

# SUBSCALE COMPOSITE LIQUID OXYGEN TANK TESTING

Neil A. Graf  
Kevin Davis  
Michael McBain

Lockheed Martin Space Systems Company – Michoud Operations  
New Orleans, LA 70129

## ABSTRACT

Lockheed Martin Space Systems Company recently completed a two-year fabrication and test program on subscale composite liquid oxygen (LO<sub>2</sub>) tanks. The goals of this program included the development of fabrication and inspection techniques, cryogenic acceptance testing of composite articles, and demonstrating oxygen compatibility under launch vibration loads. Two subscale diameter test bottles were fabricated using a proprietary Lockheed Martin material, known as LM21C03. The bottles were then inspected using an array of NDE techniques and then put through a cryogenic acceptance test program at Lockheed Martin. A NASA/Lockheed Martin test team then subjected a composite bottle to testing at an X-33 vibration profile for 15 minutes at use pressure. The tests were run at various LO<sub>2</sub> fill levels, with and without intentionally added debris. All tests were successful in that the composite bottle showed no signs of ignition or combustion as a result of the vibration testing. This test program is an important bridge between coupon-level and subcomponent LO<sub>2</sub> compatibility tests and full-scale composite LO<sub>2</sub> tank use.

**KEY WORDS:** Liquid Oxygen, Composites, Bottle Testing

## 1. INTRODUCTION

**1.1 Purpose:** The purpose of this report is to present information regarding the fabrication, inspection, and liquid oxygen testing of 46 cm (18 inch) diameter composite test bottles.

**1.2 Background:** Lockheed Martin Space Systems Company, Michoud Operations (hereafter known as LM) has been working with liquid oxygen compatibility experts at NASA Marshall Space Flight Center (MSFC) and NASA Johnson Space Center White Sands Test Facility (WSTF) since 1995 to evaluate liquid oxygen compatibility of composite materials. After successful testing on the coupon level, the LO<sub>2</sub> community wanted to demonstrate compatibility of composites on a subscale tank. This would then form a bridge between coupon-scale testing and use of composites in full-scale launch vehicles.

**1.3 Scope:** Two 46-cm (18-inch) diameter composite bottles were fabricated at NASA MSFC's Productivity Enhancement Center (PEC). These bottles were filament wound using preimpregnated

slit tape form of LM21C03, a proprietary Lockheed Martin material system. The bottles were then submitted to rigorous NDE and acceptance tests at LM. One bottle was then submitted to NASA MSFC for LO<sub>2</sub> vibration testing. The vibration spectrum was based on the X-33 flight profile, 15 launch cycles. It was tested at pressure and at three fill levels. At one fill level, debris was intentionally added to the tank to increase the risk of ignition and simulate a possible real-world scenario.

## 2. EXPERIMENTAL

**2.1 Objectives:** There were several objectives for this subscale test program.

- (1) Demonstrate LO<sub>2</sub> Compatibility of composite materials on a large scale, since many ignition hazards are not easily demonstrated on a large scale.
- (2) Demonstrate that a composite LO<sub>2</sub> tank could structurally withstand the vibration profile associated with an X-33 or similar vehicle multiple mission life.
- (3) Gain experience manufacturing large-scale parts with the proprietary LM21C03 material system, as well as develop a knowledge database on the system, such as behavior after multiple cryogenic cycling.
- (4) Demonstrate feasibility of various non-destructive evaluation (NDE) methods on cryogenic tanks.

**2.2 General Approach:** The general approach for this program outlined below.

- (1) Fabricate 46 cm (18 inch) bottles
- (2) Inspect bottle using various NDE techniques
- (3) Proof test bottle
- (4) Cryogenically condition bottle
- (5) Perform vibration check-outs
- (6) Perform vibration tests
- (7) Final inspection of bottle using various NDE techniques

**2.3 Bottle Fabrication:** The LM21C03 material was chosen for the subscale bottles because this material system was the leading LO<sub>2</sub> candidate after extensive LO<sub>2</sub> compatibility coupon testing in 1997. Invar was chosen as the boss material due to its inherent LO<sub>2</sub> Compatibility, excellent thermal properties, and past history of use in LM composite tanks. Similarly, Teflon® seals were utilized as the seals between the boss and the test caps for this tank due to their past history use and inherent compatibility.

The bottle was designed to approximately 46 cm (18 in) diameter by 61 cm (2 feet) long for two reasons. First, this is a common filament wound test bottle diameter in the aerospace industry. Second, a larger tank would not be feasible to use with NASA MSFC's vibration equipment due to size and weight considerations. However, the standard bottle design was modified slightly for the conditions of this test. The standard design for filament wound bottles is to wind around the boss, leaving the boss exposed to the inside of the tank. LM engineers felt that for cryogenic testing, the boss should be wedged, or knife-edged, into the composites. However, this is not feasible with the filament winding process. As a result, 3 plies of LM21C03 fabric was hand-laid in a 90°/30°/-30° pattern in the entire dome regions of the bottle. The bosses were then placed on the uncured fabric prepreg, and the filament winding was then started. Figure 1 shows this bottle configuration.

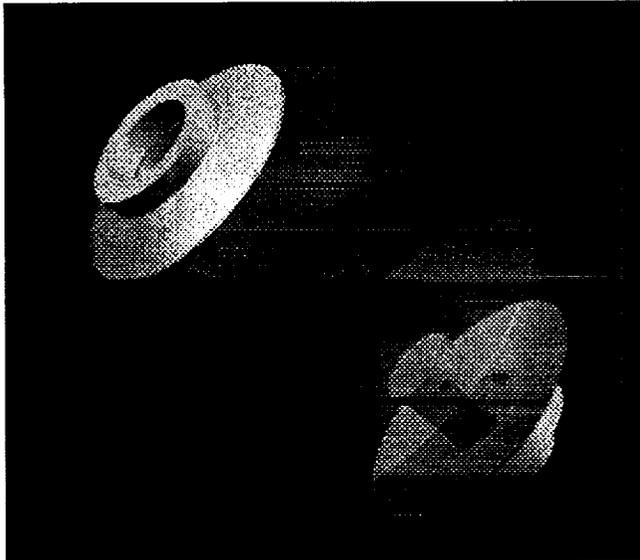


Figure 1: CATIA model of the subscale bottle design

Silicone-covered sand was chosen for the mandrel material. Sand was chosen due to its availability, low cost, and ease of removal. A NASA-owned standard mold was used to create the sand mandrel in two pieces, which were then bonded together using a sand/epoxy mixture. Mosites silicone was selected as a barrier over the sand due to prior history of use. In addition, the silicone expands slightly during cure, helping to compact the plies of the laminate. An extra layer of silicone was added in the dome area to help support the boss. Standard steel shafts were used, although the drive dogs had to be modified slightly.

Two bottles, one for vibration testing and one for a back-up or for additional future testing, were manufactured at NASA MSFC's Productivity Enhancement Center (PEC). Lockheed Martin personnel fabricated the bottles with support from NASA as required. Figure 2 is a photograph of a LM technician operating the PEC winding machine. The bottles were cured in a large autoclave in the PEC. The second of the two bottles manufactured, designated GTDP-001B, was selected to be the test bottle. The sand and silicone were removed without difficulty.

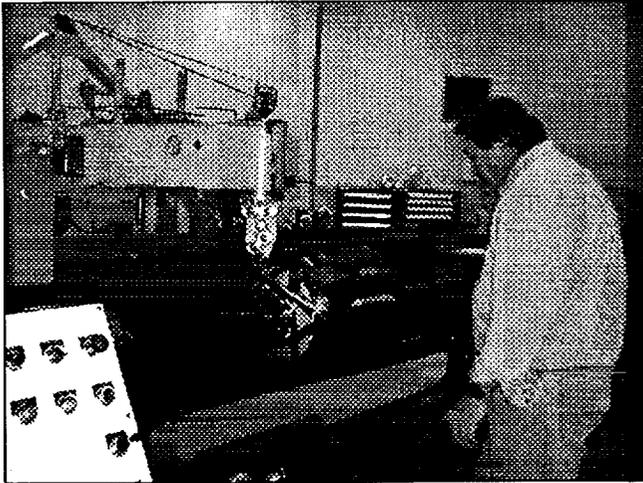


Figure 2: Winding a subscale bottle

**2.4 Bottle Inspection and Initial Acceptance Testing:** The initial inspection was performed by NASA personnel at MSFC using a fiberscope (a.k.a. boroscope). Figure 3 shows a technician inspecting the bottle.



Figure 3: Inspection of bottle with boroscope

The boroscope is a small video camera attached to a cable, which is then inserted into the bottle. Still photographs can then be made of the interior of the bottle. The initial inspection showed no gross defects. However, the boroscope only visually inspects small areas of the surface; no true determination of the quality of the laminate through the thickness can be made. The bottles were then

shipped to LM for nondestructive evaluation (NDE). Two techniques were selected for initial evaluation: thermography and shearography.

Thermography is a technique where a heat source is applied to the sample. The sample is then viewed through an infrared camera. Voids or other defects show up as light or dark areas. Figure 4 shows a thermographic scan.

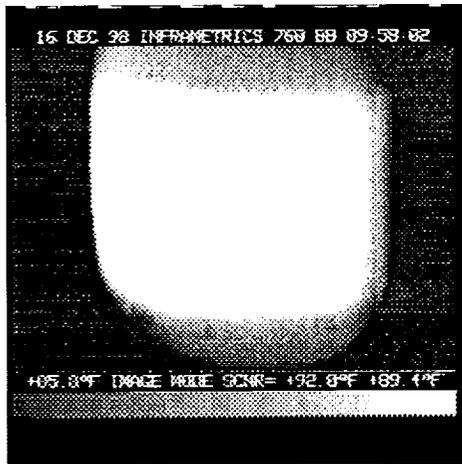


Figure 4: Thermograph of a subscale bottle

Shearography is another nondestructive technique where laser light is applied and diffracted slightly. It is then viewed through a special filter. A defect would show up as a “bulls-eye” pattern. Figure 5 is the shearography photos of the bottle. The tight fringe patterns indicate high stress level, but this is typical for filament wound tanks.

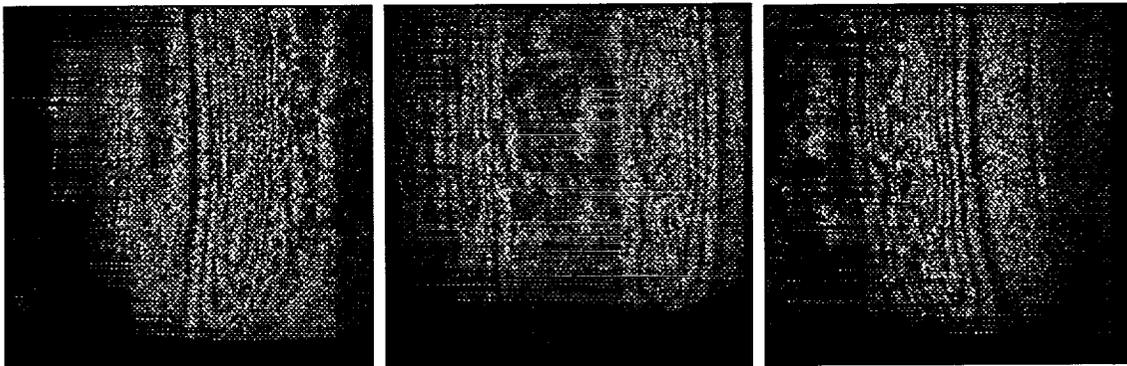


Figure 5: Shearography of the subscale test bottle

These inspections showed no unacceptable defects or anomalies. LM engineers then deemed the bottles acceptable for use.

The bottles were then put through a series of initial inspection tests. First, the bottle was subjected to an ambient temperature proof test. The bottles were partially instrumented and filled with gaseous

helium to 1.5 times the test pressure (67 psig/0.46MPa x 1.5 = 100 psig/0.69MPa). The pressure was stepped up at 20 psig/0.14MPa increments, with soap bubble checks at each increment. No leaks were detected at any increment for either bottle. Also, the strains in the tanks approximated the strains predicted by analysis. (Strain gage #4 was wired backwards; the true positive value approximates the model.) Figure 6 shows plots of the strains as well as a comparison to predicted values. Each bottle was then subjected to a 24-hour helium decay test at test pressure. Neither bottle showed any indication of pressure loss.

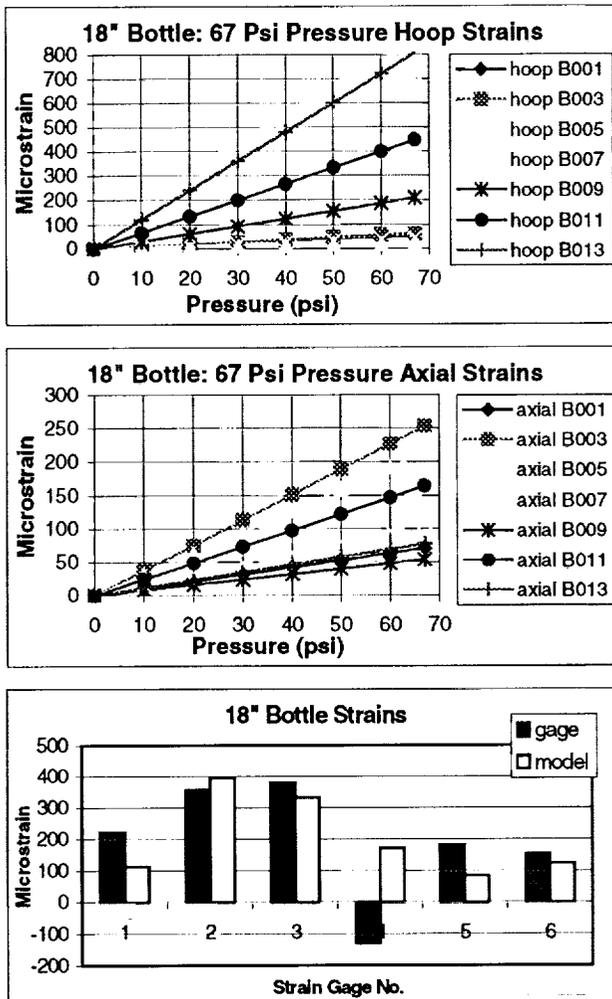


Figure 6: Test bottle strains and comparison with predicted

The bottles were then instrumented completely with strain gages, thermocouples, and an accelerometer and covered with SS1171 foam insulation, which is used on the Space Shuttle External Tank. Table 1 shows the types of acceptance tests and the order in which they were performed. The purpose of this testing was to condition the bottle for testing, as well as to verify that cryogenic cycling under load by itself would not cause the bottle to leak. No leaks were detected, nor was there any loss of pressure in

the decay tests. There was no build-up of ice on the foam, which indicated that the foam applied was sufficient. There was also no visible damage to the bottle. Figure 7 is a photograph of the bottle during a cryogenic cycle.

Table 1: Acceptance Testing Plan

Step	Test	Temperature	Pressure	Medium
1	24-Hour Decay	Ambient	0.46Mpa (67 psig)	GHe
2-6	Cryogenic Cycle	-195°C (-320°F)	0.46Mpa (67 psig)	
7	24-Hour Decay	Ambient	0.46Mpa (67 psig)	GHe

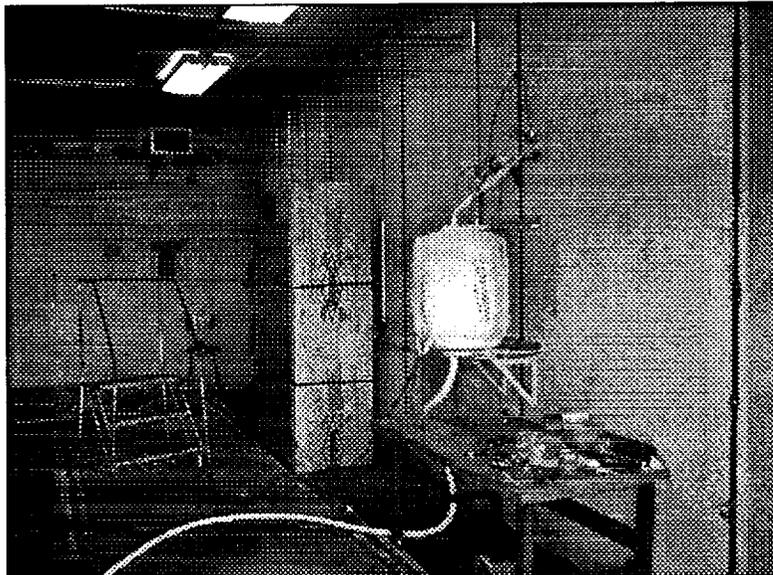


Figure 7: Bottle undergoing cryogenic conditioning

**2.5 Initial Vibration Testing:** The random vibration environments and duration were developed for the X-33 flight vehicle. At that time, vibration profiles for VentureStar or X-34 had not yet been established. Therefore, the X-33 environments were deemed acceptable for use despite differences in LO<sub>2</sub> tank material (metal vs. composite) and duration (15 one minute flights for X-33, 100 flights of unknown duration for VentureStar). Figure 8 graphically shows the vibration profile.

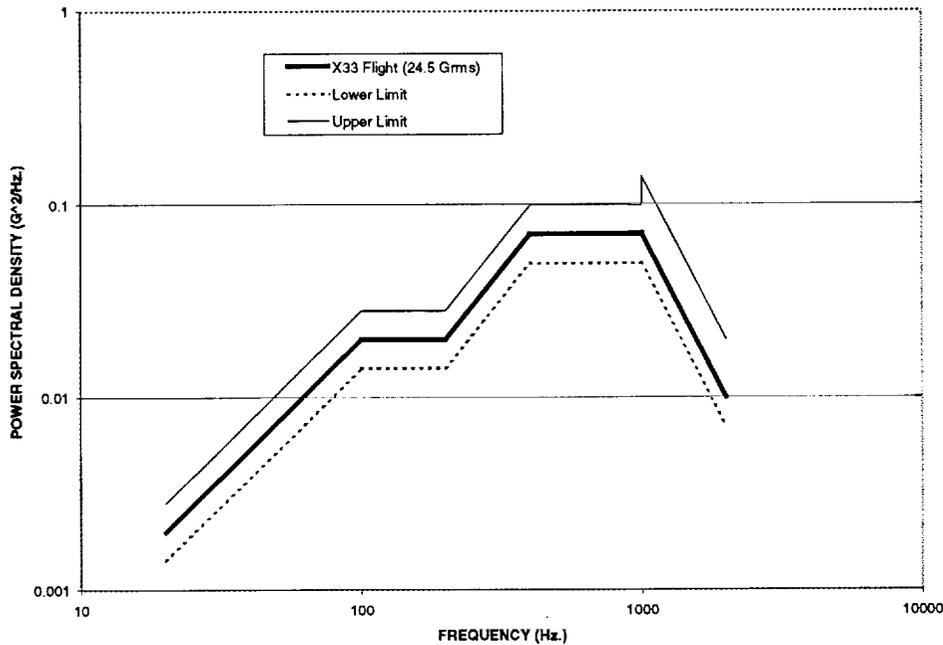


Figure 8: Vibration Profile

The other test criteria were also established by the LM-NASA team for various reasons. The test pressure (67 psi or 0.46 MPa) was based on the projected pressure in the composite VentureStar vehicle. Three fill levels were established: 90% full, 50% full, and 30% full. The 90% fill would be worst case for vibration loads, but it was felt that 30% fill would be worst case for liquid oxygen compatibility purposes. It was also decided that it would be feasible that an internal composite part, such as a slosh baffle, could come unbolted or break off. Therefore, it was decided that one run would be performed with a bolt and with a composite piece in the tank as simulated debris. It was also decided that a fixture check-out run with LN<sub>2</sub> would be prudent before the LO<sub>2</sub> runs were to begin. Table 2 outlines the order of tests.

Table 2: Planned Test Series

Run	Fill Level	Test Fluid	Debris?
Checkout	90%	LN <sub>2</sub>	No
1	90%	LO <sub>2</sub>	No
2	50%	LO <sub>2</sub>	No
3	30%	LO <sub>2</sub>	No
4	30%	LO <sub>2</sub>	Yes

The vibration test area was located at NASA MSFC. The shaker table was under a protective tent and was a minimum distance of 12.2-m (40 feet) from the nearest structure. This was to prevent any damage to buildings or equipment.

The initial fixture checkout was performed with LN<sub>2</sub>. During the fill, quite a bit of LN<sub>2</sub> was lost to boiloff due to the large heat sink present in the shaker table. The entire LN<sub>2</sub> dewar was emptied into the test bottle, but the thermocouples used to indicate fill level showed that the bottle was only approximately 50% full rather than the projected 90% full. It was decided to proceed with the testing despite the low fill level, because it was assumed that since the LO<sub>2</sub> dewars were higher pressure, it would be possible to obtain 90% fill during actual LO<sub>2</sub> testing. However, when the fill system was shut off and the bottle was pressurized to 0.46 MPa (67 psi), the fill level appeared to drop to about 30%. When the vibration run started, the accelerometer that was attached to the bottle underneath the foam came off of the bottle and stopped functioning. However, the accelerometers on the fixtures and shaker tables continued to function normally. Other than the accelerometer loosening, there appeared to be no anomalies. The bottle showed no signs of damage, and the bolts indicated no loss of preload, which would indicate a loose seal and possible leakage.

The LM-NASA team decided that despite the successful initial LN<sub>2</sub> run, a second unplanned fixture evaluation was needed. Assuming the LO<sub>2</sub> would reach 90% during test, due to the higher pressure oxygen dewars, the fixture was not adequately checked out from the standpoint of fluid weight. A second fixture evaluation was then run at ambient pressure. It was filled to 100% with deionized water, which has the same approximate mass as a 90% filled bottle containing LO<sub>2</sub>. A complete vibration run was performed. However, a post-test inspection revealed significant loss of preload on the bolts. The bolts were removed for inspection, and it was found that the bolts were bent significantly. The bottle bosses and fixture also were slightly damaged. However, there was no evidence of fluid leakage. It was determined that there was significant movement in the bottle relative to the fixture, which caused the bolts to bend. This movement was caused due to a non-flat bottle endcap.

The test bottle was then sent back to LM for additional inspection. A small delamination, approximately 8 cm by 5 cm, was found in the center of the barrel section of the bottle using hand-held ultrasonic inspection. See Figure 9. Figure 10 shows a phased-array ultrasonic inspection photograph showing the delamination. Due to the location of the delamination next to the disbonded accelerometer, and lack of indication of delaminations in earlier NDE, it was determined that the delamination was likely caused by the accelerometer rattling between the foam and bottle. Based on these results, testing was shut down in order to develop and implement a recovery plan.

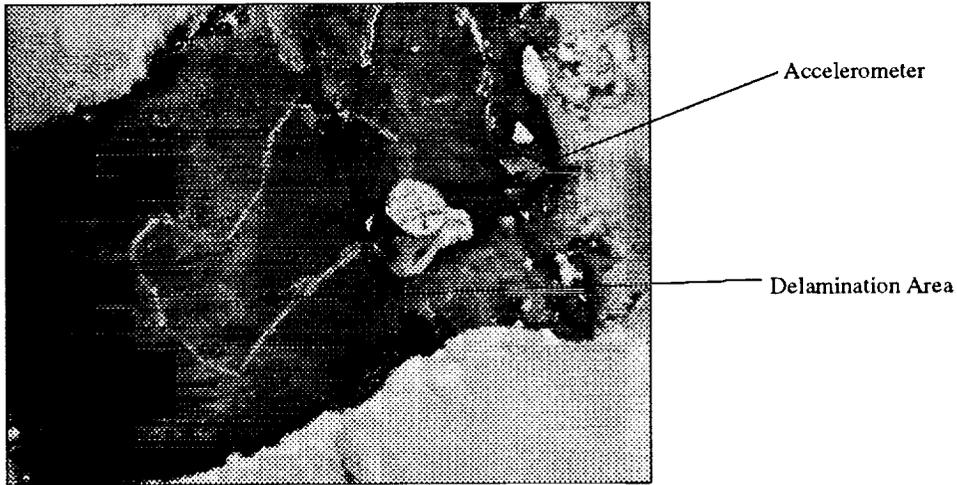


Figure 9: Photo of the delamination area, marked in yellow--note proximity to the accelerometer (metal cylinder), which became disbonded from the bottle during the first fixture checkout

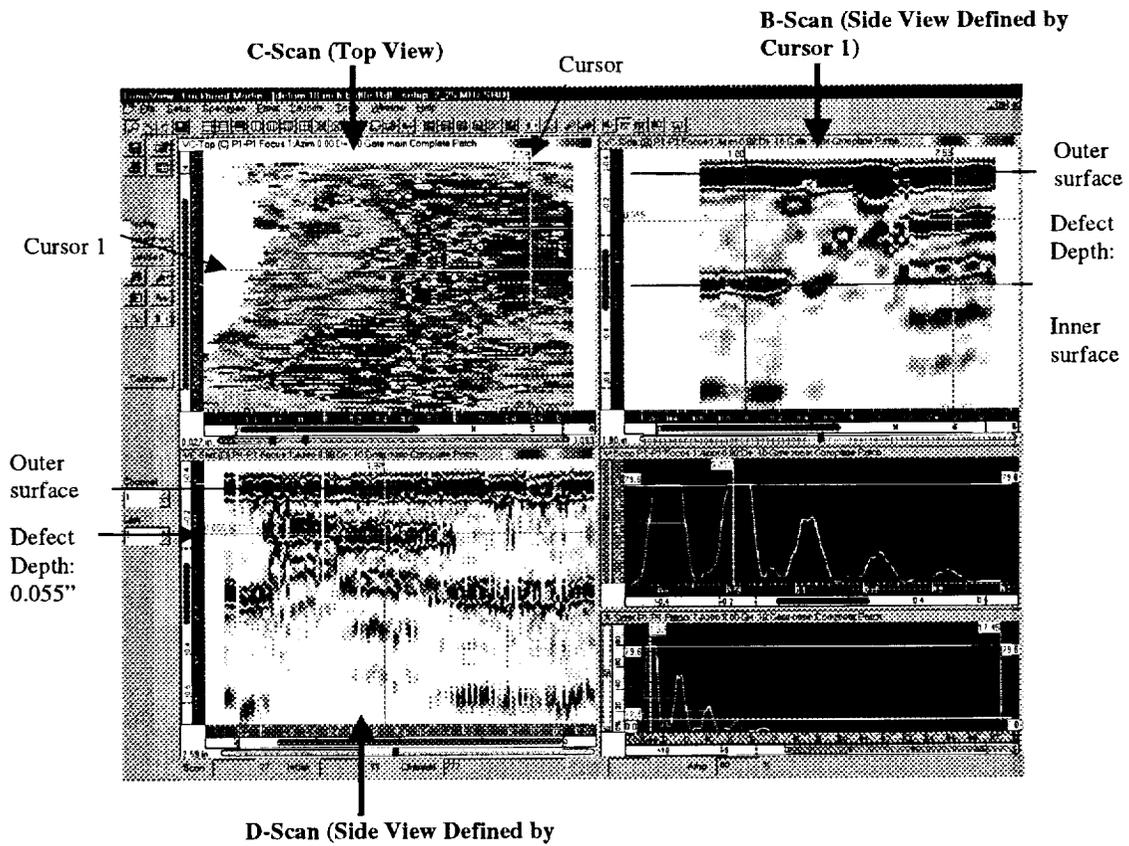


Figure 10: Phased array NDE of delamination

**2.6 Recovery Plan and LO<sub>2</sub> Vibration Runs:** The recovery plan was developed by the LM-NASA team. First, as stated above, a failure analysis indicated that the bolt bending was likely caused by non-flat endcaps. The first part of the recovery plan was to correct this issue. The bottle endcaps were re-machined flat. The bottle and test fixtures were also re-machined to remove the damaged portions. It was also decided that shear pins would be added on each side, between the fixture and the endplate and between the endplate and the bottle. This would eliminate any movement, and prevent bolt bending. Lockwire was also deemed necessary, under the assumption that the wire would help keep the bolts in place and eliminate the possibility of preload loss.

The second part of the recovery plan was to eliminate the need to perform a water test. It was decided that a second LN<sub>2</sub> run would be performed, but changes would be made to ensure higher fill levels. First, LN<sub>2</sub> lines were added to each fixture to cut off the heat sink between the bottle and the shaker table. Second, all fixturing and fill lines would be properly insulated, eliminating the heat loss to the atmosphere. Additional thermocouples were installed on the bottle to get more accurate fill measurements. Finally, new valves were installed on the LN<sub>2</sub> dewars that would allow higher pressure LN<sub>2</sub> flow into the bottle.

The third step of the recovery plan was to verify the test bottle was still acceptable for test despite the delamination. A stress analysis showed that despite the delamination, the bottle still had more than adequate safety factors. See Table 3.

A foam plug in the insulation allowed inspection of the delamination between each vibration run.

Table 3: Factors of Safety

COMPONENT	MATERIAL	FAILURE MODE	CONDITION	REQ F.S.	MIN F.S.
<b>Barrel</b>					
-Membrane Panel	16 Ply Tape	Hoop Fiber Strain	Press at Cryo	1.00 Lmt	7.49
-Delaminated Area	16 Ply Tape	Buckling	Fill and Chill	1.50 Lmt	1.85
<b>Dome</b>					
-Membrane	6 Ply Fab & 8 Ply Tape	Axial Fiber Strain	Press at Cryo	1.00 Lmt	54.51
<b>Cover Plates</b>					
-Plate	1/2" A286 Steel	Bending	Press at Cryo	1.10 Yld	11.40
-Shear Pins	3/8" dia A286 Steel	Shear & Bending	Press at Cryo	1.25 Ult	2.81
-Bolts	1/4" dia A286 Steel	Seperation (Leak)	Press at Cryo	1.10 Yld	8.97
<b>Boss</b>					
-Shear Pins	3/8" dia A286 Steel	Bearing	Press at Cryo	1.10 Yld	1.92
-Blade	Invar	Bending	Press at Cryo	1.10 Yld	3.13

The bottle was also run through re-acceptance testing at LM. This testing consisted of an ambient pressure proof test to 100 psi, a 24 hour helium decay test at test pressure, one cryogenic cycle, a second ambient pressure proof, and a second 24 hour helium decay check. This testing showed that the bottle was structurally sound and leak-free despite the presence of the delamination.

Vibration tests were then resumed. After bottle installation in the vibration fixture, the whole assembly was sprayed with SS1171 foam, and the LN<sub>2</sub> fixture chill-down lines and bottle LO<sub>2</sub> fill and drain lines attached. See Figure 12 for test configuration

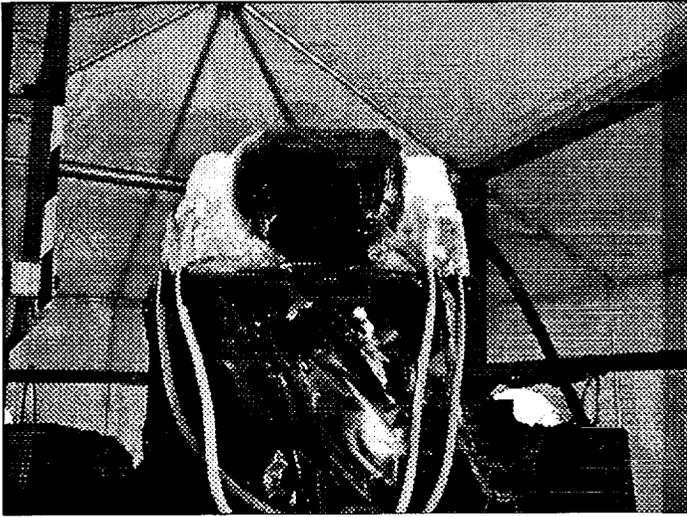


Figure 12: Bottle, wrapped in protective plastic, atop shaker table

The LN<sub>2</sub> fixture evaluation was performed without incident. The delamination area was inspected via hand-held UT, and no change in the delamination area was observed. A second unscheduled fill-only evaluation was performed to evaluate alternate fill level sensing techniques, although the original thermocouples were used for the LO<sub>2</sub> tests.

The 3 LO<sub>2</sub> runs at the 90%, 50%, and 30% fill levels were also performed without incident. There was no sign of ignition or burning, either visibly or via a pressure rise in the tank, during any of the runs. Between each run the delamination area was inspected, and no change was observed.

The bottle and fixture assembly was then partially disassembled to insert the debris. During disassembly, no significant preload loss was seen on any of the bolts or any significant bolt or bottle damage was observed. No signs of ignition (charring, etc.) were seen in the bottle interior. The debris was inserted into the bottle and the bottle and fixture were reassembled.

The 30% vibration with debris test was then performed. A 2.54 cm x 5.08 cm (1" x 2") composite piece and one stainless steel bolt were chosen to be the debris for the tests. Again, there was no sign of ignition or burning. The delamination area was again inspected, and no change was seen. The test program at MSFC was then determined to be complete.

The bottle was then sent back to LM for final NDE inspection. Phased array was performed on the delamination area, and no change was seen. The entire bottle was then again subjected to shearography, as well as a full ultrasonic inspection. (The thermal camera equipment was unavailable to repeat the thermography inspection). Other than the accelerometer delamination, no damage was seen to the bottle in either of the NDE techniques.

### 3. CONCLUSIONS

The following conclusions were drawn from analysis of test data.

- (1) The LM21C03 subscale composite tank showed the ability to withstand simulated ignition hazards (vibration, vibration with debris) to a tank without igniting or burning, thereby increasing confidence in composite LO<sub>2</sub> tanks for RLV, X-34, and other reusable launch vehicles.
- (2) The program demonstrated successful fabrication techniques with the LM21C03 material system.
- (3) The test article successfully demonstrated containment ability under repeated cryogenic and pressure cycles, which is critical for meeting multiple mission life cycle requirements for RLVs.
- (4) The test article withstood repeated vibration runs and impacts from debris with no signs of structural damage. The minor damage caused by instrumentation did not grow after repeated vibration and thermal cycles and was not a factor for containment.
- (5) The test article demonstrated the feasibility of an array of NDE techniques on composite cryogenic tanks.