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Some Notes on the Fundamentals of E.M. Initiation Towards a "Universal Sensitivity Characteristic"?

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Abstract: The stimulus needed for initiation of an energetic material is an important quantity to determine for reliable functioning and safe handling. When considering the gamma of impact, friction, spark and heating tests in use one runs into the problem of how to compare results and how to make a prediction for practical situations that an initiation certainly will happen or definitely can be excluded. The way forward is not easy. The initiation process, which in order to be successful, should be followed by sustained propagation, is not simply accessible. This paper tries to produce a contribution by considering the fundamental aspects and to show as an example some simulation results of a gas phase initiation model. Some suggestions are made for making further progress.

Keywords: initiation, chemistry simulation, gas phase ignition

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Introduction

Why looking at initiation

There are two obvious reasons for determining the sensitivity to initiation of an energetic material:

- Reliable functioning of an E.M. loaded device.
- Avoidance of any unwanted accidental explosive reaction of the material.

The relative ability of a material to show an unwanted reaction after being subjected to a stimulus is in English often referred to as sensitiveness. To quantify sensitivity we need experimental methods to increase in an incremental and measurable way the loading of an E.M. until explosion always occurs, or alternatively until the first time an explosive reaction starts, and we need criteria to decide when the desired level of reliable functioning is reached or the chance of an unwanted, hazardous reaction can be excluded. The stimulus can be heat by flame, spark, mechanical impact or shock. Over the years many methods have been developed and a huge amount of work has been done to measure sensitivity of explosives, propellants, pyrotechnic materials and their sensitiveness. The latter is also relevant for safe transport and handling of all kinds of industrial products, which can exhibit explosive behaviour. Historically one takes a hammer and hits a small sample of the material on an anvil. The higher and stronger the swing before it cracks, the less sensitive the material. In the so called mallet test the guy swinging the hammer had to do it in a standardized way! The contemporary methods are described in detail in the U.N. Recommendations for the Transport of Dangerous Goods [1]. It is not the intention to review the methods here or to go in any detail, but to discuss some common shortcomings, which hamper progress.

When one synthesizes a new energetic material one of the first things to test is the impact and friction sensitivity. The equipment to use is the BAM friction machine and drop hammer apparatus. Both produce an outcome in Joules. What can one do with the figures obtained. In absolute terms very little. The data does not give any information on what situations in practice of handling the material are safe or unsafe. It is not possible to translate the initiation energy in information of *e.g.* from what height a case with the material can be dropped to avoid any initiation. After having done the tests successfully one would only know something on a relative basis. Is it more or less sensitive than TNT or PETN. A problem may arise if the material under examination is more sensitive than a reference material in one type of test and less sensitive in another. This picture is further blurred by the fact that the test outcomes are distributed around a mean. The conditions in a test can never be controlled in such way that all variability can be excluded. Since the test procedures always vary the strength of the stimulus in incremental steps, quite sophisticated statistics have to be applied to obtain results which can be reproduced. Numerous round-robins have been organized between laboratories to compare test outcomes and to eliminate variability in assessment. Of course all experts known with the field realise this state of affairs. A new promising method for correlating sensitivity test results is pattern recognition by using neural networks [2]. However, unfortunately funding agencies embark reluctantly on programmes on a longer term to have more advanced methods developed. Instead one tries to harmonize the methods and the criteria to avoid the situation that one national authority admits a certain material while another blocks its transportation.

Explosiveness

When considering explosive reactions generated by reactive chemicals, it is instructive to consider first the three different types of explosion that can occur in gases, as well as in liquids and solids:

- Thermal explosion
    - reaction throughout the entire mass simultaneously
    - balance of rate of heat production *vs.* loss: critical temperature, mass
    - induction period (from nanoseconds upwards)
- Deflagration
    - reaction in a separate zone; propagation mechanism by heat transfer
    - burning velocity ranging from some cm h^{-1} to hundreds m s^{-1}
    - total energy release rate increases with burning surface area
    - higher pressure and temperature generally increase burning rate, hence confinement and heat insulation are important
- Detonation
    - reaction in a separate zone, propagation mechanism is shock compression
    - detonation velocity is supersonic, range: 1-9 km s^{-1}
    - below a critical diameter no propagation
    - confinement partly compensates losses.

Since Bowden and Yoffe in the forties of the past century, the thermal nature of initiation is clear. Hot spot initiation by stimuli of different nature was demonstrated in many ways. Thermal explosion is therefore key. It is further helpful to make a strict distinction between initiation and propagation. Sensitivity has in the first place to do with initiation, but only successful initiation will result in propagation. So, in fact we hit here the weak points of many methods: It is not clear what type of reaction is tried to initiate and it is not established properly whether propagation occurs after initiation. In a way propagation is a continued initiation process. To study initiation it is therefore necessary to consider the propagation processes. Beside the above, practice is complicated by the transitions in propagation mechanism. Many initial and local thermal explosions result in deflagration of the remaining of the material and in larger mass or adequate confinement deflagrations can accelerate and run-up to detonation.

Chemistry and physics of explosive processes

An aspect that is often neglected is the chemistry of decomposition and energy production, mainly because it is too complex. The explosive potential of a reactive chemical can of course be calculated still relatively simple with chemical thermodynamic and energy release codes based on Gibbs free energy minimization, *e.g.* [3, 4], but the real problem is the kinetics of the reactions. Until recently chemistry was purely empirical, and the zero-order Arrhenius law was the model par excellence to predict the overall rate of reaction. For nuances in initiation it was possible in some instances to introduce autocatalytic effects. This is however changing. Gas phase energetic processes can now be modeled in detail and hydrocarbon oxidation models contain thousand or more reactions between hundreds of species. For condensed materials the state of affairs is perhaps much less advanced, but more sensitive and precise experimental methods, supported by *ab initio* calculations and molecular dynamics will improve the situation. In solid explosives the detailed chemistry and energy transfer are being studied within the reaction zone of the detonation front [5].

As regards the physics of heat and mass transfer for developing explosive processes the situation is certainly not simpler. For gases the physics of flow interacts with the reactions. Turbulence generated may intensify mixing and therefore enhance rates but the stresses can increase to such an extent that reaction quenches. In solids and liquids during the reaction usually gases are formed which depending on the structure of the material being continuous or grained, build up pressure and start to flow. This can spread ignition rapidly. In detonation shock waves dominate the scene and the acoustic impedance of the material matters.

Analytic models of *thermal explosion* on the basis of the heat balance in differential form and simple, zero-order Arrhenius kinetics are abundant. Two extremes can be distinguished: the Semenov – no temperature gradient, ideal convection model and the Frank-Kamenetzkii – pure conduction model;

for an overview see [6]. Apart from the fact that these extremes often do not cover practical situations, usually the situation is mixed, the mathematical solution makes use of approximations which can only be realized with the zero-order exponential law. Numerical models of *e.g.* the Finite Element type are more flexible but the lack of kinetic data limits their usefulness. Also diffusion of *e.g.* gaseous decomposition products, or of oxygen and water from outside, influences the reaction rates. Since near the critical condition the temperature-time history is rather flat, it is sometimes, in particular with larger masses, quite difficult to decide whether a certain starting condition in the end will lead to an explosion or not. Induction periods of several weeks are possible.

The energy release rate in *deflagration* is determined by burning velocity and burning area. Models for the burning rate are scarce and mostly limited to propellant type of energetic materials. Geometry, confinement, physics of flow and structure of the material can have a very strong influence on the fraction of the energy generated that is used to initiate a new layer of material. If a point initiation source is applied the strong initial growth of the burning surface requires relatively much energy to sustain the front and may lead to slow down or quench of reaction. On the other hand in linear charges in all substances in (partly) confined situations a strong acceleration of the energy release rate can occur. In gases this is due to the self generated turbulence as a result of the expansion of the hot gases behind the flame front and in liquids and solids by the increase in burning rate by pressure build-up and the increase in burning area by a surface instability of the reaction front in liquids and the presence of a porous grain structure in solids. In all cases this can lead to pressure waves and reactions ahead of the flame in precompressed material and sudden initiation of detonation.

In many respects modeling and testing of detonation is conceptually simple, although the process completes in microseconds. For the models the Chapman-Jouguet overall set of equations is common knowledge. Further, the criterion for its presence is a sustained, stationary detonation wave, which can be traced either by a fragmentation pattern of confining tube, a continuous pressure sensitive probe, high speed cinematography, optical diagnostic technique as laser imaging or interferometry *etc*. Also in case of detonation geometry is important. Because of the energy diversion a point detonation is much more difficult to initiate than a detonation initiated over the full cross-sectional area of a cylindrical charge [7]. This shows up in particular when trying to initiate a low energetic material as ammonium nitrate or a so called non-ideal explosive.

Attempting to initiate over a full cross-section of a (confined) cylindrical charge ammonium nitrate (AN) can still detonate when the initiating booster only contains 10% penthrite, PETN, but a small spherical 100% PETN booster in the centre fails to do it. Of course, the process of coupling of a shock wave to the reactions it triggers and the associated energy release rate profile is still one of the great challenges to science to solve.

Overlooking these various processes one can conclude that in initiation some form of mechanical energy (impact, shock or friction), electrical energy (spark, plasma), laser light, or just thermal heating is deposited in a certain volume in a certain time, which triggers reaction. The distribution of the energy will in general not be uniform (hot spots) and by the nature of the material (slide planes, shear, crystal defects, gas bubbles) it may also work out heterogeneously. For propagation to succeed the reaction shall to a sufficient extent self-accelerate. In sustained propagation of an explosive process there is an energy flow forward initiating fresh material. In case of point initiation this energy flow is diverging. So beside a sufficient amount of initial energy input to raise temperature, there will be minimum volume required with a sufficiently large radius and surface area. The energy input shall also be within a sufficiently short time, so that heat losses do not frustrate and quench the process.

This brings us to the utopian concept of a "universal sensitivity characteristic", in which a stimulus is defined in terms of energy, time and volume of deposition. In Figure 1 schematic representations are given of the initiation energy and initiation energy density (energy deposited per unit of volume) to initiate and propagate an explosive reaction as a function of the radius of volume in which the energy is deposited. In small volumes the energy required is relatively high in particular at long deposition times because during chemical induction heat leaks away. When the volume gets large despite the losses the centre will be only slightly affected and the chemical induction time becomes determining. Hence the energy density decreases till a certain minimum level. In Figure 2 the initiation energy density as a function of time (in log scale) is schematically shown. It can again be seen how an optimum arises between energy influx, chemical response rate and energy losses to the surroundings. For a given test set-up the minimum ignition energy (MIE) apparently depends on the duration of the stimulus and the volume: Not too short, not too long; not too small, not too large.

Propagation will depend on a critical power density or energy flux to ignite the next layer. Front curvature and diameter (lateral losses) are important. In gases preferential diffusion of fuel or oxidizer species play a role. This will not be the subject of this paper.

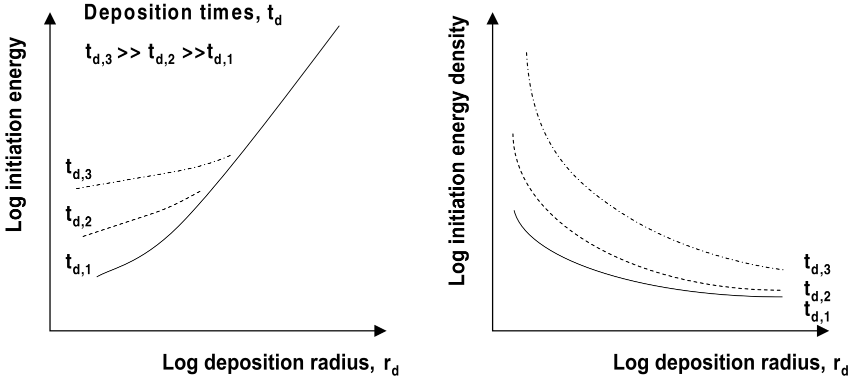

Figure 1. Initiation energy (left) and energy density (per unit of volume) (right), schematic, as a function of the deposition volume radius at three different deposition duration times. At small volume heat loss prevails. The energy density tends to a constant minimum value, although for longer deposition times at higher level.

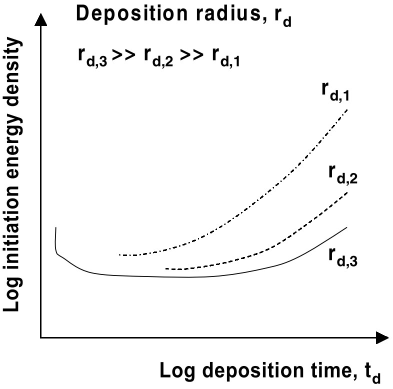

Figure 2. Initiation energy density, schematic, as a function of the deposition time at three different deposition radii. At longer times the effect of deposition volume increases. At ultra short times (nanoseconds) the chemical relaxation times are going to play a role and energy density goes up again.

Sensitivity testing: a gas phase model case

The gas phase is relatively accessible for testing. The most convenient way of initiation is by electric spark. Within certain limits energy, deposition time and to a lesser extent deposition volume can be controlled. Although many experiments have already been reported, special equipment is now installed by one of the authors (Nikolaos Zarzalis) to vary the ignition parameters as freely as possible and to study ignition also at higher pressures[1].

Since gas phase hydrocarbon oxidation kinetics are well modeled in detail one can also simulate on the basis of a simplified version of the Navier-Stokes equations, written in Lagrange coordinates. The reactions are represented by over 300 equations with more than 50 species [8]. In the ignition process the acceleration of reaction in gases under the influence of energy input is by radical branching processes (net increase in number of radicals) and self-heating. Such scheme cannot be described by a simple overall Arrhenius equation, since the oxidation mechanism changes drastically with temperature. This makes *e.g.* that at a certain temperature range induction time even becomes longer with further temperature increase. The solution is by a finite difference scheme. Below results are given of calculations made with the program INSFLA [8, 9], which fully support the schematic of Figure 1. For details of the work, see Weiss [10]. In Figure 3 the calculated ignition energies[2] and densities for stoichiometric propane-air mixtures and different deposition times are plotted against deposition radius.

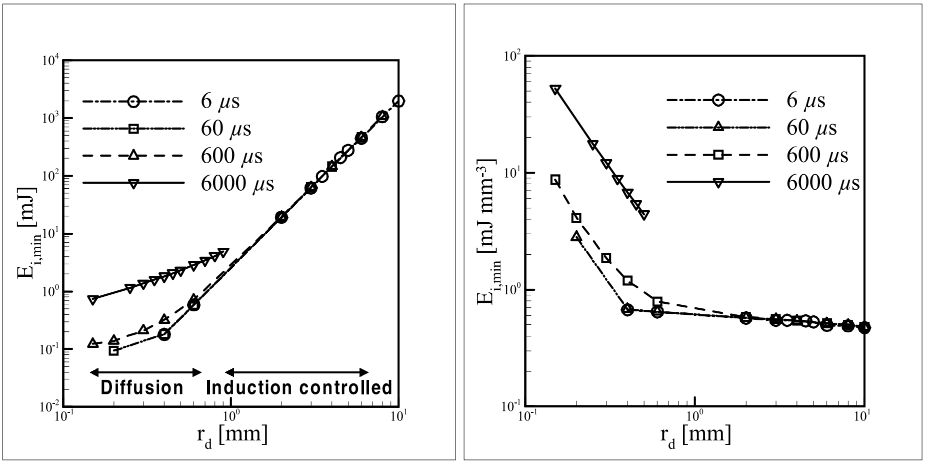

Figure 3. Ignition energies (left) and ignition energy densities (right) of propane-air mixtures *versus* energy deposition radius at different deposition times.

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[1] Financial support of this work by the European Commission within the Fifth Framework Programme on Energy, Environment and Sustainable Development, contract EVG1-CT-2002-00072, Project SAFEKINEX is gratefully acknowledged.

[2] This is the minimum energy required for successful ignition in a certain condition. The term Minimum Ignition Energy or MIE is reserved for the real overall minimum considering both volume and deposition time.

For small deposition radii the ignition energy is largely dependent on the deposition time and seems to tend to a constant value with decreasing energy deposition radius. The energy level of this constant part decreases with deposition time but this effect is slowing down in the microsecond time order of magnitude. The minimum ignition energy seems to depend therefore particularly on a certain minimum deposition time.

With longer deposition times the amount of energy for a successful ignition increases. This effect is stronger the smaller the deposition radius. This ignition region is called the diffusion controlled region. In this region the heat loss, which is mainly due to thermal diffusion, is the determining process of the ignition.

For radii larger than about 1 mm the ignition energy density reaches a nearly constant minimal value for all deposition radii and times, the minimum energy density. The ignition energy shows an asymptotic and deposition time independent behavior. It increases proportional to the energy deposition volume (3rd power of the deposition radius). This region is called the induction controlled region. In this region the ignition process is mainly controlled by the formation of a radical pool within the induction time.

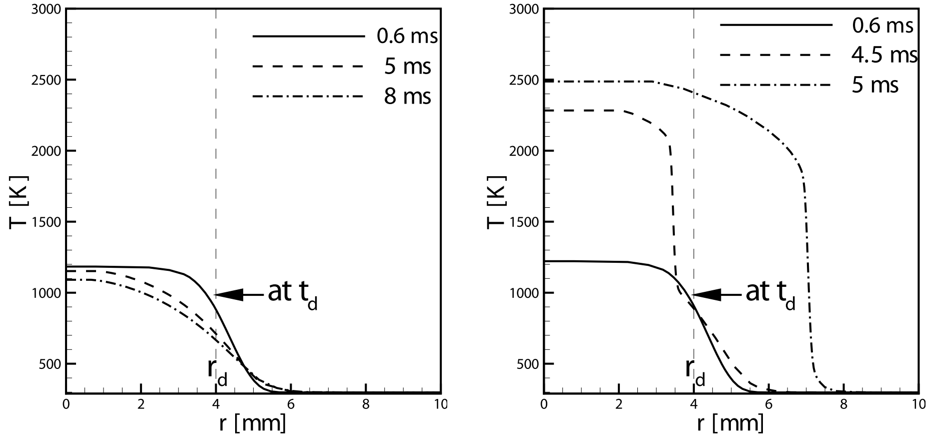

Figure 4. Induction controlled ignition (t_d = 600 μs, r_d = 4 mm) of stoichiometric propane-air at atmospheric conditions: temperature profiles at three different points in time; unsuccessful ignition at E_i = 144.3 mJ (left), successful ignition at E_i = 144.4 mJ (right).

The simulation results allow too an examination of the temperature distribution of the hot spot. In the left diagram of Figure 4 temperature profiles are shown at three different points in time for a simulation with an input energy of 0.1 mJ below the ignition energy. The deposition time, t_d has been set to a value of 600 μs and the deposition radius, r_d to 4 mm. After the deposition time near the boundary the temperature starts to decrease and the temperature profile flattens outwards. In the right diagram of Figure 4 the energy value is chosen only 0.1 mJ higher to 144.4 mJ. In the latter case the kernel temperature increases after the deposition time and the flame front starts to propagate. For large deposition radii the basic criterion for a successful ignition is that the temperature reaches a minimum ignition value after energy deposition. This is comparable with the auto ignition temperature, but the values in case of an induced ignition are slightly higher.

For large deposition radii (*induction controlled region*) the ratio of surface of the deposition volume to deposition volume is smaller than for small radii (*diffusion controlled region*). As the heat of reaction is proportional to the volume and the heat flux is proportional to the area, heat loss by diffusion in comparison to heat production by the exothermic chemical reaction is much smaller than in the diffusion controlled region. In the induction controlled region the thermal diffusion is almost negligible, compared to the diffusion controlled region, so that the ignition energy approximately depends only on the deposition radius and is independent on the deposition time. Thus the energy density is nearly constant. As one can see in Figure 4 the energy density tends for a radius larger than 1 mm toward a constant minimal value and is not depending on the deposition time.

The criterion for initiation is successful propagation. Figure 5 shows three temperature profiles at different points in time in case of ignition leading to successful and unsuccessful propagation. Looking at these graphs one can see that the temperature after energy deposition is much higher in comparison to the temperature in the induction controlled region, even if the energy amount is not high enough for a successful ignition with adjacent flame front propagation. Further one can notice a local rise of the temperature profile starting in the centre of the energy deposition volume. This is a sign of already starting exothermic chemical reaction, meaning that the exothermic reaction can start in the diffusion controlled region even if the energy input is lower than the ignition energy. Because of the small energy deposition radius the heat diffusion is high and the temperature is then decreasing rapidly in case of an energy value smaller than the ignition energy. If the ignition energy is deposited, the temperature is first decreasing. After passing a critical radius the temperature is increasing and reaches the burned gas temperature.

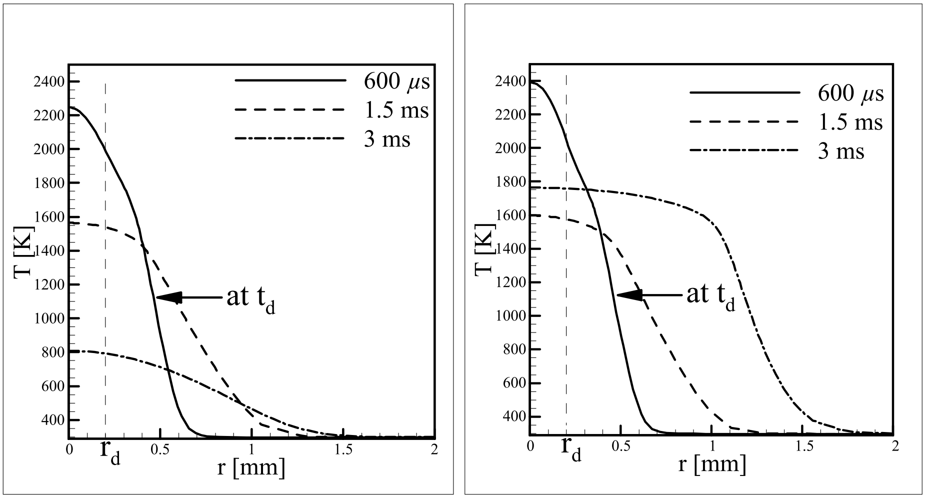

Figure 5. Diffusion controlled ignition (t_d = 600 μs, r_d = 0.2 mm) of stoichiometric propane-air at atmospheric conditions: temperature profiles at three different points in time; unsuccessful ignition at E_i= 542.1 μJ (left), successful ignition at E_i= 542.2 μJ (right).

The practice of E.M. testing

When testing it should be clear that the applied test is relevant for the situation being examined for safety, and the test effort should be efficient in view of the utility and reliability of the result obtained. This is still utopia. Because of the bad control given by the test set-up and the nature of the sample a large dispersion in result usually occurs.

The criterion for a substance tested with *e.g.* the Julius Peters fall hammer machine to be safe is no explosion (or rather no “bang”: Is any “bang” really an explosion?) at 2 Nm or 2 J drop energy, *i.e.* 1 kg hammer at 20 cm drop height [1]. Under conditions of more efficient energy transfer the same substance may be initiated by 10-100 times less energy. Basic studies have been performed amongst others by Field and coworkers [11], however more knowledge is needed to find out how much energy in what time is deposited in what volume. Further, the criterion should be met as only one explosion in 6 tests. This seems risky since for safely applying go/no-go statistics, the establishment of a 50% probability point (up- and down method assuming normal distribution of outcomes, or the Robins-Munroe method not requiring an *a priori* distribution, *e.g.* [12]) requires many tens of tests. For both reliability and safety investigation one should in fact go further and try to establish *e.g.* a 99% or a 1% probability level of an explosion event, given a certain stimulus. This would however, take several hundreds of tests.

An improvement to traditional impact testing can be found in [13], describing the Energy to Ignition test and the Ballistic Impact Chamber test. With a photo sensor the moment of ignition in a solid explosive is determined, and from the deceleration of the drop weight during the impact, one can calculate the loss in kinetic energy of the drop weight being a measure of the energy transferred to the explosive. This measure of energy is a better measure compared to the previously mentioned potential energy of the drop weight.

Correlating the thermal decomposition characteristics with impact and friction sensitivity has been attempted in view of the hot spot mechanism. This has certainly been a success. One of the possible ways is the Thermal Step Test [14]. It consists of a stainless steel capillary tube, suddenly heated by a charged capacitor discharge with the tube as part of the circuit, acting simultaneously as a heat sensing element (resistance thermometer). Consistent measurements have been possible of induction time of a millisecond to a hundred seconds at a given jump-wise-attained initial temperature level. Plotting the logarithm of induction time *versus* reciprocal absolute temperature produces straight lines down to the millisecond regime. Below this value however, anomalies occur starting at different time values for different substances, probably due to changes in heat transfer mechanism. Above this apparent energy of activation values are well in agreement with values otherwise determined. Higher sensitivity can be directly correlated with shorter induction times at a given temperature.

There are further new possibilities. Laser initiation is one of these, see *e.g.* [15, 16]. A laser can deliver in principle a well controlled amount of energy into a material with respect to power, area of energy deposition, pulse duration and total energy. It is less controllable in terms of volume in which the energy is delivered and short pulses are associated with shock. Systematic experimentation determining the energy density for ignition as a function of pulse exposure time deposition volume is scarce.

Shock initiation of solid explosives by flyer impact does provide valuable data as well. The impact flyer induces a rectangular shock wave in the explosive sample with the induced pressure related to the velocity of the impact flyer and the pulse duration to its thickness. Typically shock initiation data are given as minimum impact velocity and/or energy fluence (energy input per unit of area) as function of flyer impact thickness. Data on the initiation of pressed samples of

TATB and HNS-IV, with a density of 80% of the Theoretical Maximum Density, are given in [17] and represented here as well as energy density *versus* pulse duratione (Figure 6). From the graph of energy fluence *vs*. flyer thickness one can conclude that from energetic considerations (minimizing the kinetic energy of the impacting flyer) a flyer thickness near 125 μm is optimal for shock initiation of the TATB sample, whereas a flyer thickness of even less than 50 μm is preferred for shock initiation of the HNS-IV. Such a consideration may be important in the optimization of a detonation train. However, looking at the energy density *vs*. duration of the incoming shock one can clearly see the minimum energy density value, which is specific to the energetic material. For the TATB sample the minimum of 1.8 J mm^{-3} holds for pulse durations down to about 0.1 μs. For HNS-IV the induction controlled region extends down to pulse durations of 0.06 μs, and a corresponding minimum energy density of 0.85 J mm^{-3}.

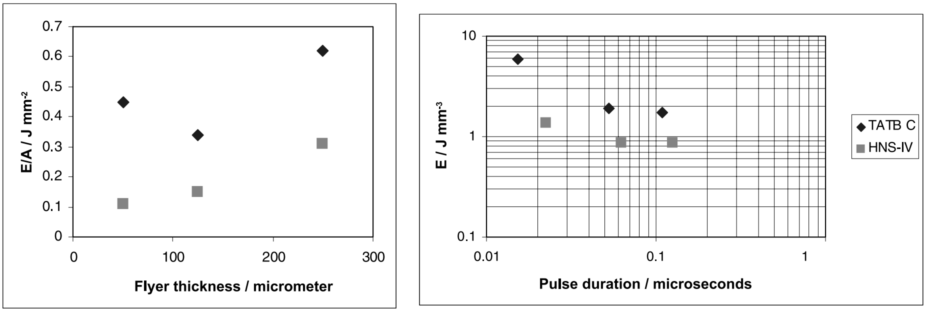

Figure 6. Minimum energy fluence *vs*. flyer thickness (left) and minimum energy density *vs*. pulse duration (right) of pressed TATB and HNS-IV explosive samples.

Conclusions

- During the last ten years considerable progress has been made in understanding mechanisms and in modeling. However, many test methods for determining sensitivity are still archaic and require too much effort given the quality and applicability of the answers.
- Rationalization and harmonization of tests and criteria is one way to go. Even more important is to unify efforts under expert leadership and create cooperative programmes to develop improved methods and to try to determine fundamentally based quantities enabling extrapolation and risk estimation in practical situations.

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