A TEST FOR IMPACT SENSITIVITY OF MATERIALS IN CONTACT WITH LIQUID OXYGEN

By B. B. Baber and F. Chang
Southwest Research Institute

ABSTRACT

A description of an impact tester, of the basic ABMA design, and the test procedure used with the tester is presented. Results of impact sensitivity tests performed with the tester are also presented. These results indicate that the impact sensitivity of two different materials is increased significantly when the sample thickness is decreased from 0.040 to 0.020 inch. Also, the repeatability of the test is shown to be within 3 in. (5 ft-lb) based upon the threshold values obtained for one fluid sample.

INTRODUCTION

It has been found that many organic and inorganic materials ignite or explode when exposed to liquid oxygen under conditions of mechanical shock. It is difficult, particularly in the missile industry, to use liquid oxygen without exposing it simultaneously to materials such as sealants, gasket materials, lubricants, and various metals, and also mechanical shock. Therefore, some method must be used to predict the hazard involved with each material.

The mechanisms involved in impact-induced reactions have not been adequately defined; thus, empirical determinations must be used to provide practical design guidance. Unfortunately, the precision of known test methods has, as yet, not proven to be entirely satisfactory. In view of this, Southwest Research Institute, under USAF Contracts AF 33(616)-6232 and AF 33(616)-7223, is currently attempting to improve the repeatability and reproducibility of the test originally devised by the Marshall Space Flight Center (then the Army Ballistic Missile Agency) for the determination of impact sensitivity of materials in contact with liquid oxygen.[1]

In order to minimize the effect of some of the variables involved, it has been necessary to employ specific equipment, and exacting cleaning and test procedures, in this program. These factors will be discussed in general terms in the following sections. The detailed specifications and procedures may be obtained from WADD.

TEST EQUIPMENT

Impact Tester

The basic test equipment used at SwRI to determine the impact sensitivity of materials in contact with liquid oxygen is the ABMA impact tester. However, some minor modifications have been made. Figure 1 presents a photograph of the tester used at SwRI. As shown in this figure, the tester consists of a plummet assembly which is guided in its vertical travel by three accurately aligned guide rails. Friction between the plummet assembly and the guide rails is minimized by the use of six small rollers, three each attached to the upper and lower plates of the plummet assembly. The plummet assembly may be held at any desired height, from 0 to 50 in., by means of an electromagnet which is supported by a fourth rail and whose position on the fourth rail is adjustable. The electromagnet support rail (fourth rail) is calibrated in inches so that the total effective drop distance may be read directly. The plummet may be released by de-energizing the electromagnet, whereupon it is allowed to drop upon a striker pin which is positioned in a specimen cup containing the test sample and liquid oxygen.

(1) Lucas, W. R., and Riehl, W. A., "An Instrument for the Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen," Army Ballistic Missile Agency Report DSN-TR-2-58, October 15, 1958.

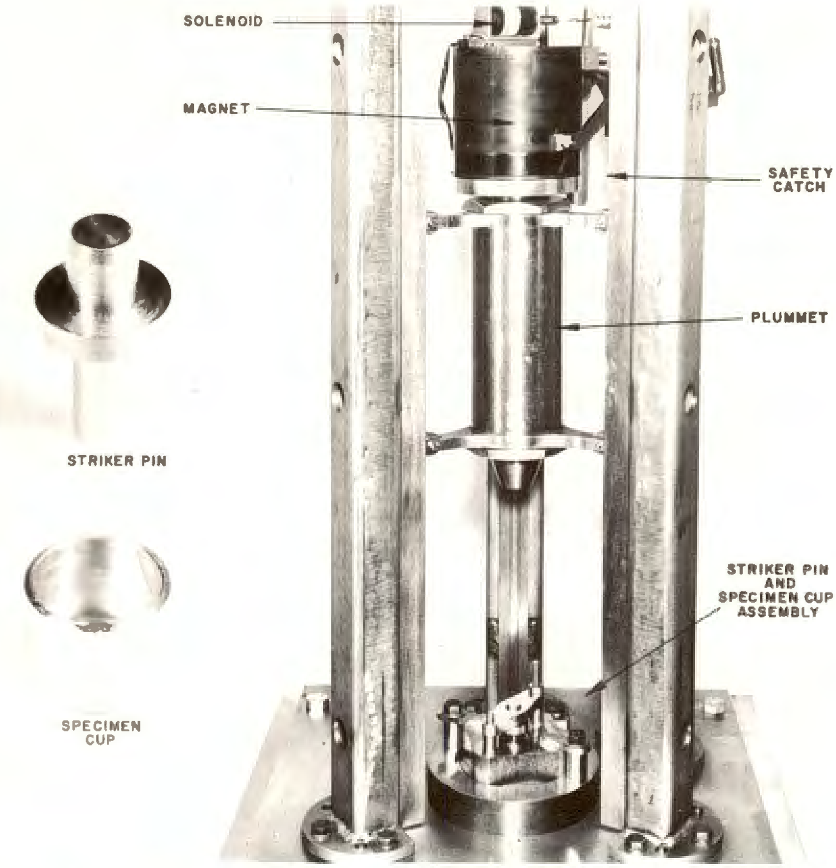

FIGURE 1. PHOTOGRAPH OF ABMA IMPACT TESTER, STRIKER PIN AND SPECIMEN CUP

Electromagnet

The electromagnet used at SwRI to support the plummet requires a 6-volt DC power supply with a power consumption of 0.5 watts.

Plummet Assembly

The plummet assembly weighs 20 ± 0.05 pounds. It is made of stainless steel, except for the top plate which is mild steel because of the magnetic requirement for the plummet support. The top plate is cadmium plated to prevent corrosion. The plummet is designed so that the nose piece may be replaced when it becomes defaced due to its repeated impacts.

Plummet Safety Catch

A positive action safety catch supplements the magnetic support of the plummet except when current is supplied to the safety catch release solenoid just prior to a test drop.

Specimen Cups

Aluminum specimen cups, Figure 2, are used to hold the material to be tested and the liquid oxygen. The dimensions of the specimen cups are closely controlled in order to insure a constant sample thickness when fluid samples are tested. The specimen cup is held in position by a stainless steel holder which in turn is positioned by a replaceable anvil plate bolted to the base plate. A new specimen cup is required for each test drop.

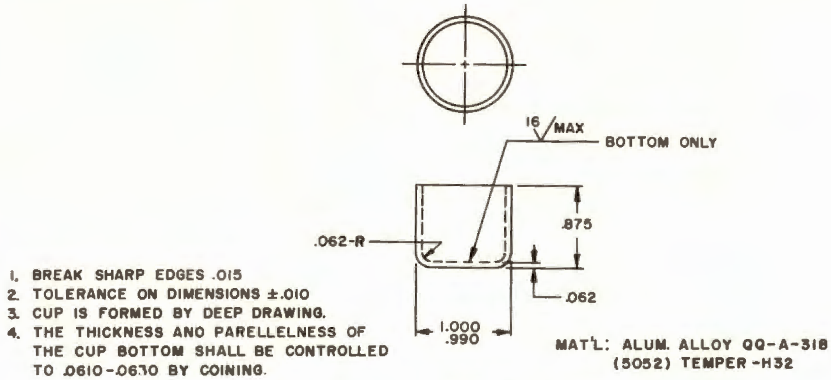

FIGURE 2. SPECIMEN CUP FOR IMPACT TESTER

Striker Pins

Stainless steel striker pins, Figure 3, are used. The striker pin is positioned in the specimen cup by a close fitting guide which is supported by two shouldered pins mounted in the specimen cup holder. The striker pins may be reused after being cleaned, provided they have not been deformed beyond certain specified limits.

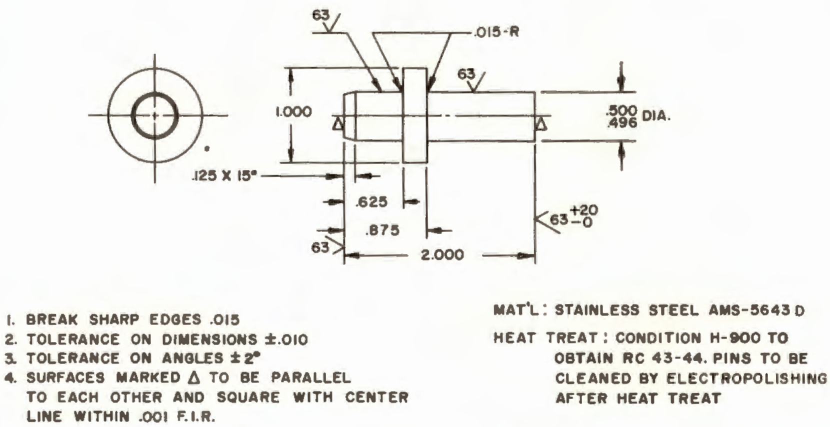

FIGURE 3. STRIKER PIN FOR IMPACT TESTER

The striker pins first used with the tester were made of Type 316 stainless steel; however, excessive deformation of these pins was encountered during impact tests. Striker pins made from three other types of stainless steel (A-286, 17-4 PH, and 17-7 PH) were tested, and all were found to be superior to Type 316 in this respect. The 17-4 PH stainless steel (AMS-5643D) was finally selected as the material for the standard striker pins on the basis of ease of fabrication, simple heat treatment, and having the smallest dimensional change after repeated impacts.

Drop Time Measurement

The drop time of the plummet is measured with a Beckman-Berkeley time interval counter. Separate circuits are used for the starting and stopping of the counter. The starting circuit is established from an insulated contact on the bottom of the electromagnet, through a resistor, a battery, the plummet guide rails, and the plummet (when it is in its raised position against the bottom of the electromagnet). Upon release of the plummet, this circuit is broken, with a subsequent change in voltage across the resistor. This voltage change starts the counter. Upon impact of the plummet, the amplified pulse from a contact microphone, located near the top of one of the guide rails, stops the counter.

TEST PROCEDURE

Equipment Preparation

The impact tester is placed in a test cell, the walls of which are lined with aluminum sheets to minimize the accumulation of dust and to facilitate cleaning. The tester assembly, specimen cups, striker pins, handling equipment, and test cell are all cleaned in accordance with strict cleaning procedures. After the specimen cups and striker pins are cleaned, they are handled with forceps or tongs and kept out of contact with the operator's hands or any other source of contamination.

Sample Preparation

For fluid samples, the volume of test fluid required to give a sample thickness of 0.050 in. in the specimen cup is determined for each fluid by trial and error. The fluid samples are then measured into the specimen cups by volume.

Solid samples are prepared by cutting a disc small enough to fit within the flat portion of the bottom of the specimen cup and larger than the diameter of the striker pin. The standard sample thickness is 0.050 in.; however, if the application product has a thickness less than 0.050 in., samples having the application product thickness are used. If the application product has a thickness greater than 0.050 in. and cannot be cut to form a disc having the required diameter, flat disc samples may be obtained from the manufacturer of the material.

Grease samples are by nature somewhat difficult to prepare to insure the desired constant sample thickness of 0.050 inch. Wright Air Development Division has investigated numerous means of obtaining constant sample thickness with greases and now accepts either of two different procedures. In one procedure, a predetermined amount of the grease sample is placed in the bottom of each specimen cup. The specimen cups containing the samples are then centrifuged for approximately one minute to give a smooth, even sample of the desired thickness. The alternate procedure requires the grease sample to be formed to the desired thickness in a "grease leveling slab," then cooled to a temperature at which disc-shaped samples may be cut with a special cutter then placed in cooled specimen cups.

Precooling Requirement

Prior to the test, all specimen cups and striker pins to be used, the anvil plate, and the specimen cup holder are precooled with liquid oxygen to prevent rapid boiling of the liquid oxygen in the specimen cups during tests. A specific procedure has been adopted for precooling the specimen cups containing the test samples to provide, as nearly as is practical, a uniform frozen sample surface.

Test Drops

After the precooling has been accomplished, a specimen cup containing the frozen test sample is filled with the liquid oxygen and transferred to the cup holder. The specimen cup is topped with liquid oxygen. A precooled striker pin is centered in the specimen cup and held in position by the striker pin guide. The test drop is then made from a preset plummet height. Observations are made and recorded as to whether or not a reaction results from the impact, the nature of the reaction, and the drop time of the plummet.

Reactions

A reaction is defined as an audible report, a visible flash, or other evidence of a reaction such as charring or pitting of the striker pin and/or specimen cup noted upon inspection of the parts after the drop test. Notations are made on the data sheet, if it is evident, whether the reaction took place on the initial impact or on the rebound of the plummet.

Blank Tests

Blank tests are performed in the same manner as the test drops, except that the specimen cup does not contain any test sample, but only liquid oxygen. These tests serve as a check on the effectiveness of the cleaning procedure. Two blank tests are performed during each of the 20 test drops; one at the beginning of each series of test drops; one at the beginning of each series of test drops and one after the first 10 test drops. If no reaction is observed from the blank tests, all the important parts are assumed to be free of contamination and tests on prepared samples are continued. If a reaction is observed, then all specimen cups, striker pins, and the impact tester are recleaned before additional test drops are made.

Determination of Threshold Value

The impact sensitivity of a material is established on the basis of its "threhold value," which is defined as the potential energy (expressed in ft-lb) for the highest of two consecutive test heights, which are 3 in. apart, at which no reactions are observed in 20 test drops.

To determine the threshold value of a material (or test sample), test drops are started at a plummet drop height of 42 in. (70 ft-lb). If no reaction is obtained within 20 test drops, the drop height is increased to 45 in. (75 ft-lb), and test drops are made from this height. If no reaction is obtained within 20 test drops, the test sample is said to have a threshold value greater than 45 in. (75 ft-lb). If a reaction is obtained before completing 20 tests at the 45 in. level, the plummet is lowered to 39 in. and test drops are made from this height. If no reaction is obtained within 20 test drops, the threshold value for that test sample is considered to be 42 in. (70 ft-lb).

If a reaction is obtained before completing 20 tests at the 42-in. level, the plummet is lowered to 21 in. (35 ft-lb) and a series of drops is made from this height. If no reaction is obtained within 20 tests, the threshold value must be between 21 in. and 42 in. and tests must be run at 6-in. intervals down from 42 in. until no reaction occurs within 20 tests. When no reaction is obtained at one of the 6-in. intervals, the plummet is then raised 3 in. from that height and the test resumed. In this manner, the threshold values may be determined to the nearest 3 in. of drop height, or 5 ft-lb of potential energy.

If a reaction is obtained before completing 20 tests at the 21-in. level, the threshold value is less than 21 in. and tests must be run at 6-in. intervals down from the 21-in. level (exactly the same as the aforementioned 6-in. step procedure, except for the heights involved). Nine inches is normally considered to be the lowest required test height.

EXPERIENCE WITH THE ABMA IMPACT TESTER

Measurement of Plummet Drop Time

The plummet drop time for different drop distances was measured, and compared with the computed theoretical time of free fall. Table 1 presents the drop time measurements for drop distances of 10, 20, 30, 40, and 50 inches. It will be noted that the repeatability of the time measurements, in terms of maximum scatter from the average value, was within one percent. The error, or the departure of the average observed drop time from the computed theoretical free fall time, was between 2 and 2.5 percent for all drop distances tested.

Effect of Sample Thickness on Sample Sensitivity

Tests were conducted to determine the effect of sample thickness on sample sensitivity. Two test fluids, with sample thicknesses of 0.020, 0.040 and 0.060 in., were used in this investigation. Twenty test drops were made in each case at drop heights of 42, 30, and 21 inches. A summary of the test results is presented in Table 2, and also in Figure 4. It will be noted that, in general, when the sample thickness was decreased from 0.060 to 0.040 in., there was a relatively small change in the number of reactions obtained. However, when the sample thickness was further decreased from 0.040 to 0.020 in., the number of reactions increased considerably. In impact tests, it is desirable that the sample thickness is such that a small variation from the specified thickness will not appreciably affect the sensitivity. Since only small differences of sample sensitivity were noted between sample thickness of 0.040 and 0.060 in., it appears that the standard sample thickness of 0.050 in. is justified by these results.

Effect of Floated Sample

One of the problems encountered in preparing the sample for impact testing has been the floating of the frozen sample in the liquid oxygen. During the process of precooling the sample to the

TABLE 1. PLUMMET DROP TIME MEASUREMENTS

Drop Test No.	Drop Time, sec 10 in.	20 in.	30 in.	40 in.	50 in.
1	0.2332	0.3297	0.4028	0.4623	0.5193
2	0.2329	0.3302	0.4034	0.4650	0.5196
3	0.2327	0.3301	0.4048	0.4670	0.5204
4	0.2343	0.3300	0.4033	0.4643	0.5200
5	0.2332	0.3300	0.4042	0.4664	0.5221
6	0.2326	0.3301	0.4047	0.4641	0.5241
7	0.2335	0.3300	0.4050	0.4672	0.5194
8	0.2325	0.3298	0.4023	0.4643	0.5244
9	0.2332	0.3298	0.4032	0.4648	0.5195
10	0.2347	0.3292	0.4028	0.4654	0.5254
Avg Drop Time, sec	0.2333	0.3299	0.4036	0.4651	0.5214
Max Scatter, %	+0.60 -0.30	+0.09 -0.21	+0.35 -0.32	+0.45 -0.60	+0.76 -0.40
Computed Time of Free Fall, sec	0.2278	0.3221	0.3945	0.4556	0.5093
Error from Time of Free Fall, %	2.4	2.4	2.3	2.0	2.3

TABLE 2. SUMMARY OF IMPACT TEST RESULTS ON TWO FLUID SAMPLES TO DETERMINE THE EFFECT OF SAMPLE THICKNESS ON IMPACT SENSITIVITY

Sample Thickness, in.	Drop Hgt, in.	No. of Tests		No. of Reactions CG-18	CG-35
0.060	42	20		2	2
	30	20		1	2
	21	20		0	0
			Total	3	4
0.040	42	20		4	1
	30	20		1	0
	21	20		2	0
			Total	7	1
0.020	42	20		12	10
	30	20		5	10
	21	20		4	3
				21	23

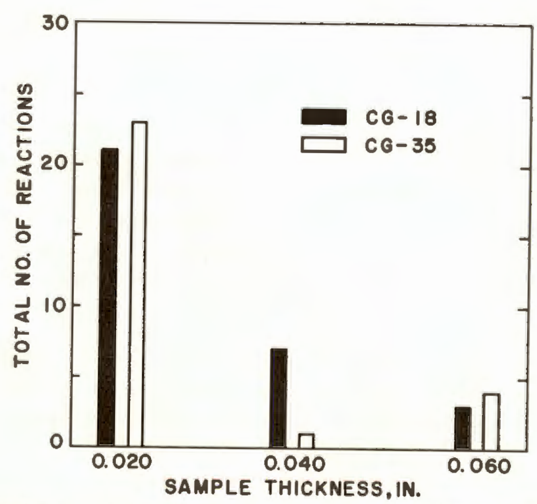

FIGURE 4. EFFECT OF SAMPLE THICKNESS ON SAMPLE SENSITIVITY

liquid oxygen temperature, the sample in the specimen cup will invariably develop cracks, but will usually maintain a relatively smooth surface. When liquid oxygen is poured into the cups containing the frozen samples, some of the frozen sample may separate from the bottom of the specimen cup and float in the liquid oxygen, leaving a void spot in the frozen sample. The area of the void spot varies from a small speck or gap to almost half of the frozen sample surface. Sometimes the entire frozen sample will crack into small pieces and float in the liquid oxygen. These floated samples are normally discarded, and additional samples are prepared if necessary. However, in order to determine what effect floated samples might have on the impact test results, comparative data were obtained with floated samples using two test fluids which had previously been tested using normal samples. Table 3 presents the results obtained. It will be noted that, in nearly every instance, a larger number of reactions was obtained with the floated samples than with the normal samples. It is believed that the effect of floated samples shown here may be a part of the reason that the reproducibility of the impact test is not as good as desired, and, of course, it may appreciably influence the repeatability of the test if the operator is lax during sample inspection.

TABLE 3. COMPARISON OF IMPACT TEST RESULTS WITH NORMAL AND FLOATED SAMPLES

Sample Thickness, in.	Drop Hgt, in.	No. of Tests	Number of Reactions: Normal CG-18	Normal CG-35	Floated CG-18	Floated CG-35
0.060	42	20	2	2	1	3
	30	20	1	2	4	1
	21	20	0	0	6	1
0.040	42	20	4	1	6	8
	30	20	1	0	7	6
	21	20	2	0	6	3

Repeatability of Threshold Value Determinations

The repeatability of threshold value determinations has been determined on only one fluid which has a measurable threshold value above 12 in. (20 ft-lb). Table 4 presents the impact test results obtained on three different occasions using one fluid sample at three different drop heights. It will be noted that the threshold values obtained for this sample vary only 3 in. (5 ft-lb).

TABLE 4. SUMMARY OF IMPACT TEST DATA SHOWING REPEATABILITY OF THRESHOLD VALUE

Drop Hgt, in.	Ratio of No. of Reactions to No. of Test Drops: May 1960	June 1960	October 1960
24	1/3	1/4	1/8
21	0/20	1/14	0/20
18	0/20	0/20	0/20
Threshold Value, in	21	18	21
Threshold Value, ft-lb	35	30	35

Repeat tests have also been conducted on other samples having threshold values greater than 45 in. (75 ft-lb), and these tests have shown good repeatability by giving no reactions. It is planned that more data will be obtained in the future on additional samples with intermediate threshold values as they become available.

Threshold Values of Fluid Samples

The threshold values of a number of different fluid samples have been determined using the ABMA impact tester. These samples were tested in an effort to find a fluid which may be used as a standard reference fluid for the impact sensitivity test. For an ideal reference fluid, the fluid should have a medium threshold value (near 30 in., or 50 ft-lb), a stable threshold value during storage, and a resistance to excessive floating after being frozen in the specimen cup.

Twelve sample fluids were selected by WADD for this investigation. A summary of the threshold values obtaining for these samples is presented in Table 5. Of the fluids tested, only the inhibited methyl phenyl silicone appears to approach the threshold value requirement of a reference fluid. In order to check on the threshold value after storage, additional sets of tests were run on this fluid after periods of approximately one month and six months. The data obtained are presented in Table 4. From these data it is apparent that the threshold value has not changed significantly within the six-month period.

TABLE 5. THRESHOLD VALUES FOR DIFFERENT FLUID SAMPLES

Sample Code	Description	Threshold Value: Height, in.	Threshold Value: Potential Energy, ft-lb
CG-18	Di-2-Ethylhexyl Azelate	12	20
CG-35	Inhibited Methyl Phenyl Silicone	21	35
CG-5	Chlorinated Phenyl Silicone	<9	<15
CG-34	Chlorinated Methyl Phenyl Silicone	9	15
CG-32	Pentaerythritol Ester	<21	<35
CG-58	Pentaerythritol Ester	<21	<35
CG-59	Pentaerythritol Ester	<21	<35
CG-63	Trimethylpelorgonate Ester	<21	<35
CG-9	Silphenylene (1958)	9	15
CG-62	Silphenylene (1960)	12	20
CG-61	Bis (Phenoxy Phenoxy) Benzene	<9	<15
CG-26	Halogenated Oil	>45	>75

CONCLUSIONS

Experience with the basic ABMA design impact tester at SwRI has been satisfactory. The selection of a new striker pin material has eliminated the need for frequent replacement of the striker pins. The expendable specimen cups are inexpensive. These factors combine to provide a test apparatus that is mechanically reliable and inexpensive to operate.

The effect of small sample thickness variations on sample sensitivity does not appear to be large in the range of the standard sample thickness (0.050 in.). However, sample thicknesses in the order of 0.020 in. may significantly affect the test results.

Two of the factors which may significantly affect the precision of the test are the condition of the frozen sample (whether a small part of it has floated or not) and the interpretation of the definition of a reaction.

On the basis of the data obtained to date, the repeatability of the sensitivity of a material, in terms of the threshold value, appears to be good (within 5 ft-lb).

Additional impact test results on materials with intermediate threshold values are needed in order to substantiate the repeatability of the test. Tests will be conducted in the near future in an effort to fulfill this need.

ACKNOWLEDGMENT

The work reported herein was conducted under the sponsorship of Wright Air Development Division, under Contracts AF 33(616)-6232 and AF 33(616)-7223. The project engineer is Mr. G. A. Beane.