Tomographic study of the structure of cast and pressed trotyl charges

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ABSTRACT

The sensitivity of condensed explosives depends strongly on the charge structure. Trotyl (TNT) is a typical example of such dependence. The pressed charges are more sensitive than the cast ones, which was confirmed in many studies including critical diameter tests and chemical spike width estimates. Our measurements of the electrical conductivity distributions in the dense TNT support the same conclusion.

In this paper, results of tomographic study of the structure of pressed and cast trotyl are presented. The distribution functions of pores larger than 4 μm were obtained using synchrotron X-raying. In cast TNT, voids varying from units to hundred microns were observed, while the pressed charge was found to be substantially more uniform and less porous. The roles of possible mechanisms of hot spots generation are discussed.

1. Introduction

The reaction kinetics, as well as the critical parameters of the energetic materials, are sensitive to the charge structure. According to the generally accepted understanding, the reaction starts within the multiple local non-uniformities, usually called hot spots [1–14].

Among the various mechanisms capable to generate hot spots under shock impact, collapse of pores is regarded as one of the most probable scenarios [3–6]. In a number of papers, such collapse is treated numerically, usually starting from geometrically regular voids. In order to understand the physics of the collapse, experimental and numerical study of void deformation in an inert material (PMMA) were performed for different amplitudes of shocks – from weak ones to those typical for detonation waves [15]. In the work [7], reaction in voids in TATB (triaminotrinitrobenzene) and HMX (cyclotetramethylenetetranitramine) was modeled at different shock pressures P_S. The dependence of critical pore size on P_S was obtained. At the typical detonation pressures, voids of less than 0.5 μm in diameter became effective. For TATB, the highest temperatures (1 500–1 800 K) are reached in collapse of small pores. The maximal size of voids in simulations was about 4 μm.

In several papers, the effect of the morphology and arrangement of voids on the sensitivity was studied [8–10]. Shock impact on the powdered HMX using real size distribution of grains was simulated

[16–18].

Other non-uniformities can be considered as hot spot precursors as well. A review of the influence of the crystal defects on the sensitivity of the energetic materials is presented in the work [11]. In work [12], structural non-uniformities are considered which can lead to ignition under shock waves. In the work [13], shocks in TATB were modeled using molecular dynamics. Nanometer-scaled shear bands were found in which the reaction barrier was somewhat lower, which should favor the ignition. In the work [14], to describe the initiation of the explosive, a multiscale model is proposed incorporating the range from molecular level to macroscale, taking the material structure into account. Other multiscale models are considered in works [4,6].

Given the progress in computational power, the modeling becomes more detailed, with increased resolution and new physical effects incorporated into codes. But, without the actual structure parameters, the capacity of the numerical simulations is obviously limited.

The variability of the critical diameter d_{cr} observed for the charges prepared using different procedures is a clear illustration of the sensitivity-structure relationship. For TNT, this phenomenon is quite pronounced. In Table I, critical diameters for dense TNT of various structures are presented. Even for the similar technology (cast charges) d_{cr} can differ by the factor 2 (lines 4, 5 and 6, 7, 9), and for higher density the critical diameter is smaller. Largest critical diameters were found in

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(a)

(b)

Fig. 1. **(a)** Critical diameter of TNT with different grain size and structure, curves 1, 2, 3, 4 corresponding to the density of 1 g/cm³. 1 – crushed fractionated TNT of a homogeneous structure [24,25], 2 – aggregate fractionated [25], 3, 4 – the charge is placed in a thin-walled glass tube, the horizontal lines show the dispersion range [20]. Color-filled areas show the range of values at close to maximum density for pressed and cast charges. **(b)** Dependence of critical diameter on density: 1, 2 – at different dispersity (data correspond to Fig. 1(a)), 3 and 4 – charge with different initial grain size is in a thin-walled glass tube [20], 5 – pressed high-density charge, 6 – cast charge, data of Table 1.

monocrystal and in liquid TNT.

The dependence of the TNT critical diameter at the bulk density of 1 g/cm³ on grain size a is shown in Fig. 1(a). 1: grains are solid, 2: grains are aggregates (powdered TNT with 0.06 mm particle size was pressed to 1.5 g/cm³, then crushed and fractionated), 3, 4: powdered TNT of different grain size, the charges fill thin glass tube. The coloured areas mark the critical diameters for dense cast and pressed TNT. The critical diameter at fixed density is smaller for small grain size. When a increases, d_{cr} falls into the area of dense insensitive cast TNT.

The dependence of the critical diameter on density is shown in Fig. 1 **(b)** for all data except monocrystal and liquid state. At the similar density d_{cr} differs for an order of magnitude in cast and pressed charges (Table 1, lines 4, 5 and 8). The range of d_{cr} values is the same, both at bulk density and at the maximal one, so it is not possible to relate the sensitivity to the density. The data in Table 1 and Fig. 1 suggest that

Table 1
Critical diameter d_{cr} of TNT charge at the different manufacturing methods.

N	Method of making a charge	ρ, g/cm³	d_{cr}, mm	ref.
1.	liquid	1.443	62.6 ± 2.6	[19]
2.	pressed in a thin-walled glass tube	1.50	2.5	[20]
3.	pressed	1.51	3 ÷ 5	[19]
4.	cast type II[a]	1.60	27.5	[21]
5.	cast "coarse-crystal"[a]	1.60	27.5	[22]
6.	creaming and casting	1.615	14.6 ± 2.0	[19]
7.	layer-by-layer casting	1.615	12.6	our data
8.	pressed	1.62	2.6 ± 0.6	[19]
9.	vacuum melting and casting	1.62	14.5 ± 0.5	[19]
10.	cast "fine crystal"[a]	1.62	15	[22]
11.	cast type I[a]	1.62	16	[21]
12.	single crystal	1.663	110	[23]

[a] Retained original name.

neither density, nor grain size or porosity, taken separately, are key factors for sensitivity. The decisive factor is the charge structure. Thus, detailed study of the charge structure is necessary to understand which nonuniformities, of which type and scale, affect the sensitivity.

Theoretical attempts to derive the pore space configuration from the initial grain size are hardly practical [20,26]. One can observe the charge surface, but its morphology is likely different from that inside the charge. The surface of the sliced or cracked specimen is also distorted (as one can see from the data presented below). For the cast TNT, the morphology of the voids depends on casting technology, the cooling rate, charge geometry, casing material, etc. and is even more uncertain. The properties of cast TNT are so sensitive to the subtle details of technology, that such details are accurately described for each charge sort [21,22,27]. So, the scanning of real inner structure of explosive is an actual and interesting problem.

This paper offers a non-destructive method to study an actual three-dimensional charge structure.

2. Experiment

2.1. Charge preparation

For the pressed charge, commercial TNT was used. Original flakes were crushed manually in a ceramic mortar. The images of grains are shown in Fig. 2. The main part of the explosive grains had the size of 10–30 μm. TNT was pressed at about 90 atm. The density was $\rho_{pres} = 1.6198 \pm 0.0099$ g/cm³, the corresponding porosity was 0.0260.

The density of liquid TNT ($\rho_{liquid} = 1.467$ g/cm³) is lower than that of solid material ($\rho_{crystal} = 1.663$ g/cm³) [28], which is a main source of defects in cast charges. To minimize the forming of large inner voids, the cast charges were prepared pouring multiple thin layers. The density of cast charge was $\rho_{cast} = 1.6151 \pm 0.0029$ g/cm³, the corresponding porosity was $m_{cast,exp} = 0.0288$.

The samples used for tomography were cylinders with a diameter of 1.5 mm, which were cut from larger pellets.

2.2. Tomography method

Numerical X-ray tomography provides three-dimensional data describing the structure of specimens under study. The data can be represented as a stack of coloured density maps. White areas will mean the maximal density. The pores have contrast boundaries which allows one to get their clear distribution.

The sketch of the measuring facility is presented in Fig. 3. The X-ray source was synchrotron radiation (SR) beam produced by wiggler incorporated into VEPP-3 accelerator (Institute of Nuclear Physics, RAS Siberian Branch). The angular spread VEPP-3 SR beam is 0.1 milliradian, and total beam X-ray power is 100 W. The small angular dispersion and high brightness of SR allows one to obtain good spatial resolution, about 2 μm for short-wave range (5–30 keV), used for explosive diagnostics.
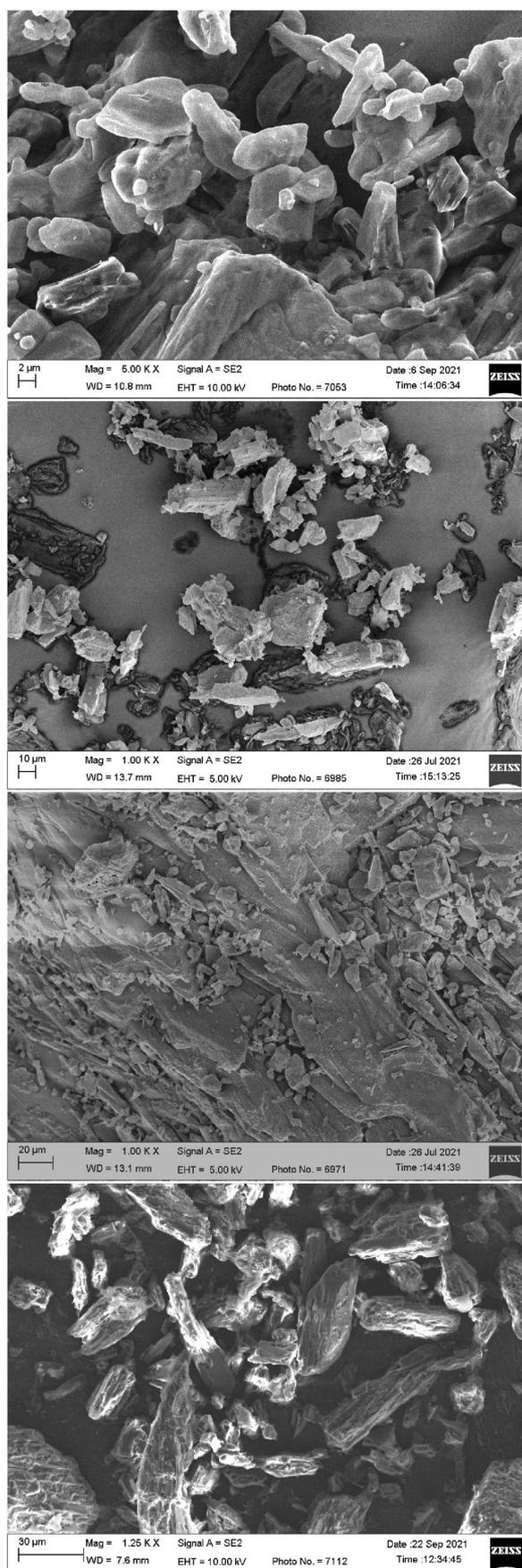
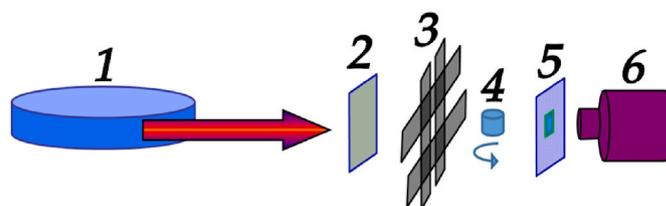**Fig. 2.** Micrographs of the initial grains used in pressing.

Fig. 3. Diagram of a tomographic study of the chargedensity: 1 - source of synchrotron radiation VEPP–3, 2 – filter, 3 – collimator, 4 – sample on a rotating stand, 5 – scintillator, 6 – detector.

Aluminium was chosen as a material for X-ray filters since it does not have any peculiarities in the used energy range. The absorption of explosive specimens was recorded using thin scintillation screen. The main sensing material was cesium iodide doped by tallium CsI(Tl), which has high luminous yield and is effective in X-ray range. A high-resolution CCD array (Hamamatsu ORCA-Flah2.8) with a size of 2048 × 2048 pixels was used as a detector. Single pixel size, taking objective magnification factor into account, was 0.6 μm. The dynamic intensity range of the detector was 16 bit. The specimen, scintillation screen and detector were positioned with 1 μm linear and 0.001 angular accuracy. To form the SR beam, 1.5 × 1.5 mm X-ray collimator was used.

The specimen was rotated, and a set of the projections was recorded, which was used to restore three–dimensional map [29,30]. The final spatial resolution was 2.6 μm.

3. Tomographic study of cast and pressed TNT: results

In Fig. 4, an example of tomographic data is presented (multimedia view contained in Supplementary data). Two cross-sections picked from the inner parts of charges correspond to cast and pressed TNT. Cast charge is highly nonuniform. One can see multiple pores up to dozens of μm in size. Some voids (marked by circles) have sufaces of concave form. The pressed specimen is more uniform, pores are generally smaller, though large pores are present in fewer number.

In Fig. 5, the tomographic sections are compared with the initial TNT grains. On the cut of the pressed charge, cracks are visible, which were formed during manipulations with the sample in the process of manufacturing and processing to the desired shape. The cavities are predominantly compact in shape, close to ideal, although there are also pores with a concave inner surface. No traces of grains can be identified in the pressed specimen. Since TNT is not very strong material, the grains are easily deformed under pressing, which leads to better uniformity and to smoothing of the boundaries of grains. The cracks visible in the pressed charge arise presumably during the extracting of the specimen from the mold.

The initial data of the tomographic examination and layer-by-layer visualization can be found at the link http://ancient.hydro.nsc.ru/tomography.

3.1. Statistic of voids

For comparative analysis, from the numerical specimens obtained by X-raying the cast and pressed TNT, two identical cylinders were cut containing 600 cells in diameter and 451 cells high. The cell size was 2.6 μm, and the total physical volume $V_0 \approx 2.24$ mm^3. Fig. 6 displays the distributions of pore numbers $N_p(d)$, volume of voids $V_p(d)$ and surface of voids $S_p(d)$ on d at Fig. 7(a). Here, d is the diameter of sphere which has the volume equal to the pore volume. For all graphs, general decrease with increasing d is observed. Maximal values correspond to the minimal displayed $d = 4$ μm. For this size, pore numbers, pore volumes and pore surface for cast and pressed specimen do not differ significantly. However, the cast charge clearly has more large pores. In the cast charge, voids which have $d > 10$ μm are present in significant
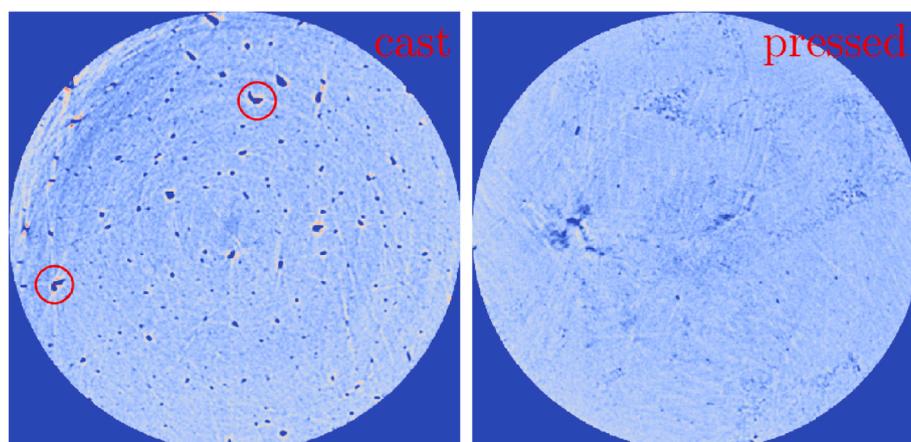
Fig. 4. Structure of charge. Sample diameter 1.5 mm, cast on the left, pressed on the right. The cast contains pores with a concave inner surface (highlighted by circles). The shape of the pores is represented by various geometrically irregular figures.
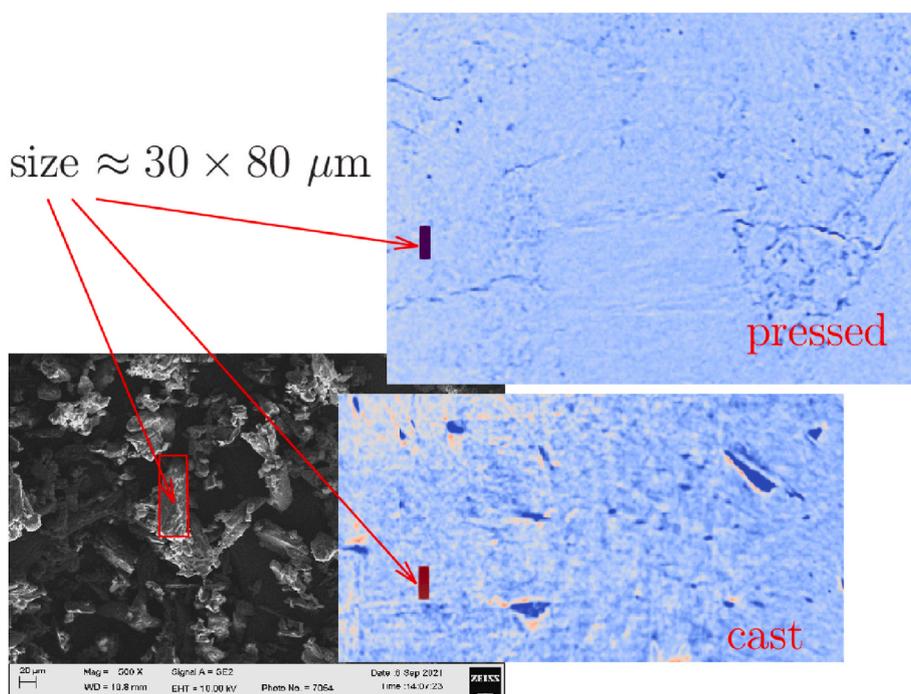

Fig. 5. Tomographic sections and a microphotograph of crushed explosive grains are shown; all photos show a 30×80 μm fragment.

number, their volume is about 10% of total pore space. The equivalent diameter of largest pores in cast charge is ≈ 70 μm while in the pressed charge the corresponding value is ≈ 20 μm.

The largest pores found in cast TNT can be fragmented behind the shock wave, forming several smaller pores. Each fragment can generate hot spot. Thus, large irregular pores potentially may be not less effective for ignition than smaller ones.

Fig. 7(b) shows the integral volume of pores calculated from minimal resolved size to the given d value. The results of the tomographic study are in good agreement with the data of direct density measurements. Some difference within error margins may occur due to limited spatial resolution (e.g., pores smaller than 4 μm are ignored) or from computational errors arising for highly irregular large pores. This problem will be addressed in the following study. For now, the tomographic method seems to be more perspective than common approaches such as observation from surface or percolation experiments [31].

Note that the tomographic method for studying the internal structure of objects finds wide practical application in medicine [32–37], for

geological exploration using a probe beam with different wavelengths, the method is suitable for studying structures of various and material properties, see Ref. [38] for example. In this work, the method of tomographic research is used in relation to explosives.

4. Electrical conductivity distribution

Previously, we developed a technique to measure electrical conductivity profile behind the detonation wave front in dense high explosives [39,40]. The consistency between the conductivity and the carbon content was confirmed [41–44]. For RDX (cyclotrimethylene trinitramine) [45], HMX, PETN (pentaerythritol tetranitrate) [40], BTF (benzotrifuroxan) [46], TATB [47], HNS (hexanitrostilbene) and TNT [48], the conductivity was found to increase with the density [49–51]. The correlation of the conductivity peaks and the chemical reaction zone was established [52–54]. This opens a perspective to use the conductivity diagnostics to study the chemical reaction kinetics in detonation waves.
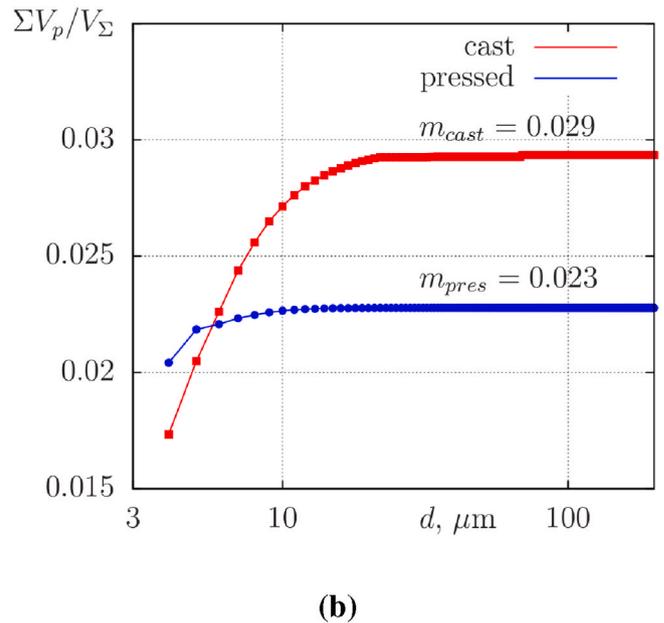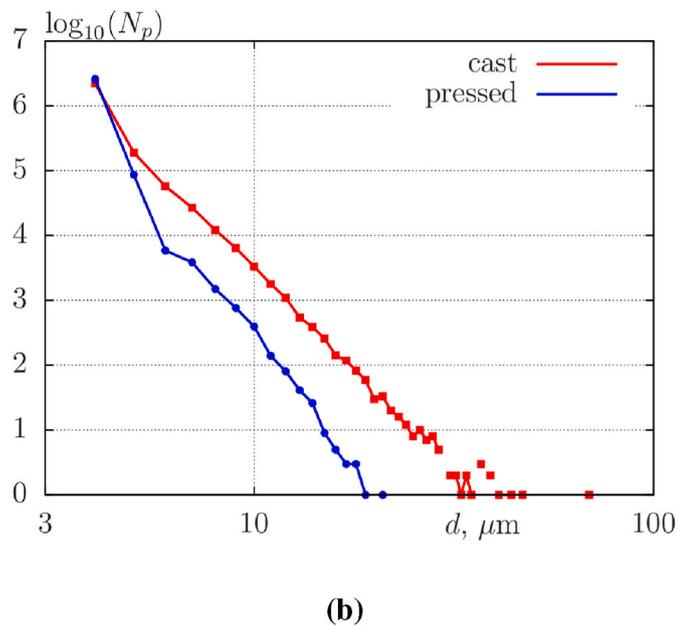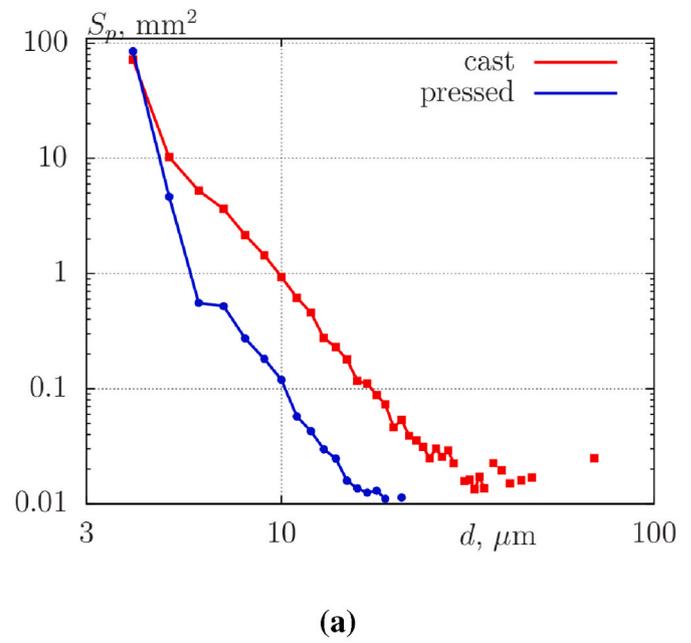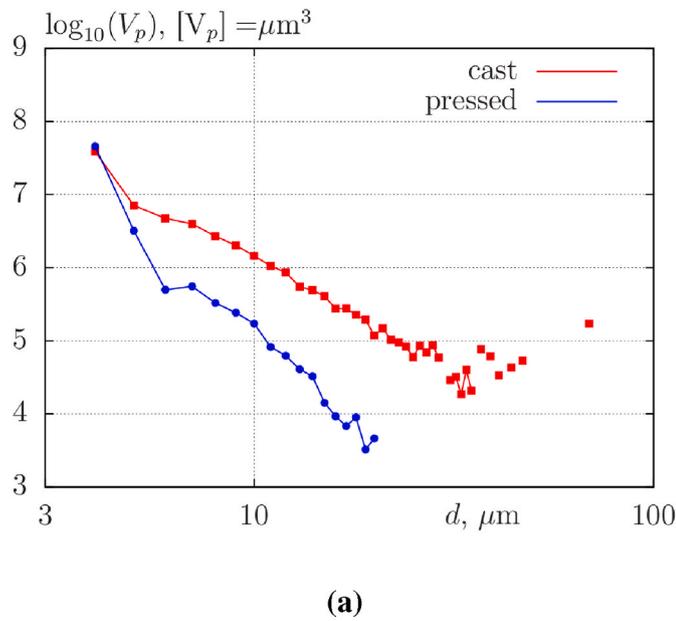
(a)

(b)

Fig. 6. The results of a tomographic study of voids in a cast and pressed charge: **(a)**: volume of pores V_p for a standard spherical pore diameter d, **(b)** – number of pores N_p.

(a)

(b)

Fig. 7. The results of a tomographic study of voids in a cast and pressed charge: **(a)** – inner surface area S_p, $S_{p,pres} = 244$ cm^2/g, $S_{p,cast} = 273$ cm^2/g, **(b)** – integral over pores normalized to the sample volume. Porosity m_{cast} and m_{pres} calculated from tomographic data.

In Fig. 8 electrical conductivity profiles in detonating TNT are shown: pressed (curve **1**) and cast (curve **2**). The experiments were performed using the same procedure as in works [39,40]. Both cast and pressed charges were prepared in a manner similar to that described in Sec. 2. The detonation velocities (see Fig. 8) are almost equal. Nevertheless, the conductivity profiles are quite different. To our opinion, sharp triangular peak ($\sigma_{max} \approx 100$ Ohm^{-1}cm^{-1}) in pressed TNT indicates higher reaction rate whereas in cast charge the conductivity is four times lower, and the reaction is slower. The high value of electrical conductivity, similar to that obtained for pressed TNT, is confirmed by the results of the works [55,56]. It is well known that the sensitivity of the pressed TNT charge is higher than that of the pressed one, this is true for both the critical diameter and the shock wave sensitivity [57–62]. Our interpretation of the electrical conductivity data on different chemical reaction rates is in good agreement with the known information on critical diameters. According to Kobylkin [63–65], critical

diameter is in inverse proportion to the reaction rate. The fact that the critical diameter of pressed TNT is smaller than that in cast trotyl means that in pressed explosive, the reaction rate is higher. The results of papers [60,61], which reported about five times higher reaction rate in pressed TNT ($\rho = 1.56$ g/cm^3) as compared with cast TNT ($\rho = 1.61$ g/cm^3) also support this view.

5. Conclusion

Sharp difference in the reaction rates of pressed and cast TNT charges is a well established fact. In the preceding sections we mentioned indicators of this difference, including critical diameter data, direct measurements of the pressure profile, and added a comparison of the electrical conductivity distributions. All evidence is in favor of the
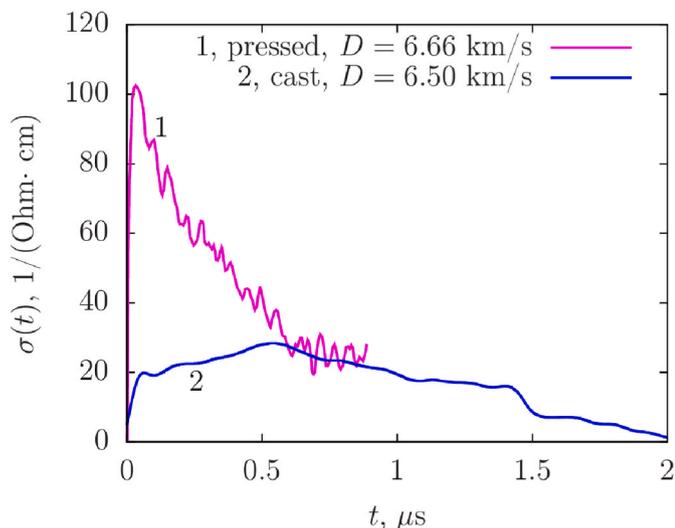
Fig. 8. Electrical conductivity profiles in detonating TNT: **1** – pressed [49], **2** – cast.

conclusion that pressed TNT is more active explosive.

Nevertheless, our tomographic data seem to contradict this conclusion. While the number of smallest resolvable pores ($d \approx 4$ μm) was almost equal, the larger voids in cast charge were sufficiently more numerous than in the pressed one. However, the pores are regarded as most probable precursors of the hot spots.

To our opinion, this contradiction can be resolved in two alternative ways. Either collapse of pores is actually the working process, but the active voids are smaller than 4 μm, and our spatial resolution was insufficient to observe them. According to different view, not pores but multiple contact boundaries between the explosive grains are the main source of hot spots in the pressed charge. The molecules which form the boundaries are in different state than those inside the microcrystals, their activation energy can be lower, and deformation effect in such locations should be more intense. Thus, the grain boundaries seem to be perspective as the ignition sites. These conclusions are in good agreement with the work [66] in which an alternative for pore collapse kinetics is proposed, based on the diagnosis of a chemical reaction zone through electrical conductivity. For now, the mechanism of void collapse seems to be more popular in the literature. But it may be rather the result of more clear physical picture and more convenient numerical modeling of this process, than of solid justification needed for an established theory.

To our information, the tomographic study is coupled with the sensitivity data for the first time in the present paper.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.rineng.2022.100621.

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