DOWNSCALING THE METHODOLOGY FOR DETERMINING DETONATION VELOCITIES APPLIED TO SMALL SAMPLE QUANTITIES OF EXPLOSIVES

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Abstract

The detonation velocity is a key parameter for characterizing the performance of explosives. The standard method at ICT, which is also used in literature, is the use of short-circuit contacts along the charge axis to detect the detonation wave propagation at distinct positions in dependence of time to derive the detonation velocity from a regression. To ensure a high accuracy, the diameter of the pins has to be negligible in relation to their distance and the total number of pins must be sufficient to ensure a high regression quality. This requires a comparatively large charge and corresponding mass of explosive. At ICT, one focus of research is the reduction of mass required to characterize energetic materials, concerning performance and safety parameters. The objective of the research is to provide a comprehensive characterization of small samples of new energetic materials produced on a laboratory scale, as well as the characterization of small energetic components such as detonators. Therefore, a new miniaturized method to measure detonation velocities is the result of the development presented in this work. The method is based on printed circuit boards, using the same principle as the short-circuit pins within the large-scale method. Through this approach, a downscaling of the method by a factor of 50 was realized. The validation of the method shows a high agreement with the results obtained by the large-scale method for HWC, as well as for HNS-pellets e.g. used in EFI applications.

Introduction

For civilian and military applications, the development of new energetic materials requires the characterization of their performance and safety properties. Fraunhofer ICT covers the entire process chain for the development as well as the characterization of new energetic materials. A key performance parameter is the detonation velocity that has an impact on the Chapman-Jouguet condition and thereby on the useable expansion work of the detonation. Compared to other performance characteristics such as detonation pressure, the detonation velocity is comparatively easy to determine and therefore an important characteristic in quality control. The measuring principle is generally based on the detection of the quasi-stationary detonation wave location as a function of time, the derivative of which represents the detonation velocity. This relation can be obtained by various methods such as ultra-high-speed imaging or based on jump off velocities recorded by an array of photon-doppler-velocimetry probes within cylinder tests. A simple but reliable approach is to use short-circuit pins arranged as an array with a defined spacing. This method defines the recent standard at Fraunhofer ICT and is usually realized for charge diameters of 21 mm and 50 mm. However, the required explosive mass for the test is high, which is a direct consequence of the resulting charge length, as a function of the circuit-pin dimensions and the number of pins required. Thereby, the diameter of the pins must be negligible compared to the distance between two pins, so that the placement of each pin can be precisely associated to a zero-dimensional point. Over the years, a factor of at least 20 has become established as an empirical value, which means 20 mm pin spacing for a short-circuit pin diameter of 1 mm and a total number of 12 pins as an array. Including an offset length for the initiation, the method requires a charge length of approx. 300 mm, resulting in a charge mass in the order of 150 g for 21 mm and up to 1 kg for 50 mm diameter, per experiment. For a given charge diameter, downscaling of the charge length is only possible to a very limited extent for the classical method due to the fixed diameter of the pins and the accuracy of their placement. This approach has already been applied at ICT[1], where a pin distance of 3 mm was realized for special applications, equivalent to a scaling factor of about 7. Since the focus of research on energetic materials at ICT is particularly on reducing the mass of explosives required for the comprehensive characterization of performance and safety parameters, this work aims to develop a new design of short-circuit contacts to realize a significant scaling of the method for the precise measurement of detonation velocities at short distances with an accuracy comparable to the classical method. A new circuit pin design is presented in detail, compared to the classical method and validated for the explosive HWC. Finally, an application for the method is demonstrated for small HNS-pellets, typically used in EFI-detonators.

Methodology

In Figure 1 a schematic of the standard method using short-circuit pins (left) is shown together with the newly designed *printed circuit board* (PCB, right). Within the classical method, the pins were inserted through drill holes to the center along the axis of the charge. The advantage is that curvature effects of the detonation wave, which expands spherically from the center of ignition, are not present and an ideal one-dimensional propagation can be assumed. However, this also leads to disadvantages, in the form that holes must be drilled in the explosive beforehand and the final placement of the pin distances cannot be validated, unless computer tomography is used. The pins also represent an inert disturbance for the detonation propagation, whereby the influence of the pins on the propagation of the wave is not known

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[1] Armin Keßler, Thomas S. Fischer, Walter Ehrhardt, and Gesa Langer, “Miniaturized Detonation Velocity Measurement, 2008: Fraunhofer Institut für Chemische Technologie (ICT),“.

and neglected in practice. This factor becomes particularly critical when the distance between the pins decreases to a level, where pin diameter and pin distance are in the same order. A standard pin distance of factor 20 times the pin diameter (1 mm) is an empirically determined parameter, which allows an accurate determination of detonation velocities using 12 pins within this configuration.

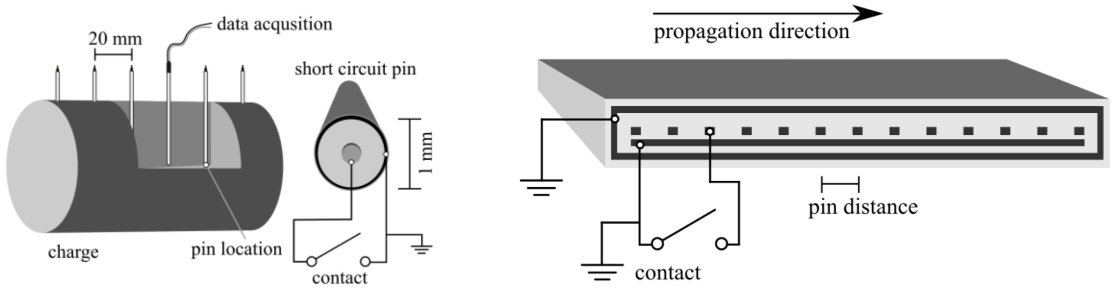
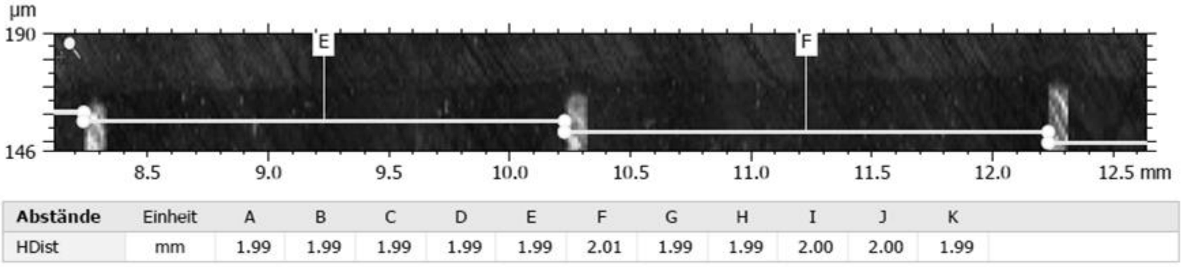


Figure 1: Standard method using shot circuit pins (left) vs. new concept using printed circuit boards (right)

For scaling down the method to reduce the required explosive mass and to be able to investigate small energetic components such as detonators, a printed circuit board design using a similar principle was developed. This allows to reduce the pin diameter by factor 10 to 0,1 mm, which equally allows to scale down the required measurement run length by factor 10 to less than 30 mm in total. Furthermore, the embedding of the pins within the PCB enables to capture the pin positions accurately beforehand the experiment using calibrated microscopes. A sample microscope image is shown in

Figure 2, where the actual pin distances can be accurately determined and used within the regression. Thereby, the observed manufacturing tolerance of 10 µm in relation to 2 mm pin distance is negligible in practice.



Abstände	Einheit	A	B	C	D	E	F	G	H	I	J	K
HDist	mm	1.99	1.99	1.99	1.99	1.99	2.01	1.99	1.99	2.00	2.00	1.99

Figure 2: Calibrated microscopy imaging for an accurate determination of pin distances

The main difference between both methods is that the small-scale method in form of the printed circuit board can only be attached to the outside of a typically cylindrical charge, whereas the pins are placed along the center axis in the case of the large-scale method. The advantage of this is that the detonation propagates completely undisturbed, as the contacts no longer have to be placed in the charge. However, this required deriving and implementing a geometrical correction for the evaluation of the small-scale method, which depends on the charge diameter and the distance of the center of ignition, which can be easily derived from geometric considerations, illustrated in Figure 3 assuming a spherically propagating detonation wave. For the classical method, the placement of the pins on the axis of the cylindrical charge lies on the normal vector of the spherical expansion, which is not the case for the outer edges within the scaled method using PCBs. While the charge diameter is a known parameter, the center of ignition requires assumptions to be modeled as a point in space, which introduces additional uncertainty. To minimize the effect, the distance between initiation and the short-circuit contacts should be generally large and the charge diameter preferably small. Since the method is generally intended to enable the reduction of the charge length, a sensible run-up distance must be selected depending on the necessary charge diameter, so that the influence of the correction is preferably small. For the investigated HWC charges of 21 mm diameter, a distance of equally 21 mm lead to an impact of the correction term of about 5 %. By doubling the distance to 42 mm, the impact decreased to 2 %. Therefore, a compromise to reduce the contribution of the correction to minimize the uncertainty of a representative center of ignition is to have a geometrical ratio between length and diameter of charge of at about factor two.

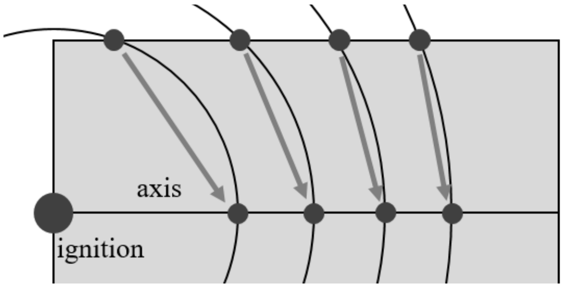


Figure 3: Illustrated spherical propagation of the detonation wave and corresponding positions on the axis and the outside of the charge

The short-circuit contacts are connected to an oscilloscope via an array of resistors to capture the generated rising signal edges. It has been proven useful to applicate oscilloscopes with at least 1 GHz band-width and 12-bit resolution and to use special low-noise high-frequency cables for signal transmission. At ICT, a new 1 GHz system with 16 analogue 50-Ohm input channels and a measurement range of +/- 10 V is in use for this purpose since 2022. A newly developed python code provides a modern flexible interface for the evaluation of the oscilloscope raw data to determine the detonation velocity. For the regression, the library statsmodels[2] is used, which allows a detailed evaluation of statistical regression parameters. The input data is defined as the time-voltage-array for each channel and the pin-position data either as a fixed distance spacing or as an array resulting from an optional

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[2] Skipper Seabold and Josef Perktold, “statsmodels: Econometric and statistical modeling with python: 9th Python in Science Conference,“ (2010).

microscopy imaging. The time-voltage data are thereby reduced to a single time point defined at a representative threshold voltage for every channel. This is achieved by validating that the time delta between the signal flanks is not dependent on the threshold voltage within a wide voltage range verified by the algorithm. From the technically point of view, this required the generation of very short signal rise times by an individually designed resistor circuit developed for this purpose.

Results and Discussion

A direct comparison for the regression curves and the derived detonation velocity for HWC (94,5 RDX, 4,5 % wax, 1 % graphite; 1,68 g/cm³, pressed at ICT) with a diameter of 21 mm is shown in Figure 4 for the full-scale method on the left and the small-scale method on the right.

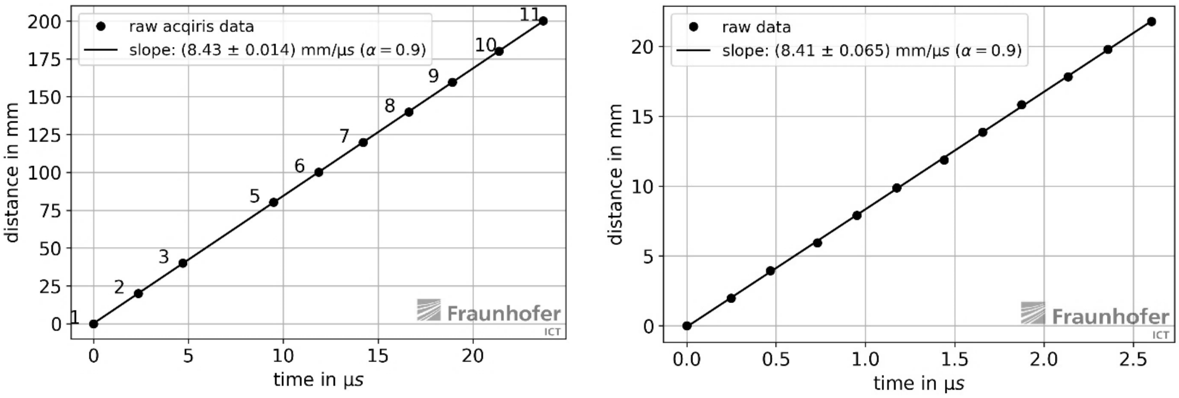
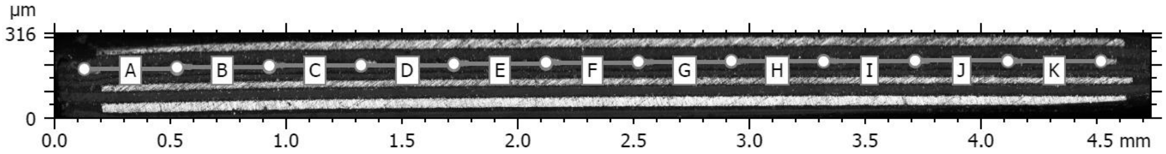


Figure 4: Regression of time-location data for HWC for the full-scale method (left) and the small-scale method (right)

To ensure a direct comparability, the short-circuit pins in the large-scale experiment were equally placed on the outside of the charge in this case. This deviates from the standard procedure, in which the pins are placed into drilled holes along the center axis. The run-up distance for both cases was more than three times the charge diameter, to ensure that the impact of the curvature correction was almost negligible, but still applied. The small-scale experiment was repeated 5 times with a resulting mean value of (8.39 ± 0.16) km/s, which is about 2 % estimated deviation for a 90 % confidence interval. The large-scale experiment was performed once. Within the regression of the data from the large-scale experiment, only 10 of the used 12 channels were included, as two signals (gauge 4 and 12) did not show distinct rising edges. Nevertheless, the goodness of fit of the regression for the large-scale method is slightly higher. The resulting detonation velocity of 8.4 km/s from both methods is thereby almost identical with the predicted detonation velocity from thermodynamic calculations by Explo 5 with 8.3 km/s as well as data from SPEED (Numerics GmbH) material database with 8.2 km/s for a similar density. The estimated variation of the mean value for the small-scale method is therefore consistent with literature data and directly comparable to the large-scale method tested for HWC. The comparison of the two methods therefore shows a high degree of comparability concerning the achieved accuracy and the detonation velocity is comparable to literature data. For an additional statistical comparison of the variation of the mean values between the two methods, repeated measurements of the large-scale method for HWC must be performed, which has not yet been done. Due to the high use of explosives and the experimental effort, the investigation of statistical influences for repeated trials within the large-scale method at ICT is generally not well known.

In the next step, the PCB has been scaled down further by reducing the pin distance while keeping the pin diameter constant. This approach suffers in principle from the same problem as the large-scale method, since the error in the local assignment of the pin positions then increases. However, in contrast to the large-scale method, the measurement of the pin spacing by means of microscopy allows the actual deviation due to manufacturing tolerances to be easily included in the evaluation. This approach made it possible to fit 12 contacts into a PCB of less than 5 mm length. The pin distance was thereby reduced to only 400 µm, which means a scaling factor of 50 compared to the large-scale method. A sample microscopy image is shown in Figure 5. In addition to the prototype in Figure *2*, additional layers for an effective electromagnetic shielding were added. The measured distance still only deviates from the specified distance by a maximum of approx. 2 % and is generally lower.



Abstände	Einheit	A	B	C	D	E	F	G	H	I	J	K
HDist	mm	0.400	0.399	0.395	0.400	0.398	0.398	0.401	0.398	0.395	0.401	0.401

Figure 5: Calibrated microscopy imaging for determination of pin distances. The additional layers represent an electromagnetic shield.

The method was then used to determine detonation velocities of HNS-pellets with a diameter of 5 mm and a length of 5 mm. The shock wave generated to initiate the pellets was transferred through a PMMA gap and can be assumed of almost planar shape. In Figure 6 an example for the time-location correlation is shown for an HNS-pellet, where the first half of the data points indicate a non-stationary region where initiation occurs, whereas the second half shows a uniform propagation. Using only the last 5 data points (corresponding to a length of 2 mm), the detonation velocity was determined.

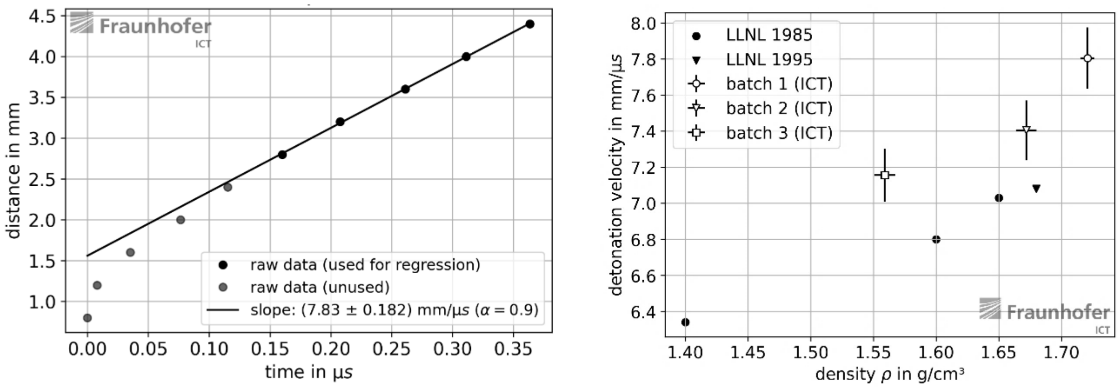


Figure 6: Linear regression for time-location data of a HNS-pellet of 5 mm length and 5 mm diameter (left) and the resulting averaged detonation velocities for 3 batches of HNS-pellets (right).

Due to the very small length to evaluate the detonation velocity and the very short pin distance, a stable mean value required repeated measurements and averaging. The resulting mean values are derived from less than 10 trials per batch and were shown for three different densities of HNS-pellets in Figure 6 (right). The errors in y-direction again indicate the variation of the mean value in the order of ± 2 %. The errors in x-direction result from the accuracy of the method used to determine the density using nano-computer-tomography. The obtained mean detonation velocities for three different densities thereby correlate with the density increase, while the absolute values are slightly higher compared to literature[3] [4] data.

Conclusion

In summary, the method for measuring the detonation velocity was downscaled by factor 50, which makes it possible to measure detonation velocities within a distance of only 2 mm, making it applicable for small charges and detonators. The principle of using short-circuit contacts has been retained, but in the form of printed circuit boards that allowed significant scaling of contact diameters and distances. By using modern oscilloscopes, low-noise cables and high-frequency shielding of the printed circuit board, distinct and high quality signals can be obtained to determine a detonation velocity. By placing it on the outside of the charge, the application effort is very low compared to the standard method and the propagation of the detonation wave is not disturbed by the pins otherwise placed in the charge within the standard method so far. By maintaining a ratio of charge length to charge diameter greater than two, the influence of the geometric correction and the associated uncertainty is low. The new method has been validated for HWC (21 mm) and HNS (5 mm) and shows high agreement with literature data and own measurements.

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[3] B. M. Dobratz and P. C. Crawford, “LLNL Explosives Handbook, Properties of Chemical Explosives and Explosive Simulants,“ (1985).

[4] P. C. Souers, B. Wu, and Haselman, L. C., Jr., “Detonation Equation of State at LLNL, 1995: Lawrence Livermore National Laboratory, UCRL-ID119262 Rev 3“ (Lawrence Livermore National Laboratory, 1996).