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Lap Shear Testing Of Candidate Radiator Panel Adhesives

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Abstract. During testing of a subscale radiator section used to develop manufacturing techniques for a full-scale radiator panel, the adhesive bonds between the titanium heat pipes and the aluminum face sheets failed during installation and operation. Analysis revealed that the thermal expansion mismatch between the two metals resulted in relatively large shear stresses being developed even when operating the radiator at moderate temperatures. Lap shear testing of the adhesive used in the original joints demonstrated that the two-part epoxy adhesive fell far short of the strength required. A literature review resulted in several candidate adhesives being selected for lap shear joint testing at room temperature and 398 K, the nominal radiator operating temperature. The results showed that two-part epoxies cured at room and elevated temperatures generally did not perform well. Epoxy film adhesives cured at elevated temperatures, on the other hand, did very well with most being sufficiently strong to cause yielding in the titanium sheet used for the joints. The use of an epoxy primer generally improved the strength of the joint. Based upon these results, a new adhesive was selected for the second subscale radiator section.

Keywords: **a**dhesives, epoxy resin, adhesive bonding

INTRODUCTION

There exists a need for a low-cost, easily manufactured radiator to provide heat rejection for NASA Glenn Research Center's Technology Demonstration Unit (TDU). Such a radiator can provide end-to-end verification of the TDU and assist in modeling the behavior of the system. Titanium water heat pipes were chosen to utilize the most probable heat pipes for a future flight radiator, but expensive composite face sheets were replaced with inexpensive aluminum face sheets. To minimize the cost of manufacturing, simple adhesive joining was selected. A subscale radiator section was produced and tested (Ref. 1), but failures occurred at the titanium/aluminum interface. Subsequent analysis of the thermally induced shear stresses (Ref. 1) combined with lap shear testing of representative samples demonstrated that the original two-part epoxy used had insufficient shear strength. This testing was undertaken to find a higher strength adhesive that could be used to manufacture the full-scale radiator.

EXPERIMENTAL PROCEDURE

Adhesive Selection

A review of the manufacturers' literature was conducted first to identify candidate adhesives. The primary properties of interest were the shear strength, the shear modulus, the cure temperature and the glass transition temperature. Some manufacturers provided additional useful information such as the shear strength for aluminum-to-aluminum joints and elevated temperature properties. From this review and consultations with the manufacturers, several new adhesives were selected.

Two-part epoxies that cure at room temperature were preferred for ease of manufacturing, which is why the MasterBond EP30HTLO was selected originally. MasterBond also recommended EP21TDCHT-LO as a lower strength but lower modulus alternative. Based upon the thermal stress analysis (Ref. 1, 2), a lower modulus adhesive would act like a compliant layer and lower the shear stresses.

In addition, the original MasterBond EP30HTLO two-part epoxy was tested in several conditions to examine if the problem lay with the original adhesive lot and preparation. A new lot was procured to ensure that the epoxy was fresh and using epoxy that had exceeded its recommended shelf life was not the cause of the failures. The original tests were done on adhesive joints cured at room temperature, and the adhesive had silver added to it to attempt to enhance its thermal conductivity. Elevated temperature cures and removing the silver, since it did not seem to greatly decrease the thermal resistance at the bond were examined to improve the baseline EP30HT adhesive.

In aerospace, a class of adhesives called structural film adhesives are used to adhesively bond load-bearing structures together. These adhesives are uncured resin sheets, with or without fiber reinforcement. Film adhesives require an elevated temperature cure, but they are generally stronger and allow better control of the adhesive thickness. An elevated temperature cure also can be beneficial since the zero stress state for the bond will be at the cure temperature, and stresses at the operating temperature will be minimized. The prior analysis (Ref. 1) had established that a thicker bond, while detrimental to the thermal performance of the joint, would lower the shear stress. By using a thicker adhesive film, the thickness of the adhesive layers could be correspondingly increased in a controlled manner. Film adhesives also offer some advantages in manufacturability in that they are easier to place in the desired position and have much longer working lives (days versus hours or even minutes for two-part epoxies). Of the available film adhesives, 163-2 K (3M), FM 300-2 (Cytec) and HYSOL EA 9695 (Henkel) appeared to have the best combinations of properties and were selected for testing.

Finally, several adhesive manufacturers provide epoxy primers as a way to improve the bond strength. EW-5000 primer (3M) was procured and used with some of the film adhesives to determine if the bond strength could be improved this way.

Two-part epoxy can be cured at room temperature or elevated temperature. MasterBond also recommends a 343 K post-cure to maximize the strength. The EP30HT two-part epoxy was tested in the as-room temperature cured, room temperature cured plus 343 K post cure treatment and 398 K cured conditions. The EP21TDCHT-LO epoxy was tested only in the room temperature cured plus 343 K post cure treatment and 398 K cured conditions. The film adhesives were cured using their manufacturers' suggested curing temperature. The complete list of adhesives and sample conditions is given in Table 1.

In addition, to simulate a thermal cycle in vacuum, all but a single set of samples (Condition 23) were also given a 398 K vacuum exposure for one hour followed by an oven cool. The vacuum was provided by a mechanical roughing pump, but it was sufficient to help volatilize any volatile compounds and remove any dissolved gasses.

Specimen Design and Manufacture

Since analysis indicated that thermally indicated shear stresses were the most likely cause of failure in the original radiator, a test designed to measure the shear stress that could be supported by the adhesive was selected. Single lap shear testing per ASTM Standard D1002 (Ref. 3) develops the shear stress by pulling on the ends of two pieces of metal bonded in the center. From the maximum load and the area of the joint, the maximum shear stress experienced by the adhesive can be determined. Failure could be in the bond or at either adhesive/metal interface and would indicate the relative strength of each.

The lap shear joints were made from dissimilar metals as will be experienced in the radiator. To simulate the titanium heat pipes, Grade 2 commercially pure (CP) titanium sheet 0.90 mm thick was used. To simulate the aluminum face sheets and manifolds that may be adhesively bonded, Grade 1100 (commercially pure) aluminum 1.54 mm thick was used. The titanium thickness matched the thicknesses of the material used in the actual TDU heat pipe while the aluminum thickness was more representative of the manifolds than the face sheets. The thicker material was used because it represented a "worst case" scenario based upon the stress analysis of the joint (Ref. 1).

The metal surfaces were prepared by first sand-blasting the surfaces to produce a matte, white metal surface. The sand-blasting removed the existing oxide layer and cleaned the surface. The surfaces were wiped down with ethanol using a clean cloth once to remove any debris and dirt, allowed to dry completely, and wiped down a second time with ethanol to remove any remaining grease or oil.

Samples that used the EW-5000 primer coating were dipped in the primer, inverted so that the coated end was at the top of the piece when held vertically, and allowed to air dry for 30 to 45 minutes. When dry, the pieces were cured at 300 K for 60 minutes and oven cooled. This produced a primer layer approximately 0.02 mm thick.

Table 1. Test Conditions

Condition	Adhesive	Cure Temperature	Thermal Exposure	Test Temperature
1	Old MasterBond EP30HT-LO w/silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	RT
2	Old MasterBond EP30HT-LO w/silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	398 K
3	Old MasterBond EP30HT-LO w/silver	398 K	398 K / 1 H/ Vac / FC	RT
4	Old MasterBond EP30HT-LO w/silver	398 K	398 K / 1 H/ Vac / FC	398 K
5	New MasterBond EP30HT-LO w/silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	RT
6	New MasterBond EP30HT-LO w/silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	398 K
7	New MasterBond EP30HT-LO w/silver	398 K	398 K / 1 H/ Vac / FC	RT
8	New MasterBond EP30HT-LO w/silver	398 K	398 K / 1 H/ Vac / FC	398 K
9	New MasterBond EP30HT-LO w/o silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	RT
10	New MasterBond EP30HT-LO w/o silver	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	398 K
11	New MasterBond EP30HT-LO w/o silver	398 K	398 K / 1 H/ Vac / FC	RT
12	New MasterBond EP30HT-LO w/o silver	398 K	398 K / 1 H/ Vac / FC	398 K
13	MasterBond EP21TDCHT-LO - Low Modulus	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	RT
14	MasterBond EP21TDCHT-LO - Low Modulus	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	398 K
15	MasterBond EP21TDCHT-LO - Low Modulus	398 K	398 K / 1 H/ Vac / FC	RT
16	MasterBond EP21TDCHT-LO - Low Modulus	398 K	398 K / 1 H/ Vac / FC	398 K
17	3M 163-2 K Film Adhesive + EW-5000 Primer	383 K	398 K / 1 H/ Vac / FC	RT
18	3M 163-2 K Film Adhesive + EW-5000 Primer	383 K	398 K / 1 H/ Vac / FC	398 K
19	Cytec FM 300-2 Film Adhesive	393 K	398 K / 1 H/ Vac / FC	RT
20	Cytec FM 300-2 Film Adhesive	393 K	398 K / 1 H/ Vac / FC	398 K
21	Henkel Hysol ea 9695 Film Adhesive	393 K	398 K / 1 H/ Vac / FC	RT
22	Henkel Hysol ea 9695 Film Adhesive	393 K	398 K / 1 H/ Vac / FC	398 K
23	Old MasterBond EP30HT-LO w/ silver	RT	None	RT
24	Cytec FM 300-2 Film Adhesive + EW-5000 Primer	393 K	398 K / 1 H/ Vac / FC	RT
25	Cytec FM 300-2 Film Adhesive + EW-5000 Primer	393 K	398 K / 1 H/ Vac / FC	398 K
26	New MasterBond EP30HT-LO w/ silver (Repeat of Condition 7, data sets combined)	RT + 343 K Post Cure	398 K / 1 H/ Vac / FC	RT
27	New MasterBond EP30HT-LO w/silver (Repeat of Condition 8, data sets combined)	398 K	398 K / 1 H/ Vac / FC	398 K
28	Henkel Hysol ea 9695 Film Adhesive + EW-5000 Primer	393 K	398 K / 1 H/ Vac / FC	RT
29	Henkel Hysol ea 9695 Film Adhesive + EW-5000 Primer	393 K	398 K / 1 H/ Vac / FC	398 K

RT = Room Temperature (~293 K)

For the two-part epoxies, the end of one piece was marked to indicate the 25 mm long section of the sample that would make up the adhesive joint. The epoxies were mixed according to the instructions provided by MasterBond, and the epoxy brushed onto the piece. For the film adhesives, a 25 mm x 37 mm piece was cut from the provided sheet of adhesive and affixed to the end of one piece. The second piece was joined to the first. A clamping force between 69 and 103 kPa was used to hold the pieces together without pressing the adhesive out of the joint. The specimens were given the thermal treatments listed in Table 1. The area of the adhesive was measured using a Focus optical comparator from Optical Gaging Products to measure the lengths of the joints and calipers to measure the widths.

Lap Shear Testing

Lap shear testing was conducted using an Instron TT-series load frame upgraded to computer control and data acquisition. Per ASTM Standard D1002 (Ref. 3), the specimens were gripped at each end and pulled in tension at a crosshead rate of 12.7 mm/min while the load was monitored using a load cell. To ensure that the loads were uniaxial, shims were used to counter the offset of the lap joint and position the samples in a vertical position. The peak load and the area of the bond were used to calculate the shear strength of the joints. Three tests for each condition were conducted.

Testing was conducted at room temperature and at 398 K. For the 398 K tests, a three-zone furnace approximately 150 mm tall was placed around the sample. Thermocouples placed on the top, middle and bottom of the adhesive joint were used to monitor the joint temperature. The samples were heated to 398 K in about five minutes typically.

Once the test temperature was achieved, the samples were allowed to soak for 5 minutes to attain full thermal equilibrium and ensure that the thermally induced stresses were maximized. The samples were tested in the same manner as the room temperature specimens.

RESULTS AND DISCUSSION

The results of the lap shear tests are presented in Figure 1. The bars represent the average shear strength. The error bars are equal to plus and minus one standard deviation and give an indication of the scatter of the data points. No tests were deemed to be outliers, so all tests are included in the calculation of the averages and standard deviations.

t was noted during testing that several of the samples showed evidence of yielding in the load-displacement curves such as is seen in Figure 2 for Samples 15-2 (room temperature test) and 25-2 (398 K test). Samples without yielding of the titanium reached peak stress and failed, so they do not have the tail on the right of these two curves. Post-test evaluation of the samples indicated that the titanium had necked and, in some cases, failed as shown in Figure 3. The titanium sheet stock was sufficiently thinner than the aluminum so as to be the weaker of the two halves of the specimen. When at least one specimen yielded or failed, it was noted in the figure. Once yielding occurred, the stress-strain curve indicated that the behavior of the specimen was dominated by the titanium rather than the adhesive. The peak load was still used to calculate the adhesive shear stress since the adhesive did have to withstand that load. The values reported likely represent a lower bound for the adhesive shear strength since the yielding of the titanium sheet produced a biaxial stress condition in the adhesive joint instead of the desired uniaxial test condition. This is probably representative of the manifolds but is not representative of the face sheets where thinner aluminum sheet is used. Retesting with aluminum sheet samples the same thickness as the actual face sheets would likely lead to a change to yielding in the aluminum. Either way, the load bearing capability of the adhesive exceeds that of at least one of the metals.

Figure 4 shows more typical adhesive joint failures. The pictures cover most of the adhesives used in the testing and are representative of the class as well as the specific adhesive. Samples tested at both room temperature and 398 K are presented. The sample on the left in each pair is the aluminum half of the specimen, and the darker sample on the right is the titanium half of the sample. Most samples failed at the adhesive/metal interface. This was expected since titanium and aluminum both form tenacious oxide layers that are largely chemically inert. A few cases did show evidence of failure in the adhesive. Most notably, the failure of the HYSOL EA 9695 with EW-5000 primer in the adhesive with near complete coverage of both the titanium and aluminum (Figure 4 g and h) indicated the best adhesive bond to the metals.

The MasterBond EP21DCHT-LO was expected to have a lower strength than the EP30HTLO. However, it proved to be a very good performer at room temperature with a shear strength of 11.3 MPa, which is comparable to those of the epoxy film adhesives. That was sufficiently strong to cause the titanium sheet to undergo yielding. Unfortunately, at 398 K, the strength degraded to about 2 MPa. The low elevated temperature strength made the EP21DCHT-LO two-part epoxy unacceptable since the maximum stress in operation was expected at 398 K with a room temperature cure epoxy system. The EP20DCHT-LO also suffered in manufacturability because the epoxy's pot life was only 20 minutes and the mixed epoxy had a very high viscosity, which made it difficult to apply it to the parts quickly and easily.

The MasterBond EP30HTLO showed uniformly lower than required strength regardless of silver addition, curing temperature, post cure treatment, thermal cycle or test temperature. The best result at room temperature was obtained for the fresh epoxy without a silver addition. The same condition performed well at 398 K but was equaled by the new EP30HTLO samples given a 398 K cure. Based upon the lower than required shear strength, EP30HTLO was deemed unsuitable for use in this application.

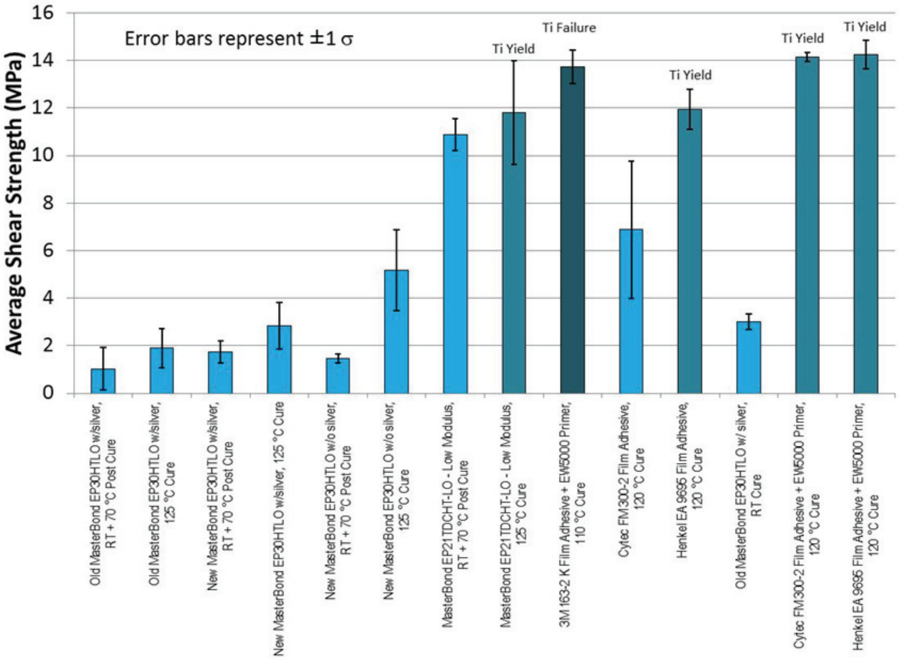
As can be seen in Figure 1, the highest shear strengths were obtained by epoxy film adhesives. With the exception of the FM 300-2 adhesive without a primer, all developed a bond sufficiently strong at room temperature to cause yielding in the titanium sheet. In the case of the 163-2 K epoxy, the bond shear strength exceeded the ultimate tensile strength of the titanium sheet.

The high strengths of the film adhesives generally persisted at 398 K. As can be seen in Figure 1, the film adhesives with the exception of the 163-2 K had shear strengths in the 9.6 to 11 MPa range. This was sufficient to cause yielding to varying degrees in the titanium sheet.

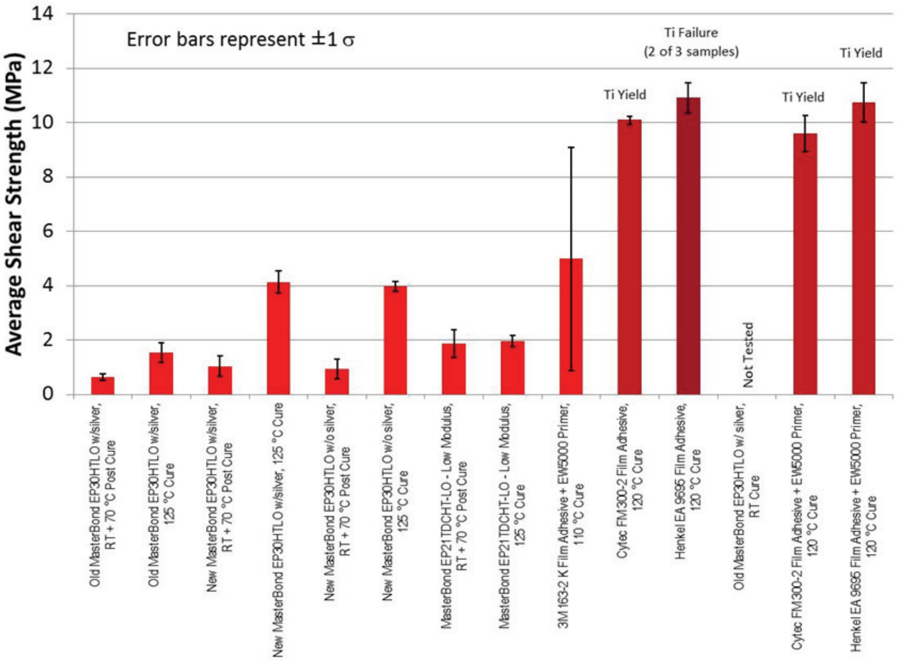
Because the film adhesives must be cured at elevated temperatures near the radiator's planned operating temperature, it is assumed that the zero stress state will be near 398 K while the most stress for the film adhesive bonds will be experienced at room temperature. Because of this, the 163-2 K cannot be ruled out entirely, but the FM 300-2 and HYSOL EA 9695 adhesives appear to be better overall choices.

In terms of manufacturability, all of the film adhesives were easier to apply accurately and cleanly than the two-part epoxies, but they require an elevated temperature cure and hence a very large oven for a full size radiator panel. Alternative curing techniques such as operating the heat pipes at the epoxy cure temperature are under consideration for the large panels that will alleviate the need for a large oven. The 163-2 K was the easiest film adhesive with which to work since the protective films on both surfaces released easily when desired. The FM 300-2 was next easiest to apply. The paper protective film had a tendency to fall off, which could prematurely expose the epoxy adhesive, but the polyester film was difficult to remove. The HYSOL EA 9695 was the hardest of the three film adhesives to use since the polyester protective film on both sides was difficult to remove. The 163-2 K had the added advantage that it had a tacky surface and would stay in place well once adhered. The HYSOL EA 9695 and FM 300-2 did not adhere as well and could slip. The application of low heat to develop a tack bond is an option for both.

The role of an epoxy primer can be seen when comparing the shear strength of the FM-200 and HYSOL EA 9695 film adhesives with and without the EW-5000 primer. At room temperature, there is a dramatic increase in the shear strength of the bond for FM 300-2 from 6.9 MPa to 13.8 MPa. The HYSOL EA 9695 also shows a substantial increase from 11.9 MPa to 14.1 MPa. The benefits were not as clear at 398 K since both adhesives reached the yield strength of the titanium sheet with and without the primer, but the HYSOL EA 9695 in particular showed enhanced bonding as evidenced by more deformation in the titanium sheet and failure in the adhesive rather than at the metal/adhesive interfaces. It appears that for the radiator application, an epoxy primer as a bond coat will enhance the bond and improve the probability of successful bonding.



(a) Room Temperature



(b) 398 K

Figure 1. Adhesive Shear Strengths

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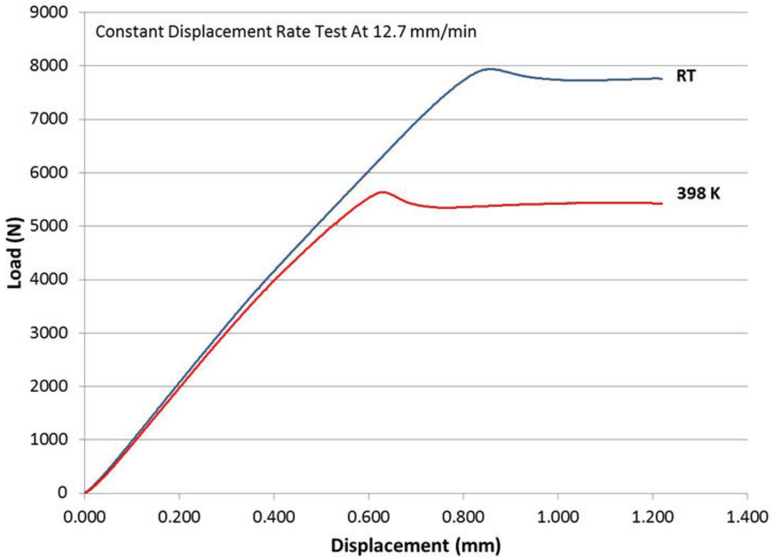
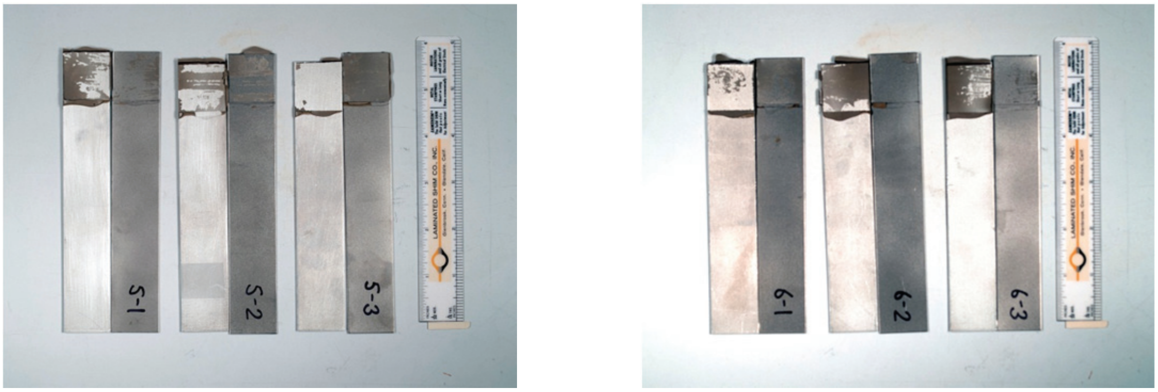


Figure 2. Typical Shear Load-Displacement Curves Demonstrating Titanium Yielding Behavior (Room Temperature - Sample 15-2, 398 K – Sample 25-2)

(a) 163-2 Tested At Room Temperature
(Condition 17)

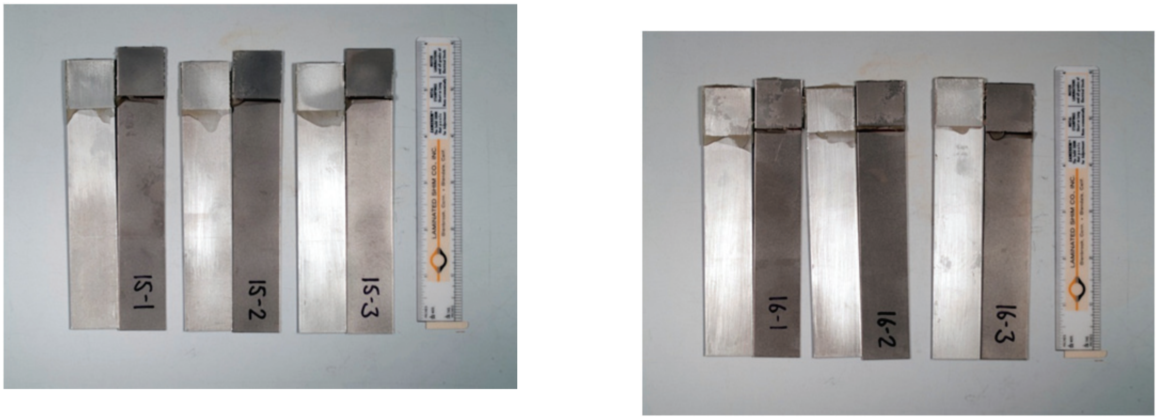
(b) EA9695 Tested At 398 K
)Condition 22)

Figure 3. Lap Shear Samples That Failed In The Titanium:



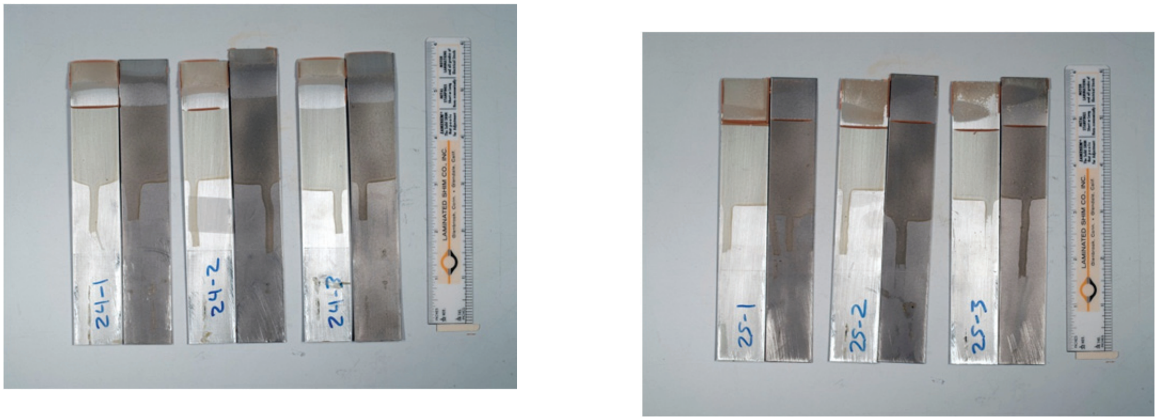
(a) EP30HT-LO, With Silver, RT Cure + 343 K Post Cure Treatment (Condition 5), Room Temperature Test

(b) EP30HT-LO, With Silver, RT Cure + 343 K Post Cure Treatment (Condition 6), 398 K Test



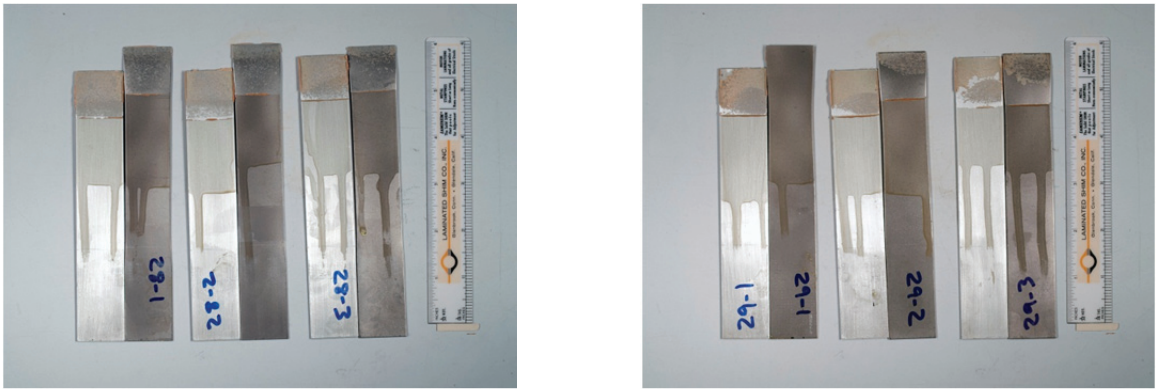
(c) EP21DCHT-LO, 398 K Cure (Condition 15), Room Temperature Test

(d) EP21DCHT-LO, 398 K Cure (Condition 16), 398 K Test



(e) FM 300-2, 393 K Cure (Condition 24), Room Temperature Test

(f) FM 300-2, 393 K Cure (Condition 25), 398 K Test



(g) HYSOL EA 9695 With EW-5000 Primer, 393 K Cure (Condition 28), Room Temperature Test

(h) HYSOL EA 9695 With EW-5000 Primer, 393 K Cure (Condition 29), 398 K Test

Figure 4. Representative failure surfaces of lap shear joints

SUMMARY

Based upon the combination of strength and manufacturability, the epoxy film adhesives clearly are the preferred adhesives of those tested. Yielding of the titanium in many instances indicated that the film adhesives likely had even higher shear strengths than those reported here. The 163-2 K had very good room temperature strength, but did not perform as well as the FM 300-2 and HYSOL EA 9695 adhesives at elevated temperature. The best performance for the radiator application of those adhesives appears to be the HYSOL EA 9695 epoxy film adhesive with the EW-5000 primer, while the FM 300-2 epoxy film adhesive with the EW-5000 epoxy primer was a close second.

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