2024-T3, all from the same lot of material, into sets of five single lap shear joints as seen in Figure 1 below.

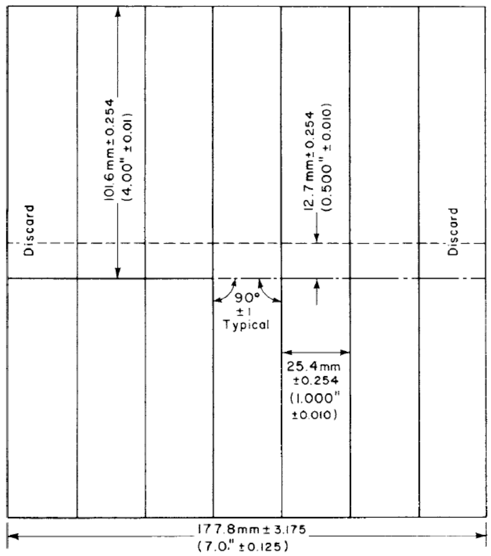

Figure 1. Standard Test Panel for ASTM D 1002

The surface is cleaned and primed per the requirements of this study and then the set of lap shear coupons are mounted into a bonding fixture to ensure alignment of the pair. The adhesive is mixed using the manufacturer recommendations and then applied to the cleaned specimens. After the adhesive has been cured, the sets are cut into individual samples. Samples were given an elevated temperature cure at temperatures up to 93ºC depending on the adhesive. This was to bring bond strength data more in line with results to be expected from hardware bonded joints that had undergone elevated temperature exposure.

2.2.1.2 Test Selection

According to ASTM D 1002 [1], this test is used for easily comparing the different parameters that can be changed within a test. These include surface preparations, primer, environments and adhesive. For this test, the surface preparation and primer remained fixed, allowing for only the adhesive and different temperature to be the defining parameters producing change in properties.

2.2.1.3 Sample Fabrication and Surface Preparation

Since the study required that only temperature be the defining difference in each test, all the specimens were prepared in the same way. Aluminum 2024-T3 was used as the substrate for all the ASTM D 1002 tests performed. The lap shear panels were created from the same lot of material to ensure that the lot to lot differences would not affect the data. The aluminum was cut using a water jet technique to cut down on oils that can be contributed to the surface during the machining process. The panels were then deburred and cleaned.

The surface preparation for these panels was detailed to ensure that each surface was as clean as possible, creating an ideal substrate surface for bonding. First, the panels were cleaned per JPL's specification for preparation of surfaces for adhesive bonding; including an elevated temperature alkaline cleaning. Then, the bonding surfaces were primed with BR-127 and cured for one hour at 125°C.

The panels were then cut apart. After they were machined, the samples were filed down to remove sharp edges and then they were ready to be tested in the Instron Machine.

2.2.1.4 Tensile Test Method

The lap shear coupons were then pulled to failure using an Instron machine. Two chambers were used for this test due to the large volume of specimens that needed to be tested; Instron 1331 with a Bemco 1.8 model thermal chamber and an Instron MTS with a Thermatron F-2 model thermal chamber. All equipment that was used for this test was calibrated at the time of the test.

For the test, the thermal chambers were set to the necessary temperature for the test and then the sample was loaded into the machine using bolt and clevis fittings with the 9.5mm diameter holes drilled into the end of each adherend. The Instron machine pulled the samples at a rate of 1.27mm per minute and the load vs. displacement results were recorded using a Labview data system.

After a sample failed, it was inspected to determine what type of failure had occurred. Failure types included adhesive, cohesive and mixed mode, which were recorded in percentages.

2.2.1.5 Procedure

For this study, a total of 10 single lap shear specimens were made up for each adhesive at each test temperature. Average bond strengths were recorded; peaks values were not calculated. Results were averaged and B-basis design allowables were computed using a Visual Basic program known as Stat 17.

2.2.1.6 Concerns with ASTM D 1002

With using this procedure, there was a concern for some anomalies to be introduced into the data. First, the single lap shear fingers were made to be 3.2mm thick. This was done to minimize issues related to adherend bending and twisting during the pull test.

Additionally, there was also a problem with this test at cold temperatures. When the specimens were in the grips at cold temperatures, they were unable to hold onto the specimens correctly rendering them unable to pull the specimens to failure. To fix this problem, 9.5mm holes were drilled into the ends of the lap shear specimens. With this modification, the coupons were held in the Instron machine with a bolt and clevis fitting.

2.3 Dynamic Mechanical Analysis (DMA)

2.3.1.1 Overview of DMA

To supplement the lap shear joint work, the study also included DMA testing. DMA is a dynamic method of characterizing the viscoelastic properties of a material (see Figure 2 below). A sinusoidal force (stress) is applied to the material at a set frequency and the response (strain) to this input is measured. The ratio of peak stress to peak strain gives a complex modulus from which storage modulus (G') and loss modulus (G") are obtained. The storage modulus is related to the energy stored by the material per cycle. A storage modulus curve is presented in Figure 2 below. Stiff and "glassy" materials have a high storage modulus. The loss modulus is related to the energy dissipated or lost by the material during the cycle, and it will go through a significant decrease in the glass transition temperature region of the sample. Tan Delta (Tan δ) is the ratio of the loss modulus to the storage modulus and is commonly referred to as the loss factor and is related to the viscoelasticity and also the damping (how well the material can disperse energy) of the material. The maximum in the Tan Delta peak is commonly used as the glass transition temperature (Tg).

2.3.1.2 Specimen preparation

To create samples for the DMA testing, each adhesive was mixed and cured per the manufacturer's recommended practice. The adhesive was cured as a flat panel and then cut down into 25mm x 75mm x 6mm bars. These were provided to the Analytical Chemistry Lab at JPL to be cut down further into approx 17.5mm x 13mm x 3 mm pieces using a fine hack saw. DMA was performed on a TA Q800 DMA instrument configured in a single cantilever clamp mode and run from -130 ºC to +150 ºC at a constant frequency (1 Hz) and constant amplitude (50 µm). Storage modulus, loss modulus, tan delta were graphed after the analytical runs were completed and results are presented in Figures 6-11.

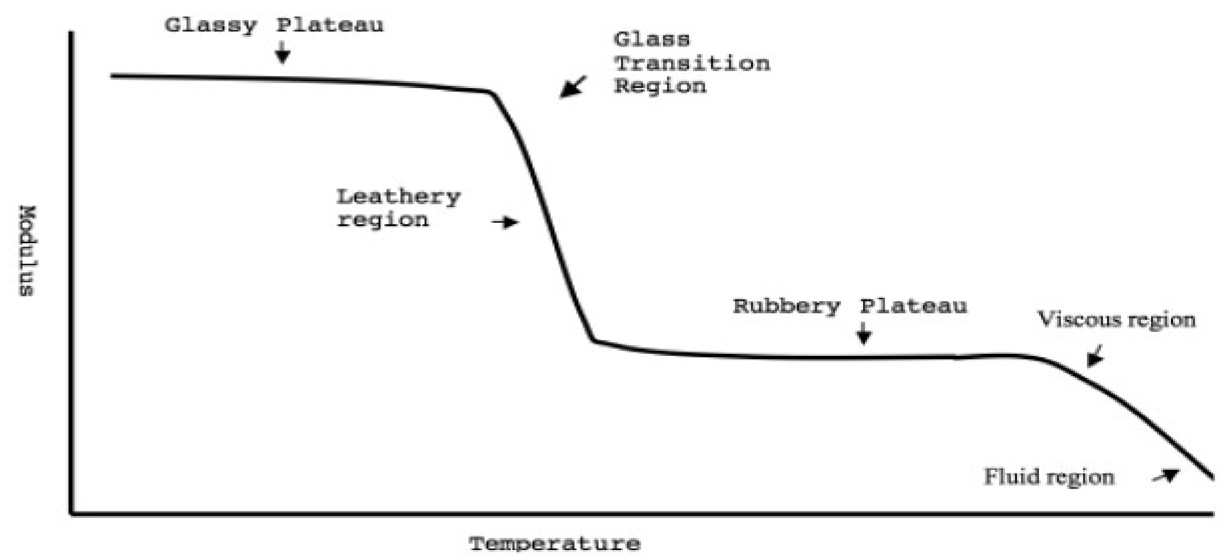

Figure 2. Storage Modulus Curve from Viscoelastic Regions of Polymers.

3. RESULTS

3.1 Results from ASTM D 1002

Results were averaged and B-basis design allowables were calculated using a visual basic program known as Stat 17. Relative standard deviation of test results was approximately five percent or less. These results were considered to be satisfactory and are shown in comparative graphs below. The graphs were plotted using Excel. We used a rule of thumb that the upper service temperature of a material was the temperature above which the adhesive bond strength dropped below 7MPa.

3.1.1.1 Stycast 2850 with Different Catalysts

Stycast 2850 is used for its relatively high thermal conductivity (>1 w/mºK) and successful application at cryogenic temperatures. In this study, we compared Stycast 2850 with Catalyst 9 vs. Stycast 2850 with Catalyst 24LV

The results shown in Figure 3 indicate the effect of the difference in catalyst over the testing temperature range for these adhesives. The 2850-24LV, which is cured at 65ºC, performs better than 2850-9 at low temperature. However, the ultimate strength drops off significantly around 50ºC and the 2850-9 drops off more slowly, maintaining 10.5MPa up to 100ºC.

We did not arrive at a definitive reason for the multiple peaks in the scans. In addition to glass transitions, there may have been secondary polymer transition related to polymer blends in the material or inhomogeneities. Further discussion is found in the DMA results section.

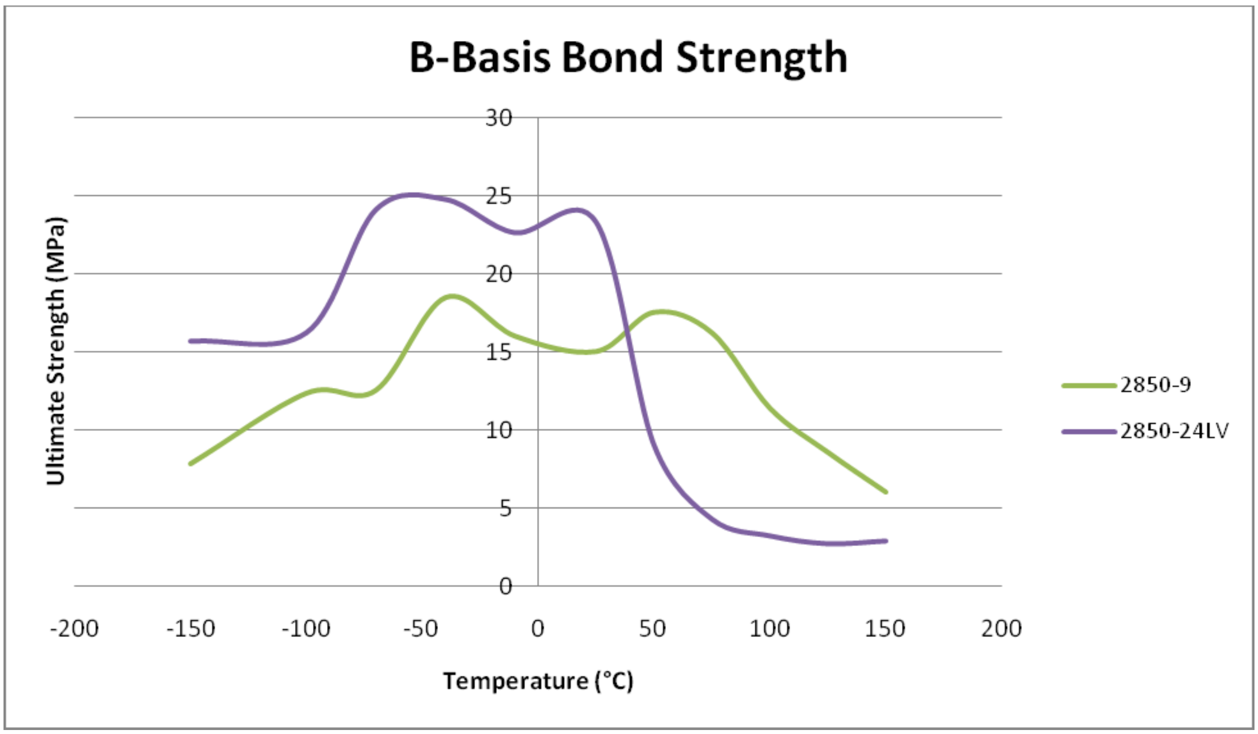

Figure 3.Comparison of Stycast 2850 with Different Catalysts

3.1.1.2 Lower Service Temperature Adhesives

EC2216, EA9309.3 and EA9361 are frequently used at JPL for applications where temperatures of 80ºC or less will be encountered, bonding of optical components, being one example. Figure 4 provides a comparison of the bond strengths of these adhesives as a function of temperature. EA9309.3 provides the best overall performance, but EA9361 appears to perform extremely well at temperatures of -100ºC and less.

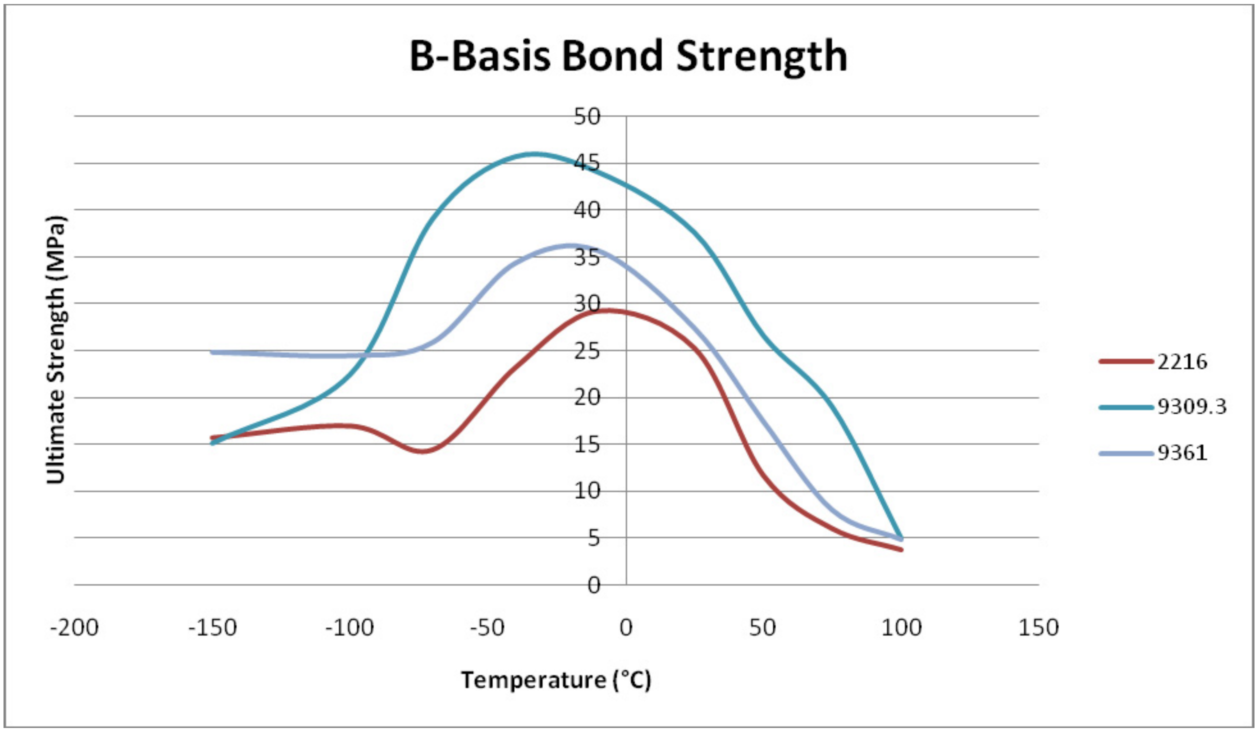

Figure 4. Comparison of Lower Service Temperature Adhesives

3.1.1.3 Higher Service temperature Adhesives

EA9394 and EA9360 are used for applications where use temperatures exceed 100ºC, as seen in Figure 5. In the case of EA9394, the material has been successfully used where application temperatures exceed 150ºC. While the limit service temperature of EA9360 is only in the 110ºC range, overall performance is excellent. Combined with the high peel strength of the material and its processability, EA9360 has been successfully used for missions such as Mars Science Laboratory, the JPL rover set to launch in September 2011.

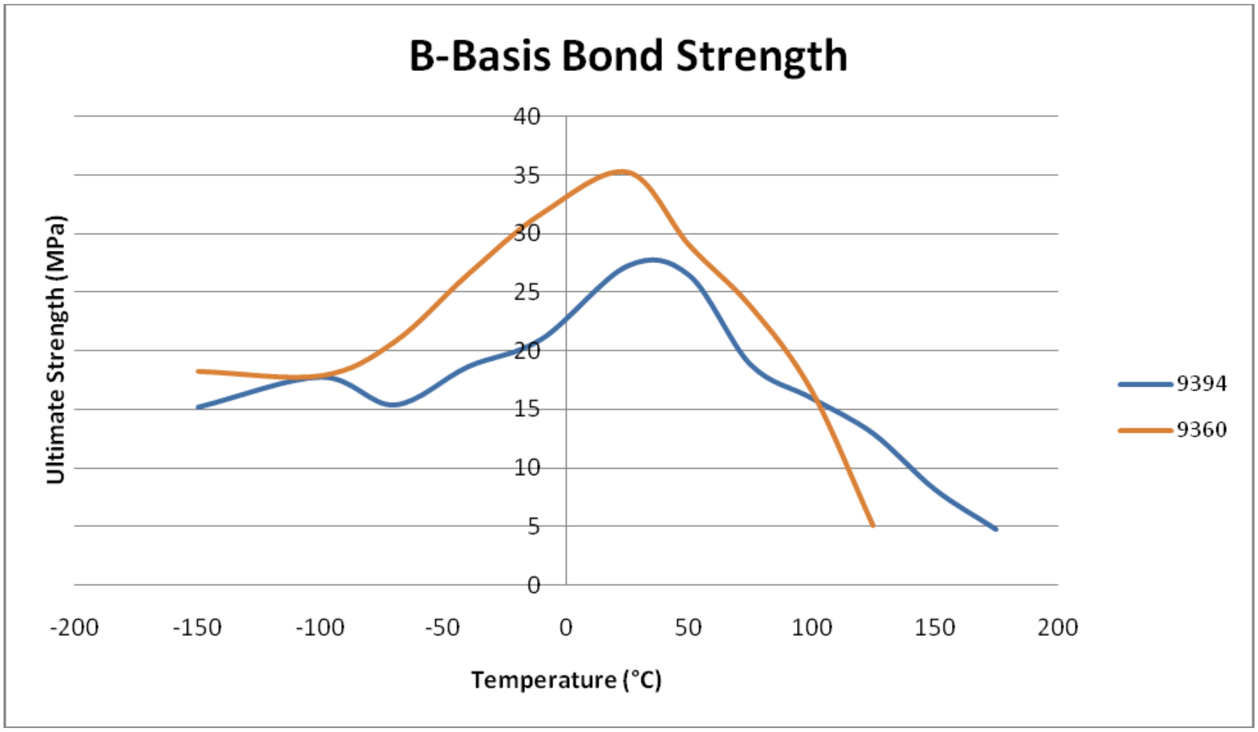

Figure 5. Comparison of High Temperature Adhesives

3.2 Results from DMA

Dynamic Mechanical Analysis was run on neat, cured specimens of the adhesive resins. To provide modulus and glass transition temperature data, the results are shown in Table 1 and in Figures 6-11.

Sample	**1st Tg (°C)**	**2nd Tg (°C)**	**3rd Tg (°C)**	**1st Loss Peak (°C)**	**2nd Loss Peak (°C)**	**3rd Loss peak (°C)**
9394	-33.6	106.1	NA*	-41.6	101.3	NA*
2216	-57.1	55.3	NA*	-60.2	39.1	NA*
2850 Cat 24LV	-41	74.	NA*	-50.7	66.9	NA*
2850 Cat 9	-39.4	97	NA*	-46.8	87.6	NA*
9309-3	-55.6	-37.0	80.2	-58.6	-43.7	73.2
9360 (blue color)	-62.3	-27.3	101.9	-63.8	-34.0	89.7
9360 (dark grey)	-56.2	58.0	NA*	-59.5	40.7	NA*

NA* = not applicable

Table 1. Glass Transition and Loss Modulus Temperatures

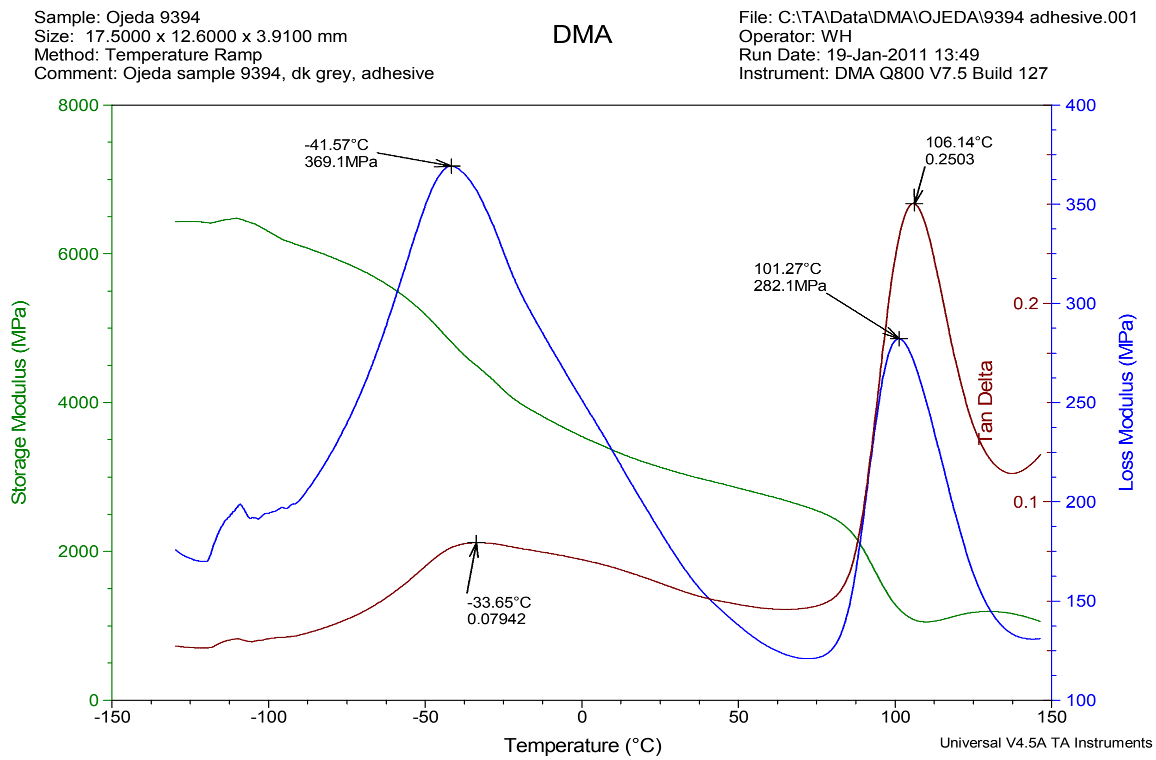

Figure 6. DMA of Sample 9394

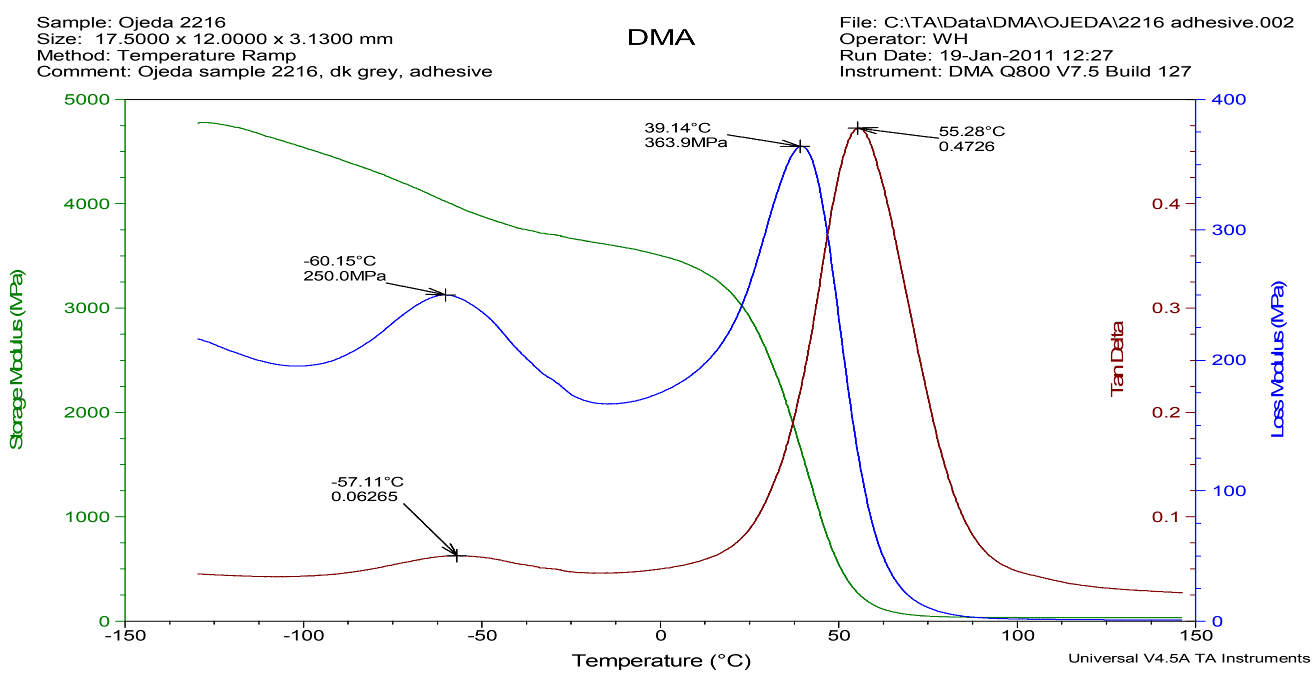

Figure 7. DMA of Sample 2216

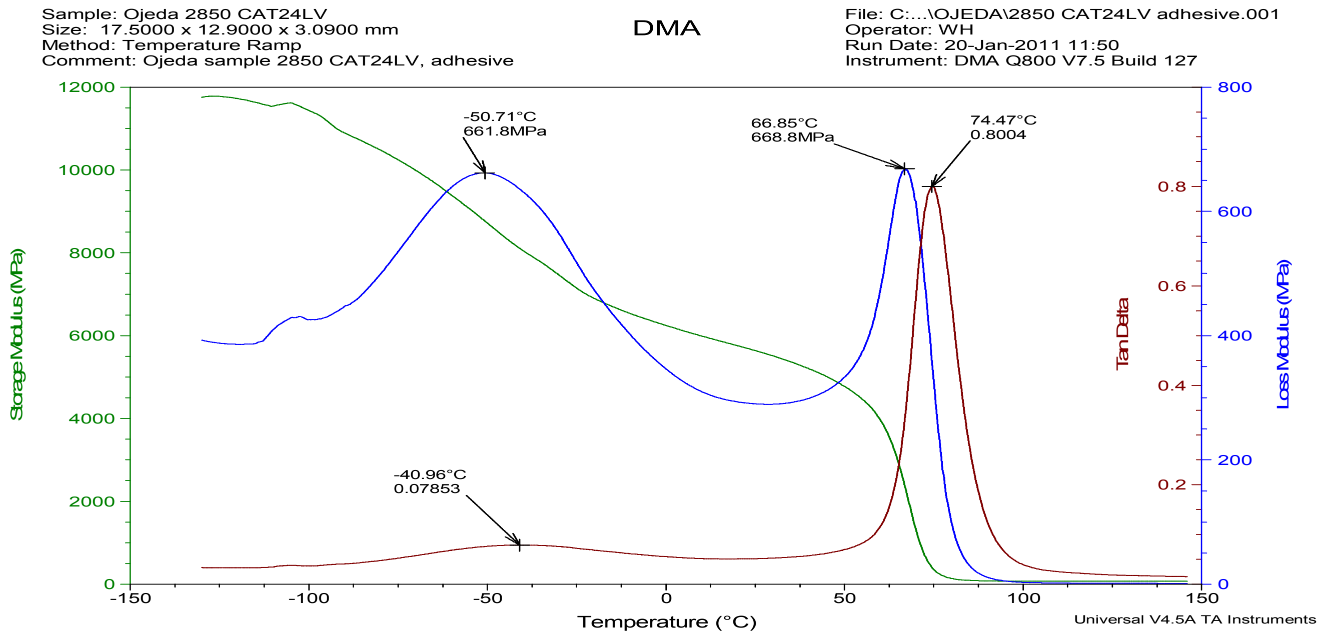

Figure 8. DMA of Sample 2850 CAT 24LV

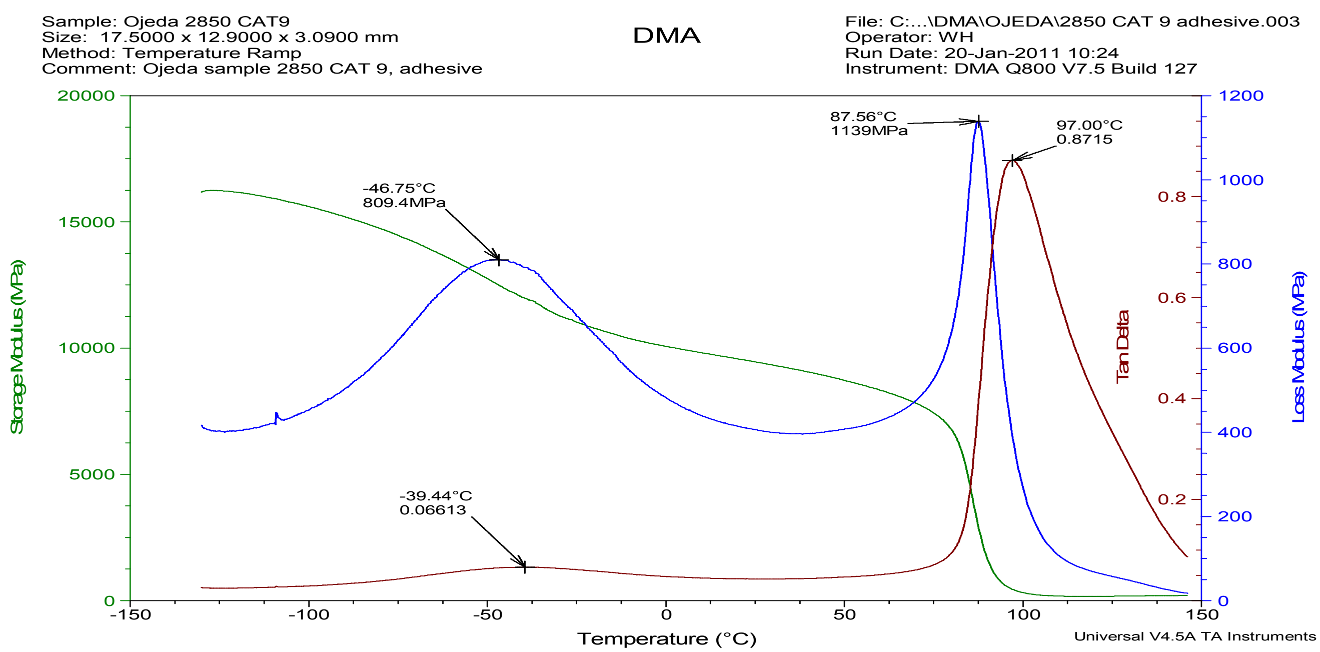

Figure 9. DMA of Sample 2850 CAT 9

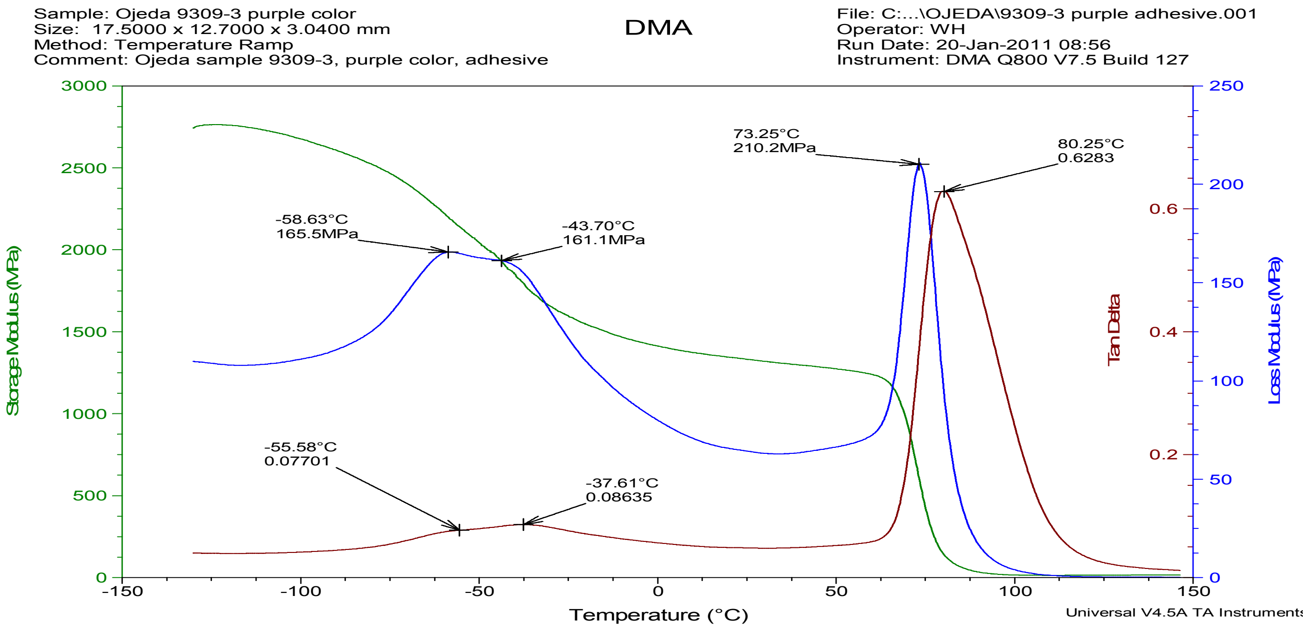

Figure 10. DMA of Sample 9309-3

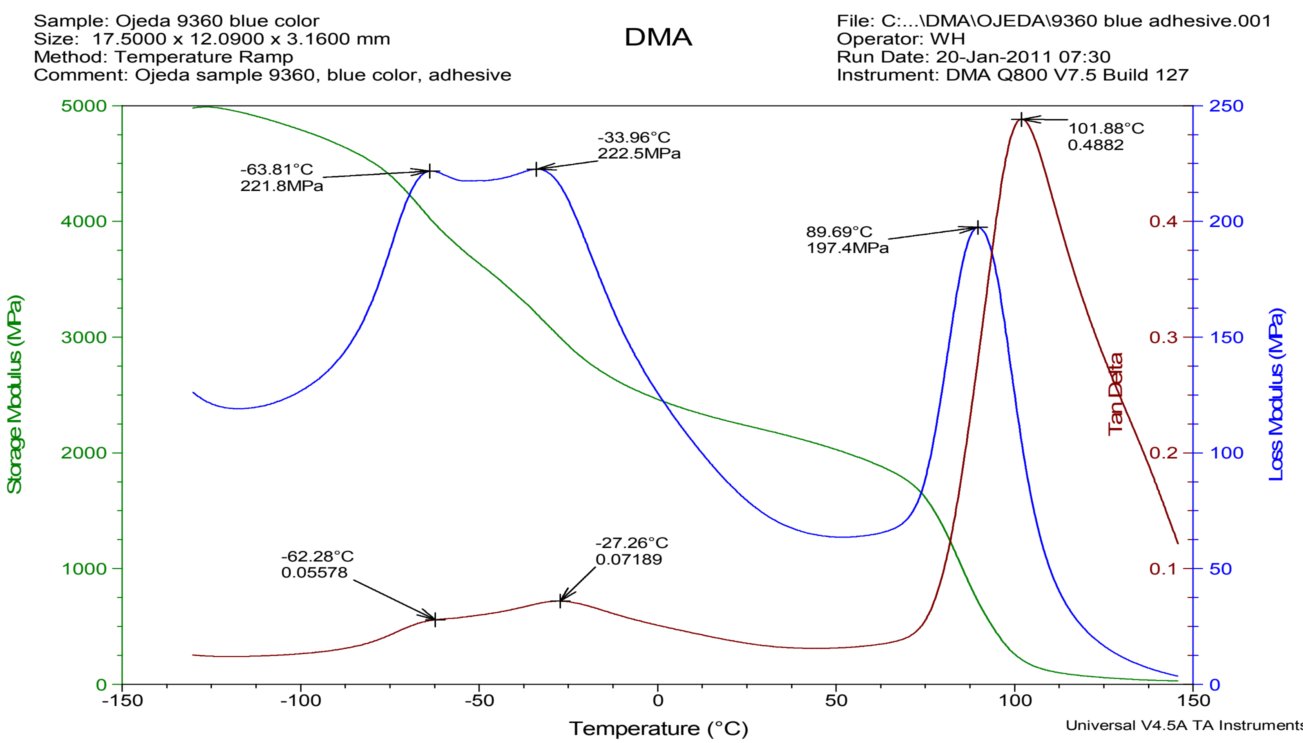

Figure 11. DMA of Sample 9360

3.2.1.1 Discussion of DMA Graphs

The fact that there are several Tan Delta and Loss Modulus peaks for each sample is indicative of samples that have morphological inhomogeneities. This is most commonly due to a sample that is comprised of a polymer blend, polymer "alloy" or a polymer that has additives (such as chain extenders or cross-linking agents). The rheological result of the mixture or blend is the generation of several (two or more) Loss Modulus and Tan Delta peaks throughout the DMA analytical run that apparently arise from the individual components of the blend. In general, the modulus results correlated well with the variations in adhesive bond strength as a function of temperature.

4. CONCLUSIONS

4.1.1.1 ASTM D 1002 and DMA Conclusions

The results show that lap shear testing at different temperatures effect the ultimate strength of adhesives. Each temperature variance has an effect on the performance of the adhesive. It is therefore important to know the application the adhesive is being used in. If it is a high temperature application, it is recommended that Hysol 9394 or Hysol 9360 are used. While for lower temperatures, EC 2216, Hysol 9309.3 and Hysol 9361 would be a better selection. In the future, we will study how substrate changes, combined with temperature effects, affect the performance of these commonly used adhesives.

From the DMA figures, it is apparent the temperature, in addition to affecting the ultimate strength, also affects the modulus of the adhesive. There is a correlation in the change in modulus with the change in ultimate strength. DMA is an inexpensive, quick method for determining how an adhesive's modulus will change over a temperature range.

Overall, it can be summarized that this work does present a good overall picture or guideline of variations in bond strength and modulus of an adhesive as a function of temperature. Careful consideration should be given when selecting an adhesive for a particular application. While this is not a complete B-basis database, it does provide a look into the changes in the properties of commonly used adhesives over a broad temperature range.

In a future paper, we will present results on the effects of different adherend materials and will present adhesive tensile modulus data obtained using ASTM D 638 for these same adhesives over these temperature ranges.

5. REFERENCES

[1] *ASTM D 1002 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to- Metal).* 01 Oct. 2010. IHS Standards. 03 Jan. 2011 <http:/standards.nasa.gov>.