

Linear Correlation Between Confined Explosive Quantity and Dent Volume of an Underlying Aluminium Block Using the SSRT Setup

Lukas Bauer,^[a] Maximilian Benz,^[a] and Thomas M. Klapötke*^[a]

Abstract: The SSRT setup gives smart access to test various properties of explosive materials and requires only little substance quantities. Unlike the standardized SSRT, we studied the bulge development from the blast of different amounts of PETN (200–1300 mg). The bulge of the corresponding aluminum blocks was evaluated with the help of a profilometer (Keyence VR-5200). This device, which measures the volume of the dents using the offset of structured light projected on the object, has allowed us to analyze the differences precisely. Despite the experimental limitations

and the resulting undirected explosion direction, a throughout linear correlation between the respective amounts of PETN and the resulting dent depth could be determined. Our study thus forms an illustrative development of how the explosion behavior, represented by the dent of an aluminum block, of compressed energetic materials behaves in with increasing filling quantity, which is transferable to larger experimental setups as well as to other explosives.

Keywords: Correlation · Detonation Properties · Explosives · PETN · SSRT

1 Introduction

Initiating explosives in the Small Scale Shock Reactivity Test (SSRT) is a method, which can be used to investigate how explosive a substance is, and provides an initial estimate of the detonation behavior of the substance – even if the test is carried out below the critical diameter [1]. This test was developed to combine the advantages of the classic lead block and gap tests [2,3].

The SSRT test uses a similar procedure as the plate dent test [4,5]. In this test, the explosive under investigation is initiated by a commercial detonator and the depth (volume) of the dent formed in a solid aluminum block after initiation of the explosive material is measured using appropriate methods. The volume of the dent correlates with the power of the explosive.

The volume of a crater (massive dent) formed by the detonation of an explosive charge lying freely on the ground does not scale linearly with the mass of the explosive [6–9]. However, it has remained unclear how the dent volume in a SSRT test scales with the mass of explosive. To answer this question, we carried out the study presented in this paper.

2 Experimental Section

CAUTION! *Despite the large quantities that were handled, PETN is classified as a primary explosive and therefore extremely sensitive toward any kind of external stimuli and can*

detonate as a result of improper handling. Safety precautions and equipment (such as wearing a leather coat, face shield, Kevlar sleeves, Kevlar gloves, earthed equipment, and ear-plugs) must be used during all manipulations.

A 100 mL double jacked vessel, connected to a circulating cooler and equipped with a mechanical stirrer, was loaded with white fuming nitric acid (99%, 50 mL, 75 g, 5 Parts). At $15 \pm 3^\circ\text{C}$ 2,2-bis(hydroxymethyl)propane-1,3-diol (15.0 g, 110 mmol, 1 Part) was added portion-wise over 25 min. Then the reaction was left for completion at 5°C for 30 min. The suspension was filtrated over a PTFE membrane filter (10 μm). The solid was transferred into diluted nitric acid (11%, 150 mL) stirred for 15 min, and filtrated again. The acidic PETN was dissolved in acetone and ammonia was bubbled through the solution until neutralization was achieved. An excess of water was added slowly to yield

[a] L. Bauer, M. Benz, T. M. Klapötke
Department of Chemistry, Inorganic Chemistry, Chair of Small Molecule and Energetic Materials Research, LMU Munich
Butenandtstraße 5–13, 81377 Munich
*e-mail: tmk@cup.uni-muenchen.de

Supporting information for this article is available on the WWW under <https://doi.org/10.1002/prop.202100332>

© 2022 The Authors. Published by Wiley-VCH GmbH. This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution, and reproduction in any medium provided the original work is properly cited.

PETN after filtration as a fine powder (33.5 g, 106 mmol, 96%, grain size: 100–200 μm (85%)).

DTA (5°C min^{-1} , onset): 143°C (melt) followed by decomposition; Sensitivities: BAM drop hammer: 3.5 J, friction tester: 54 N; IR (ATR) $\tilde{\nu}$ (cm^{-1}) = 2986 (w), 2904 (w), 1638 (s), 1630 (s), 1473 (m), 1396 (w), 1305 (m), 1283 (s), 1266 (s), 1036 (m), 998 (s), 939 (m), 833 (s), 752 (s), 701 (s), 619 (s), 458 (m); Elem. Anal. ($\text{C}_5\text{H}_8\text{N}_4\text{O}_{12}$, $316.15 \text{ g mol}^{-1}$) calcd.: C 19.00, H 2.55, N 17.72%. Found: C 19.01, H 2.28, N 17.47%; ^1H NMR ($\text{DMSO-}D_6$, 400 MHz, ppm) $\delta = 4.70$ (s, 8H); ^{13}C NMR ($\text{DMSO-}D_6$, 101 MHz, ppm) $\delta = 70.3, 40.8$; ^{14}N NMR ($\text{DMSO-}D_6$, 29 MHz, ppm) $\delta = -45$.

3 Results and Discussion

3.1 Sample Preparation

For our experimental determinations, we used a classic setup as described for SSRT (small-scale shock reactivity test) experiments [1b]. The experimental setup consists of a cylindrical aluminum block (height: 25.0 mm, diameter: 50.0 mm, EN AW-2007) with a matching steel block (height: 25.0 mm, diameter: 50.0 mm, hot-rolled mild steel) placed on top of the aluminum block. The steel block is provided with a 7.5 mm wide hole in the center, into which the substance to be tested is later filled. For easier handling, both blocks are temporarily tied together with the help of a crepe tape (Figure 1). Subsequently, the appropriate amounts of PETN were poured into the hole of the steel block with a funnel.

Particularly with filling quantities of 1000 mg or more, the material had to be pre-compacted manually by tapping to accommodate the entire quantity. A suitable steel pin (diameter: 7.45 mm) was then placed in the hole on top of the PETN and pressed with the aid of a hydraulic press at a pressure of 3 tons. This was repeated twice in order to obtain the maximum possible compression. After removal of the steel pin, a thin PETN layer remained on the inner wall

of the steel block bore. This was removed with the aid of a pipe cleaner to ensure a maximum insertion length of the detonator later on.

The blocks with the compressed PETN were then placed in a steel plate with a matching recess at the bottom. A corresponding steel lid with an opening around the steel hole was then placed on top of the blocks. The two steel plates are each provided with four holes, allowing matching threaded rods to be used for fixation. The respective screws for fastening were hand-tightened (Figure 2–3). This setup was designed to prevent the blocks from slipping and counteract the force of the explosion and project the maximum of the released energy onto the aluminum block.

The detonator (Dynadet-C2) was then inserted into the bore of the steel block and placed as close as possible to the compressed PETN with the appropriate force. After initiation, the screws of the fastening device were loosened and the aluminum block was removed. In the case of smaller quantities of PETN, it happened that parts of the alumi-

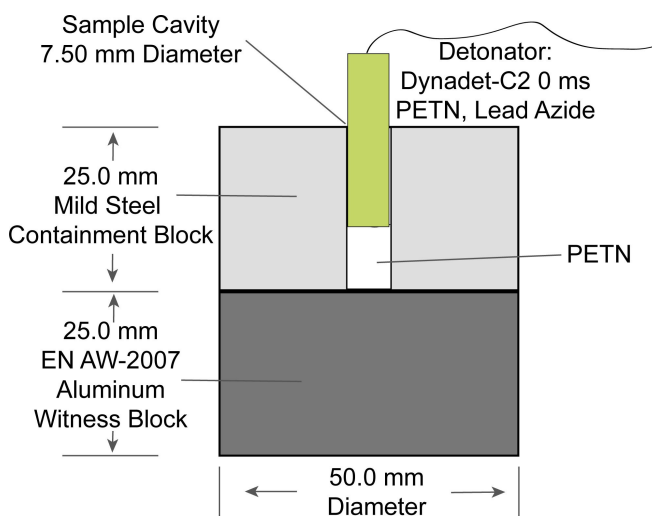


Figure 2. Schematic setup of the experiment as displayed in Figure 3 (left).

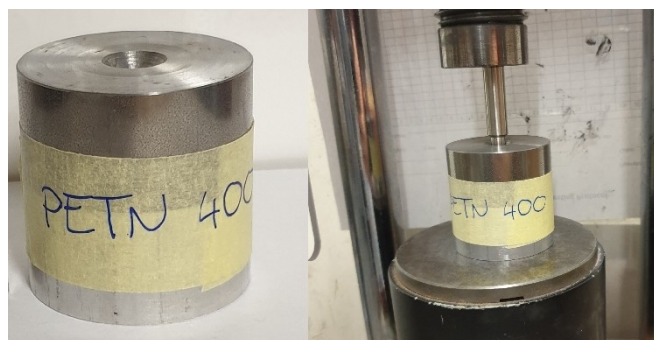


Figure 1. Experimental setup consisting of an aluminum block and steel block with hole connected by crepe tape (left), Test setup during the pressing process (right).

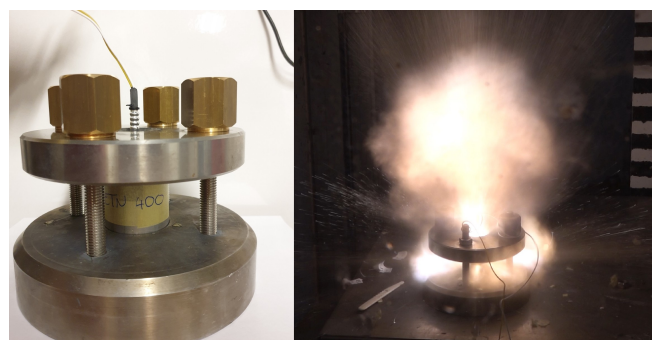


Figure 3. Test blocks in fixing device with detonator inserted in the steel block hole (left), Moment of detonation of 400 mg PETN (right).

num sleeve of the detonator were blasted into the block. These could be easily removed with pliers. The block was cleaned of any soot present by washing it with water and acetone.

3.2 Analysis

The obtained aluminum blocks were analyzed through non-contact measurements with the XYZ-axis motorized 3D profilometer VR-5200, produced by Keyence (Osaka, Japan; Figure 4). The measuring range is 200 mm × 100 mm × 50 mm with a resolution of up to 1 μm [10]. Structured light is emitted by two angled telecentric transmitter lenses and projected on the surface of the aluminum block. The offset of the structured light is detected by the receiver lens and appears banded and bent based on the changes of topography on the surface of the block [11]. The offset of the structured light is then used to calculate the dimensions of the block and the dent depth using the light sectioning method and triangulation [10, 12].

No anti-reflective spray was needed for these measurements. Up to six blocks were measured in one pass, then turned by 90° and measured again to avoid blind spots in

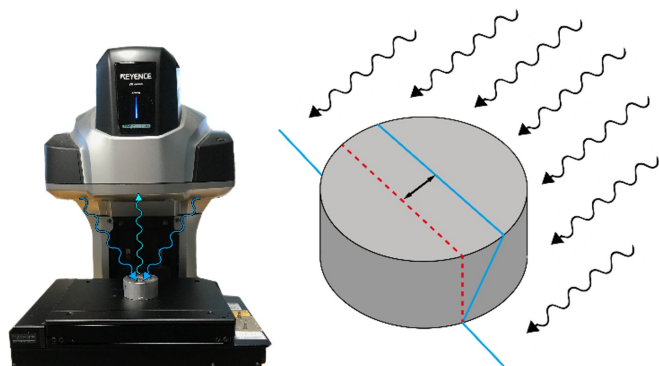


Figure 4. Profilometer with indicated light emitting from the angled lenses and detected by the receiver lens in the middle (left); Illustration of the light sectioning method and triangulation (right).

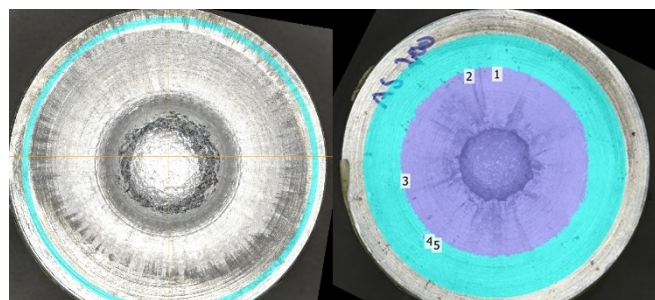


Figure 5. Reference area indicated in cyan (left); Measured area indicated in cyan and detected indentations indicated in violet and numbered.

the measurement. The volume of the dent was determined as follows: In the analysis software, a ring of 1 mm thickness and an average diameter of 47 mm was placed on the block. The resulting area was used as a reference area to determine the reference height of the block (Figure 5).

The blocks were then aligned and the depth of the dent inside the area of the ring was determined. Finally, the two values for the first second measurement were averaged.

3.3 Method and Evaluation

For our test series, we decided on quantities of PETN starting from 200 mg (minimum compressible quantity) in 100 mg intervals progressively up to 1300 mg (maximum filling quantity of the borehole). The tests with the respective PETN quantities were performed twice to be able to form an average value of the dent volumes and thus minimize the error and deviation. Since the detonator used contains a considerable amount of explosive material and thus has an effect on the depth of the dent, this effect was investigated separately. A blank test, containing $(\text{NH}_4)_2\text{SO}_4$ instead of PETN was carried out for each filling quantity and thus for each filling height since it was to be expected that the influence of the detonator decreases with increasing distance to the aluminum block. Finely ground ammonium sulfate was used as filler material for the blank samples, as it has the same ideal crystal density as PETN (1.77 g cm^{-3}), is not particularly moisture sensitive or hygroscopic, not ignitable, thermally stable, and can be readily compacted in fine powder form. The respective samples were then prepared and ignited as described in section 3.1.

As expected, the dent depth decreases with increasing filling level of unreactive material. The blue fitting line included in Figure 6 illustrates an exponential regression of the dent depth. It is nevertheless remarkable that a filling quantity of 1300 mg of ammonium sulfate still results in a similarly strong bulge as with 1000 mg since the detonator was only a few millimeters inside the steel block and thus the explosion energy was able to escape uncentered and distributed in all directions.

The values obtained were identified as zero points for the experiments with PETN to illustrate only the corresponding influence of the explosive force of PETN on the dent depth. The values, as well as the graph for the absolute blast depth, can be found in the Supporting Information.

The blocks were measured twice and rotated by 90° in-between measurements to capture any unexposed areas. The volumes obtained were averaged for the values of blast 1 and blast 2. To obtain the adjusted volumes, the dent depth values caused by the detonator obtained by the fitted exponential regression curve were subtracted from the absolute averaged volumes displayed in Table 1.

The values obtained from the PETN measurements, corrected by the ammonium sulfate experiments, result in the

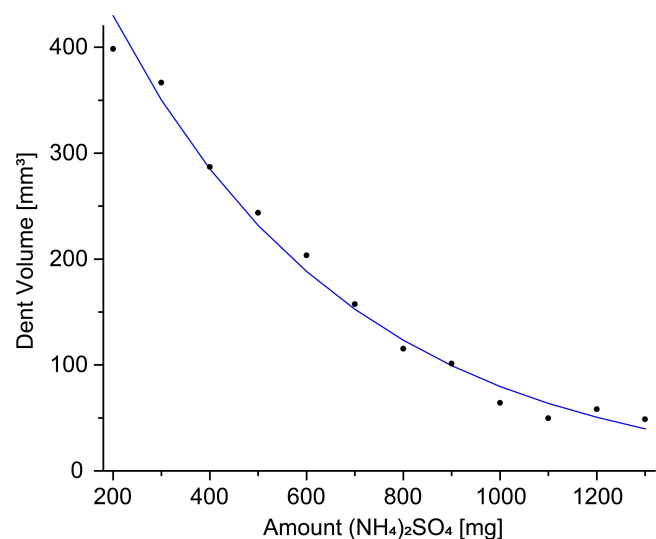


Figure 6. Dent depth caused by the detonator depending on the distance from aluminum block ensured by different amounts of ammonium sulfate as filler material. The blue line corresponds to an exponential regressive fit with the equation $y = 658.21 \cdot e^{-0.002x}$.

Table 1. Values obtained for the dent depth of the two blasts with respective PETN charges, deviation of the values from each other, averaged absolute volume of blast 1 and 2, and adjusted blast volume of the PETN charges calculated as average volume minus the dent volume generated by the detonator.

Amount PETN [mg]	Blast 1 [mm ³]	Blast 2 [mm ³]	Deviation [%]	Average Volume [mm ³]	Adjusted Volume [mm ³]
200	874,25	937,21	3,48	905,73	497,95
300	1072,27	1010,01	2,99	1041,14	617,18
400	1074,13	1137,51	2,87	1105,82	736,41
500	1151,01	1238,23	3,65	1194,62	855,64
600	1262,35	1233,94	1,14	1248,15	974,87
700	1263,54	1244,03	0,78	1253,79	1094,1
800	1260,68	1343,54	3,18	1302,11	1213,33
900	1377,05	1333,14	1,62	1355,10	1332,56
1000	1543,19	1519,20	0,78	1531,19	1451,79
1100	1624,01	1688,12	1,94	1656,06	1571,02
1200	1694,67	1776,06	2,35	1735,37	1690,25
1300	1807,79	1909,65	2,74	1858,72	1809,48

graph shown in Figure 7 with a linear fit added as a blue line. Considering some errors, there is a linear relationship between the amount of explosive and the dent depths, for the chosen amounts of explosive. We did not expect a linear dependence but rather a sloping curve. We arrived at this assumption because, as the quantity of PETN filled in increased, we reached the capacity limit of the steel block and the explosive contained in it could no longer just emit a targeted pressure wave in the direction of the aluminum block, but could also increasingly decay upwards.

We consider several factors to be responsible for the deviation of some of the values from the ideal blue line. First,

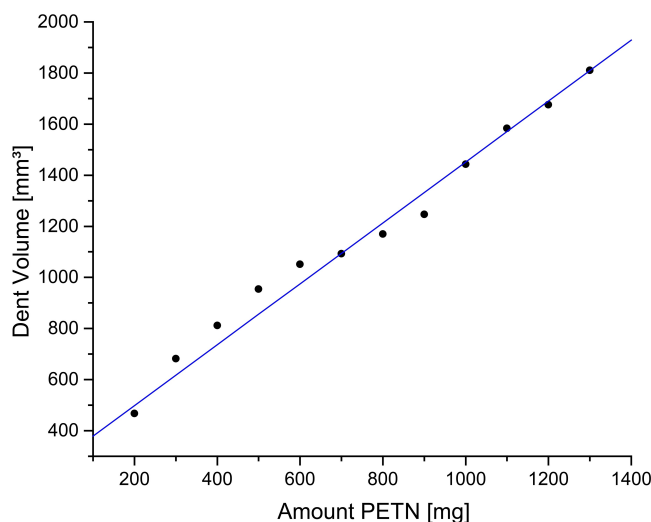


Figure 7. Aluminum block buckling caused by PETN subtracting the pre-determined fitted base buckling caused by the detonator. The blue line corresponds to a linear fit with the equation $y = 1.1923x + 259.49$.

each set of PETN amounts was shot only twice, which gives a useful tendency but cannot be called statistically exact. The material used was weighed, filled, and pressed manually, whereby inaccuracies and deviations in the quantity may occur. In addition, despite the compression method used, there is no guarantee that the PETN used has compacted ideally. Should this not have been the case in a batch, this may have led to an increase in porosity and thereby the addition of small cavities. This, in turn, would reduce the resulting dent volume, as not all energy is transferred to the witness block but is also used for the compression of the sample and gases in the cavities. Despite the chosen SSRT setup, the detonation also releases some of the energy into the deformation of the steel block. Figure 8 shows a steel block before detonation and below a steel block after the detonation of 1300 mg PETN. The force of the detonation caused the borehole to widen from 7.5 mm to 11.7 mm. We assume that with an increase of the amount of explosive proportionally more energy is released in other directions. Larger explosive quantities will likely result in a flattening of the curve.

4 Conclusion

In summary, we investigated the correlation between the amount of PETN (200 mg–1300 mg) filled in a SSRT setup and the resulting dell depth of the associated aluminum block after the ignition of the explosive. The evaluation of the respective depths of the aluminum blocks after the explosion was carried out with the aid of a profilometer. The resulting extremely precise measurement data made it possible to compare the blocks with each other and to work



Figure 8. Steel block (left) and aluminum block (right) before (top) and after (bottom) the detonation of 1300 mg PETN.

out a trend. Taking into account some experimental errors and the influence caused by the detonator, a linear relationship could be drawn. The results of this study could be applied to other energetic materials as well and serve as an experimental basis for future theoretical calculations or possible larger test quantities.

Acknowledgement

For financial support of this work by Ludwig-Maximilian University (LMU), the Office of Naval Research (ONR) under grant no. ONR N00014-19-1-2078 and the Strategic Environmental Research and Development Program (SERDP) under contract no. W912HQ19C0033 are gratefully acknowledged. The authors are indebted to thank Tobias Lenz for the synthesis of PETN. Open Access funding enabled and organized by Projekt DEAL.

References

- [1] a) J. E. Felts, H. W. Sandusky, R. H. Granholm, Development of the smallscale shock sensitivity test (SSST), *AIP Conf. Proc.*

- 2009**, 1195, 233; b) H. W. Sandusky, R. H. Granholm, D. G. Bohl, Small-Scale Shock Reactivity Test (SSRT), IHTR 2701, Naval Surface Warfare Center, Indian Head, MD, 12 Aug 2005; c) R. H. Granholm, H. W. Sandusky, SMALL-SCALE SHOCK REACTIVITY AND INTERNAL BLAST TEST, *IP Conference Proceedings* 845, 1257, **2006**; <https://doi.org/10.1063/1.2263553>.
- [2] T. M. Klapötke, T. G. Witkowski, 5,5'-Bis(2,4,6-trinitrophenyl)-2,2'-bi(1,3,4-oxadiazole) (TKX-55): Thermally Stable Explosive with Outstanding Properties, *ChemPlusChem* **2016**, 81, 357–360.
- [3] K. V. Domasevitch, I. Gospodinov, H. Krautscheid, T. M. Klapötke, J. Stierstorfer, Facile and selective polynitrations at the 4-pyrazolyl dual backbone: straightforward access to a series of high-density energetic materials, *New J. Chem.* **2019**, 43, 1305–1312.
- [4] N. Dhawan, B. D. Majumdar, B. Abraham, A. G. Rajendran, Comments on the dent test vs. metal acceleration, *9th Intl. High Energy Materials Conf. and Exhibits, HEMCE-363, SVVC, Trivandrum, India, Feb. 13–15, 2014*.
- [5] B. T. Neyer, Use of VISAR to replace detonator dent tests, *31st AIAA/ASME/SAE/ASEE Joint Propulsion Conference and Exhibit, San Diego, CA, 1995*.
- [6] I. D. Bjelovuk, S. Jaramaz, P. Elek, D. Micković, L. Kričak, Estimation of the explosive mass based on the surface explosion crater on asphalt, *Tehnički vjesnik* **2015**, 22, 227–232.
- [7] D. Ambrosini, B. Luccioni, R. Danesi, Craters Produced by Explosions on the Soil Surface, *Meccanica* **2003**, XXII, 678–692.
- [8] T. M. Klapötke, *Chemistry of High Energy Materials*, 6th edn., chapter 1.3, Walter de Gruyter, Berlin/Boston, **2022**.
- [9] R. Mayer, J. Köhler, A. Homburg, *Explosives*, 5th edn., Wiley-VCH, Weinheim, **2002**, 148.
- [10] Keyence, <https://www.keyence.de/landing/lpc/vrprofilometer.jsp> (accessed November 2021).
- [11] M. A. A. Neil, R. Juškaitis, T. Wilson, Method of obtaining optical sectioning by using structured light in a conventional microscope, *Opt. Lett.* **1997**, 22, 1905–1907.
- [12] T. Takatsuji, A. Kirita, T. Kurosawa, A simple instrument for measuring edge angles using a light sectioning method, *Meas. Sci. Technol.* **1997**, 8, 782–786.

Manuscript received: November 16, 2021
 Revised manuscript received: January 11, 2022
 Version of record online: February 25, 2022