Eleventh International Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres

Mechanical Impact Testing – A Statistical Measurement

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Abstract

In the decades since the 1950s, when NASA first developed mechanical impact testing of materials, researchers have continued efforts to gain a better understanding of the chemical, mechanical, and thermodynamic nature of the phenomenon. The impact mechanism is a real combustion ignition mechanism that needs understanding in the design of an oxygen system. The use of test data from this test method has been questioned due to lack of a clear method of application of the data and variability found between tests, material batches, and facilities. This effort explores a large database that has accumulated over a number of years and explores its overall nature. Moreover, testing was performed to determine the statistical nature of the test procedure to help establish sample size guidelines for material characterization. The current method of determining a pass/fail criterion based on either light emission or sound report or material charring is questioned.

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Abstract

In the decades since the 1950s, when first the Army and then NASA first developed mechanical impact testing of materials, researchers have continued efforts to gain a better understanding of the chemical, mechanical, and thermodynamic nature of the phenomenon. The impact mechanism is a real combustion ignition mechanism, which must be considered and understood in the design of an oxygen system. The use of test data from this test method has been questioned because of the lack of a clear method of application of the data and variability found between tests, material batches, and facilities. This effort explores a large database, which has accumulated over a number of years, and explores its overall nature. Moreover, testing was performed to determine the statistical nature of the test procedure to help establish sample size guidelines for material characterization. The current practice of reporting reaction frequency data at dropped energy rather than energy the sample receives does not offer a way to compare data between facilities.

Keywords: Mechanical impact, oxygen compatibility, ignition, nylon 6/6, Lexan\textsuperscript{\textregistered} FR 700-701, neoprene, silicone, Teflon\textsuperscript{\textregistered} and Viton\textsuperscript{\textregistered}

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Introduction

Materials used in habitable flight compartments must be tested to determine their flammability, upward flame propagation, heat and smoke release rates, flash points, odor, and offgassing products. These materials must also be tested to establish their sensitivity to mechanical and pneumatic impact in both liquid oxygen (LOX) and gaseous oxygen (GOX) environments (Ref. 1). The required mechanical impact tests are primarily carried out in National Aeronautics and Space Administration (NASA) test facilities at Marshall Space Flight Center (MSFC) and White Sands Test Facility (WSTF).

Work presented in this effort focuses on a review and evaluation of mechanical impact test data for six nonmetallic materials in high-pressure LOX and GOX and in ambient LOX environments. The nonmetallic materials considered in the report are nylon 6/6, Lexan® FR 700-701, neoprene, silicone, Teflon® polytetrafluorethylene (PTFE), and Viton®. The test data were collected by NASA over a period of years since the early 1970s and are stored in its Materials and Processes Technical Information System (MAPTIS) database.

The purposes of the study were to:
1. Examine the characteristics of mechanical impact data
2. Identify the potential need to increase the number of test runs for each material to reflect the probabilistic nature of the test
3. Identify potential patterns and trends in the data that might lead to improved prediction techniques and test planning
4. Develop potential benchmarks that can be used as calibration standards.

Background

In the decades since the 1960s, when NASA first developed mechanical impact testing of materials, researchers have continued efforts to gain a better understanding of the chemical, mechanical, and thermodynamic nature of the phenomenon. Looking at the chemical processes taking place during impact testing, Glassman (Ref. 2) presents a good review of the basic combustion principles associated with impact reactions; the works of Vilyunov and Zarko (Ref. 3) present a comprehensive review and detailed examination of all aspects of the ignition process. Yet, difficulties remain in predicting the sensitivity of materials to impact testing based on their chemical properties, as demonstrated in the studies of Tapphorn, Shelley, and Benz (Ref. 4), which attempted to correlate the auto-ignition temperature of materials with their subsequent sensitivity to impact.

To establish the potential effect of test apparatus design and test procedures on the sensitivity of materials to impact, researchers deQuay and Scheuermann (Ref. 5) looked exclusively at the mechanical design of impact test apparatus, while Barthelemy, Roy, and Mazloumian (Ref. 6) investigated the effect of both material contaminants and apparatus design on the resulting sensitivity of materials to impact. Nguyen and Pham (Ref. 7), Reed, Simon, and Berger (Ref. 8), and McColskey, Reed, Simon, and Bransford (Ref. 9) also conducted extensive studies that established differences between the test apparatus designs at MSFC and WSTF that would lead to differences in the impact test
results. Measurements for penetration depths of metal samples were reported by Bransford, et al. (Ref. 10) comparing different mechanical testers.

Because of the thermodynamic nature of the ignition process, researchers Barragan, Wilson, and Stoltzfus (Ref. 11) also investigated thermal aspects of the ignition process resulting from compression, while Simon and Reed (Ref. 12) looked at the effective temperature rise of impacted materials related to material deformation and fracture. Additional studies by Bowden and Yoffe (Ref. 13) focus on the production of localized hot spots that lead to the detonation of impacted materials. Researchers Janoff, Pedley, and Barnford (Ref. 14) conducted studies of pressure effects on the ignition sensitivity of materials in similar pneumatic impact tests.

Based on the results of these and many other research studies, it is apparent that significant difficulties still remain in efforts to develop a mechanical impact calibration standard or a theoretical model of material sensitivity to impact. Thus far, test data have suggested that different results for each material being tested can be achieved, depending upon the specific test facility and sample. These differences can result from possible batch effects, contaminants, flaws in the samples being tested, roughness effects, errors in testing procedures, differences in apparatus design, energy transfer rate, local hot spot formation, and the probabilistic effects inherent in all chemical reactions. The current investigation was conducted, in part, to determine if available test data can be used to better understand these differences.

Mechanical Impact Test Methodology

Ambient and high pressure test procedures are governed by NASA Standard 6001, Flammability, Odor, Offgassing and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion. Tests for ambient pressures in LOX are governed by Test 13A procedures and tests for high pressures in LOX and GOX are governed by Test 13B procedures described in NASA-STD-6001. Data from both test procedures are examined herein.

In the Test 13B impact test apparatus, a 20-lb plummet is released from rest and falls freely from heights (not exceeding 3.6 ft) that correspond to specific impact energy levels. An alignment rail guide controls the plummet free fall with minimal losses related to friction during the descent. At the bottom of its free fall, the plummet impacts a striker pin, which is set above the material specimen in a high-pressure GOX or LOX test chamber. When impacted by the plummet, the striker pin accelerates into the material specimen. Before testing, the material sample is cleaned in accordance with prescribed procedures and placed in a cup within the test chamber. A balance chamber is incorporated into the apparatus to offset the lifting force created by the high pressure in the sample test chamber. To keep the plummet from impacting the specimen more than once, a device catches the plummet after the first impact.

Criteria used by NASA to judge the sensitivity of the materials to impact are based entirely upon the detection of a flash of light, an audible detonation “report,” or the charring of the test sample during impact. In tests conducted at the NASA test facilities, the number of tests made for each material at each test condition varies significantly.
based upon the outcome of preceding tests and upon whether a reaction has already been observed. The passing criteria for a material for use in manned spaceflight operations are no reactions observed in 20 test cases at any energy level or a maximum of one reaction observed in 60 samples tested at a maximum energy level of 72 ft-lb. Test conditions controlled during mechanical impact testing include chamber pressure and temperature, specimen thickness, impact energy based on initial plummet height, gas species concentration as a percent, and type and thickness of any specimen substrate.

**Test Data Examination**

Impact test data for nylon 6/6 and Lexan® FR 700-701 were obtained as part of the current effort using the ambient pressure method of 13A for LOX environments. High-pressure (13B) LOX and GOX test data from MSFC and WSTF were obtained from archives in the MAPTIS database at MSFC for nylon 6/6, neoprene rubber, silicone rubber, Teflon® PTFE, and Viton® rubber. The reaction frequency was correlated for each test condition based on variations in the impact energy, test chamber pressure and temperature, and sample thickness. In cases where multiple tests were conducted for the same relative test conditions, a statistical mean reaction frequency was established. The mean was established by adding the total number of reactions occurring in all of the test cases at any given test condition and dividing the sum by the total number of impacts at these test conditions. When data regarding the test conditions were missing, data for the test case were excluded from the analysis. When the data were limited and differences in the absolute temperature were small, temperature effects were excluded from the analysis. In most cases, test conditions in which less than 9 impact samples tested were excluded from the data presented herein.

**Ambient Tests in LOX (Test 13A)**

**Nylon 6/6:** Nylon 6/6 was used as a potential candidate to be considered as a reference material in impact testing. Consequently, a large data set was obtained using the 13A test method. Tests were conducted in LOX for five material thicknesses over a range of energy levels. The reaction frequency as a function of impact energy is shown in Figure 1. Each data point represents the frequency determined from 60 samples. The standard procedure in Test 13A is to allow the plummet to impact and rebound. Any observed reaction is recorded along with the impact number at which the reaction occurs. Figure 2 shows the reaction frequency as a function of material thickness for the first impact and cumulatively for all impacts. A large difference is evident between the single and multiple impact data. Observation of the material samples shows that the first impact shatters the specimen into an aggregate of particles ranging in size from small to large. The subsequent rebound impacts of lower energy strike this aggregate, and small particles have a higher propensity to ignite. Figure 1 shows cumulative impact data.

Figure 3 shows the dramatic effect of multiple strikes on the cumulative reaction frequency, which is normally reported. The cumulative reaction frequency is the sum of the first impact; the first, second, and third rebounds are shown as a function of initial impact energy at a constant material thickness. At 72 ft-lbf, the reaction frequency for
the first strike and first rebound are essentially equal. At initial impact energies of 40 ft-lbf or less, only the first impact is significant.

The number of reactions is recorded as a function of sample number during the process of the test and is available for each of the conditions shown in Figure 1, where each point represents percent reactions that occurred with the 60 samples tested. Figure 4 shows the running percent reactions as a function of sample number for a single thickness at six energy levels. If the probability of an impact to cause a reaction is sought from this testing, it is evident that 20 twenty samples is inadequate. These data support the 1991 recommendation by McClolskey, Reed, Simon, and Bransford, "We recommend that a increased number of tests (at least 60) be conducted in order to determine the material reactivity threshold with a higher confidence level" [Ref. 9].

**Lexan® FR 700-701:** Lexan® FR 700-701 was tested at ambient conditions in LOX at six impact energy conditions for a constant thickness sample. At each energy level, 70 drops were performed. These data are shown in Figure 5, which also includes data for nylon 6/6 for comparison. The reaction frequency is linear in a semi-log plane of energy level. Lexan® FR 700-701 is less reactive than nylon 6/6. Correction for the slight thickness difference is expected to further separate the curves based on trends from Figure 2.

The thickness trend from Figure 2 suggests that a thicker material reacts less. To examine the effect of thickness with increased surface area, the Lexan® samples were doubled by using two sheets of the same 0.0248-in. material. The results are shown in Figure 6, in which the reaction frequency curve is increased by a constant shift of about 25 percent at a given energy level. It is speculated that sliding friction between sheets or possibly bubble formation on the surfaces and subsequent adiabatic compression provided hot spots for initiation of combustion that were not present with the single sheet.

**High-Pressure Tests in GOX and LOX (Test 13B)**

Data from the MAPTIS archives were extensively reviewed and evaluated for five nonmetallic materials subjected to mechanical impact tests in high-pressure and varied-temperature GOX and LOX environments. Over 2,800 test cases were identified in the MAPTIS database and divided into GOX and LOX files. The GOX and LOX files were subdivided into groups based upon the material type, use, and sample thickness. These data groups were then sorted by pressure, impact energy, and temperature. A predominate number of the tests were conducted in GOX environments.

Data for nylon 6/6, neoprene rubber, silicone rubber, Teflon® PTFE, and Viton® rubber were reviewed and are summarized in a set of tables provided herein (Tables 1 to 13). Test conditions in which fewer than 20 drops were performed were considered statistically suspect. Data conditions in which fewer than 9 samples tested were usually dropped. This provided a large data set of data primarily from Marshall Space Flight Center (MSFC) and White Sands Test Facility (WSTF). Even though the data set is large for each material, it is not systematic in terms of independent variables covered. Moreover, comparable data between Centers is somewhat limited. On examination of data between test Centers, it became evident that the reported data for the same tests...
conditions did not agree when statistically large sample sizes were compared. Previous comparisons of testers between MSFC and WSTF revealed (Ref. 10) that the same energy was not delivered to a sample for the same dropped energy level [U (ft-lb)]. To attempt to adjust for this difference, the penetration depth data of Reference 10 was used. By assuming that the energy delivered to the sample is directly related to the penetration depth \( F = d^2 \) recorded on a metal sample, a relationship was developed for the ratio of the energy imparted to a sample between the two facilities.

\[
\text{Temperature} | \text{Pressure (psia)} | \text{F/U mm}^2/\text{kg-M} \\
\text{MSFC Amb | 14.7 | 0.184807} \\
\text{MSFC Amb | 4995.0 | 0.165881} \\
\text{WSTF Amb | 14.7 | 0.139128} \\
\text{WSTF Amb | 4995.0 | 0.107795}
\]

Assuming the pressure effect is linear, the following relation was developed for the high-pressure systems using GOX. Insufficient data are available to develop a similar relation for LOX.

\[
\text{Uw/Um} = -2.0848E-5(P - 14.7) + 0.752829, \text{ where } P = \text{pressure (psia)}.
\]

If the MSFC input energy is used as the reference, then

\[
\text{Um} = U, \text{ the MSFC drop energy value, and} \\
\text{Up} = \text{Uw, the WSTF penetration equivalent to the MSFC drop energy,}
\]

yielding:

\[
\text{Up} = [-2.0848E-5(P - 14.7) + 0.752829] U \text{ for WSTF data}
\]

This estimate of the facility normalizing penetration equivalent energy is provided in the tables for GOX reaction frequency data along with the facility reported drop energy, U.

**Nylon 6/6:** Reaction frequency data for nylon 6/6 are presented in Tables 1 and 2, for GOX and LOX, respectively. Nylon 6/6 reacts in GOX and LOX with rather high frequency for most pressure levels and with high impact energy (Figure 7). Lowering the impact energy significantly lowers the reaction frequency, as seen in ambient testing. This trend can be seen more clearly in Figure 8, in which data from Figure 7 were interpolated at a constant pressure of 1250 psia. The data form a reasonable distribution versus energy level, with the MSFC and WSTF data being consistent within the data pattern. The LOX data are for a larger thickness (Th) than the GOX test data. This type of inconsistency is prevalent throughout the test data, making comparisons and establishing trends quite difficult.

**Neoprene:** Reaction frequency data for neoprene sheet and O-rings tested in GOX are presented in Tables 3 and 4, respectively. Only one test condition was recorded for neoprene tested in LOX; consequently, it is not reported here. All of the data shown are from tests conducted at WSTF. Neoprene appears quite compatible with GOX and shows a high reaction frequency at the highest energy condition. The sample size of eight may have significantly influenced the reported reaction frequency of 25% at \( U = 72 \text{ ft lb} \).
Silicone: Reaction frequency data for silicone sheet and O-rings tested in GOX are presented in Tables 5 and 6, respectively. The sheet configuration generally has a lower reaction frequency than the O-ring. The highest reaction frequency shown in Table 5 is 0.667, which appears for a limited sample size of nine at 3000 and 3300 psia. The worst condition tested for sheet silicone is the MSFC 72 ft-lb input at 3000 psia, which recorded a reaction frequency of 28% using 100 samples. Silicone O-ring data are given in Table 6 and shown in part in Figure 9. The reaction frequency is higher than the sheet values. The MSFC and WSTF penetration impact adjusted data appear consistent. The lower pressure condition (1050 psia) realized higher reaction frequencies than the higher condition (3000 psia), which is not expected. The reaction frequencies in LOX (Table 7) at the much lower pressure condition of 170 psia are comparable to the high-pressure conditions in GOX.

Teflon®: Reaction frequency data for Teflon® tape and tubing tested in GOX are presented in Tables 8 and 9, respectively. Reaction frequencies are quite low for the data from very thin tape from both facilities and appear high only for conditions in which the sample size is relatively low. Reaction frequencies above 5% appear to occur at pressures above 4000 psia. Many other tests were conducted on Teflon® products such as gasket sealants, felt, mold compounds, and sealant compounds. The number of samples tested at each condition was small.

Viton®: Reaction frequency data for Viton® sheet in GOX and LOX are given in Tables 10 and 11, respectively, and data for Viton® O-rings in LOX and GOX are given in Tables 12 and 13, respectively. Viton® sheet reaction frequencies in GOX exceed 10% only for 2 of 18 test conditions. The peak of 23.3% is found for a high sample size and intermediate test condition (1250 psia). Viton® sheet reaction frequencies in LOX exceed 10% only at one condition in which the sample size is questionable. Viton® O-ring reaction frequencies in GOX exceed 10% only at one pressure (Figure 10) for maximum input energy of 72 ft-lb. Viton® O-ring reaction frequencies in LOX exceed 12.5% at pressures above 1000 psia and are quite high at 5000 psia.

The lack of comparable data sets with significant sample sizes makes ranking and comparing materials difficult. Figure 11 shows neoprene, silicone, and Viton® reaction frequencies in GOX for O-ring configurations at 1050 psia as a function of impact energy. The data show no reactions for neoprene, small reaction frequencies for Viton® at two thicknesses, and significant reaction frequencies for silicone, which increase with increasing impact energy. Figure 12 compares silicone and Viton® sheet reaction frequencies at 3000 psia and demonstrates that Viton® is quite superior to silicone.

Throughout the review of the high-pressure data tables and plots, the observation that the high reaction frequency points that stood out appeared to be those obtained with small sample sizes. Consequently, plots were made of the reaction frequency versus number of samples tested. All materials and all groupings appeared to have the same feature: a few high frequency data points at small sample sizes that did not appear at high sample sizes. A typical plot is shown in Figure 13, in which silicone data are plotted for sheet and O-ring configurations. The energy level was restricted but pressure was not. This plot shows 5 suspect data points for sample sizes below 36. This is not conclusive proof of inadequate sample size data, but it is highly suspicious. Only additional testing can
verify this suspicion; however, this behavior and the results shown in Figure 4 provide the empirical evidence that a larger sample size should be used.

Conclusions and Recommendations

1. All materials examined exhibit a probability to react caused by impact while in an oxygen environment. The probability of reaction is a function of the environment and geometry tested.
2. Nylon 6/6 and Lexan® FR 700-701 exhibit well-behaved reaction frequency versus impact energy curves when 60 or 70 samples are used to establish the reaction frequency at one test condition.
3. Significantly different results are observed for single and multiple impact conditions of Test 13A for LOX ambient conditions. Multiple impact data should be applied only for those physical conditions in which multiple impact conditions are expected. Moreover, multiple impact conditions should be reexamined for the realism of the rebound impact energy imparted by current testers.
4. Even though a large database exists for high-pressure LOX and GOX conditions, a systematic comparison of different materials over a range of impact energies and pressures is difficult because of the lack of overall test planning. The practice of testing for only a user’s test conditions provides only a patchwork of data without a context.
5. The current practice of declaring that a material passes for application if no reactions are observed in 20 samples tested appears questionable. Many passes given are probably because insufficient samples have been tested. In batch testing of the same material, many batches which passed and many batches that failed may have had the same low reaction frequency characteristics. It is just as likely that a low reaction frequency is observed in the first 20 samples as a high value.
6. An approximate method was introduced to compare data from WSTF and MSFC, based on the penetration depth test data provided in Reference 10. Round robin tests between facilities should include metal disks for measuring penetration depth. Without this or some other comparable method being available, the data included in MAPTIS and open literature are labeled with the same drop energy but, in actuality, do not represent the same energy delivered to the sample.
7. The data and analysis of this work strongly support the recommendations made in 1991 by McClolskey, Reed, Simon, and Bransford, “We recommend that a increased number of tests (at least 60) be conducted in order to determine the material reactivity threshold with a higher confidence level.” (Ref. 9).

References


### Table 1. Nylon 6/6 in GOX

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<th>U (ft-lbf)</th>
<th>P (psia)</th>
<th>T (°F)</th>
<th>Th (in.)</th>
<th>Number Impacts</th>
<th>Reaction Freq.</th>
<th>Test Facility</th>
<th>MSFC Penetration</th>
<th>Equiv. Energy (UP) (ft-lbf)</th>
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<td>75</td>
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Table 5. Silicone in GOX

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<th>Reaction Freq.</th>
<th>Test Facility</th>
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### Table 6. Silicone O-Ring in GOX

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<th>Reaction Freq.</th>
<th>Test Facility</th>
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### Table 7. Silicone in LOX

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### Table 8. Teflon® Tubing in GOX

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### Table 12. Viton® O-Ring in LOX

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Figure 1. Nylon 6/6 Ambient LOX Impact Reaction Frequencies

Figure 2. Nylon 6/6 Thickness Effect on First and Cumulative Impact Reaction Frequency
Figure 3. Nylon 6/6 Reaction Frequency as a Function of Impact Sequence

Figure 4. Sample Number Running Reaction Frequency Data
Figure 5. Lexan® FR 700-701 and Nylon 6/6 Reaction Frequency Comparison

Figure 6. Lexan® FR 700-701 Single and Double Sheet 13A Impact Data
Figure 7. Nylon 6/6 Reaction Frequencies in GOX at Elevated Pressures

Nylon 6/6 in GOX
T = 75 °F,
Th = 0.057-0.077 in. (0.0145-0.196 cm)

- UP ~ 72 ft-lb (97.62 J)
- UP ~ 65 ft-lb (88.13 J)
- UP ~ 60 ft-lb (81.35 J)
- UP ~ 55 ft-lb (74.57 J)
- UP ~ 50 ft-lb (67.79 J)
- UP ~ 45 ft-lb (61.01 J)
- UP ~ 40 ft-lb (54.23 J)

Pressure (psia)
(0 MPa) 400 800 1200 1600 2000 (13.79 MPa)
Reaction Frequency

Figure 8. Nylon 6/6 Interpolated Data from Figure 7 at 1250 psia

Nylon 6/6 in GOX
P = 1250 psia (8.62 Mpa), T = 75 °F,
Th = 0.057-0.077 in. (0.0145-0.196 cm)

- MSFC
- WSTF
- MSFC/WSTF

Impact Energy (UP) [ft-lbf]
10.00 100.00 (13.56 J) (135.6 J)
Figure 9. Silicone O-Ring Reaction Frequencies in GOX at Elevated Pressures

Figure 10. Viton® O-Ring Reaction Frequency in GOX
Figure 11. Reaction Frequency Comparisons for Neoprene, Silicone and Viton® O-Rings in GOX

Figure 12. Comparisons of Silicone and Viton® Sheet Reaction Frequencies
Figure 13. Reaction Frequency versus Number of Samples Tested