EXPLOSIVE SAFETY TESTING AT NEW MEXICO TECH: THE BROWER ADIABATIC COMPRESSION TEST

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ABSTRACT
A new hazard test has been developed at the Research Center for Energetic Materials at New Mexico Tech in which bubble compression ignition of liquid or solid material is simulated under well defined conditions of temperature and pressure. Small samples of the test explosive are placed in a piston-cylinder apparatus and a drop weight, usually 1 kg, is used to initiate rapid compression ignition. A series of drop heights can be used to vary the ignition conditions. The rebound energy imparted to the drop weight from the ignition is measured. The temperature and pressure conditions, typically in the ranges 800-2000 K and 200-1200 atm, are determined from the compression ratio and nature of the gas in the chamber. Chemical analysis of the residual explosive or product gases can be performed to elucidate the reaction mechanism of the ignition.

INTRODUCTION
Adiabatic compression of gas bubbles is generally accepted as a potential ignition source of liquid explosives and propellants. Indeed, gas bubbles have been known as primary sensitizing agents of liquid energetic materials since the early days of nitroglycerin production. Nitromethane, for example, can be made sensitive to a No. 8 cap by the addition of 1.5% of glass bubbles. Bubble compression is relevant to hazards in such varied practical applications as hydrazine transfer to space vehicles, operation of liquid propellant guns, and pumping of commercial emulsion explosives. Initiation of solid explosives in gun projectiles by compression of voids, the so-called setback problem, has been the subject of several investigations.

No single drop weight apparatus for measuring the sensitiveness of liquid explosives is generally used. The NATO AOP-7 manual gives a small scale test which has been discussed in terms of its use for hazard classification of liquid propellants. This test is performed using an ASTM test apparatus in which a small amount of liquid is placed in an O-ring in the bottom of a cylindrical cavity and covered with a steel diaphragm. A 2 kg weight is dropped from various heights onto a steel ball in contact with a steel striker on top of the diaphragm and sample. The air trapped inside the O-ring with the sample is heated by compression to ignite the sample. A positive event is indicated by a ruptured diaphragm.
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The conditions that cause ignition of samples in this apparatus are not well established, and events are only classified as go or no-go ignitions. A similar "Liquid Explosive Impact Test" is listed in Reference 7.

Several authors have analytically studied the process of ignition by compression of voids using numerical simulation techniques\(^3\). For voids contained in solids, the physical properties of the material are found to be important since heating also occurs (in addition to gas phase compression heating) as the result of viscoplastic work, inviscid plastic work, and/or condensed phase compression.

A new compression ignition test has been developed by Brower and coworkers\(^8\) in which small samples of liquid (or solid) are subjected to potential ignition by hot compressed gas at pressures up to about 1200 atmospheres and temperatures up to or greater than 2000 K. Conditions can be varied by the choice of gas and the drop height. The energy release from partial or complete ignitions can be measured and product samples can be taken for chemical analysis. Heating mechanisms other than by gas compression are eliminated since the sample is not significantly deformed. This test and some results are discussed below.

**EXPERIMENTAL**

The apparatus consists of a hardened steel O-ring sealed piston and cylinder of 1.3 cm bore with a small diameter side arm tube for filling with gases other than air and for withdrawing product samples. Figure 1 shows a schematic diagram of the apparatus. This assembly is held in an aluminum block (which can be heated) on top of a lead brick. A 1 kg drop weight from heights of up to 120 cm is used to drive the piston. The drop weight has a mechanical catch which arrests its motion at the point of maximum rebound height. A rebound of less than 4 cm is obtained from tests on inert samples. The rebound height from test sample ignitions can be used to calculate the mechanical work released by the sample. Expansion of the gas upon rebound of the piston quenches the chemical reactions and limits the duration of the event to about 500 \(\mu\)s.

The experimental test conditions have been determined using a variety of diagnostics. The compression ratio is determined from the known initial volume of the system and measurements of the minimum clearance between the piston and cylinder. This has been measured by placing a small lead sphere inside the apparatus and measuring its final thickness. The compression ratio was also measured using a magnetic velocity sensor on the weight (differentiated to give acceleration). Pressures derived using the two methods agreed\(^9\) within 6%. Assuming all of the work done by the piston goes into heating the gas, the ratio of initial and final volumes can be related to final pressures and temperatures.
using the ideal gas law. The following relationships are obtained:

\[ P_f = P_i \left( \frac{V_i}{V_f} \right)^\gamma \]

\[ T_f = T_i \left( \frac{V_i}{V_f} \right)^{\gamma^{-1}} \]

where the subscripts \( i \) and \( f \) refer to initial and final (maximum compression) states and \( \gamma \) is the ratio of heat capacities, \( C_p/C_v \), of the gas (mixture). It can be seen from the exponents that the temperature ratio increases less rapidly with increasing compression ratio than the pressure ratio. For argon, air, \( \text{C}_2\text{H}_6 \), and \( \text{SF}_6 \), \( \gamma \) has values of about 1.67, 1.40, 1.25, and 1.09 respectively. Figure 2 shows final pressure and temperature ratios calculated using the above equations for air compression at various \( V_i/V_f \) ratios. Figure 3 shows the difference in final temperatures obtained for compression of air at two initial temperatures, 298 and 398 K. The different temperatures obtained using three gases of different heat capacity ratios are shown in Figure 4. It can be seen that polyatomic explosive vapors with \( \gamma \) approaching one greatly reduce the heating by adiabatic compression, whereas a monatomic gas such as argon gives maximum heating. This effect can be used to advantage in selecting test conditions, but can be a complicating factor if test sample vapors alter the \( \gamma \) of the gas atmosphere in the apparatus. When necessary, the apparatus and sample are cooled to reduce the sample vapor pressure and so to achieve high final test temperatures.

In practice, the compression ratio for a given test series can be measured as a function of drop height from lead shot measurements. These data are then used to calculate maximum pressure and temperatures for given initial conditions. A compression ratio of 100, for example, gives 630 atm and 1880 K when air is used in the apparatus. Test samples consist of \( \approx 10-20 \) mg of liquid (or solid). The Bruceton up/down method can be used to determine the drop height for a given threshold rebound height or degree of sample decomposition.

**RESULTS**

This apparatus and technique were initially developed for a study of nitromethane decomposition and the effects of various additives\(^8\). Partial ignitions were obtained for neat nitromethane using drop heights as small as 20 cm, with complete consumption of the small sample when the drop height was about 70 cm. Some nitromethane data\(^8\) are shown in Figure 5 which shows that a variable response from partial ignitions is obtained. This ability to get a graded response is a distinct advantage over tests in which only a go or no-go is determined, since it makes it possible to study the early stages of the ignition process and allows a better differentiation of the sensitivity of various materials.

Figure 6 shows test results\(^9\) for solid and liquid TNT (melting temperature = 81°C) obtained at constant drop height. Not only does the fraction of tests that are positive ignitions increase with increased temperature, but the average rebound height also increases.
(12 cm rebound at 70°C and 35 cm rebound at 90°C). This can be compared to normal drop weight impact tests on TNT as a function of temperature where the impact sensitivity greatly decreases at temperatures above 75°C because the soft or melted sample is not heated as efficiently upon mechanical impact. The data from the compression ignition test show that, in fact, TNT is more sensitive to ignition when hot (as is reasonable).

Another example application of this test procedure to solid material is illustrated in Figure 7 for mixtures of ammonium perchlorate with two different hydrocarbon fuels. The drop height, gas, and initial temperature were held constant in these tests and the composition of the samples was varied. A stoichiometric mixture of AP with hydrocarbon will contain about 9 weight percent hydrocarbon. The data in Figure 7 indicate that fuel lean (less fuel than stoichiometric) mixtures are more sensitive to ignition by hot compressed air than the stoichiometric mixture. The maximum pressure and temperature conditions for these tests were about 2050 K and 700 atm. Figure 8 shows additional rebound data for three mixtures of AP with hydrocarbon taken at various initial drop heights. The shape of the rebound data with drop height is seen to be similar to that of the temperature obtained as a function of drop height (c.f., Figure 2 and 3).

CONCLUSIONS
A new hazard test has been developed in which ignition from hot, rapidly compressed gas, such as occurs in bubble or void collapse, of liquid or solid material is simulated under well defined conditions of high temperature and pressure. Small samples of explosive are tested in a piston-cylinder apparatus using a drop weight to initiate rapid compression ignition. A series of drop heights and compression of different gases can be used to vary the ignition conditions and to obtain a gradual increase in response with increasing drop height. The rebound energy imparted to the drop weight from the ignition can be used to measure the energy release from the sample. The apparatus is simple, inexpensive to build, and can be used in the laboratory environment without unusual safety precautions.

This test procedure and apparatus give much greater control and knowledge of the conditions to which samples are subjected than other impact tests for liquids. Having knowledge of the pressure, temperature, and duration of the exposure of the energetic material to hot gases allows better analysis of the results and possibly extension to other situations of larger scale.
REFERENCES


FIGURE 1. ADIABATIC COMPRESSION APPARATUS
FIGURE 2. CALCULATED TEMPERATURE AND PRESSURE RATIOS FOR AIR COMPRESSION.
FIGURE 3. CALCULATED FINAL TEMPERATURES FOR AIR COMPRESSION

Ti = 398 K
Ti = 298 K
FIGURE 4. CALCULATED FINAL TEMPERATURES FOR COMPRESSION OF DIFFERENT GASES
FIGURE 5. REBOUND HEIGHTS FOR NITROMETHANE IGNITION
Data adapted from Reference 8. Sample size = 12 μl.
Energy release at 70 cm drop height is ca. 1500 J/g.
FIGURE 6. COMPARISON OF LIQUID AND SOLID TNT IGNITIONS
Drop height = 40 cm. Data from Ref 9. The average rebound height for ignitions at 70°C was 12 cm compared to 35 cm at 90°C.
FIGURE 7. REBOUND HEIGHTS FOR AP/HYDROCARBON MIXTURES. Drop height = 90 cm; gas = air at ambient temperature and pressure.
FIGURE 8. REBOUND HEIGHTS FOR AP/HYDROCARBON MIXTURES. Gas = air at 100°C and ambient pressure. 
○ = 1% fuel; △ = 2% fuel; □ = 4% fuel.
Figure 10. Radiograph of LO₂ jet into the pool.
Figure 11. Mixing zone impingement contours for LN$_2$ into LH$_2$. 
Figure 12. Jet penetration velocity after impingement.
Figure 13. Mouth temperature variation for three similar tests.

Figure 14. Mass flowrate at the mouth of the dewar for three similar tests.