A Joint MSFC/STTF Test Activity

Cycom 977-2 Composite Material Impact Test Results

Marshall Space Flight Center
Materials Combustion Research Center

Dr. Carl Engel - Qualis Corporation
Stephen Herald - ICRC
Casey Watkins - Qualis Corporation

Material Supplied by
Tod Palm & Jim Bohlen of
Northrop Grumman
Outline

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Four Phase Testing Program
Description

Phase I: Ambient (13A) tests of Cycom 977-2 impact characteristics by the Bruceton and statistical method at MSFC & WSTF

Phase II: Repeat (13A) test of tested Cycom from Phase I at MSFC to expanded testing statistical database

Phase III: Conduct High-Pressure tests (13B) in LOX and GOX at MSFC and WSTF to determine Cycom reaction characteristics and batch effects

Phase IV: Conduct expanded Ambient (13A) LOX test at MSFC and High-Pressure (13B) testing to determine pressure effects in LOX. Expand 13B GOX database.
Phase I Objectives

Phase I: Ambient (13A) tests of Cycom 977-2 impact characteristics by the Bruceton and statistical method at MSFC & WSTF

- Establish Cycom 977-2 reaction characteristics using Bruceton and statistical testing methods
- Using the forgoing data, provide a data set for comparison of facility results
## Phase I Test Matrix

### Ambient 13A LOX Ambient Pressure Multiple Impact

Note: MSFC Nominal Pressure = 14.7 psia  
WSTF Nominal Pressure = 12.4 psia  
Average Pre test sample Wt. = 0.774 g  
Average sample thickness = 0.0895 in

<table>
<thead>
<tr>
<th>Energy Level ft-lb</th>
<th>MSFC samples</th>
<th>1st impact reactions</th>
<th>all Impact reactions</th>
<th>Wt Change w/Reaction</th>
<th>Wt Change w/o Reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>27.2</td>
<td>40</td>
<td>6</td>
<td>18</td>
<td>-0.70%</td>
<td>0.43%</td>
</tr>
<tr>
<td>31.2</td>
<td>30</td>
<td>4</td>
<td>17</td>
<td>-0.37%</td>
<td>-0.10%</td>
</tr>
<tr>
<td>35.8</td>
<td>30</td>
<td>6</td>
<td>15</td>
<td>-0.38%</td>
<td>0.30%</td>
</tr>
<tr>
<td>40.8</td>
<td>30</td>
<td>13</td>
<td>28</td>
<td>-0.55%</td>
<td>-0.19%</td>
</tr>
<tr>
<td>54.5</td>
<td>60</td>
<td>51</td>
<td>59</td>
<td>not available</td>
<td>not available</td>
</tr>
<tr>
<td>Subtotals=</td>
<td>190</td>
<td>80</td>
<td>137</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Energy Level ft-lb</th>
<th>WSTF samples</th>
<th>1st impact reactions</th>
<th>all Impact reactions</th>
<th>Wt Change w/Reaction</th>
<th>Wt Change w/o Reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>31.2</td>
<td>30</td>
<td>10</td>
<td>23</td>
<td>-0.31%</td>
<td>-0.46%</td>
</tr>
<tr>
<td>35.8</td>
<td>30</td>
<td>22</td>
<td>29</td>
<td>0.12%</td>
<td>-0.13%</td>
</tr>
<tr>
<td>40.8</td>
<td>30</td>
<td>25</td>
<td>30</td>
<td>0.30%</td>
<td>-</td>
</tr>
<tr>
<td>Subtotals=</td>
<td>90</td>
<td>57</td>
<td>82</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Single Impact

<table>
<thead>
<tr>
<th>Method</th>
<th>Samples</th>
<th>Energy Level</th>
<th>Wt Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSFC</td>
<td>34</td>
<td>50% energy</td>
<td>52.08 ft-lb</td>
</tr>
<tr>
<td>WSTF</td>
<td>34</td>
<td>50% energy</td>
<td>46.65 ft-lb</td>
</tr>
</tbody>
</table>

### Multiple Impact

<table>
<thead>
<tr>
<th>Method</th>
<th>Samples</th>
<th>Energy Level</th>
<th>Wt Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSFC</td>
<td>34</td>
<td>50% energy</td>
<td>24.18 ft-lb</td>
</tr>
</tbody>
</table>
Post Test Samples

62.9 FT LBS/14.7 PSIA
BRUCE TON METHOD POST-TEST SAMPLE

Many post-test samples were found intact

35.9 ft-lb
Weight Change Data

Phase I
Ambient 13 A LOX, Ambient Pressure
Multiple Impact

Data taken before new weighing procedure

- MSFC Wt% w/ reaction
- MSFC Wt% w/o reaction
- WSTF Wt% w/ reactions
- WSTF Wt% w/o reactions

Weight loss for both with and without reactions was found to be small.
Mass Loss vs. Test Sequence
MSFC Data

Phase I 13 A Ambient LOX, Ambient Pressure
Multiple Impact

Weight loss appears to be random and within measurement precision

Data prior to implementing new weight determination procedure.

- ▲ Set 1 40.8 ft-lb
- □ Set 2 35.8 ft-lb
- △ Set 3 31.2 ft-lb
- ● Set 4 27.2 ft-lb
First Strike Reaction Frequency

Ambient Impact Test 13A
Carbon Fiber/Epoxy Resin (0.0895 in. thick)

- MSFC Data
- WSTF Data

Phase I

Energy (ft-lb)
Phase I Conclusions

- MSFC and WSTF reaction frequency data have been compared, revealing a shift between facilities.
- The Bruceton and statistical methods appear consistent.
- MSFC and WSTF weight loss due to reactions is quite small for Phase I conditions and appear consistent between facilities.
- For conditions that initiated a reaction, no samples exhibited propagation of the reaction by consuming a significant portion of the material. The initiated reaction was quenched.
- The Phase I test data reveal a characteristic of this composite, which offers the acceptance of this material as impact resistant if the technical community accepts an alternate pass criterion.
- Phase I results encouraged the development of a more accurate methodology for measuring pre- and post-test sample weights and additional testing.
New Sample Weighing Procedure

- Samples are placed within a humidity chamber at 40% for 24 hours.
- After 24 hours, the samples are weighed via (mg) scale.
- Pretest weights are recorded for all samples.
  - Sample is removed from the humidity chamber.
  - The chamber is closed.
  - The sample is weighed.
  - The sample is placed into its designated bag and the bag is closed.
  - The next sample is removed from the humidity chamber and the weighing procedure is repeated with this sample.
- Immediately after all weights are obtained, standard testing begins.
- After each sample is tested, the entire cup with sample is placed into the humidity chamber at 40% for 24 hours.
- Note: The samples are tested in order and placed on their proper identification labels on a foil sheet inside the humidity chamber after testing.
- The time that each sample is placed into the humidity chamber is noted.
- After 24 hours within the chamber, each sample is weighed in the same manner as the above procedure and the post-test weights are recorded.
Phase II Objectives

Phase II: Repeat (13A) test of tested Cycom from Phase I at MSFC to expand testing statistical database

- Retest tested Phase I samples that remained intact from Phase I testing to expand the database with minimal material available
- Implement new accurate pre- and post-test weighing method to determine if the weight loss was from the test procedure or from reaction of the material
## Phase II Test Matrix

### Phase II Material Retest

**Ambient 13A LOX Ambient Pressure**

<table>
<thead>
<tr>
<th>Energy level ft-lb</th>
<th>MSFC samples</th>
<th>Sample source (Note: 1, 2, 3)</th>
<th>1st impact reactions</th>
<th>all impact reactions</th>
<th>Wt Change w/Reaction %</th>
<th>Wt Change w/o Reaction %</th>
</tr>
</thead>
<tbody>
<tr>
<td>23.5</td>
<td>65</td>
<td>Prev Multiple Impact Bruceton, Prev Single Impact Bruceton, Prev 27.2</td>
<td>6</td>
<td>8</td>
<td>-0.40%</td>
<td>-0.16%</td>
</tr>
<tr>
<td>31.2</td>
<td>15</td>
<td>Prev. 31.2 samples</td>
<td>0</td>
<td>6</td>
<td>-0.54%</td>
<td>-0.14%</td>
</tr>
<tr>
<td>35.8</td>
<td>15</td>
<td>Prev. 35.8 samples</td>
<td>2</td>
<td>Catcher</td>
<td>-0.84%</td>
<td>-0.63%</td>
</tr>
<tr>
<td>40.8</td>
<td>14</td>
<td>Prev. 40.8 samples</td>
<td>8</td>
<td>Catcher</td>
<td>-1.01%</td>
<td>-0.38%</td>
</tr>
<tr>
<td></td>
<td>109</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Note 1:** Post Phase I samples hand selected for completed disk. Samples are weighed. Samples greater than 0.72 gm were selected.

**Note 2:** Use Std cleaning but keep individual sample identification.

**Note 3:** Store 24 hr in 40% humidity before testing. Weigh sampled before testing after storage at controlled humidity. Place samples in controlled 40% humidity for 24 hours after testing. Weigh sampled and package samples.
Average Weight Change Data

Phase II
Testing Ambient 13 A LOX Ambient Pressure

After accurate weight measurement process was implemented:

- Some mass is lost without reaction due to the open cup testing method.
- A small amount of mass is apparently consumed by the reaction initiation.
- The small amount consumed implies the reaction is quenched.
First Strike Reaction Frequency Data

Ambient Impact Test 13A
Carbon Fiber/Epoxy Resin (0.0895 in. thick)

- Phase I MSFC Testing
- Phase II MSFC Retesting

\[ y = 2.54 \times 10^{-5} \times 2.55 \times 10^{00} \]

\[ R^2 = 9.21 \times 10^{-01} \]

Bruceton Method
Phase II Conclusions

- The data from the retested material appears consistent with the Phase I material, which had not been tested. The data have been combined with Phase I data.

- The new mass loss measurement techniques and improved care of the post-test samples appear to have improved accuracy of mass loss measurements.

- The mass loss with reactions is small, i.e., less than 1.1%, indicating the reaction initiation did not propagate for any sample.

- The testing process and sample recovery process produce a small mass loss.
Phase III Objectives

Phase III: Conduct High-Pressure tests (13B) in LOX and GOX at MSFC and WSTF to determine Cycom reaction characteristics and batch effects

- Determine Cycom reactivity in LOX at 100 psia
- Determine Cycom reactivity in GOX at 100 psia
- Examine batch sensitivity in LOX at 100 psia
- Examine batch sensitivity in GOX at 100 psia
- Compare MSFC and WSTF 13B data for Cycom in LOX and GOX
## Phase III Test Matrix

### Phase III Testing 13 B Bruceton Method

<table>
<thead>
<tr>
<th>Batch # (ASDL #)</th>
<th>Sample Number</th>
<th>Test</th>
<th>Pressure psia</th>
<th>LOX/GOX</th>
<th>Reactions</th>
<th>No Reactions</th>
<th>50% Energy ft-lb</th>
<th>Wt Change w/reactions %</th>
<th>Wt Change w/o reactions %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Set A, 18110</td>
<td>34</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>LOX</td>
<td>4</td>
<td>30</td>
<td>67.23</td>
<td>-14.56%</td>
<td>-0.19%</td>
</tr>
<tr>
<td>Set B, 18109</td>
<td>34</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>LOX</td>
<td>4</td>
<td>30</td>
<td>64.77</td>
<td>-26.25%</td>
<td>-0.01%</td>
</tr>
<tr>
<td>Set A, 18110</td>
<td>34</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>GOX</td>
<td>0</td>
<td>34</td>
<td>-</td>
<td>-</td>
<td>0.00%</td>
</tr>
<tr>
<td>Set B, 18109</td>
<td>34</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>GOX</td>
<td>0</td>
<td>34</td>
<td>-</td>
<td>-</td>
<td>-0.008%</td>
</tr>
</tbody>
</table>

### WSTF

<table>
<thead>
<tr>
<th>Batch # (ASDL #)</th>
<th>Sample Number</th>
<th>Test</th>
<th>Pressure psia</th>
<th>LOX/GOX</th>
<th>Reactions</th>
<th>No Reactions</th>
<th>50% Energy ft-lb</th>
<th>Wt Change w/reactions %</th>
<th>Wt Change w/o reactions %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Set A, 18110</td>
<td>20</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>LOX</td>
<td>0</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>-0.06%</td>
</tr>
<tr>
<td>Set A, 18110</td>
<td>25</td>
<td>13 B Bruceton Method</td>
<td>100</td>
<td>GOX</td>
<td>0</td>
<td>25</td>
<td>-</td>
<td>-</td>
<td>-0.03%</td>
</tr>
</tbody>
</table>
Weight Change in LOX at 100 Psia at MSFC

Phase III
13B Bruceton Method LOX 100 psia

![Graph showing weight change over sample number](image)

- Reactions

- 100 psia Set A LOX (13B)
- 100 psia Set B LOX (13B)
Phase III Conclusions

- Cycom does not support initiation of reactions or propagate of reactions in GOX at 100 psia based on tests at MSFC and WSTF at 72 ft-lb impact energy.

- No batch effect was identified in LOX or GOX.

- WSTF show no reactions at 72 ft-lb and 100 psia in LOX whereas MSFC showed 4 of 34 reactions (11.7%) for both batches tested.

- Six of the eight reactions in LOX (72 ft-lb at 100 psia) supported initiation and propagation of reaction as indicated by the large amount of mass loss (ave. = 26%) by the impact promoted reaction.
Phase IV Objectives

Phase IV: Ambient (13A) test at MSFC to examine average time of sample in test well effects and (13B) testing to determine pressure effects in LOX. Expand 13B GOX database

Determine the pressure effect on reaction frequency:
Hypothesis: At reaction frequency is a function of the number of entrained Bubbles, then higher pressure will reduce the number of bubbles and lower the reaction probability.

Note: Since no reactions were observed with GOX at 100 psia, the adiabatic compression of bubbles is considered an initiation mechanism for LOX impact.
# Phase IV Test Matrix

## Ambient 13A (LOX)

<table>
<thead>
<tr>
<th>Batch (ASDL #)</th>
<th>Energy Level ft-lb</th>
<th>MSFC samples</th>
<th>Pressure psia</th>
<th>1st impact reactions</th>
<th>all impact reactions</th>
<th>Average Time s</th>
<th>Wt change w/ reaction g</th>
<th>Wt change w/o reaction g</th>
</tr>
</thead>
<tbody>
<tr>
<td>18109</td>
<td>72</td>
<td>30</td>
<td>14.7</td>
<td>26</td>
<td>catcher</td>
<td>40</td>
<td>-5.88%</td>
<td>0.96%</td>
</tr>
<tr>
<td>18110</td>
<td>72</td>
<td>30</td>
<td>14.7</td>
<td>20</td>
<td>catcher</td>
<td>80</td>
<td>-8.39%</td>
<td>-5.40%</td>
</tr>
<tr>
<td><strong>Subtotals</strong></td>
<td></td>
<td><strong>60</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Average time is defined as the time between the sample being placed into the sample well to the time the sample is impacted. Forty, 40, seconds is the average time for a 13A sample at MSFC under normal conditions.

## 13B (LOX)

<table>
<thead>
<tr>
<th>Batch (ASDL #)</th>
<th>Energy Level ft-lb</th>
<th>MSFC samples</th>
<th>Pressure psia</th>
<th>Reactions</th>
<th>No reactions</th>
<th>Average Time</th>
<th>Wt change w/ reaction g</th>
<th>Wt change w/o reaction g</th>
</tr>
</thead>
<tbody>
<tr>
<td>18109</td>
<td>72</td>
<td>30</td>
<td>50</td>
<td>13</td>
<td>17</td>
<td>-</td>
<td>-57.07%</td>
<td>-5.75%</td>
</tr>
<tr>
<td>18110</td>
<td>72</td>
<td>30</td>
<td>200</td>
<td>18</td>
<td>12</td>
<td>-</td>
<td>-51.51%</td>
<td>-0.44%</td>
</tr>
<tr>
<td>18110</td>
<td>72</td>
<td>2</td>
<td>500</td>
<td>2</td>
<td>0</td>
<td>-</td>
<td>-37.33%</td>
<td>-</td>
</tr>
<tr>
<td><strong>Subtotals</strong></td>
<td></td>
<td><strong>62</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Phase IV Test Matrix Continued

<table>
<thead>
<tr>
<th>Batch (ASDL #)</th>
<th>Medium</th>
<th>Energy Level (ft-lb)</th>
<th>MSFC (samples)</th>
<th>Pressure (psia)</th>
<th>1st impact reactions</th>
<th>Wt change w/ reaction g</th>
<th>Wt change w/o reaction g</th>
</tr>
</thead>
<tbody>
<tr>
<td>18109</td>
<td>GOX</td>
<td>72</td>
<td>30</td>
<td>500</td>
<td>22</td>
<td>-6.223%</td>
<td>-0.147%</td>
</tr>
<tr>
<td>18109</td>
<td>GOX</td>
<td>72</td>
<td>30</td>
<td>200</td>
<td>4</td>
<td>-0.395%</td>
<td>0.208%</td>
</tr>
<tr>
<td>Subtotals</td>
<td></td>
<td></td>
<td>60</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Combined First Strike
Reaction Frequency Data

Ambient Impact Test 13A, Cycom
Carbon Fiber/Epoxy Resin (0.0895 in. thick)

\[ y = 6.94E-05x^{2.26E+00} \]
\[ R^2 = 8.27E-01 \]

- MSFC Phase I, II & IV

Bruceton Method

14.7 Psia
Mass Loss in LOX Testing

Phase IV
13B Cycom Impact Testing in LOX @ 72 ft-lbs and -297 F

Weight Change [%]

Sample Number

Reactions below this Line

-100%

-90%

-80%

-70%

-60%

-50%

-40%

-30%

-20%

-10%

0%

10%
No Reaction and
Non-Quenched Reaction Comparison

Cycom 977-2 13B LOX 50 psia 72 ft-lbs

Sample 18 No Reaction
Sample 6 Reaction
Pressure Effect on Reaction Frequency

Cycom LOX Impact Tests at 72 ft-lb

Is the reduction with pressure due to reduced entrained bubbles for adiabatic compression ignition sites?

Not statistically reliable due to low sample size.

- Reaction Frequency
- Total Samples

Graph showing the relationship between reaction percentage and pressure (psia) with total samples tested.
Average Weight Loss with Reactions at 72 ft-lb

Cycom LOX Impact Tests at 72 ft-lb (MSFC)

Higher Pressure Appears to Promote Reaction Propagation when ignition occurs.

Pressure (psia)

Weight Loss with Reactions (%)
Reaction Frequency in GOX at 72 ft-lb

Cycom GOX Impact Test @ 72 ft-lb

- Reaction Percentage
- Weight Change

30 samples at each pressure level
LOX/GOX Reaction Frequency Comparison

Cycm 977-2 13B Impact Testing @ 72 ft-lb

Reaction Percentage

Pressure [psia]
Phase IV Conclusions

- Phase IV 13A results were combined with Phases I to III data to provide a more complete reaction frequency data set.

- A small, but statistically insignificant, reduction in reaction frequency was observed by testing at double the sample dwell time before testing.

- Phase III and IV LOX data were combined to show a decrease in reaction frequency with increasing pressure and a corresponding increase in mass loss due to combustion.

- Cycom reaction frequency was shown to increase with pressure from 0% at 100 psia to xx% at 500 psia in GOX.
Overall Observations and Conclusions

- The reaction frequency data from 13A testing by MSFC and WSTF appear well behaved for the sample number used by each and exhibit the same type of energy level dependency. The reaction frequency shift in energy level is unexplained at this time.

- All the 13A data suggest that only a small amount of material is consumed when reactions take place.

- At ambient pressure, most if not all reactions are quenched as indicated by the small mass loss.

- As test pressure is increased in LOX, using 13B results,
  - The impact initiation has a greater probability of propagation.
  - The probability of ignition is reduced.

- Cycom does not support initiation of reactions or propagation of reactions in GOX at 100 psia based on tests at MSFC and WSTF at 72 ft-lb impact energy. Reactions do occur at higher test pressures.

- No batch effect was identified in LOX or GOX.
Recommendations

- The technical community consider accepting a material for use, which exhibits consistent and universal quenching of impact induced reactions over the potential energy levels at the planned material use thickness.

- The technical committee formulating changes to NASA-STD- 6001 consider altering the testing method to require measurement of pre- and post-test weights.

- Impact combustion initiation be considered a quenched reaction when the mass loss is consistently below 10% of the pre-test mass when a reaction is observed.

- A quenched reaction condition be considered an acceptable risk similar to a flammability test burn length of less than 6.0 inches.
In case we forget that initiation to sustained reaction of Cycom 977-2 can produce significant energy release, see the following slide.
Impact Tester Vent Tube Burn Through
(Cycom with LOX @ 500 psia & 72 ft-lb)

Stainless Steel vent tube

Note: Less than 0.4 gm of Cycom consumed.