MULTIVARIATE REGRESSION ANALYSIS OF GASEOUS AND LIQUID OXYGEN MECHANICAL IMPACT TEST DATA

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Mechanical impact (MI) data of materials tested in liquid and gaseous oxygen according to ASTM G86[1] were modelled and analyzed using multivariate logistic regression methodology. Models were developed from material test data published in "ASTM Manual 36" (Manual 36).[2] Oxygen system designers rely on standard test data published in Manual 36 for material selection. Further, Oxygen Hazards and Fire Risk Analysis (OHFRA) performed on oxygen systems often rely on the material test data published in Manual 36. Proper assessment of the hazards and fire risk is critical to avoiding fires and ensuring the safety of personnel[3], and the data analysis technique presented is useful in the assessment of that risk.

The logistic regression model was used to characterize the material's probability for reaction (ignition) at a given pressure and impact energy level. The authors have developed a process of determining metrics based on ASTM G86 material result criteria modelled using a Bernoulli distribution. Applying these metrics to a multivariate logistic regression model allows designers to assess the reaction probability of a material across a wide range of possible pressure and impact energy conditions (including conditions that have not been tested) using characteristic plots. Characteristic plots are provided for Vespel® SP-1, Vespel® SP-21, Kel-F® 81 (CTFE), and Teflon® (PTFE) which are common non-metal soft goods used in oxygen systems.

I. INTRODUCTION

An approach was developed for characterizing a material's sensitivity to ignition due to mechanical impact in ambient or elevated pressure liquid oxygen (LOX) or gaseous oxygen (GOX). A generalized approach to the numerical processing requirements is presented, so that other researchers can recreate the analysis using statistical modelling tools. A numerical processor that has a generalized linear modelling algorithm capable of performing multivariate logistic regression is recommended. The authors utilized the R programming language and processor for statistical computing.[4]

WHA International, Inc. (WHA) performs ASTM G86[1] testing, and the following describes the test method and test analysis method utilized. Analysis of test data for Vespel® SP1, published in ASTM Manual 36, is detailed in order to demonstrate the required steps for the analysis. Following that are finalized plots for other non-metallic materials with data listed in ASTM Manual 36. Characteristic plots are provided for Vespel® SP-1, Vespel® SP-21, Kel-F® 81[a] (CTFE), and Teflon® (PTFE), which are common non-metal soft goods used in oxygen systems.

II. ASTM G86 MECHANICAL IMPACT TESTING

The ASTM International[b] (ASTM) G86 test method has been used extensively in industry to determine and select oxygen compatible materials and to conduct batch/lot testing for materials already determined to be oxygen compatible. WHA conducts G86 testing as an integral part of its oxygen compatibility testing and analysis efforts. Furthermore, most publicly available G86 test data was accumulated through testing conducted by the National Aeronautics and Space Administration (NASA) for

[a] Kel-F® 81 (CTFE) was heavily used in the 80's and early 90's by NASA but hasn't been available since 95. A typical formulation of CTFE used today is Daikin Neoflon™ CTFE.
[b] Formally American Society for Testing Materials.

the qualification of materials for oxygen service in the Apollo and Space Shuttle program. Primarily, the ASTM G86 test standard was designed to determine the relative reaction (ignition) sensitivity to mechanical impact of materials in ambient or elevated pressure liquid oxygen (LOX) or gaseous oxygen (GOX) environments and thereby create a relative ranking of materials for use in oxygen systems.

II.A. Test Method

The G86 test method utilizes a plummet dropped from designated heights to impact a striker pin which delivers the impact energy to a test sample contained in either an open cup sample holder (ambient pressure) or a pressurized test chamber (up to 68.9 MPa [10,000 psig]). For each test, the test sample is fully immersed in either LOX or GOX and then impacted. Determination of sample reaction is based on several real-time test observations including pressure increase (pressurized test only), temperature increase, light emission, and sustained burning along with post-test observations such as char or burn residue present on the sample cup or the remains of the test sample.

The passing result criteria of ASTM G86 requires that a material exhibit no more than one reaction in sixty tests (1 in 60) at the 98 J (72 ft·lbf) energy level or no reactions in twenty tests (0 in 20) for any energy level lower than 98 J. Test conditions can be varied over impact energy or test pressure to determine thresholds levels for either energy or pressure with a given material. The lowest energy level or test pressure at which a test material meets the passing result criteria is determined to be the energy or pressure threshold respectively for that material. Furthermore, pressurized gaseous oxygen testing can be conducted at elevated temperature conditions.

II.B. Test System

ASTM G86 provides specific test system requirements but allows for various mechanical configurations to meet those requirements. Critically, all G86 test systems must utilize a 9.07 kg (20 lbm) plummet and a test tower capable of drop heights up to 1.10 m (43.3 in). Additionally, for pressurized test systems, the pressure chamber must be capable of providing a net force of 222 N (50 lbf) on the test sample. The pressurized mechanical impact test system designed and utilized by WHA is detailed in Fig. 1, and similar test systems are used by other test facilities including NASA Johnson Space Center, White Sands Test Facility (WSTF), and NASA Marshall Space Flight Center (MSFC). The energy level for each test is set by adjusting the initial drop height of the plummet to levels corresponding with TABLE I. The test chamber in the WHA test system is a two-piece design with an inline counter-balancing piston similar to reference designs shown in ASTM G86.

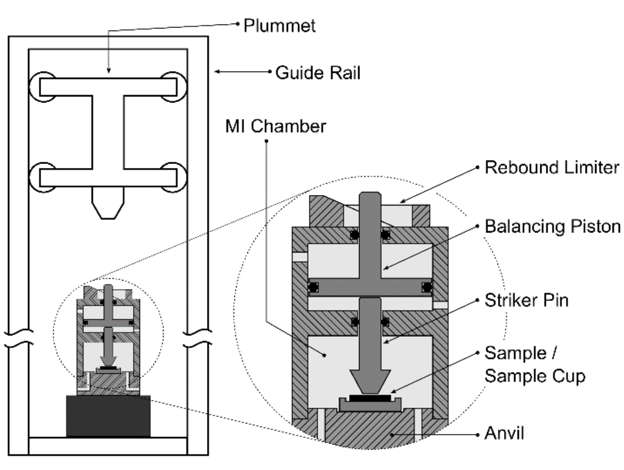


Fig. 1. WHA mechanical impact drop tower and test chamber mechanical configuration

TABLE I. Drop Height Schedule for Energy Threshold Value Determination Using a 9.07-kg (20-lb) Plummet

Energy		Drop Height	
Joules	**Ft·lbf**	**Meters**	**Inches**
98	72	1.10	43.3
88	65	0.99	39.0
81	60	0.91	36.0
75	55	0.84	33.0
69	50	0.76	30.0
61	45	0.69	27.0
54	40	0.61	24.0
48	35	0.53	21.0
41	30	0.46	18.0
34	25	0.38	15.0
27	20	0.31	12.0
20	15	0.23	9.0
14	10	0.15	6.0

II.C. Test Results and Standard Analysis

The analysis and ranking method outlined by ASTM G86 is limited to the determination of the energy or pressure threshold for a given test material or simply whether it passes at a specific threshold, often the 98 J energy level. This determination is made through a simple binary analysis of whether the test material meets the passing result criteria or not. ASTM G86 does not specify or require any additional statistical analysis of the test results. The determined thresholds for a given material are then used to compare and rank that material with other materials that have undergone G86 testing. Often, a material is determined to be acceptable for oxygen service if its energy threshold is higher than another material that has already been classified as safe for oxygen service.

III. STATISTICAL TREATMENT OF ASTM G86 RESULTS

The following describes a numerical process for the treatment of ASTM G86 data by modeling the likelihood of test reaction probability based on the pass criteria in the test standard (specifically, only 1 ignition in 60 tests). Metrics based on the mean response of a 1 out of 60 result were modelled as a Bernoulli trial outcome and then applied directly to the fitted multivariate logistic regression curve in order to determine the pressures and energy levels consistent with the pass criteria of the test standard. The process presented below is adaptable to other user-defined metrics that meet their risk assessment needs.

III.A. Development of Metrics Based on the Bernoulli Distribution

The material under test may be observed to react or to not react under a number of different measures described above. The outcome is only one of two possible values, and therefore follows a Bernoulli trial outcome that can be modelled with a Bernoulli distribution. A discretized form of the Bernoulli distribution is presented in Eq. (1), with N as the number of tests, z is the number of observed reactions, and θ is test reaction probability. Kruschke derived this from the continuous Bernoulli distribution in Ref. (5). Eq. (2) defines θ_i as a discretized sample space between 0 and 1.

$$p(z, N|\{\theta_i\}) = \theta_i^z(1-\theta_i)^{N-z} \quad (1)$$

$$\{\theta_i\} = [0, \ldots, 1] \quad (2)$$

$$p(z=1, N=60|\{\theta_i\}) = \theta_i^1(1-\theta_i)^{59} \quad (3)$$

$$\sum_0^{\theta_x} \frac{p(z=1, N=60|\{\theta_i\})}{\sum p(z=1, N=60|\{\theta_i\})} \leq 0.95 \quad (4)$$

$$\mathrm{R_{U08}} = \theta_x \times 100\% \approx 8\% \quad (5)$$

$$\mathrm{R_{M02}} = z/N \times 100\% = 1/60 \times 100\% \approx 2\% \quad (6)$$

The Bernoulli distribution in Eq. (3) models the likelihood for a material to react given 1 observed reaction in 60 tests. Fig. 2 is a plot of the likelihood for possible values of test reaction probability, θ. A 95% measure of the spread can be assessed using a one-tailed 95% confidence interval. Solving for θ_x in the inequality equation Eq. (4) provides the upper θ value for the confidence interval. The denominator in Eq. (4) normalizes the discretized form of the Bernoulli distribution such that its summation is 1.

The metrics are defined in Eq. (5) and Eq. (6), and are annotated in Fig. 2.

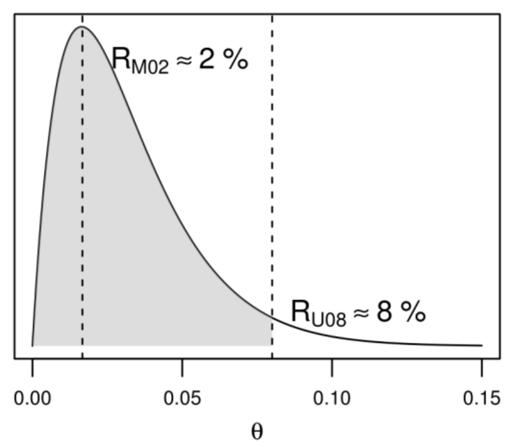


Fig. 2. Bernoulli likelihood function for $z = 1$, $N = 60$ with annotated $\mathrm{R_{M02}}$ and $\mathrm{R_{U08}}$ metrics

III.B. Multivariate Logistic Regression Model

The data used to demonstrate the procedure for the analysis steps is in TABLE II. The “Outcome” column was determined by analyzing the ASTM G86 test criteria with the given data. In every test series there was only one failure, therefore, the outcome status was considered ‘pending’ if the energy level was 98 J (72 ft·lbf) since additional testing may have been performed to determine if the material passed or failed after sixty tests at that level according to the ASTM G86 test standard. This categorization is just for the benefit of the scatter chart, and is not used in the analysis; all of the raw data from each test series was used in the analysis.

Fig. 3 contains a scatter plot based on the outcome of the testing. The scatter plot presents data obtained but demonstrates the great difficulty in specifying the compatibility of the materials due to the scatter evident in the data. Therefore, additional statistical analysis, such as by logistic regression, provides a way of understanding the implications of the data to where safe use of the material might be expected.

A single logistic regression analysis may provide some insight if test series were grouped together. For example, all of the data between 6.89 MPa (1000 psi) and 7.34 MPa (1065 psi) could be treated as the same pressure with an analysis performed using energy as a predictor. Similarly, all of the 98 J (72 ft·lbf) energy level data may be grouped together and an analysis performed using pressure as the predictor variable. However, a multivariate logistic regression analysis is a better technique that considers all of the data. Single logistic regression is easily extended to more than one predictor. Neter et. Al, states that, "in fact, several predictor variables are usually required with logistic regression to obtain adequate description and useful predictions."[5]

For this analysis, pressure and energy were used as predictors for test reaction probability. Each predictor was treated independently with no interactions. Fig. 4 contains a three-dimensional plot of the regression analysis results showing the upper 95% confidence interval (single tailed) and the mean response.

TABLE II. Vespel® SP-1, GOX MI ASTM G86 Results

Pressure (MPa)	Energy (J)	Reactions (Count)	Tests (Count)	Outcome
1.38	98	0	20	pass
3.45	75	0	20	pass
3.45	81	1	1	fail
6.89	98	1	1	pending
7.24	41	0	20	pass
7.24	47	1	27	fail
7.34	41	0	20	pass
7.34	47	1	3	fail
10.3	98	1	1	pending
13.8	98	1	1	pending
17.3	98	1	1	pending
37.9	14	0	20	pass
37.9	75	1	14	fail

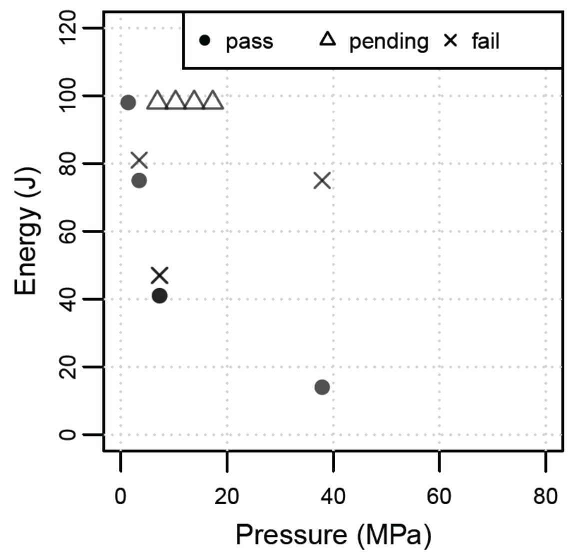
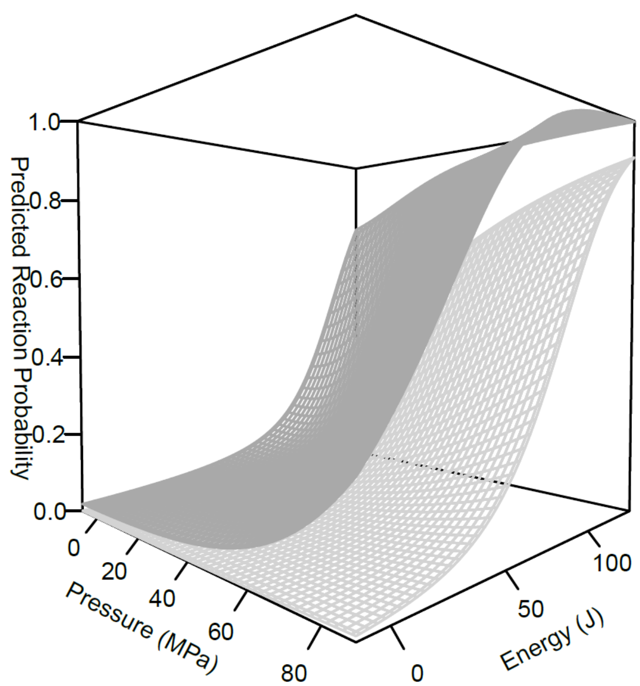


Fig. 3. Vespel® SP-1 ASTM G86 Scatter Plot



$\beta_{intercept}$ = -6.490 log odds
$\beta_{pressure}$ = 0.028 log odds/MPa
β_{energy} = 0.047 log odds/J

Fig. 4. Multivariate Logistic Regression Model; showing upper 95% confidence interval (single tail) and mean response

III.C. Application of RM02 and RU08 Metrics to Regression Model

In section III.A, metrics were developed by analyzing the test pass criteria of ASTM G86 by way of determining the likelihood of test reaction probabilities. The application of those metrics to a multivariate logistic regression model is presented in this section.

The R_{U08} and R_{M02} metrics, Eqs. (4) and (5), were traced out on each response curve. Fig. 5 and

Fig. 6 are energy range plot traces with pressure held at a constant value, 60 MPa and 10 MPa, respectively. The R_{M02} and R_{U08} values are indicated in both figures. When plotted on a two-dimensional pressure versus energy plot, where R_{U08} and R_{M02} intersect, the distribution would look very similar to the one presented in Fig. 2. This circumstance only exists at the point of intersection, so it is important to pick the more conservative of the two metrics in the analysis. In this context, the conservative metric will always be at the lower energy value.

In general, if the number of tests is low, R_{U08} will be more conservative than R_{M02}. An example of this is shown in Fig. 5 in which the metric with the lowest value is R_{U08} at 21.6 J. The mean response value at 21.6 J has test reaction probability of 1.2%. This is less than the mean response of a 1 out of 60 result which is 1.7%. Furthermore, if the number of tests is high, R_{M02} will be more conservative than R_{U08}. An example of this is in

Fig. 6. where the lowest value metric is R_{M02} at 51.4 J. The upper confidence interval at 51.4 J is at 6.3%. This is less than the upper confidence interval of a 1 out of 60 result which is 7.6%.

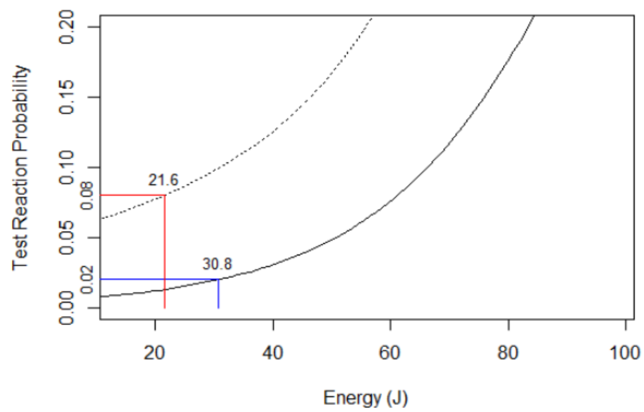


Fig. 5. Slice of regression plot at 60 MPa showing upper 95% confidence interval (single tail) and mean response. R_{M02} and R_{U08} shown in blue and red respectively

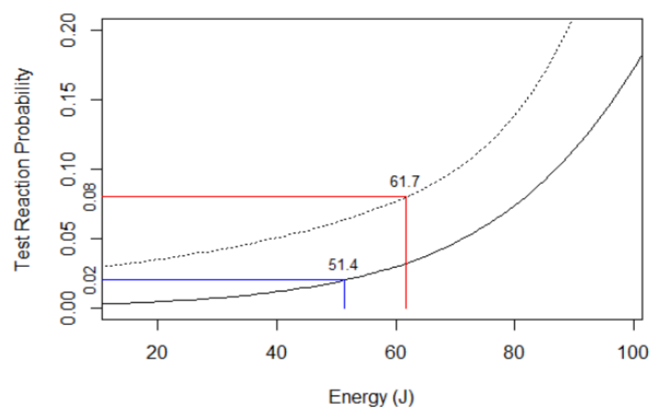


Fig. 6. Slice of regression plot at 10 MPa showing upper 95% confidence interval (single tail) and mean response. R_{M02} and R_{U08} shown in blue and red respectively

If a material was tested at conditions that supported a test reaction probability at 2%, there would be a 67% chance of passing the ASTM G86 with twenty no-reaction observations [$P(n = 20) = (1 - 0.02)^{20} = 0.67$].

III.D. Characteristic Plots

Using a numerical processor, the energy and pressure values at the R_{M02} and R_{U08} metrics were consolidated onto a characteristic two-dimensional plot. Fig. 7 shows the resulting characteristic plot for Vespel® SP-1. It is noteworthy that although the lines are extrapolated to 0 J in Fig. 7, additional testing may reveal that high pressure applications of the material are reasonable so long as mechanical impact energies remain minimal. The gray area defined by the R_{M02} and R_{U08} lines is a space where the likelihood of test reaction probability values defines a distribution that is equivalent or more conservative than the Bernoulli distribution presented in Fig. 2.

The authors compared other materials with data published in Manual 36 and compiled a comparison of characteristic plots for Kel-F® 81 (CTFE), Teflon® (PTFE), and Vespel® SP-21 in addition to the Vespel® SP-1. For comparison, Fig. 8 shows a combined plot of all the characteristic plots for these materials along with annotated lines denoting the extent of the test data. The predictions for all the materials shown in Fig. 7 and 8 extend to a 0 J energy threshold at some pressure. This behavior is not physically expected since a certain minimum amount of impact energy would still be required for ignition even at higher pressures (i.e., nonmetallic materials do not self-ignite when exposed to static, high-pressure oxygen). The prediction trend towards 0 J is artificial and likely related to the lack of data at higher pressures and lower energies. Instead of reaching 0 J, an asymptote would be expected to develop at a minimum ignition energy.

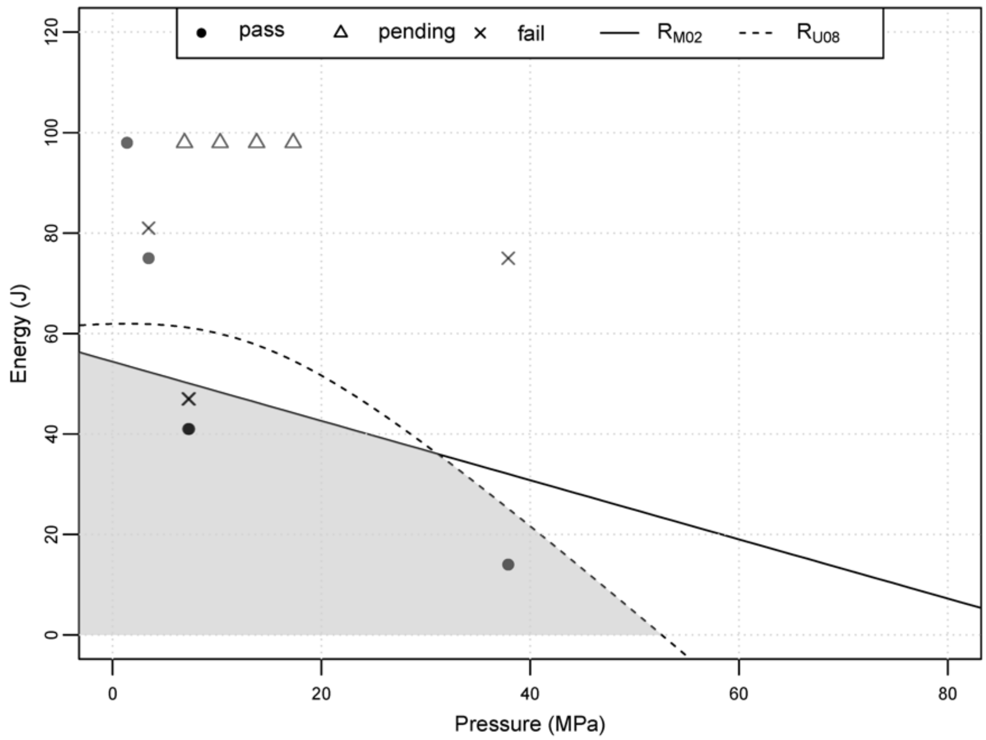


Fig. 7. Vespel® SP-1, GOX MI ASTM G86 Results

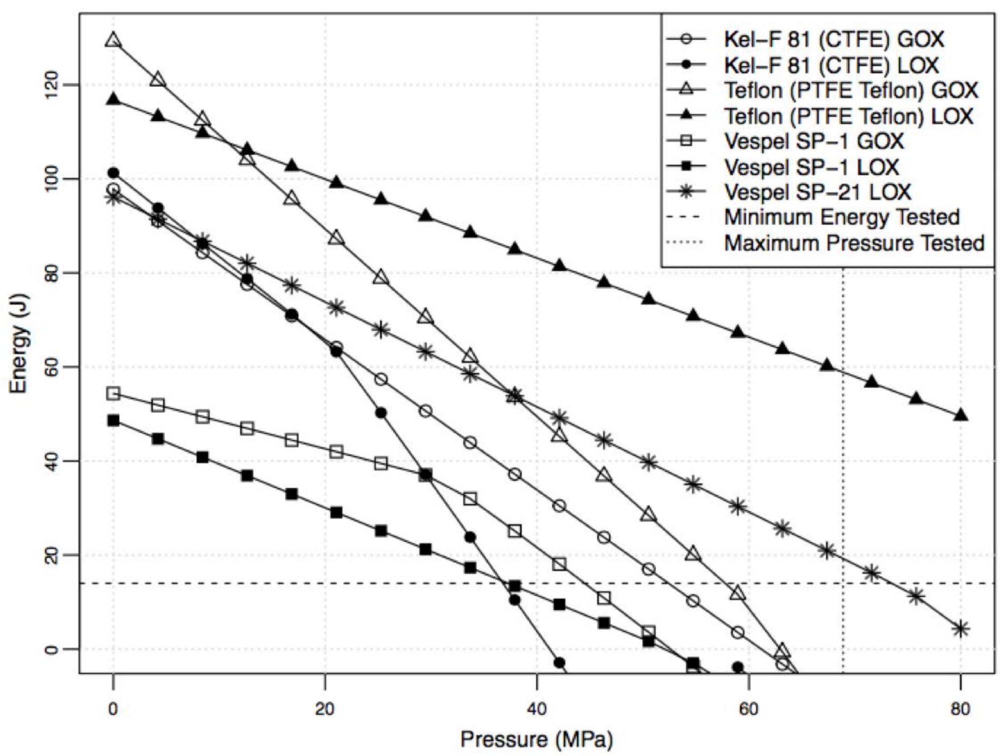


Fig. 8. Combined GOX MI ASTM G86 Results for Kel-F 81 (CTFE)®, Teflon®, Vespel® SP-1, and Vespel® SP-21

IV. CONCLUSIONS

Multivariate logistic regression is useful for analyzing data sets with multiple explanatory variables (i.e. pressure, temperature, energy, etc.) with the condition that the explanatory variables selected must be independent. Plotting R_{U08} and R_{M02} metrics from a multivariate logistic regression analysis on a two-dimensional plot of the explanatory variables is a useful way at determining zones that have test reaction probability distributions (probability ranges) consistent with or more conservative than a 1 out of 60 test result when considering a 95% confidence interval.

This approach utilizes all data (reactions and non-reactions) to describe the behavior of the material and allows designers to pick their material based on the conditions of use and the historical behavior of the materials that have been tested.

These plots allow a discrimination in oxygen compatibility between materials for a range of energy, pressures, and other conditions that might be modelled using the technique presented. Other explanatory variables such as sample thickness and temperature could be useful in making meaningful predictions. Other ignition and flammability tests with pass/fail criteria such as ASTM G74[7], ASTM G124[8] etc. can be analyzed with the same approach.

When considering the analysis of the materials presented, Fig. 8 demonstrates the ignition trends in the data far better than TABLE II or Fig. 3 scatter plots. Fig. 8 also demonstrates that for all materials except Kel-F 81 (CTFE)®, the energy required for ignition is higher in LOX than in GOX, indicating that GOX presents a more severe environment for ignition on average than LOX. Fig. 8 demonstrates a general ranking of the compatibility of the materials analyzed in LOX and GOX with Teflon® (PTFE) being the most compatible.

The analysis method presented demonstrates that a screening approach that considers multiple variables (pressure, energy, sample thickness, temperature, etc.) that are critical in determining a materials sensitivity to ignition in a GOX or LOX environment will likely result in the production of data that can be used to *characterize* a material in those environments. This would provide for a far more robust analysis of the material than a simple pass/fail criterion.

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