New Device for Evaluating Reactivity of Materials With Propellants

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Over the past several years, increased interest in the detonation characteristics of materials with highly reactive propellants has resulted in the need for a reliable, inexpensive method for determining these characteristics. This paper discusses the investigation that the Reaction Motors Division of Thiokol Chemical Corporation conducted to develop a simple drop weight type of tester. The discussion covers the problems associated with some early testers and the important features that were incorporated into the present RMD tester. Photographs of the prototype and final tester are presented. The critical parts of the tester are discussed in detail, and some of the data that have been accumulated with this equipment on liquid propellants are presented graphically. Information is also presented which indicates the reliability and sensitivity of the equipment. An oil contamination system is described which should be very helpful in exploring the general impact problem as it concerns the industry, in that it will 1) permit data conversion between different style testers over the complete sensitivity range, and 2) serve as a standard for tester calibration.

With the advent of the rocket engine and missile into industrial prominence, the need for development of higher performance propellants has been a necessity. The latter development has resulted in a wide variety of propellants, which in many cases are highly reactive. The propellants are reactive not only with other propellants but also with metal, ceramic, plastic, and liquid materials with which they may come into contact during construction or operation of the rocket engine or missile.

Several years ago, when the rocket engine industry began using highly reactive oxidizers such as liquid oxygen and concentrated hydrogen peroxide, the basic method of evaluating the compatibility of materials was to immerse the material or component in the propellant, to determine reactivity through decomposition of the propellant, or to examine the effect of the propellant on the material surface and drop a weight of some magnitude on the material when in contact with the propellant. Most of the concern with detonability was centered around liquids, greases, or paint systems. However, as more experience was gained, it became evident that many of the plastic, elastomeric, and metal materials were also susceptible to detonation when in contact with liquid oxygen, hydrogen peroxide, or the newer inhibited red fuming nitric acid. At the same time, it became evident that just dropping arbitrary weights on pieces of material in propellant was not good enough to determine the detonability characteristics.

Early in 1953, the Reaction Motors Division began a study of detonability characteristics on solids such as silicone rubber, Teflon, natural rubber, and some metals and ceramics. At the same time, a program was initiated to evaluate the various types of impact testers available to the industry and to select one that was safe, reliable, and relatively inexpensive to build and operate. Most of the test equipment in use, which included the Reaction Motors Division impact tester, was bulky and awkward and usually required a separate room where testing personnel would not be exposed to the abnormally large blasts resulting from impact sensitive materials. Another objection to the equipment was the poor reproducibility of data from the drop weight testers. The use of striker pins having a relatively large area was investigated, and it was found that the pin did not strike normal to the surface. Generally, the larger the area to be covered during impact, the greater the possibility of misalignment that yielded very erratic test data. Also, with large striker areas, it was necessary to use large plummet weights (20 to 70 lb).

The card gap test (1)[2] for shock sensitivity measures the minimum hydrodynamic shock necessary to produce propagation of a high order detonation in a specified diameter steel pipe. A test of this type had been used for about 15 yr in the testing of the sensitivity of solid and liquid propellants toward shock waves. However, for the present purposes, the tests appeared to be unsatisfactory for two reasons: 1) large quantities of detonable materials had to be mixed for each test, and, consequently, personnel within the immediate area of the tester would be in a hazardous area; and 2) the test was very time consuming. Furthermore, it was felt that, for solid materials, this type of test did not apply a sufficient number of parameters associated with a mechanical impact in actual service.

Some other testers, such as the JANAF (2) impact tester, are not, at the writing of this article, capable of testing solid material with cryogenic propellants. Their design precludes the use of a machine similar to the RMD tester since there is no way to replenish the propellant supply in the sample area. The latter tester was originally designed to evaluate propellants by themselves rather than solids with these propellants.

Equipment Development

It was decided finally that a drop weight type of tester be developed. It was further considered mandatory that the test require only a few drops of propellant and sample so that optimum safety conditions would be realized. Furthermore, it was recognized that, with small contact areas between striker pin and the anvil, the effect of eccentric loading on the sample during impact could be minimized.

A drop weight tester was designed with several important features in mind:

1) The rail system was aligned accurately to guide the plummet on a perpendicular course to the anvil.

2) The bed plate and supporting structure were constructed of materials with low damping capacity.

3) Arrangements were made for accurate temperature control.

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[2] Numbers in parentheses indicate References at end of paper.

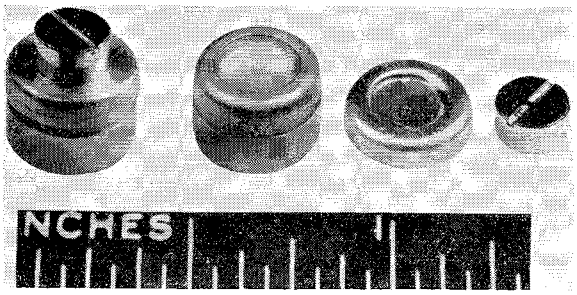

Fig. 1 Sample cup assembly, cup, cap, and striker pin

4) A shatterproof, transparent casing enclosed the equipment to protect personnel observing the test, as well as to exclude dust.

5) In addition to the forementioned, a most important item was the design of the sample cut assembly that holds the propellant and the test sample. A simple, tiny cup was used to handle liquid propellants. Propellant and sample were placed into the shallow cup. The cup was covered with a thin sheet metal cap, and a striker pin was centered in a recess on the cap. The detail parts and complete assembly are shown in Fig. 1. For liquids with higher boiling points, such as hydrogen peroxide, the sample area is covered by the cap. As the plummet hits, a high pressure is instantaneously created, along with mechanical working of the sample as the striker pin is projected into the sample below.

When testing materials with liquid oxygen, a cap having small holes is used to admit liquid oxygen as well as to vent gas bubbles. Liquid oxygen is poured into a reservoir surrounding the anvil until the level is somewhat above the striker pin. The plummet is dropped when the cup assembly is cooled to the temperature of liquid oxygen.

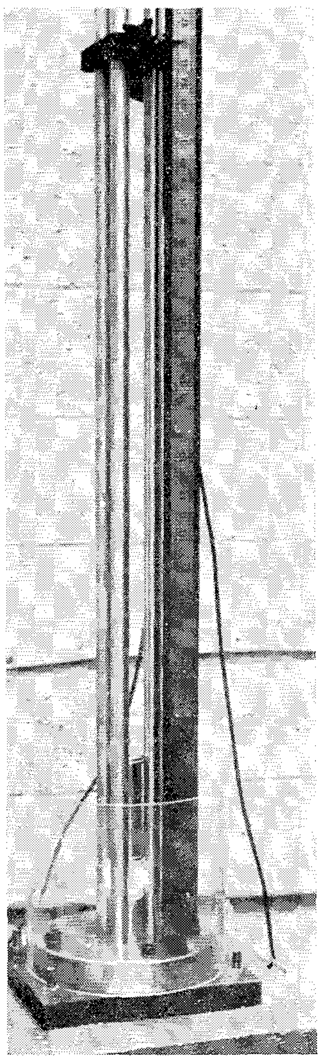

Fig. 2 **RMD impact tester in its final design, with detonation taking place**

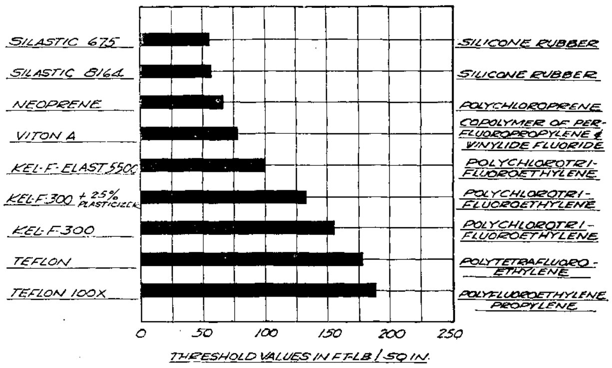

Fig. 3 Shock sensitivity threshold values in LOX (nine plastics)

The sample cup is designed with a slight curvature for two reasons: 1) it compensates for small misalignments of the striker pin; and 2) it traps liquid beneath a solid type of material so that both hydrodynamic pressure and mechanical work are created at the same time.

Positive reactions are recorded by audible detonation, flashes, and the presence of burning in the cup. The shock sensitivity threshold value of any material is determined by starting the testing at a maximum of 36 in. and lowering the starting position of the weight by a few inches with each successive drop (if a positive reaction occurs) until the highest value is obtained, where 10 successive drops may be made without causing a detonation or reaction. Thus, inert materials have very high threshold values, whereas active materials have very low threshold values. Data are recorded in foot-pounds per square inch of contact area and calculated from the striking surface of the striker pin. In determining detonability, the sensitivity in drop height of this impact tester is ± 1 in. or ± 11 ft-lb/in.[2]

As mentioned before, low damping capacity materials have been used for the tester bed plate, and the same applies to the cup and striker pin. High damping materials would tend to absorb the shock of impact by the plummet and striker pin and, consequently, lessen the sensitivity of the materials to detonate. Low alloy high strength steel in the hardened condition has been used as base material. For compatibility purposes, the low alloy steels have been plated with chromium. Recently, tests run in the Reaction Motors Division laboratory, indicate that the 300 series stainless steels will be satisfactory for the cups.

The unit in its final design is shown in Fig. 2. For convenience, a 2-kg plummet is used, except for occasional checks on the equipment. Other weights as low as 500 g have been used with no change in the resultant test values.

The results of testing some plastics and liquids with liquid oxygen are shown in Figs. 3 and 4, and the results of testing

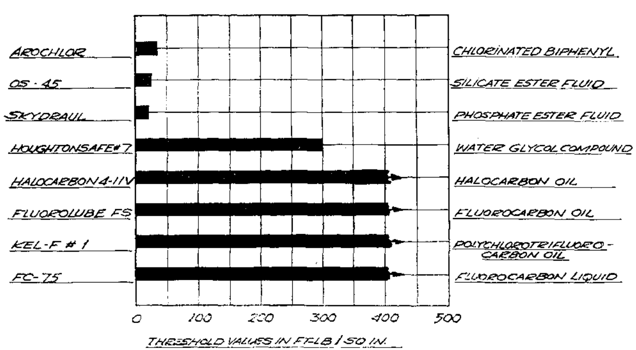

Fig. 4 Shock sensitivity threshold values in LOX (eight hydraulic fluids)

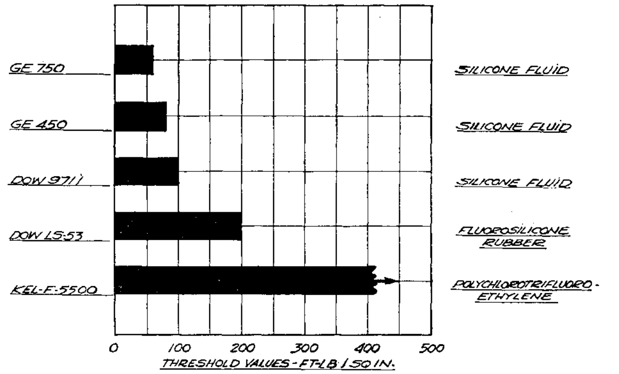

Fig. 5 Elastomer materials vs shock sensitivity threshold values in 90% H_2O_2

some plastics with 90% hydrogen peroxide are shown in Fig. 5. In general, it has been found that the sensitivity of detonable materials increases twofold or more when testing in liquid oxygen than in 90% hydrogen peroxide.

In a test program that was performed in the Reaction Motors Division laboratory, it was attempted to determine the sensitivity of the equipment by increasing the contaminant in what previously had been determined to be a completely compatible material. In this program, several blends of Halocarbon 4-11 V and a polyalkylene glycol oil were tested for impact sensitivity with liquid oxygen. The blends ranged from 5 to 100% Halocarbon oil.

The results of this impact sensitivity study are presented in Fig. 6. The threshold value having once been established, a series of 10 tests were performed for each point on the curve. Note that, as the Halocarbon oil is added, the mixture becomes less sensitive to impact (the threshold value rises) and that a straight line energy relationship results when plotted on a semilogarithmic scale. The straight line holds only within the range shown. As the Halocarbon percentage is lowered or increased at each end, the threshold value respectively drops or increases sharply. These data have been reproduced many times over the past three years, even when the solutions were stored for several months prior to testing. Other data, some of which have been presented in this paper, have also been retested with repetitive results.

Conclusion

In the past few years, a considerable quantity of data on this equipment has been accumulated. These data have been used by the Reaction Motors Division design personnel as design data. Experience with the Reaction Motors Division rocket engines has proved that the data can be used as one would use the tensile properties of materials for design purposes, and so, though a material might have a threshold value for detonation of 300 ft-lb/in.^{2} in liquid oxygen, it does not necessarily mean that the material cannot be used in liquid oxygen.

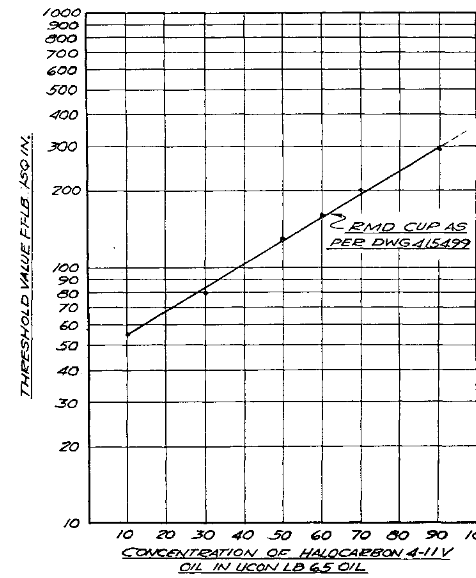

Fig. 6 Threshold values vs concentration

In general, the author's experience with the tester has indicated its reliability and reproducibility and has allowed the use of a greater variety of materials with reactive propellants than might otherwise be used.

With the Halocarbon oil system described previously, it is possible to obtain any threshold value within the range shown. This system should be very helpful in exploring the general impact problem. It will permit data conversion between different styles of testers over the complete sensitivity range, and it will serve as a standard for tester calibration.

At the present time, many types of testers are being used, and a mass of widely varying data is being accumulated. Some effort by the government and by industry is being made through the Aerospace Industries Association and the Aeronautical Systems Division of the Air Force Systems Command to establish standards for procedure and to specify testing equipment required.

It is the author's recommendation that a tester and an alternate, if favorable comparable data for the alternate exist, be established as quickly as possible as standards for the industry so that data between various agencies can be correlated properly for this very important phase of materials compatibility testing.

References

1 "Liquid propellant test methods, test number 1, card gap test for shock sensitivity of liquid monopropellants," Liquid Propellant Info. Agency, Appl. Phys. Lab., Johns Hopkins Univ., Silver Spring, Md. (March 1960).

2 "Liquid propellant test methods, test number 4, drop-weight test," Liquid Propellant Info. Agency, Appl. Phys. Lab., Johns Hopkins Univ., Silver Spring, Md.