Final Test Report
for
Purchase Order PE005392-W

Through-Thickness Insulation and Copper Tensile/Adhesion Tests

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Prepared for:
Princeton Plasma Physics Laboratory
P.O. Box CN-17
US Rte 1 North at Sayre Drive
Princeton, NJ 08543

Prepared by:
Composite Technology Development, Inc.
2600 Campus Drive, Suite D
Lafayette, CO 80026

Report Signature Page

Prepared by: __________________________
Sam Grandlienard, Engineer

Reviewed by: __________________________
Paul E. Fabian, Sr. Program Manager

Approved by: __________________________
Fred L. Beavers, Sr. Contracts Manager

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Introduction

This report documents the results of the fabrication and mechanical testing performed for Princeton Plasma Physics Laboratory (PPPL) by Composite Technology Development, Inc. (CTD) on Purchase Order PE005392-W. CTD fabricated through-thickness tensile test specimens, composed of two copper (Cu) end pieces bonded to CTD-101K/S-2 Glass composite insulation and tested them at 77 K. A schematic of the Cu/composite test specimen is shown in Figure 1.

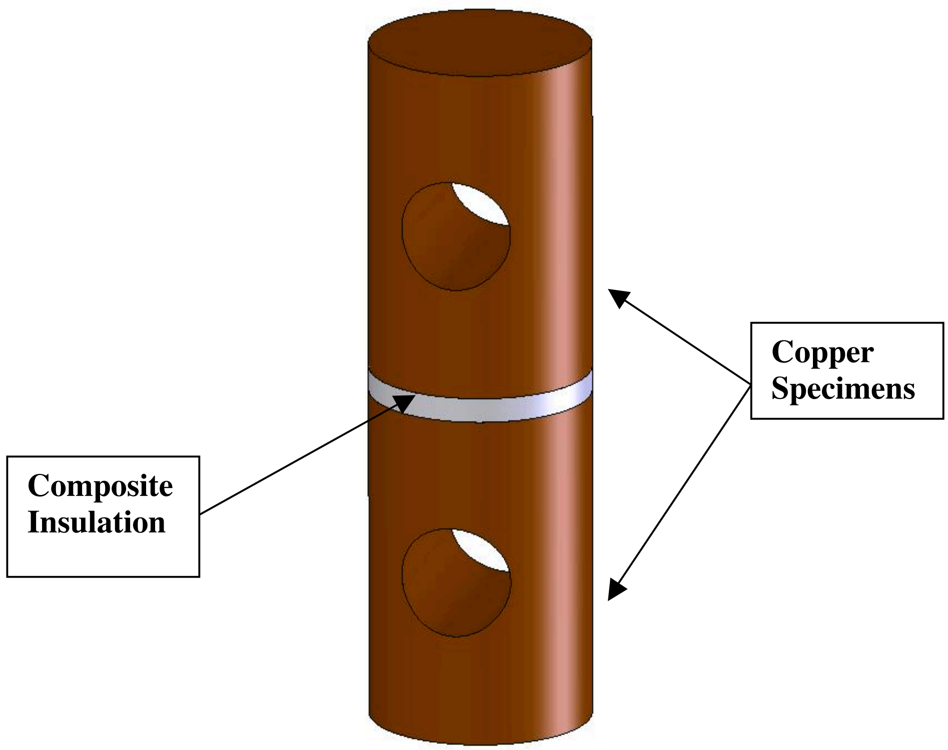

Figure 1. Cu/Composite through-thickness tensile/adhesion test specimen.

Specimen Fabrication

The Cu/composite tensile specimens were fabricated using two, 0.498-in. diameter copper end pieces, approximately 0.750 inches long, that sandwiched a layer of CTD-101K/S-2 Glass fabric composite, nominally 0.050-inch thick. The bonding surfaces of the copper end pieces were prepared according to directions provided by PPPL.

The specimen mold used by CTD can produce 16 specimens in a single molding run. Four different surface preparation methods were evaluated, with four replicates of each method produced in the single processing run.

The surface preparation methods evaluated in this program included:

1) Set #1: Cleaning with MicroClean™ EC2-I cleaner, RDZ-1673 (RD Chemicals, Mountain View, California), followed by ambient air exposure for 7 days.
2) Set #2: Cleaning with the MicroClean™ cleaner.
3) Set #3: Cleaning with the MicroClean™ cleaner, followed by the application of ChemBond™ EC2-I, RDZ-1674 black oxide coating (RD Chemicals, Mountain View, California).
4) Set #4: Grit blasting, followed by cleaning with the MicroClean™ cleaner and application of the ChemBond™ EC2-I black oxide coating.

The MicroClean™ cleaner was applied following the manufacturer's directions by immersion of the copper for 1-2 minutes in the cleaner at 50-70°C, followed by a water rinse. The ChemBond™ EC2-I black oxide was applied following the manufacturer's directions by immersing the copper for 3 minutes at 40°C, followed by a water rinse. Grit blasting was performed using 100 grit Al_2O_3 media applied at 80 psi, followed by a clean, filtered compressed air blast of the surface at 100 psi. The four surface preparation methods evaluated are summarized in Table 1.

Table 1. Surface Preparation Treatments

Set #	Grit Blasting	Microclean (1 week age)	Microclean	Chembond
1		X		
2			X	
3			X	X
4	X		X	X

Following surface preparation, the copper end pieces, along with six layers of dry S-2 Glass fabric, 6781 style weave, were loaded into a closed mold, and the glass fabric impregnated using standard Vacuum Pressure Impregnation (VPI) processing procedures with CTD-101K epoxy resin. Major steps in the VPI process included:

1) The mold was degassed at 60°C for 3.5 hours at 115 mTorr.
2) The resin was degassed at 60°C for 0.5 hours at 117 mTorr.
3) The resin was transferred at 60°C for 45 min.

The cure profile used was as follows:

- 1 hour ramp to 90°C
- 4 hour hold at 90°C
- 1.5 hour ramp to 135°C
- 1.5 hour hold at 135°C
- 2 hour ramp to 25°C

During cure, a positive pressure of 20 psi of nitrogen gas was maintained in the mold to minimize the formation of bubbles or void. Figure 2 shows the mold and specimens following cure. Specimens were removed from the mold by a press, pushing vertically. Figure 3 shows a completed specimen after cleanup of excess resin.

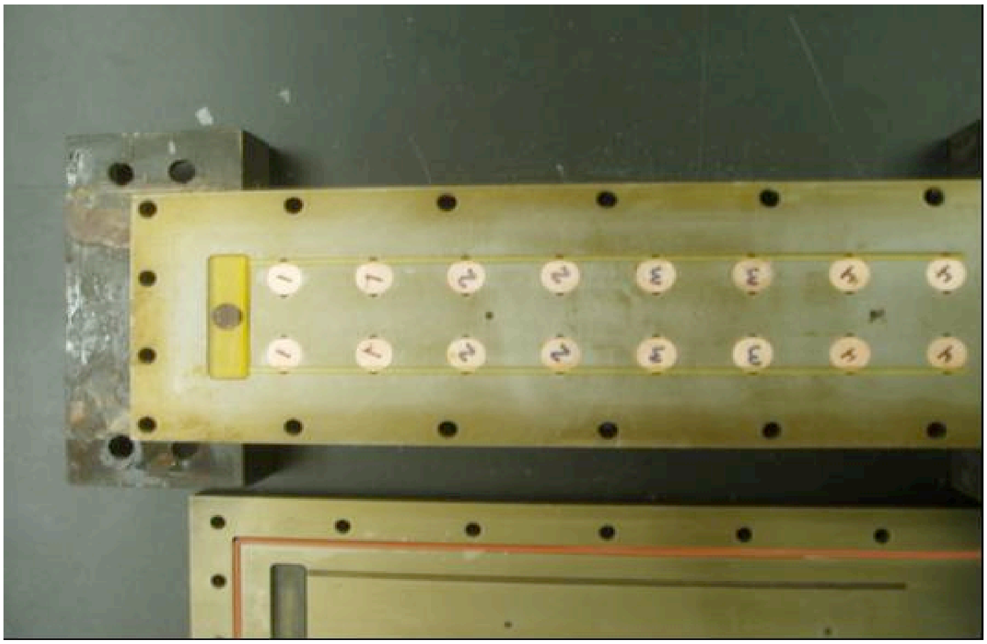

Figure 2. Cu/composite specimens in mold following VPI.

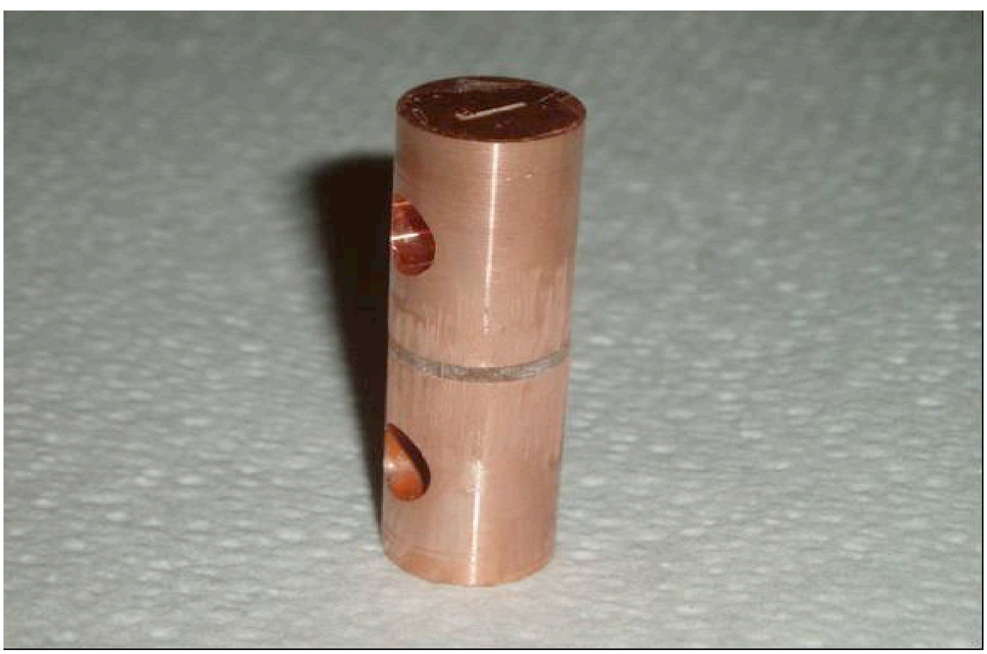

Figure 3: Cu/composite test specimen prepped for testing.

Two of the specimens in Set #1 and one of the specimens in Set #3 had surface adhesion failures while being pressed out of the mold. Further surface adhesion failures became apparent when the excess CTD-101K resin was removed from the outside of each sample. Additional specimens from Sets #1, #2 and #3 experienced surface adhesion failures during the resin removal process. Figure 4 shows some of the failed specimens that were not tested.

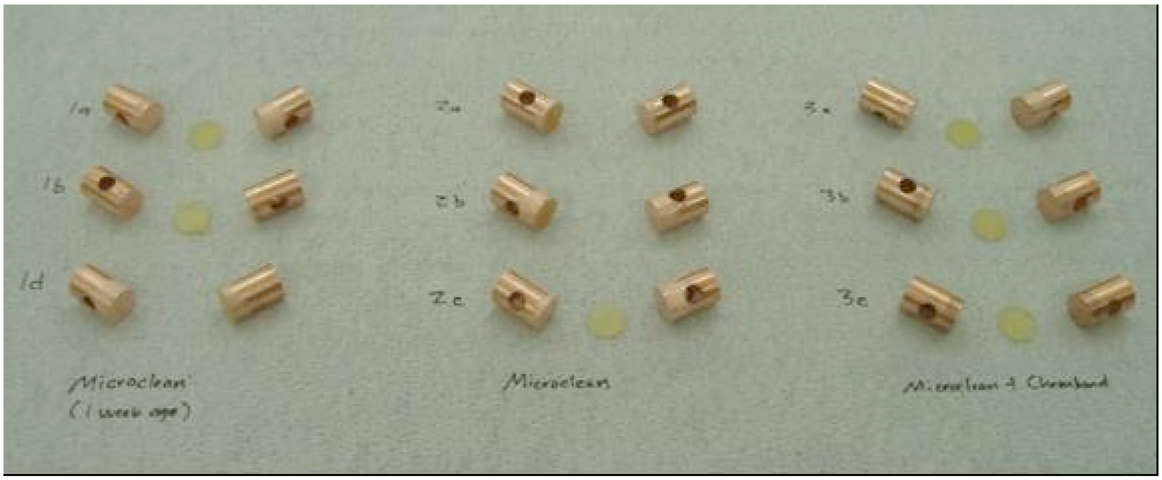

Figure 4. Failed specimens from Sets #1, #2, and #3 after removal from mold.

Adhesive-type failures at the metal surface are shown in Figures 5 and 6. Set #4, which was the only set to use a grit blasted surface, was the only complete set of samples without pre-test failures. Figure 7 shows the difference in surface roughness between a grit blasted and non-grit blasted copper surface. Additionally, only one specimen from Set #2 was available for testing.

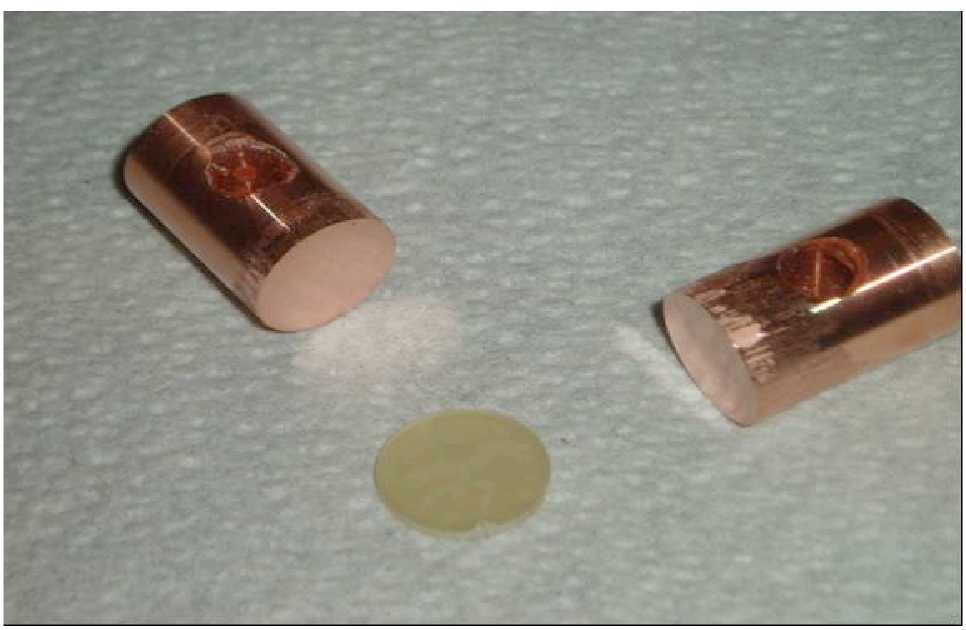

Figure 5. Typical adhesion to metal surface failure.

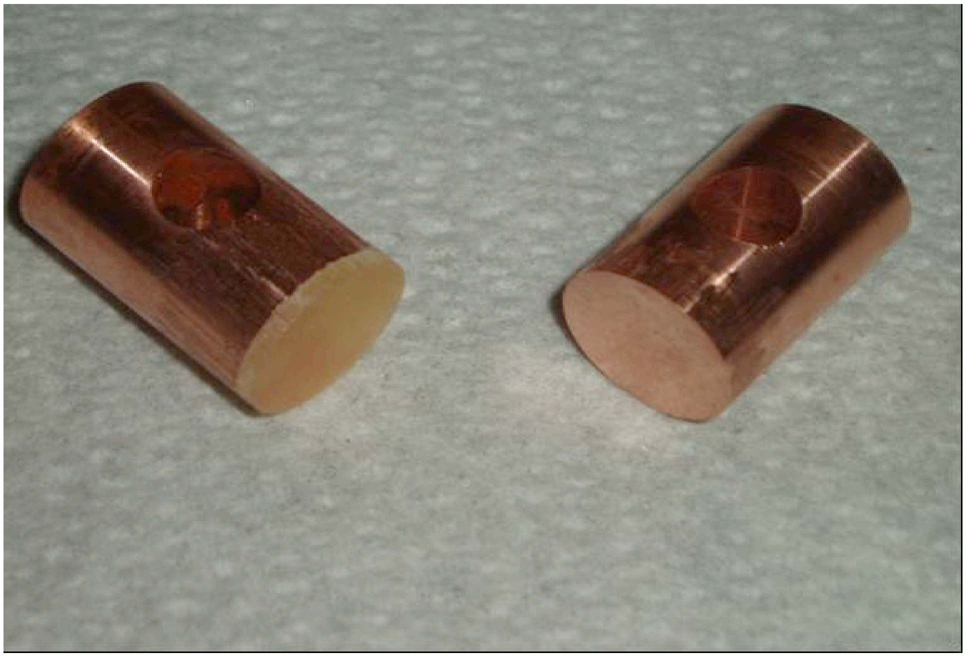

Figure 6. Adhesion to metal surface failure.

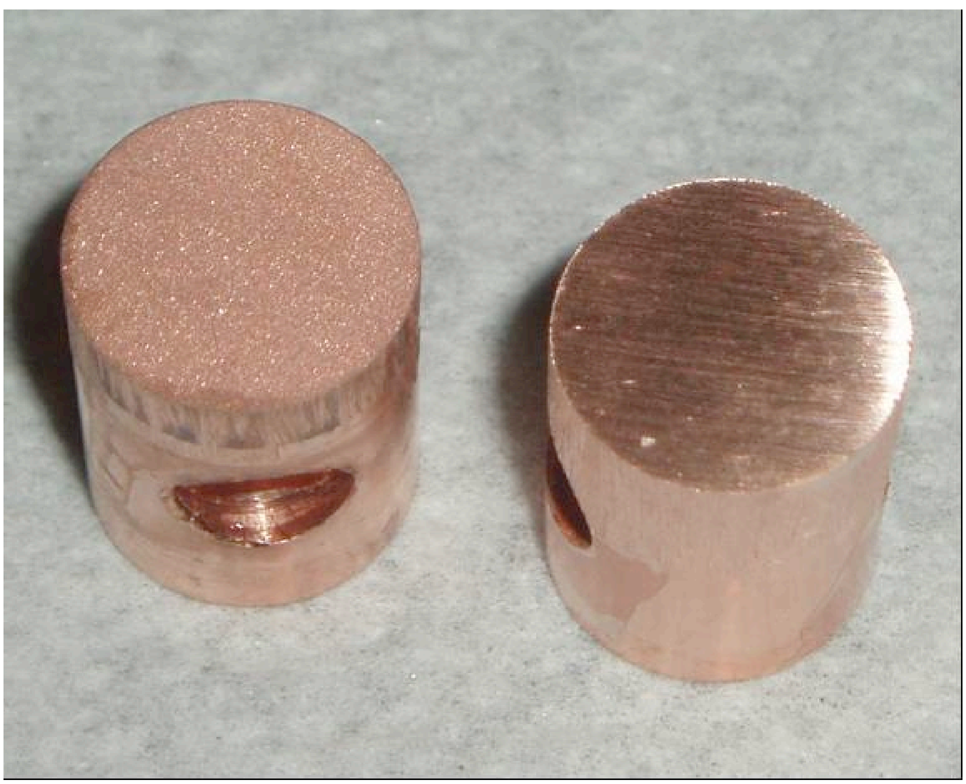

Figure 7. Copper surface comparison. Grit blasted (left), Non-grit blasted (right).

Test Results

All tests were performed on a MTS Systems Servo-Hydraulic test machine with a 100 kip load capacity. The test loads were measured using a 1000-lb. load cell, while the displacement was measured by an LVDT located on the MTS machine hydraulic actuator. Load and displacement were recorded through a data acquisition system designed by CTD. The data sampling frequency was 10 Hz. The original data files recorded during the test and all modified (for data analysis purposes) data files are provided on a CD with this report. Table 2 identifies each piece of equipment used for this test program as well as the date of calibration.

Table 2. Test Equipment

Description	Model Number	Serial Number	Calibration Date	Comments
MTS Systems Corp.				
1000 lb. Load Cell	1210BJS-1K-B	117628A	1/19/2004	±1000 lb. range card
LVDT	LVDT	106826	1/19/2004	±0.5 in range card

Test specimens were gripped using a custom-made clevis grip system utilizing 0.25-in. diameter cross-pins to react the tensile loads. In addition, a lower spherical seat was used to help maintain proper vertical alignment of the specimen during testing. Once loaded into the clevis grips, specimens were slowly submersed into liquid nitrogen (76 K) and held at the test temperature for 5 minutes prior to the start of the test. The test specimen was then loaded in tension, using a crosshead displacement rate of 0.00033 in/s, until failure.

Test results for Sets #2 and #4 are shown in Tables 3 and 4, respectively. As previously mentioned, all specimens from Sets #1 and #3 failed during fabrication processing, and were not able to be tested. All specified dimensions refer to the insulation layer of the copper test specimens. Tensile modulus was determined after calculating the strain in the composite insulation material by using the MTS machine LVDT measured displacement and the composite thickness as the gage length. Machine compliance was not accounted for in these measurements and therefore it is expected that the actual through-thickness modulus should be slightly higher than the values reported here.

Table 3. Set #2 Test Results

Tension Test Results-7090 Copper Adhesion
Set #2: Microclean

TEST CONDITIONS

Matrix System:	**CTD-101K**	*Load Range Card:*	**±1 Kip**
Reinforcement:	**S2-Glass (6781)**	*Stroke Range Card:*	**±0.5 in.**
		Temperature Controller:	**Lakeshore 330**
Specimen Type:	**Tension**	*Temperature Sensor:*	**Si Diode #D47355**
Specimen Reference:	**Set #2**		
Speciment Prep:	**Microclean**		
CTD Program #:	**7090**		
Load Rate:	**0.00033 in/s**		
Test Fixture:	**Tension fixture**	*Test Temperature:*	**76 K**
Test Date:	**11/16/2004**	*Temperature Hold Time:*	**5 minutes**

TEST RESULTS

Specimen #	Height (in)	Diameter (in)	Area (in^2)	Ultimate Load (lbs)	Tensile Strength (ksi)	Modulus (ksi)	Failure Mode
2d	0.055	0.498	0.195	189.1	1.0	15.2	A

A = adhesion to metal failure

	Tensile Strength	
	ksi	MPa
Value	1.0	6.7

	Compression Modulus	
	ksi	MPa
Value	15.2	104.5

Table 4. Set #4 Test Results

Tension Test Results-7090 Copper Adhesion
Set #4: Grit Blast + Microclean + Chembond

TEST CONDITIONS

Matrix System:	**CTD-101K**	*Load Range Card:*	**±1 Kip**
Reinforcement:	**S2-Glass (6781)**	*Stroke Range Card:*	**±0.5 in.**
		Temperature Controller:	**Lakeshore 330**
Specimen Type:	**Tension**	*Temperature Sensor:*	**Si Diode #D47355**
Specimen Reference:	**Set #4**		
Speciment Prep:	**Grit Blast + Microclean + Chembond**		
CTD Program #:	**7090**		
Load Rate:	**0.00033 in/s**		
Test Fixture:	**Tension fixture**	*Test Temperature:*	**76 K**
Test Date:	**11/16/2004**	*Temperature Hold Time:*	**5 minutes**

TEST RESULTS

Specimen #	Height (in)	Diameter (in)	Area (in^2)	Ultimate Load (lbs)	Tensile Strength (ksi)	Modulus (ksi)	Failure Mode
4a	0.055	0.498	0.195	262.5	1.3	21.4	A
4b	0.054	0.498	0.195	200.4	1.0	17.9	A
4c	0.055	0.498	0.195	188.0	1.0	17.5	A
4d	0.053	0.498	0.195	340.7	1.7	21.7	A

A = adhesion to metal failure

	Tensile Strength	
	ksi	MPa
Average	1.3	8.8
Std. Dev.	0.4	2.5
CV:	0.28	0.28

	Compression Modulus	
	ksi	MPa
Average	19.6	135.2
Std. Dev.	2.2	15.5
CV:	0.11	0.11