

# Development of a method for quantification of hot-spots

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## Abstract

A system has been developed that allows the power and duration of a hot-spot to be specified within an energetic material. A diode laser is used to create a hot-spot in a cell containing an energetic material that can be pre-pressurised and heated. A range of diagnostics are used to provide data ideally suited for modelling the ignition characteristics of the material. The data produced have implications for assessing the safety of energetic materials. A preliminary study into the critical parameters for ignition hot-spots in isopropyl nitrate (IPN) is described. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Isopropyl nitrate; Ignition; Hot-spot

## 1. Introduction

Since 1940's, it has been generally accepted that hot-spots are important in the ignition of reaction in energetic material, in the propagation of fast reaction and in problems such as, thermal cook-off. Bowden and Yoffe [1,2] led the field in the qualitative descriptions of the various mechanisms by which hot-spots could be formed. Mechanical energy is converted into localised thermal energy through the interaction of the pressure waves with inhomogeneities. The inhomogeneities can exist prior to the impact as is the case with cavities or can be created during the impact as, for example, adiabatic shear bands are. Alternative mechanisms such as, electrical discharge and laser heating have also been successfully used to ignite energetic materials. A review of the subject is given by Field [3]. For ignition processes, Bowden and Yoffe showed that typical temperature, size and duration are 700 K, micron dimensions and 10  $\mu$ s–1 ms, respectively.

Clearly if duration, for example, is reduced then temperature or size has to increase. During shock propagation, time is very short, say from  $10^{-6}$  to  $10^{-9}$  s, and so the temperature would need to be very high and the mechanism producing the hot-spot capable of operating at this very short time. We have argued [4] that this almost certainly favours void collapse processes rather than adiabatic shear band formation or frictional heating. In cook-off, rather larger times are clearly involved.

The common link between the various mechanisms for producing hot-spots is that all of them produce a localised region of the material at a higher temperature than the bulk and as such can be considered to be thermal rather than mechanical phenomena. Although the mechanisms that cause hot-spots have been well-described, quantitative information regarding the critical pressure, temperature and duration of hot-spots required for ignition is still incomplete. Various attempts to develop a system for isolating these factors have been made and most have met with some limited success.

Isopropyl nitrate (IPN) is a detonable liquid that has found wide use in rocket technology as a

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monopropellant. Research has been carried out into its thermal properties by a number of groups [5–8]. It was chosen as the subject for the preliminary study outlined in this work as data on the vapour pressure and calorimetric responses suggested that ignition would be relatively easy to obtain.

Clearly the set-up described, below with a diode laser can only investigate a narrow range of hot-spot conditions. The general approach, with a range of lasers, has wide validity for hot-spot studies for a wide range of situations.

## 2. Experimental

An ideal system for the study of the critical parameters required for ignition would not perturb the energetic sample at all and yet would allow the introduction of a hot-spot of a known size, temperature and duration. Control over the pressure and temperature of the sample prior to the introduction of the hot-spot would be desirable. The diagnostics would give accurate and rapid information for the temperature, pressure and level of reaction at all points within the cell; again without adding any perturbation to the system being studied.

During the course of the study described, a system was developed capable of introducing purely thermal hot-spots into an energetic medium. A steel cell was built with a 10 mm long, 10 mm diameter cylindrical chamber. Through the lid of the chamber, both the hot-spot source and the diagnostics were introduced. The initial pressure and temperature of the cell are controlled. A schematic of the cell is shown in Fig. 1 and a complete diagram of the experimental arrangement is given in Fig. 2.

### 2.1. Laser

The hot-spot was provided using a 1.2 W fibre-coupled diode laser operating at 980 nm (Applied Optonics). A 200  $\mu\text{m}$  diameter fibre from the diode was fed into the cell. On the fibre-tip a thin layer of graphite was fixed to absorb the laser energy. The power of the laser pulse is controlled by a custom-built diode driver which provides a constant current when an external trigger is applied. The trigger pulse is provided by a Thurlby Thandar TGP 110

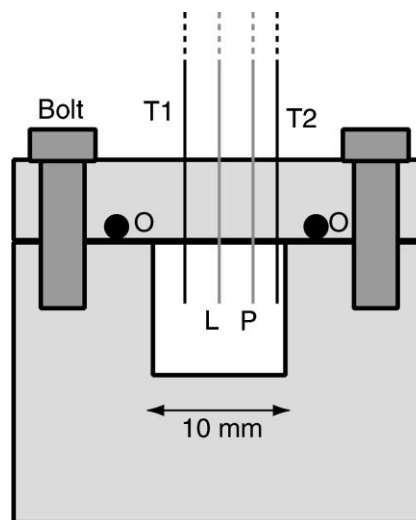


Fig. 1. Steel ignition cell: thermocouples (T1 and T2); optical fibre from diode laser (L); optical fibre to photodiode (P); viton O-ring (O).

pulse generator capable of producing single pulses of duration between 100 ns and 10 s. Calibration of the laser power was carried out using a Coherent Lasemate Q power meter filtered with a Barr & Stroud UG1 band pass filter. In the experiments outlined below the power used was 1.0 W and the duration of the pulse was 5 s.

### 2.2. Diagnostics

Two thin type K thermocouples were introduced to the cell at distances of approximately 2.5 and 4 mm from the tip of the optical fibre. These have a response of  $15 \mu\text{V K}^{-1}$  and the output from these was fed through an amplifier to a Tektronix TDS 460 digitising oscilloscope.

In some of the experiments outlined in this paper, an Electro-Optics ET2010 visible light silicon photodiode was used in order to record the light levels within the cell. The output from the photodiode was also recorded on the Tektronix oscilloscope.

All of the fibres and thermocouples that were led into the cell were sealed in place using Permabond fast cure epoxy. The quality of the seal was tested by pressurising the cell prior to an experiment as any loss of cell integrity would allow the material within the cell to escape.

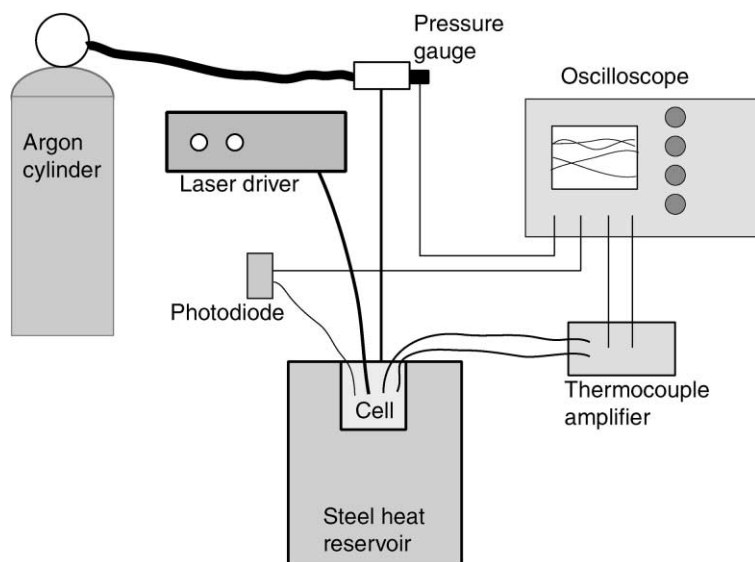


Fig. 2. Experimental arrangement.

### 2.3. Initial parameter controls

The initial temperature of the system could be varied between room temperature and 200 °C. This was achieved through placing the whole system within a large piece of heated steel. Variations in the heating rate were shown not to affect the experiment significantly and a rate of approximately 0.3 °C s<sup>-1</sup> was typically used. The upper limit on the initial temperature was due to the temperature range of the epoxy glue used. If higher temperatures are required this would be changed to a more robust material, in these experiments, however, this was not a problem.

The initial pressure was controlled using a cylinder of argon that fed through a regulator into a pipe attached to the lid of the cell. Pressures up to 10 bar can be easily achieved using the regulator and cell as currently configured. Higher pressures can be obtained using a different regulator and a more robust method for sealing the thermocouples and optical-fibres into the cell. A pressure transducer was used to monitor the pressure in the cell prior to ignition. The transducer used was a piezo-resistive bridge obtained from RS components (stock number 235-5863). Due to the chemical environment and the potential for high temperatures during an ignition event, the pressure transducer was attached to the

top of the pipe leading from the regulator to the cell. This prevented a complete pressure history of the material within the cell after ignition, but the pressure prior to ignition was recorded on the digitising oscilloscope.

The choice of gas used to pressurise the cell has been shown [7] to have an effect on the thermal characteristics of IPN; only argon was used during the course of this study.

### 2.4. Materials

The IPN ((CH<sub>3</sub>)<sub>2</sub>CHONO<sub>2</sub>) used in the preliminary study was 99% pure and supplied by Aldrich Chemicals (catalogue number 24,195-4). No further purification was carried out.

## 3. Results

Fig. 3 shows a typical set of traces recorded by the digitising oscilloscope. The initial gauge pressure for this experiment was  $6.6 \times 10^5$  Pa and the initial temperature within the cell was 148 °C. The period for which the laser was on is shown by the shaded region of the graph. As can be seen from the traces, the onset of exothermic reaction at the position of the closest

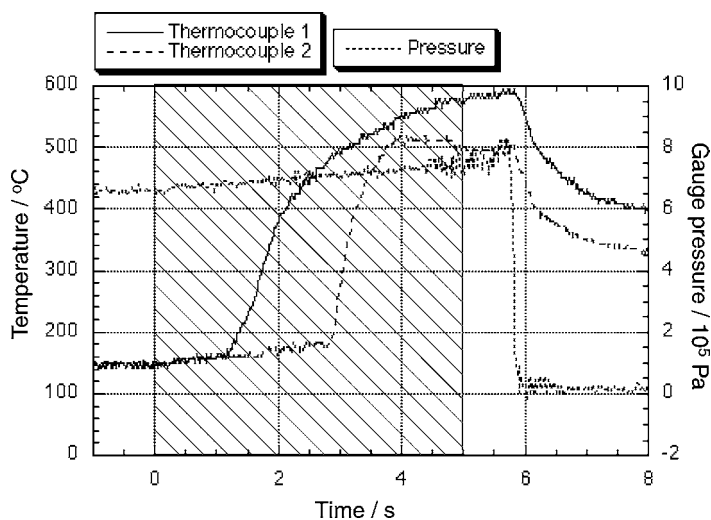


Fig. 3. Traces recording the temperature at the thermocouple positions and the pressure. Initial temperature 148 °C; initial pressure  $6.6 \times 10^5$  Pa. Shaded region corresponds to time when laser firing.

thermocouple (2.84 mm) occurs 1.1 s after the laser is operating. The reaction front reaches the second thermocouple (at 4.61 mm) 1.8 s later. This gives a mean reaction velocity of  $0.98 \text{ mm s}^{-1}$ . Due to their own thermal masses, the temperatures measured during reaction by the thermocouples can only be considered approximate, however, the time of arrival of the combustion wave as indicated by the onset of the

temperature rise is given very accurately. The reaction was quenched when it became apparent that the ignition had led to a self-sustaining reaction by releasing the pressure to atmosphere. The act of quenching allows a far higher rate of experimental repetition and doesn't detract significantly from the aim of this study which is to quantify the parameters affecting production of sustained reaction.

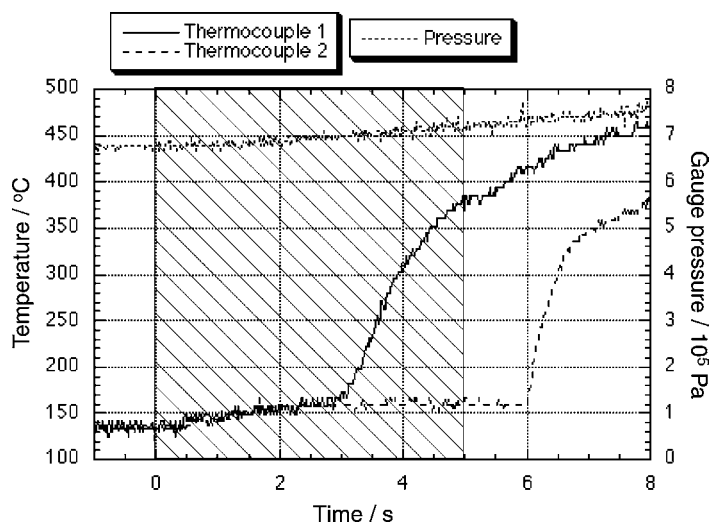


Fig. 4. Trace showing the temperature and pressure as the laser is fired at an initial temperature of 132 °C and an initial pressure of  $6.8 \times 10^5$  Pa. The shaded region corresponds to the time when laser firing.

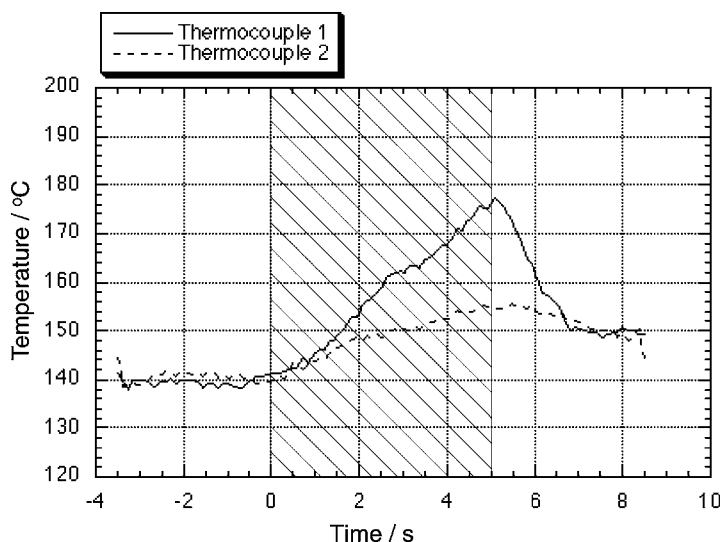


Fig. 5. Thermocouple traces from a shot that was fired at an initial temperature of 140 °C and an initial pressure of  $5.2 \times 10^5$  Pa. The reaction was quenched when the laser was turned off.

Fig. 4 shows another experiment carried out at a similar initial pressure ( $6.8 \times 10^5$  Pa) but lower initial temperature (132 °C) than shown in Fig. 3. As can be seen, the onset of exothermic reaction takes significantly longer (3.0 s) to reach the first thermocouple and travels at a slower rate ( $0.59 \text{ mm s}^{-1}$ ) through the cell to reach the second thermocouple.

Fig. 5 shows the thermocouple records relating to an experiment in which the laser was fired at a temperature of 140 °C but at the lower pressure of  $5.2 \times 10^5$  Pa. In this experiment it can be seen that although some exothermic reaction is occurring leading to a temperature rise at the nearer of the thermocouples of nearly 40 °C it died out after the end of the laser pulse and did not propagate to the second thermocouple. In this instance the energy released during the exothermic reaction that occurred was not sufficient to sustain the combustion wave.

The trends from the experiments carried out show that at lower initial pressures higher initial temperatures are required in order that sustained ignition can occur if other parameters are kept constant. It has also been found that the speed at which the combustion wave propagates through the cell is heavily dependent on the initial temperature and has some dependence on the initial pressure.

#### 4. Discussion

Although the results described above for the ignition of IPN are only preliminary, they do show the scope of this line of research to lead to a truly quantitative method for measuring the critical parameters of hot-spots. The critical energy and duration of the hot-spots required to ignite IPN across a range of initial temperatures and pressures have been measured.

The trends that have been measured regarding the effect of initial temperature and pressure on the burning rate of the material are in agreement with the trends found by many workers in other propellant materials [9–11].

#### 5. Conclusions

The system used in this study has a far wider range of application than simply the study of the ignition of IPN. It has been designed to operate with a range of energetic materials both solid and liquid. Accurate control and measurement of the initial temperature and pressure have been achieved in a situation where single purely thermal hot-spots are introduced to the

system. The range of diagnostics available for use in this type of study is wide. High-speed spectroscopy and high-speed photography are both planned as future steps to look at the nature of the reaction as it progresses. The former can be carried out through the fibre that is currently fed in to the photodiode and the latter will require a small redesign of the cell in order to get an optical pathway through to the energetic medium.

It is also intended to increase the size of the cell in order that boundary conditions where the sample is in contact with the walls of the cell become less of an issue. An increase in the volume of material will also act to reduce the effect of the perturbations caused by the presence of the optical fibres and thermocouples within the energetic medium.

The simplicity of the approach used in this research together with the accuracy and range of the measurements made aids modelling. Currently computer-based models struggle to accurately reproduce ignition type experiments unless they are sufficiently empirical in nature that the computer is simply following a pre-determined curve. With a high level of control over the variables affecting the ignition process, the experimental technique described should be perfect for modelling almost from first principles. Basic models to describe the cell are currently being developed but have not yet reached a stage where they can be reported.

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