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Full Paper

A Device for Testing Thermal Impact Sensitivity of High Explosives

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Abstract

A drop-weight instrument is described to investigate the impact sensitivity of high explosives at elevated temperatures. This test is typically performed to discover potential safety aspects of either newly synthesized materials of unknown behavior. Normally, drop-weight impact sensitivity tests are conducted at room temperature, since high explosives are often stored or handled at room temperature. The instrument described here has the capability of heating the samples from ambient to 300 °C prior to impact. The thermal impact sensitivities of PETN, TATP, HMX, and silver azide (AgN_3) as a function of temperature are obtained. The phase transition of HMX can clearly be observed by a significant decrease in the impact height in situ.

Keywords: Drop-Weight Machine, HMX, PETN, Thermal Sensitivity Analysis

1 Introduction

In order to test the sensitivity of the potential explosive compounds, they must run through a series of small-scale safety tests to determine the sensitivity to various stimuli, such as friction, static spark, and impact [1]. Impact testing is a direct and simple step in evaluating the sensitivities of high explosives and is used to determine the response of an explosive when it is impacted by a moving mass. This test simulates impact conditions such as in processing operations, wherein an explosive is subjected to a collision between moving components of the processing equipment. The impact test is also relevant in everyday handling of energetic materials. Generally, explosive safety testing results on one material are qualitatively compared with other materials used in the same test at same conditions. An eventual goal would be to correlate the results from a variety of small-scale safety tests together to aid in the prediction of bulk properties.

Ignition of energetic materials include several possible mechanisms, such as friction, pore collapse, and adiabatic shear postulated by Field et al. [2]. These mechanical processes focus on localized heating to initiate the explosives. Bourne and Milne [3] proposed three main features of the collapse that provide a means for ignition: formation of a high-speed jet and elevated velocities in the convergent flow around the cavity; the shock-heating caused by an asymmetric collapse of jet-impact; and the compression of the gaseous content of the cavities. The hot-spot theory [4] has been widely accepted that detonation in solid explosives is facilitated by void collapse [5,6]. The collapse of cavities within a material is one of the most important of the hot-spot production mechanism. The size of the voids showed a very significant effect on ignition and ensured reactions. A burning front of explosive may be quenched or may accelerate so that a transition to detonation may occur according to the confinement of the material. The output performance of the explosive has a significant dependence on the microstructures of the explosives. Usually, the cavity size with diameter ranging from micrometer to hundreds of micrometer may be added to commercial explosives to render them more sensitive, which is also a consideration for adding gas bubble in the commercial explosives. For example, Frey [7] reported that the void with size of 0.1–10 μm form critical hot spots from which propagate the reaction in a time duration of 10 μs to 1 ms at room temperature. Therefore, sensitivity depends on not only the chemical properties but also the heterogeneity of the material and these inhomogeneities may be affected by temperature.

In the drop-weight testing, the lowest drop height where you get a "GO" is considered the "reaction threshold" and typically involves several experiments to determine [8]. In order to quantify the sensitivity of the explosives, Moore and Ray [9] described a statistical method for sensitivity and performance analysis of complex computer simulation experiments to calculate Dh50, which is "50% impact height" of the "GO" sound level of reaction, to characterize the explosives. However, the most used method is Bruceton method described originally by Dixon and Mood [10], which relies upon the first stimulus and the step size. In this method, a series of stimulus (height) have been recorded with equal log spacing of the step size. The results are tabulated and analyzed though Bruceton analysis to provide estimates of the mean and standard deviation.

Up to date, a variety of impact devices have been used in the explosives sensitivity analysis. For example, the Steven impact test [11] results have increased the fundamental knowledge and practical predictions of impact safety hazards for confined and unconfined explosive charges. The procedure of the Steven impact test is that a gas gun accelerates the steel projectile head to hit the explosives, which was confined in a thick steel plate on the impact face. A Teflon ring around the explosive provides radial confinement. The overpressure transient data [12], coming from the overpressure gauge, are used to calculate an equivalent point source energy for samples that react. Also, ERL Type 12 Drop Weight Sensitivity Apparatus [8] has been successfully applied in energetic materials, such as pentaerythritol tetranitrate (PETN), cyclotrimethylenetrinitramine (RDX), Comp-B3, and trinitrotoluene (TNT), by Lawrence Livermore National Laboratory in 1956. In this test, a small sample (~35 mg) of explosive is placed between the strikers and a microphone is used to record events. Although these devices paid much attention on the confinement of the explosives, these designs did not provide sample heating, which limited the application in high temperature sensitivity testing.

In this study, the heated-anvil drop hammer machine is used to test the thermal sensitivities of high explosives. The Dh50 were calculated with the up-and-down method [13] (Bruceton method). The high explosives, PETN, triacetone triperoxide (TATP), cyclotetramethylene-tetranitramine (HMX), and silver azide (AgN_3) were analyzed by thermal drop hammer at temperatures of 25, 35, 45, 55, and 60 °C. Also, the thermal drop-weight tests of HMX samples with sample temperatures ranging from 150 to 210 °C have been done to demonstrate the phase change sensitivity at elevated temperatures [14].

2 Apparatus Design and Setup

The thermal drop-weight machine used in this experiment is shown in Figure 1. This apparatus comprises two guide rails, an anvil connected to the lower region of the frame upon which a specimen will be placed for testing, two strikers, and an adjustable drop-weight mechanism which is connected with one striker to apply a vertical impact force on the anvil. The device is leveled by the leveling screws. The guide rails are connected with the steel base and aligned to facilitate unhindered movement of the

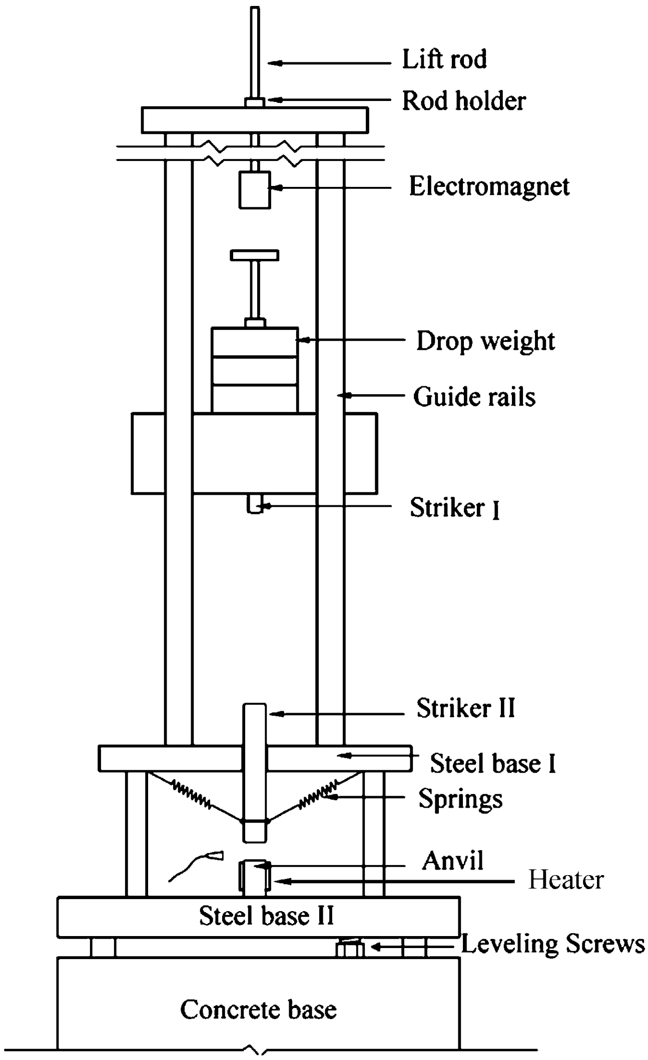

Figure 1. The diagram of laboratory-made thermal drop-hammer instrument.

weight assembly. The mass of the weight assembly can be modified from 0.5 to 6.8 kg. The guide block is rigidly mounted to the weight assembly and is able to slide down the guide rails using nylon bushings.

In order to obtain consistent data, two strikers are introduced in this design, which provide the accurate impact position during the experiments and increased reproducibility. A lift rod suspends the drop weight at a desired height, up to 100 cm in the current design. A manual safety clamp is used to stop the drop weight falling down during loading of the energetic compounds. An electromagnet is used to hold the weight assembly, which has maximum mass holding 100 kg (Industrial Magnetics, Inc., Boyne City, MI). A containment box surrounds the anvil holder and striker to shield the operator from flying debris and any gasses produced by the samples.

The sample heater is comprised of three parts: an aluminum heating block, a ring heater, and an isolation ring (made of clay with thickness 2.5 cm and length and width

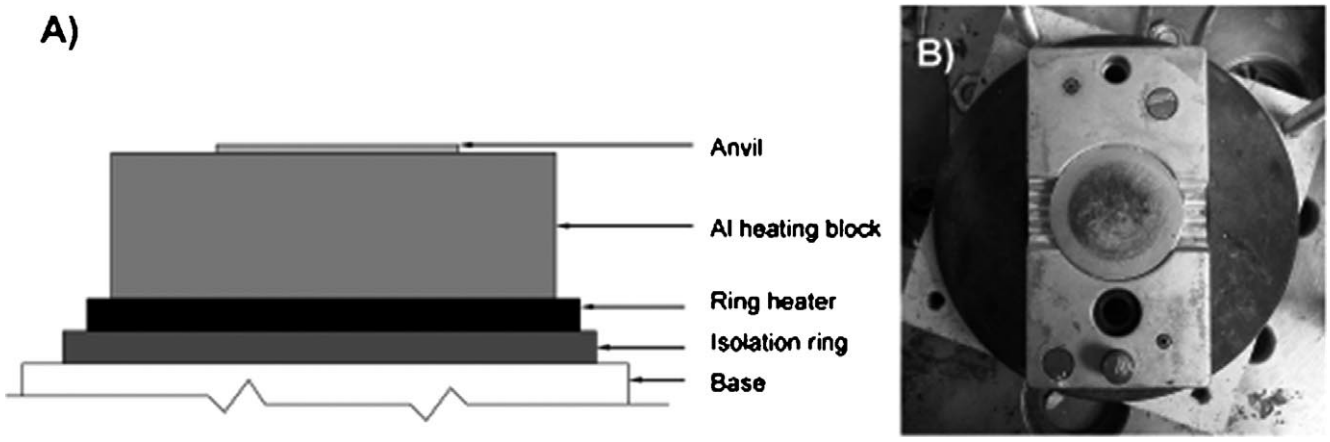

Figure 2. The sample heater setup. (A) Side view illustration of sample heater and (B) top view of the heater setup.

of 8 cm) as shown in Figure 2. The anvil can slide up and down without disturbing the heater during experiments. The heating block provides a wide heating range (20–300 °C) and the temperature is controlled by a feedback control system.

3 Experimental

PETN powder from Lawrence Livermore National Laboratory with purity >99 % is used in present experiments without further purification. HMX was obtained from Sandia National Laboratory with purity >95 %. TATP is an extremely sensitive explosive to impact, temperature, and friction. TATP was synthesized at a scale of 0.1 g by the procedure shown in Ref. [15]. AgN_3 is a colorless primary explosive, which is prepared by treating an aqueous solution of silver nitrate with sodium azide [16]. Both TATP and AgN_3 have low solubility in water, and the products are collected by precipitation from the solution and were used without further purification.

In order to investigate how relative humidity can influence the drop-weight values, TATP mixtures were prepared with a commercial humidity chamber (Associate Environmental Systems Inc., Ayer, MA), which can control the humidity of the sample from 10 to 100 % at room temperature or lower.

The drop weight used in these experiments included 0.5, 1, 2, and 5 kg of machined steel. The samples were prepared with a laboratory-made pelleting machine with 25–50 mg explosives. In order to keep the sample shape and thickness consistent, the samples of explosives weighing ±2 mg are charged into a die and cold pressed for 3 min to make a pellet. The final thickness of the pellet depends upon the material density obtained. In order to obtain an acceptable initial drop-height, the calculation based on the laws of the pulse and energy conservation [17] has been applied to this study.

4 Results and Discussion

In this study, we used the same procedure as reported by Simpson and Foltz [8]. The Dh50 values of explosives were analyzed with Bruceton method. In this procedure, a series of testing levels is chosen with equal log spacings between drops, the first level is chosen by guessing an initial height. The "GO" or "NO-GO" height will be used to calculate Dh50 and its standard deviation. The Bruceton method is a statistical method and relies upon two parameters: initial stimulus and step size. Generally, the accurate impact testing of explosive needs thousands of runs, however, 15–20 runs for each temperature were collected to calculate drop height and standard deviation with Bruceton method which is indicated by our large error bars.

Figure 3 shows the drop-height change of PETN, TATP, AgN_3, and HMX with various temperatures. Figure 3A shows the impact results of PETN at different temperatures with 5 kg drop weight. The drop height shows a slow drop with increasing temperature; the drop height shows a significant decrease around 60 °C, which is probably due to the changes on size of PETN particles and microstructures. As reported, significant sublimation of PETN can occur as the temperature is increased above 318 K [18].

Figure 3B shows the drop height of TATP at various temperatures with drop weight 2.5 kg. There are many reports of TATP based explosives and their power decrease significantly with increasing of the water content in the mixture to more than 30 % [15]. As shown in Figure 3B, the drop height of TATP water mixture increased to 10 cm at room temperature as the water content increased. As the temperature is increased, the drop height of the samples slightly drops. The drop height tends to converge as the temperature reached 70 °C, which is probably due to the evaporation of water in the samples. Also, the melting point of pure TATP is 91 °C [19] and the TATP easily sublimes, both of which limit the upper temperature range.

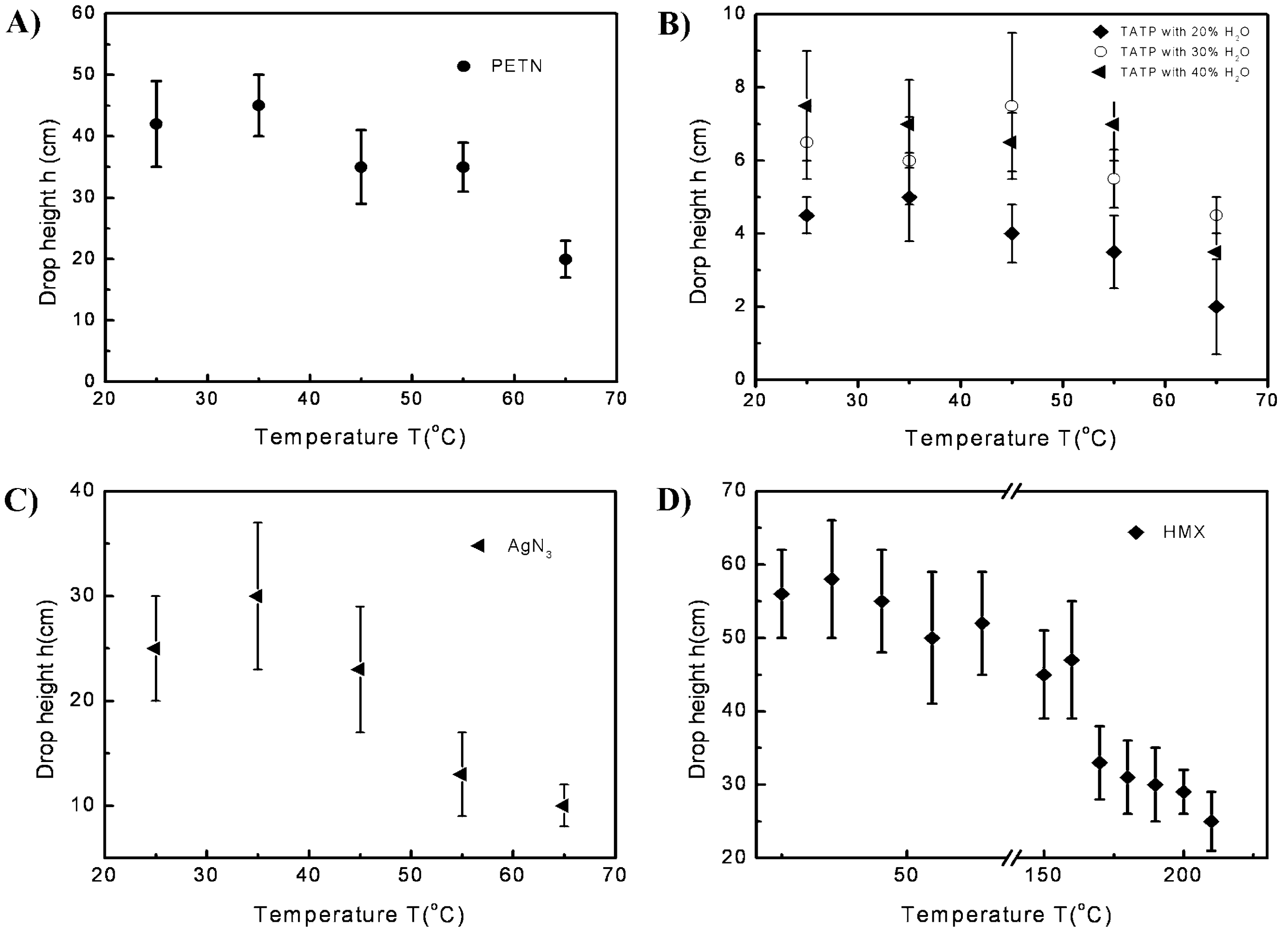

Figure 3. The drop heights changing with temperatures of TATP, AgN_3, HMX, and PETN. (A) Drop weight 2.5 kg, 15 mg; (B) drop weight 5 kg, 30 mg; (C) drop weight 7.5 kg, 50 mg; and (D) drop weight 5 kg, 50 mg.

Figure 3C shows the drop height of AgN_3 with drop weight 5 kg. AgN_3 is a typical non-nitrate inorganic high explosive. While AgN_3 is very sensitive to heat, spark tests show that AgN_3 is not sensitive to spark. The drop height shows a dramatic drop near 45 °C, also, the drop height at each temperature shows a big deviation, which may be due to the size distribution changing as the temperature is increased.

The drop-height of HMX with increasing temperature is shown in Figure 3D. As the temperature is increased from room temperature to 150 °C, the drop height shows a slight decrease. When the temperature is increased above 175 °C, there is a dramatic drop which shows a good consistency with the phase transition temperature of HMX from β- to δ-HMX. The phase transition of HMX can be clearly observed with the impact sensitivity.

Generally, HMX has several phases α, β, δ, ε, ϕ, etc. α-HMX has a limited range of stability and is not readily observed at ambient condition, and ε,ϕ-HMX are only seen under very high pressure conditions [20]. Of the polymorphs, β-HMX has the highest density and is stable at room temperature and pressure, which is the form in which HMX is normally produced and used. When HMX is heated above 435 K, a transition of β-HMX to the δ phase can be observed at atmospheric pressure [21,22].

The δ phase is reported to be a more impact sensitive form than the β phase [23]. The phase transition with drop-weight impact on HMX [24] has been investigated at room temperature with second harmonic generation (SHG) [23]. However, the in situ phase transition by impact test has not been previously reported. In our tests, the phase transition can be clearly observed at ~430 K where this is a sharp decrease in the drop height required for initiation. The drop height of HMX shows a dramatically drop when the temperature is increased to 433–443 K, which indicated that the sensitivity of HMX has been obtained at the phase transition of HMX.

In explosives sensitivities tests, some uncontrolled factors enter the testing procedure, which increase the spread of the distribution and the number of tests required to accurately determining Dh50 point will also increase. Such factors might include temperature, the degree of confinement, and the heterogeneity of the explosive form (either compact solid or powder). In this study, the temperature control is maintained with accuracy of ±0.5 °C by a feedback heating system. The effect of temperature of 3 °C upon impact has proved to be undetectable. Therefore, the temperature is not likely a significant source of error.

The confinement of the samples during the striking will introduce uncertainty into the data. It is very hard to make certain the samples were placed in exactly the same site for multiple experiments, which may result in unequal forces applied on the samples. In addition, the material defects from the surface of the striker and anvil will introduce errors with multiple experiments. As the experiments indicated, the striker surface was indented about 0.5 mm after more than ten runs of the testing and therefore it is necessary to re-grind the striker and anvil surface occasionally.

The most important variation in the results of such experiments is caused by the heterogeneity of explosive compound. For example, the compact solid contains much smaller voids, which is easier to produce hot-spots as the shock wave is applied, however, in a powder sample, the microstructures and voids are not well controlled, and may show relative low sensitivity during the impact testing. As the temperatures of the samples are increased, the morphology or phase change could also influence the impact sensitivity as discussed above.

5 Conclusion

In this work, we presented a new design of a thermal drop-weight machine, which provides the in situ testing of high explosives. The drop testing on HMX has illustrated that this device can be used to obtain the information how the physical status of materials influence the impact sensitivity. The accuracy of this device could be limited by the material properties of the striker and anvil, however, this device can be used to do the testing on newly synthesized explosives and important impact tests for explosive under harsh conditions, such as deep oil well and mining applications. Similar studies could be performed below ambient temperature by incorporating a peltier stage for cooling.

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