

# Testing Tensile and Shear Epoxy Strength at Cryogenic Temperatures

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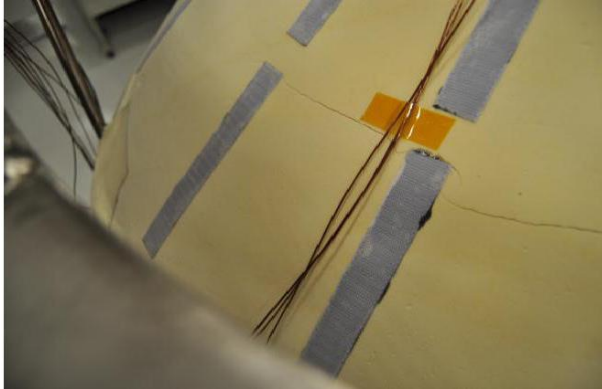
**Abstract.** This paper covers cryogenic tensile and shear testing research completed on 15 epoxies used in cryogenic applications. Epoxies are used in many different applications; however, this research focused on the use of epoxy for bonding MLI standoffs to cryogenic storage tanks and the loads imparted to the tank through the MLI. To conduct testing, samples were made from bare stainless steel, aluminum and primed aluminum. Testing involved slowly cooling test samples with liquid nitrogen (LN2) then applying gradually increasing tensile loads to the epoxy. If the sample passed tensile testing, shear testing was then performed in a similar fashion. The testing evaluated the strength and durability of epoxies at cryogenic temperatures which showed that some epoxies withstood the harsh conditions while others failed. The three epoxies yielding the best results were CTD Cryobond 621, Masterbond EP29LPSP and Scotchweld 2216. For all metal surfaces tested, each of these epoxies had zero failures for up to 18 kg of mass.

## 1. Introduction

Structural methods need to be refined in order to attach MLI blankets to spacecraft in a manner to survive a combination of acceleration, acoustic, and venting loads while minimize the parasitic heat load to the tank. The attachments serve as direct heat loads to the tank and often are a significant portion of a tank applied heat load [1]. There have been instances where MLI was not appropriately attached to spacecraft and has been lost or damaged, compromising the mission. [2, 3]. Based on a review of typical attachment methods, most use plastic (nylon or ultem) holders to minimize conduction loss through a blanket. However, these plastics have a much larger coefficient of thermal contraction and often contract 1% or more than most base metals (most metals contract between 0.3 – 0.4% between room temperature and 77 K and plastics can contract up to 1.5% or greater) [4]. As such, an epoxy must be able to handle the differential contraction between the two materials and also handle the many other forces that it may encounter.

A typical insulation system for a cryogenic upper stage would include spray-of foam insulation (SOFI) underneath the MLI blankets to prevent air liquefaction. The polyetherimide standoffs would be attached to the tank and protrude through the SOFI to provide points of attachment for the MLI blankets. Previous attempts to attach the MLI directly to SOFI has induced cracking in the SOFI as shown in Figure 1 [5].

For reference, Figure 2 shows a possible configuration of a standoff with an MLI blanket. The flat, disk portion of the standoff is bonded to the tank wall with epoxy. The strength of that epoxy bond at cryogenic temperatures is important since failure of that bond would cause the MLI to separate from the tank and increased the boil-off of cryogenic propellants. Therefore tests were conducted to assess the tensile and shear epoxy strength to aluminium, primed aluminium, and stainless steel samples at cryogenic temperatures.



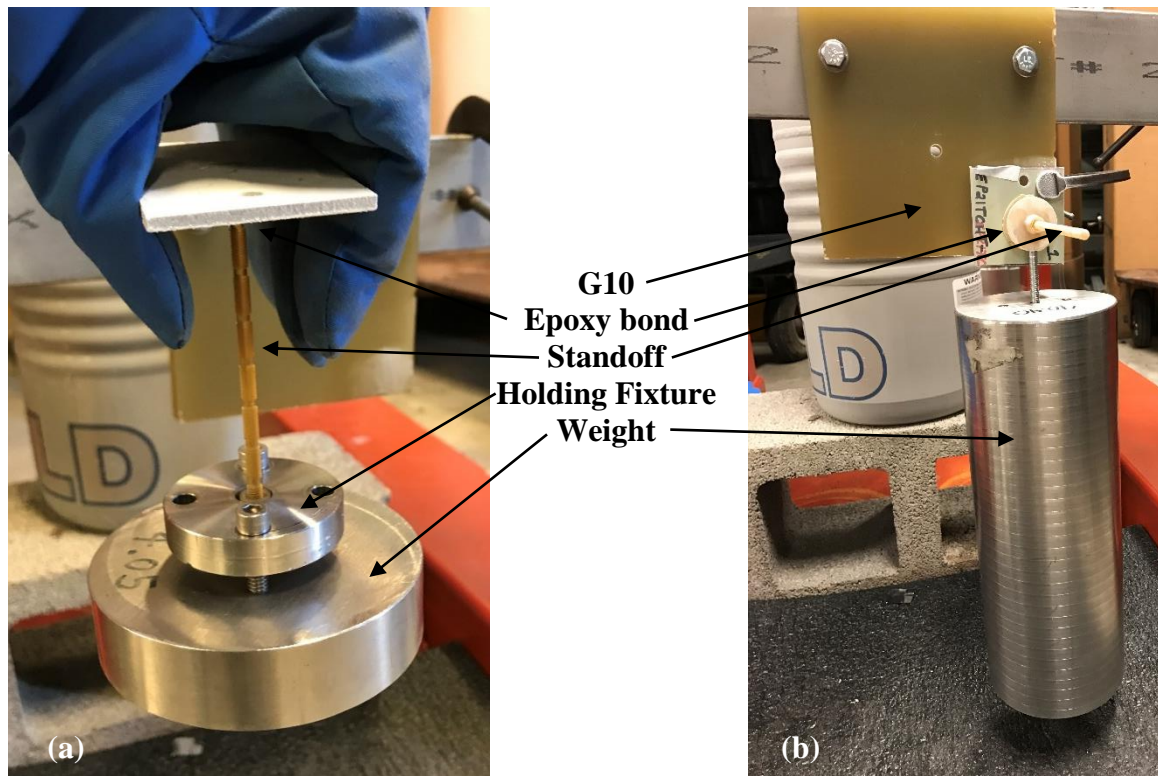
**Figure 1.** SOFI after MLI was directly attached to the surface.



**Figure 2.** An MLI standoff holding the MLI blanket and foam insulation to the metal surface.

## 2. Testing

Shear and tensile epoxy testing was conducted at the Creek Road Cryogenic Complex facility at NASA Glenn Research Center in six rounds over the course of 8 months. Each epoxy tested was bonded to aluminium, primed aluminium, and stainless steel samples. Testing consisted attaching a type E thermocouple to the back of a sample and slowly cooling it with liquid nitrogen (LN<sub>2</sub>) over the course of approximately 30 minutes to minimize the time dependant thermal stresses in the sample and avoid thermal shock. Once at a temperature of 86K, the tensile strength of the epoxy bond was tested by hanging weights from the end of the standoff shown in Figure 3a. After testing one or two weights, the sample was returned to the liquid to minimize warming and thermal cycling of the sample. The mass hanging from the standoff was gradually increased from 1.29kg up to 11.82kg. The maximum weight was determined by the rated strength of the polyetherimide standoff [6]. Samples which survived tensile testing were then tested in shear by hanging the weights at the base of the standoff as shown in Figure 3b. For shear testing, the sample was attached to a piece of G10 to minimize heat transfer. Also, a standoff mount was used near the base of the standoff to keep the weight as close to the base of the standoff as possible to reduce moment effects. Testing did not strictly follow ASTM standards, however ASTM E8 and D1002, D3163, and D4896 were used as guide in developing the test procedures [7-10].



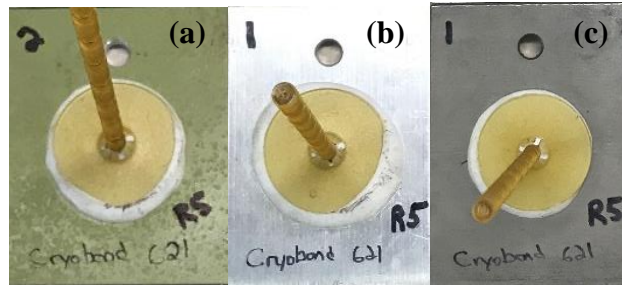
**Figure 3.** (a) Tensile epoxy test configuration with weight sample. (b) Shear epoxy test configuration with weight sample.

## 2.1. Epoxies for Testing

The fifteen epoxies tested included Altheris C1, Altheris EA-2A, Cryobond 621, GE Varnish, Huntsman EPIBOND, HYSOL EA9369, HYSOL EA9430, Masterbond EP29LPSP, Masterbond EP21TCHT-1, Masterbond EP30-2, Masterbond SUP12AOHT-LO, M-bond AE-10, Scotchweld 2216, STYCAST 1266, and STYCAST 2850FT.

## 2.2. Samples for Testing

The samples were made by cutting out rectangular pieces of metal, roughly 4 x 5 centimeters. A hole was drilled centered on the end of the metal to be used as an attachment point for LN2 cooling. After the surface was deburred and wiped with acetone, the MLI standoff was epoxied to primed aluminium 2219, aluminium 6061 or stainless steel 304 as shown in Figure 4 and cured per the manufacturer's instructions. Additionally, the surfaces to be used for Altheris and Masterbond AE-10 epoxies were scoured with 320 grit sandpaper prior to application of the epoxy and the standoff. The epoxies varied in cure time and several required a period of heating in an oven complete the cure. When multiple cure options were available, the lowest temperature cure was selected. The epoxied standoff to the metallic based is hereto referred to as a test sample. As frequently as possible, multiple samples were made using the same epoxy/metallic base combination.

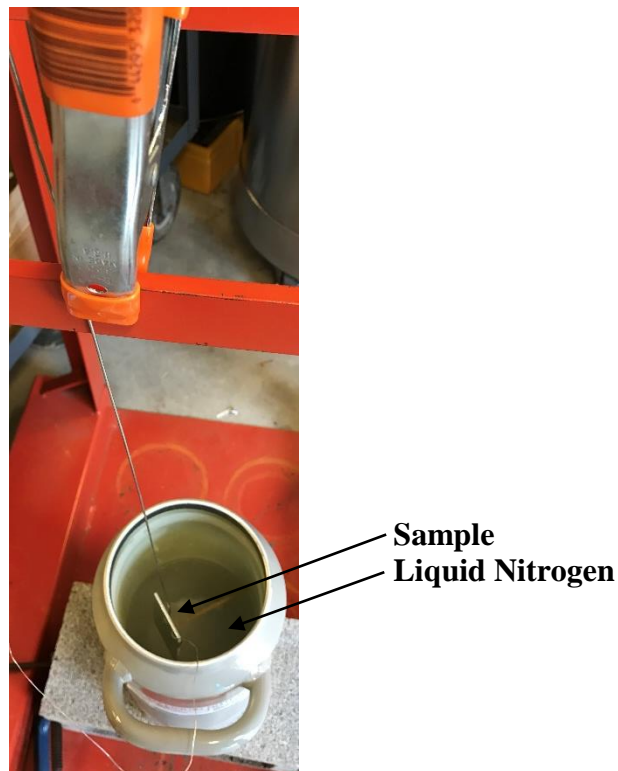


**Figure 4.** (a) Primed 2219 aluminium sample. (b) Aluminium 6061 sample. (c) Stainless steel 304 sample.

A thermocouple was taped to the back of each sample for monitoring the sample temperature during chill down and testing. The 10 weights used were cut from stainless steel bar stock and ranged from 1.11 to 11.6 kg. Three holes were tapped on the end of each weight to allow for mounting of a stainless steel flange fitting or a threaded hook for tensile and shear testing, respectively.

### 2.3. Testing Procedure

In order to minimize the time dependant cooldown stresses and prevent thermal shock, each sample was slowly cooled by hanging the sample above a Dewar of LN2 via a hook and clamp as shown in Figure 5. Using 5 minute increments, each sample was kept at approximately 25 mm above, 12 mm above, just touching, and fully submerged in the LN2. The sample temperature was monitored using a type E thermocouple taped to the back of the sample.



**Figure 5.** Setup for sample cooldown.

Once the sample reached the minimum temperature of 86 K, it was removed from the LN2 bath and attached to the appropriate weight via screws, see Figure 3a. The weight was then lifted by pulling

straight up on the metal sample. With each weight applied, observations were made and temperatures were monitored. After testing two weights, the sample was removed from the attaching hardware and re-submerged in LN2. Typically samples warmed up to 125 K during this time period. Once the sample was back to approximately 86K, the process was repeated using the next highest weight. Tensile weights tested were 1.3, 2.0, 3.6, 5.3, 6.9, 8.0, 10.5, 11.5, 16.0, and 18.5 kg. If a sample passed the 18.5 kg mass, it was then tested in shear starting at the lowest weight, per Figure 3b. Shear masses tested were 1.1, 1.8, 3.4, 5.1, 6.7, 7.8, 10.3, 11.6, 15.9 and 18.3 kg. The 0.18 kg difference in mass between the tensile and shear testing is due to the added weight of the modified flange for tensile testing. A sample tensile and shear test is shown in Figure 3.

### 3. Results and Discussion

The tensile and shear testing results are compiled in Table 1. Each column represents a different weight and testing in either shear or tension. The green check mark and red "X" indicate the passing and failure of the epoxy, respectively. From Table 1, one can see the epoxy bond results are fairly binary in that the bond typically broke at a lower mass or survived to the largest mass. Several of the epoxies (Huntsman EPIBond, HYSOL EA9369, Masterbond EP21TCHT-1, STYCAST, 2850FT, and STYCAST 1266) which failed on the stainless steel and aluminium, performed well on primed aluminium samples.

In the case of an epoxy failure, with the exception of the Altheris C1, the epoxy removed "cleanly" from the metal sample and remained primarily on the standoff as shown in Figure 6. The STYCAST 2850FT, Masterbond EP29LPSP, and CTD 621 were all noted as easy to work with given the opaque nature and thicker consistency of the epoxies. Later rounds of testing recorded observations of loud popping and cracking noises during sample chill down, in particular when the sample was lowered to touching the LN2. When noise observations were recorded, the popping noises were heard primarily during the sample cool down. Although not guaranteed, the occurrence of these popping noises provided good indicators for future epoxy and standoff failure. Additionally, 5 out of 6 standoff failures occurred above 11.8 kgs. Of the 65 samples tested, 24 samples, not including standoff breaks, survived tensile and shear testing.



**Figure 6.** Epoxy failures on stainless steel samples.

The CTD Cryobond 621, Masterbond EP29LPSP, and Scotchweld 2216 were least sensitive to the sample material while providing good epoxy bond strength at cryogenic temperatures. It is worth noting that the Masterbond EP29LPSP required elevated temperatures to cure, while the CTD Cryobond 621 and Scotchweld 2216 cured at room temperature.

**Table 1: Cryogenic tensile and shear epoxy testing results.**

Epoxy Brand		Coupon Material	Sample #	Popping	Cryogenic Epoxy Strength Testing (Coupons maintained at approx. 86 K)		Epoxy Brand	Coupon Material	Sample #	Popping	Cryogenic Epoxy Strength Testing (Coupons maintained at approx. 86 K)		
					Tensile (kg)	Shear (kg)					Tensile (kg)	Shear (kg)	
Altheris	C1	AL - 6061	1	*	16.0 X	-	Masterbond	EP29LPSP	AL - 2219 primered	1		✓	✓
			2	*	✓	18.3 X				2		✓	18.3 *
		SST 304	1	*	3.6 X	-			3		✓	✓	
	2		*	1.3 X	-	AL - 6061			1		✓	✓	
	EA-2A	AL - 6061	1		✓				✓	2		✓	✓
			2		✓				✓	SST 304	1		✓
		2		1.3 X	-	2			✓		✓		
CTD	621	AL - 2219 primered	1		✓	✓		EP21TCHT-1	AL - 2219 primered	1		✓	✓
			2		✓	✓				2		6.9 *	-
		AL - 6061	1		✓	✓			SST 304	1	**	1.3 X	-
			2		✓	✓				2		1.3 X	-
GE	Varnish	AL - 2219 primered	1	**	1.3 X	-			EP30-2	AL - 2219 primered	1		6.9 X
			2		11.8 *	-	2					5.3 X	-
Huntsman	EPIBond	AL - 2219 primered	1	*	✓	✓	SUP12AOHT-LO	AL - 2219 primered	1		1.3 X	-	
			2	*	✓	✓			2		1.3 X	-	
HYSOL	EA 9430	AL - 6061	1	*	1.3 X	-	STYCAST	2850FT	AL - 2219 primered	1	*	3.6 X	-
			2	*	1.3 X	-				2		5.3 X	-
Masterbond	AE-10	AL - 6061	1		✓	✓	1266	AL - 2219 primered	1		✓	✓	
			2		11.8 *	-			2		✓	✓	
		SST 304	1		✓	18.3 *			AL - 6061	1		1.3 X	-
			2	*	1.3 X	-	2			16.0 X	-		

✓ Successful Test    X Epoxy failure    ■ Standoff Break  
 \* Popping noises while cooling    \*\* Sound observations not recorded

#### 4. Conclusions

Fifteen epoxies were tested for tensile and shear bond strength at cryogenic temperatures. The purpose of this testing was to determine which epoxy would provide a good bond strength at cryogenic temperatures for polyetherimide (PEI) standoffs holding MLI on standard tank materials. The testing method developed used a slow chill down period for each sample before testing was conducted. Each sample was made by bonding an MLI standoff to a metal surface. A thermocouple was taped to the backside of each sample to allow the temperature to be monitored throughout the testing process. The test was then conducted by hanging weights of increasing mass from the sample as observations were recorded.

The results showed that of the 15 epoxies tested only CTD Cryobond 621, Masterbond EP29LPSP, and Scotchweld passed all the weights in tensile and shear testing for multiple sample materials. It is worth noting that the Masterbond EP29LPSP required elevated temperatures to cure, while the CTD Cryobond 621 and Scotchweld 2216 cured at room temperature. In general, these results provide useful insight to epoxy selection for attaching MLI systems to cryogenic tanks.

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## References

- [1] D. Plachta, J. Feller, et al., "Liquid nitrogen Zero Boiloff Testing", NASA/TP – 2017-219389, 2017.
- [2] D. J. Daniels, P.I Kawano, et al., "An evaluation of Orbital Workshop Passive Thermal Control Surfaces", *40<sup>th</sup> International Conference on Environmental Systems*, 1974.
- [3] A. Ferrero, R. Palestro, et al., "Qualification Campaign on ATV Enhanced Design MLI Blankets", *40th SAE/AIAA/ASME/ASMA/AIChE International Conference on Environmental Systems*, 2010.
- [4] Barbosa, Nicholas. "Cryogenic Material Properties Index." *NIST*. N.p., 21 Sept. 2016. Web. 11 May 2017.
- [5] W. L Johnson, J. G. Valenzuela, et al., "Tank Applied Testing of Load-Bearing Multilayer Insulation (LB-MLI)", *Propulsion and Energy Forum 2014*; 28-30 Jul. 2014; Cleveland, OH; United States.
- [6] Private communications with Dave Frank, Dave Oberg and ClickBond personel.
- [7] ASTM E8/E/M-16a, Standard Test Method for Tension Testing of Metallic Materials, ASTM International, West Conshohocken, PA, 2010.
- [8] ASTM D1002-10, Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading, ASTM International, West Conshohocken, PA, 2010.
- [9] ASTM 3163-01, Standard Test Method for Determining Strength of Adhesivly Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading, ASTM International, West Conshohocken, PA, 2010.
- [10] ASTM D4896-01, Standard Test Method for Useof Adhesive-Bonded Single Lap-Joint Specimen Test Results, ASTM International, West Conshohocken, PA, 2010.