

Effect of Titanium and Zirconium Hydrides on the Detonation Heat of RDX-based Explosives – A Comparison to Aluminium

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Abstract: Detonation heats of RDX-based non-ideal explosives containing 15, 30 and 45 % of Al, (Al/ZrH₂), TiH₂ and ZrH₂ were measured with a water calorimeter set. To get more information on the behaviour of the additives in the detonations performed in an argon atmosphere, the solid post-detonation products extracted from the calorimetric bomb were analysed to determine their elemental and phase compositions. The calorimetric heats were compared with the detonation energies obtained from thermochemical calculations. All the tested explosives produce more en-

ergy during detonation in the bomb than RDX itself, but only aluminium positively influences the total energy release. The results of thermal and XRD analyses indicate that post-detonation products contain mainly carbonaceous materials, metal oxides, unreacted hydrides and/or the metals. Unexpectedly TiH₂ is the least reactive additive. At TiH₂ content of 45 %, the heat effect is close to that of calculated with an assumption of complete inertness of the additive. Moreover TiH₂ and metallic titanium are present in the bomb residues in a significant amount.

Keywords: non-ideal explosives • metal hydrides • detonation heat • solid residues

1 Introduction

C. E. Needham in his excellent book titled *Blast Waves* defines a non-ideal explosive as “an explosive or detonable mixture of chemicals that releases some of its energy after the passage of the detonation front” [1]. Typically such explosives include metal powders, among which aluminum, magnesium, titanium, boron, zirconium or mixtures or alloys of these metals are widely used as admixtures [1,2]. They may react both with the detonation products like water or carbon dioxide (anaerobic reactions) and with ambient atmospheric oxygen when it mixes with the detonation products (aerobic reactions). How much of the metal admixture (and during which stage of explosion) burns, depends not only on the oxidizer availability but also on the metal chemical and physical properties (reactivity, melting and boiling points), particle size and morphology, and the presence of coating layer on the particle surface. Moreover the burning efficiency of the metal particulates in expanding detonation products is affected by charge size, charge casing, as well as volume and confinement of the test structure [1].


In the search for effective fuel supplements in non-ideal explosives, metal hydrides have also received significant attention [3–21]. They are promising additives to energetic materials, because of their chemical activity, high heat of combustion, and generation of low molecular weight decomposition/combustion products. Among all the metal hydrides, MgH₂ and AlH₃ have the highest hydrogen storage capacity (7.6 and 10.1 %, respectively), and mainly for that

reason they are best suited candidates for components of rocket propellants [3–6], high explosives [7–13], and even pyrotechnic compositions [14].

Energetic materials containing titanium and zirconium hydrides have not been tested so frequently as those with magnesium and aluminum hydrides [15–21]. Titanium hydride has found applications as a component of pyrotechnics [17–19]. In a mixture with potassium perchlorate, it was also utilized in low-energy flaying plate detonators, as the composition detonates when exposed to firing current of 3.5 amp. [17]. While heating up, TiH₂/KClO₄ compositions start to decompose above 500 °C [18], and under room conditions they can be safely stored for a long time [19]. So that TiH₂ is compatible with strong oxidizers and can be mixed with organic high explosives such as RDX or HMX.

Improved high explosive compositions with metal hydrides (including zirconium and titanium hydrides) were disclosed in 1981 by F.R. Walker and R.J. Wasley [15]. However the additives of the invention constituted only a minor portion of the composition and served not as additional high-

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energy fuel components but as a source of low molecular weight radicals or ions that could improve detonation characteristics, i.e. initiation sensitivity, detonation velocity, brisance, etc. [15]. Titanium hydride was also proposed as a minor amount additive to solid rocket propellants to increase their burning rate [16].

Decomposition and ignition characteristics of TiH_2 at high heating rates were investigated by G. Young et al. [20]. The ignition experiments in air at a heating rate of approx. $1.3 \times 10^5 \text{ K/s}$ for pressures ranging from 0.1 to 7 MPa revealed that the ignition temperature decreased linearly from approx. 1700 to 1475 K with the increasing pressure. Post combustion particles consisted of titanium, oxygen and nitrogen. The subject of the study and discussion of the obtained results suggest that the experiments were performed to check the possibility of TiH_2 applications in high explosives and propellants.

The first paper that directly introduces non-ideal explosive mixtures of RDX (passivated with wax) and titanium hydride was published in 2015 [21]. The tested compositions contained from 10 to 20% of TiH_2 powder with mean particle size of 4.6 and 45 μm . Pressed charges of 20 g in mass were detonated in an explosion vessel of approx. 25 m^3 in volume, filled with air. The results obtained showed that TiH_2 significantly increases parameters of blast waves generated in the test chamber and reduces the detonation velocity. The peak overpressure, duration, and specific impulse were higher by 6, 9, and 23%, respectively, as compared to RDX/wax. The highest improvement of the blast performance was recorded for explosives containing smaller TiH_2 particles [21]. Detonation velocity linearly decreased with increasing TiH_2 content, and the TiH_2 particle size had no influence on this parameter. Titanium dioxide was found in the explosion products of RDX/ TiH_2 = 50/50 explosive, but TiH_2 was not detected by XPS method. On this basis, the authors conclude that decomposition and oxidation of TiH_2 happens behind the detonation wave, and the energy released due to TiH_2 oxidation causes the observed better blast performances [21].

In the present work RDX-based explosive compositions containing 15, 30, and 45% of titanium and zirconium hydrides are tested. For comparison, explosives with aluminium and ZrH_2/Al = 50/50 mixture are also examined, as aluminium is most widely used in high blast explosives [22]. Measurements of detonation heat in pre-compressed argon atmosphere are performed for all the explosives. The composition of the solid products recovered from the calorimetric bomb is also determined.

2 Experimental

2.1 Materials

Explosive mixtures were made of RDX passivated with wax (CH_2)_n and the powders of aluminium (Al), titanium hydride

(TiH_2), and zirconium hydride (ZrH_2). Commercial grade passivated RDX (RDXph) was used to prepare the tested mixtures. This explosive contains 94% of pure RDX and 6% of wax. TiH_2 and ZrH_2 powders were purchased from Sigma-Aldrich. They have purity higher than 98% and particle sizes lower than 44 μm (325 mesh). Al powder contains 99.5% of active metal with particle sizes below 44 μm .

All the additives were used as obtained, but the shape and size distribution of particles were determined applying scanning electron microscopy (SEM) and infrared particle sizer (IPS). SEM images of TiH_2 , ZrH_2 , and Al powders are shown in Figure 1.

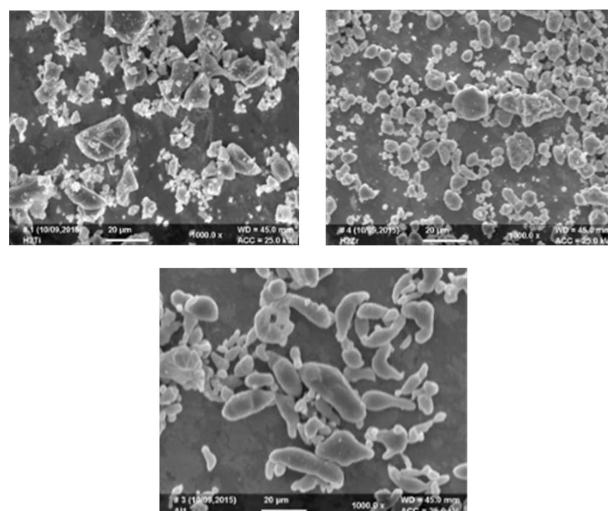


Figure 1. SEM images of TiH_2 , ZrH_2 (top, from the left side) and Al particles (bottom).

TiH_2 particles have the most irregular shapes and sharp edges whereas particles of ZrH_2 are more rounded. Al particles are elongated and have oval shapes. The particle sizes of all the additives are higher than 4 and lower than 40–45 μm , with the maximum of size distribution between approx. 13 (Al and ZrH_2) and 20 μm (TiH_2), so that the powders have similar size distributions and their particle sizes are in accordance with the declarations of suppliers.

The samples of RDX/Wax/Additive mixtures were prepared by granulation of RDXph/Additive mixture in the presence of hexane. Hexane was used to permanently bind the additive particles (TiH_2 , ZrH_2 , Al) with RDXph particles, as it partly dissolves wax layers. First the ingredients, taken in a designed ratio, were dry-mixed by hand in a container. After obtaining an optically homogeneous mixture, hexane was poured into the container, and the resulting, putty-like substance was kneaded/rubbed to obtain powdery consistency. The mixture was then left for 12 hours at ambient temperature to evaporate completely the solvent.

For the calorimeter experiments, the powdery samples were pressed (at 300 MPa) into 25-g cylinder pellets of

25 mm in diameter. The charges were weighted and measured to calculate their density. The measurements were performed twice, one and eighteen hours after production of the pellets. Results were identical. They are presented in Table 1.

Table 1. Composition and density of the charges tested

Explosive	Contents [%wt.]				Average density [g/cm ³]
	RDXph	Al	ZrH ₂	TiH ₂	
RDXph	100				1.66
+ 15 % Al	85	15			1.75
+ 30 % Al	70	30			1.86
+ 45 % Al	55	45			1.98
+ 15 % Al/ZrH ₂	85	7.5	7.5		1.80
+ 30 % Al/ZrH ₂	70	15.0	15.0		1.97
+ 45 % Al/ZrH ₂	55	22.5	22.5		2.17
+ 15 % ZrH ₂	85		15		1.85
+ 30 % ZrH ₂	70		30		2.09
+ 45 % ZrH ₂	55		45		2.37
+ 15 % TiH ₂	85			15	1.79
+ 30 % TiH ₂	70			30	1.95
+ 45 % TiH ₂	55			45	2.12

The charges are characterized by a good mechanical strength, and their density increases with increasing content of the metal additive – from 1.75 g/cm³ for the mixture containing 15 % of Al to 2.37 g/cm³ in the case of composition with 45 % of ZrH₂.

2.2 Calorimetric Measurements

A spherical steel bomb with an internal volume of 5.6 dm³ was the main element of a water calorimeter used for the measurements. The bomb was placed in a polished stainless steel calorimeter bucket that held 27000 ± 1 g of water. The bucket was surrounded by a constant-temperature jacket whose temperature was maintained at 20.0 ± 0.1 °C. The thermal equivalent of the instrument, determined by burning certified samples of benzoic acid with purified oxygen at a pressure of 2.0 MPa, was found to be 163.7 ± 1.2 kJ/K. This error of ± 0.73 % indicates the ultimate precision of the instrument.

The 25-g charges were ignited with military fuses (electric blasting caps). A mass of 1.2 g of PETN can be assumed as an energetic equivalent of the secondary output charge and firing composition of the fuse. Argon at a pressure of 2.0 MPa was used as an atmosphere in each experiment. At least three measurements were performed for each explosive. To estimate the energy released by the fuse, TNT charges of different mass were detonated in the bomb filled with argon. Assuming that the detonation heat of the fuse does not depend on the mass of the TNT charge, the dependence of the total heat effect on the TNT mass should be a linear one. This enabled us to determine the heat re-

leased by the fuse. It was found to be 11.7 ± 0.45 kJ. To calculate the detonation heat of an explosive, the difference between the measured total heat effect and the heat released by the fuse was divided by the mass of the tested charge. After each test, residues were collected for the post detonation analysis.

2.3 TG/DTA Analysis

TG/DTA measurements were performed using a Labsys TG/DTA/DSC apparatus (SETARAM). A powdery samples of the residues recovered from the calorimetric bomb were heated up from 20 to 1000 °C, at a 10 °C/min heating rate, with an oxygen dynamic atmosphere (flow rate of 50 cm³/min.). The sample was placed in an open alumina crucible.

2.4 Powder X-ray Diffraction

X-ray diffraction (XRD) spectra of the explosion products were collected (D500 Diffractometer, Siemens, CuK_α radiation) in the range of 2θ from 10° to 80°. For the analysis of the spectra obtained, ICDD database PDF4 + 2015 of standard diffraction patterns was used.

3 Results and Discussion

If any detonation heat measurement result out of the three first ones differed from the arithmetic mean by more than 4 %, two consecutive measurements were performed. The lowest and the highest values were omitted, and the remaining three were averaged and rounded to the nearest tens. Results of the detonation heat measurements are collected in Table 2, and the dependences of detonation heat on additive contents are shown in Figure 2. The straight dashed-dotted line was plotted assuming that only RDXph decomposes and releases energy, and the additives are completely inert substances. It means that the value of detonation heat at an additive content of 45 %, was calculated as 55 % of the detonation heat of RDXph.

Table 2. Results of detonation heat measurements

Explosive	Average detonation heat [J/g]	Explosive	Average detonation heat [J/g]
RDXph	4990 ± 70		
+ 15 % Al	6100 ± 30	+ 15 % ZrH ₂	4980 ± 80
+ 30 % Al	7190 ± 70	+ 30 % ZrH ₂	4660 ± 90
+ 45 % Al	6900 ± 180	+ 45 % ZrH ₂	4560 ± 120
+ 15 % Al/ZrH ₂	5550 ± 20	+ 15 % TiH ₂	4670 ± 180
+ 30 % Al/ZrH ₂	5800 ± 70	+ 30 % TiH ₂	4080 ± 150
+ 45 % Al/ZrH ₂	6280 ± 50	+ 45 % TiH ₂	3100 ± 50

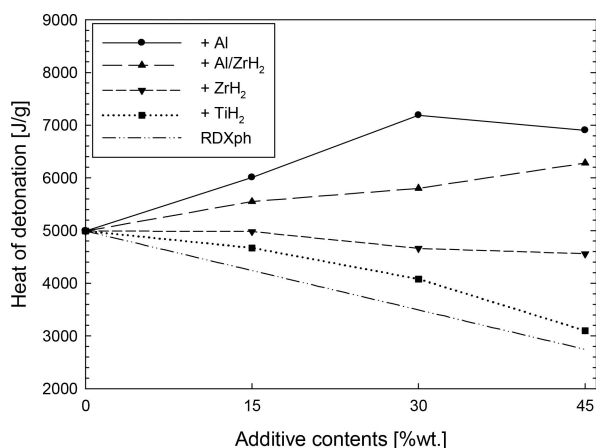


Figure 2. Dependence of detonation heat on additive contents.

All the tested explosives produce more energy during detonation in the calorimetric bomb than RDXph itself, but only aluminium positively influences the total energy release. The maximum heat of detonation of RDXph/Al composition corresponds to the Al content of 30%. If a half of Al additive is replaced with ZrH₂, the energy effect is proportionally lower, but still it linearly increases with the increasing Al/ZrH₂ content, up to 45%.

In the case of RDXph/ZrH₂ mixtures, increasing ZrH₂ content causes a slow reduction in the total heat effect, but a part of the additive exothermically reacts with RDXph detonation products, and the released heat nearly compensates for decreasing amount of RDXph.

The least reactive additive is titanium hydride. At TiH₂ content of 45%, the heat effect is close to that of calculated with an assumption of complete inertness of the additive.

The calorimetric detonation heats were compared with the theoretical detonation energy obtained from thermochemical calculations. The detonation energy is a sum of mechanical and thermal energies. The mechanical energy is equal to the expansion work of detonation products and it is determined for the volume of the products corresponding to the pressure of 1 atm. The thermal energy represents the amount of energy locked up as heat of detonation products at 1 atm. [23]. For calculations, the composition of detonation products is usually frozen at the temperature of 1800 K on the isentrope beginning at the Champan-Jouguet point [24]. Thermochemical calculations in this work were performed by the use of CHEETAH code [23]. The BKWS set of parameters [25] for the Baker-Kistiakowsky-Wilson equation of state and covolume factors for gaseous species were applied. The calculations were performed assuming that additives were chemically active, and also with an assumption of Al and TiH₂ chemical inertness. The results of calculations are presented in Figure 3.

For all tested explosives, the detonation energy, calculated with an assumption that the additives fully participate in the chemical reactions, is higher than the measured deto-

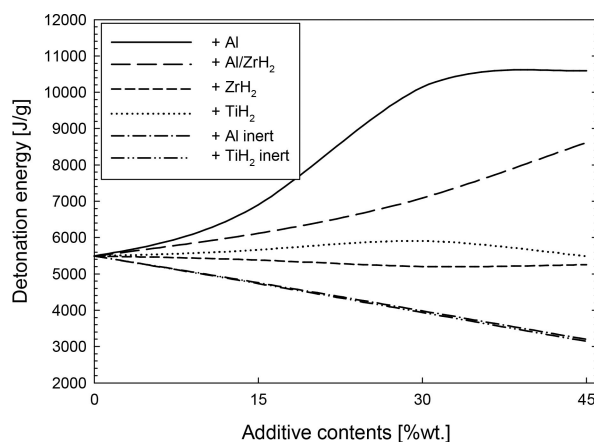


Figure 3. Dependence of theoretical detonation energy on additive contents.

nation heat. This means that Al, ZrH₂ and TiH₂ only partly burns during explosions in the calorimetric bomb filled with argon. However, the qualitative consistency of dependences of the calculated and experimental parameters on the additive contents can be seen except for RDXph/TiH₂ composition. In the whole percentage range, the theoretical detonation energy of the explosive containing TiH₂ is higher than that of RDXph/ZrH₂, while measurements give the inverse relationship (Figure 2). This suggests a particularly low chemical activity of titanium hydride under the experimental conditions. Obviously the detonation energy, calculated assuming chemical inertness of the additives, is the lowest and linearly decreases with increasing additive contents (Figure 3).

The solid residues of detonation of 25-g charges of the RDXph/TiH₂ and RDXph/ZrH₂ explosives in the calorimetric bomb filled with argon were analyzed to determine their composition. In each case, the residue extracted from the bomb was first sieved (to remove fragments of detonator and its electric leads), dried for 24 h at 100 °C and then weighted. The products were black and dusty like soot.

The thermograms of detonation products of explosives containing TiH₂ and ZrH₂ are presented in Figures 4 and 5.

From an analysis of the thermograms in Figure 4, it follows that first amorphous carbon (soot) oxidizes. The process begins at approx. 280 °C, and for RDXph/TiH₂ explosive is accompanied by a mass reduction of 3.5 to 15.4% (out of the initial sample mass). At approx. 500 °C titanium or TiH₂ begins to oxidize, what is evidenced by the slow increase in mass and the presence of a broad, exothermic peak on the DTA curves. The sample mass increases (by 17.1–37.7%) because oxygen is fixed by titanium giving TiO₂.

Also in the case of RDXph/ZrH₂ (Figure 5), soot oxidizes in a temperature range of 280–420 °C. The sample mass decreases, and the mass reduction is inversely proportional to the content of ZrH₂ in the explosive. An endothermic peak, corresponding to aluminum melting (at 655 °C), can be seen

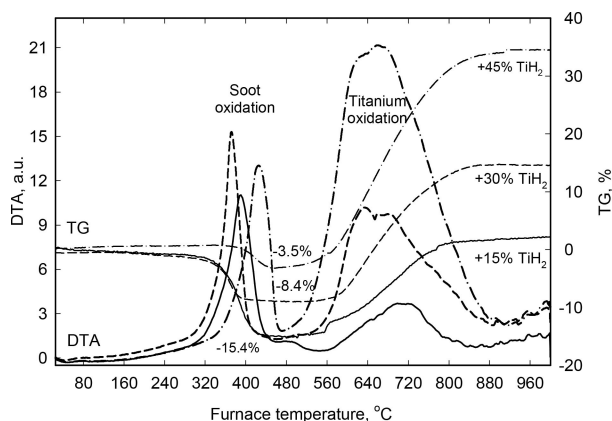


Figure 4. TG/DTA curves of residues extracted from the bomb after detonation of RDXph/TiH₂ charges.

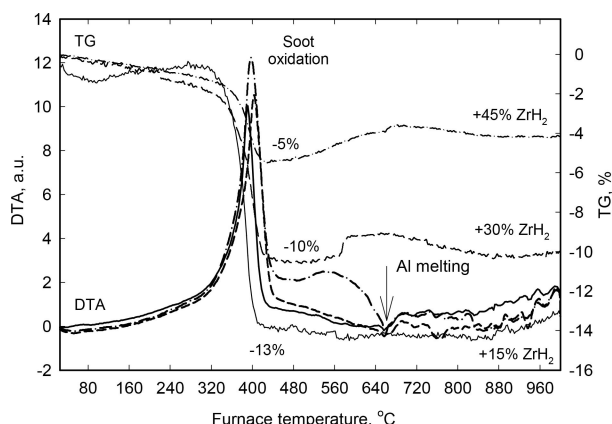


Figure 5. TG/DTA curves of residues extracted from the bomb after detonation of RDXph/ZrH₂ charges.

on the DTA curves. Small amount of aluminum is present in the tested sample, because the fuse cup was made of this metal.

The substance remaining in the crucible after soot gasification is stable up to 1000 °C in oxygen atmosphere. It might be zirconium oxide or unreacted ZrH₂ or metallic zirconium covered with zirconium oxide layer. This uncertainty induced us to perform the phase analysis of chosen detonation products. The recorded spectra for samples of detonation products of RDXph + 30%ZrH₂ and RDXph + 30%TiH₂ charges in argon atmosphere are presented in Figures 6 and 7.

Results of the phase analyses unambiguously confirm that both of the tested samples contain the metal oxides: ZrO₂ (00-036-0420, baddeleyite) and TiO₂ (00-021-1276, rutile), respectively. Moreover complex aluminum-zirconium oxide (Al_{0.52}Zr_{0.48}O_{1.74}) and iron-titanium oxide (Fe₃Ti₃O₁₀) systems are also present. Pure aluminum phase was also detected in both the residues. Iron oxide is formed as a product of oxidation of the calorimetric bomb internal elements.

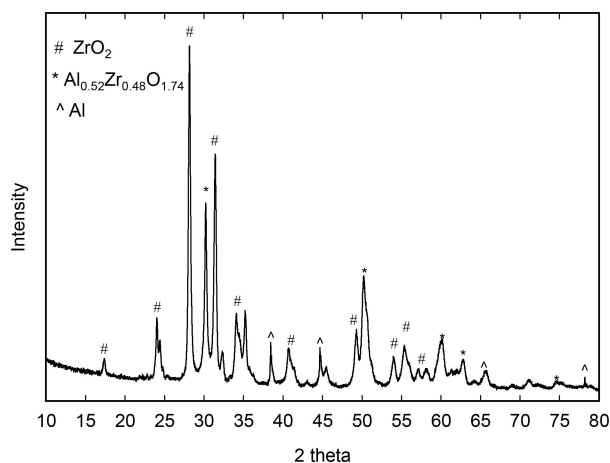


Figure 6. XRD pattern of the residues extracted from the bomb after detonation of RDXph + 30%ZrH₂ composition.

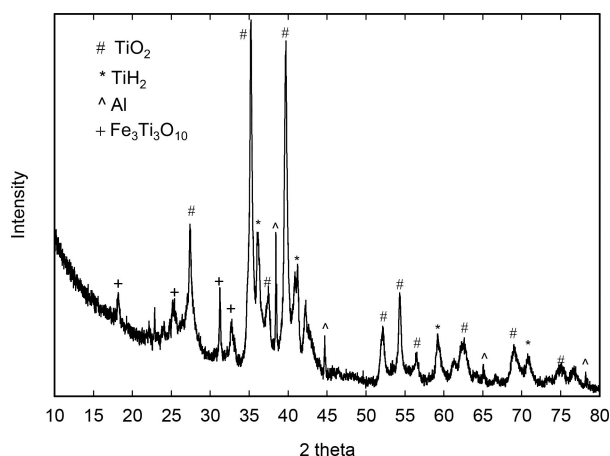


Figure 7. XRD pattern of the residues extracted from the bomb after detonation of RDXph + 30%TiH₂ composition.

Summing up, the thermal analysis results indicate that detonation of RDXph/TiH₂ and RDXph/ZrH₂ charges in argon atmosphere gives solid products containing mainly carbonaceous material, metal oxides, unreacted hydrides and/or the metals (Zr/Ti/Al). This means that a part of zirconium and titanium is oxidized by gaseous detonation products of RDX. Results of XRD analysis show that unreacted titanium hydride is present in the RDXph/TiH₂ solid detonation products.

4 Conclusions

All the tested explosives produce more energy during detonation in the calorimetric bomb than RDXph itself, but only aluminium positively influences the total energy release. If a half of Al additive is replaced with ZrH₂, the energy effect is proportionally lower, but still it linearly increases with in-

creasing Al/ZrH₂ content up to 45%. In the case of RDXph/ZrH₂ mixtures, increasing ZrH₂ content causes a slow reduction of total heat effect, but a part of the additive exothermically reacts with RDXph detonation products, and the released heat nearly compensates for decreasing amount of RDXph. The least reactive additive is titanium hydride. At TiH₂ content of 45%, the heat effect is close to that of calculated with an assumption of complete inertness of the additive. The results of thermal and XRD analyses indicate that detonation of RDXph/TiH₂ and RDXph/ZrH₂ charges in the calorimetric bomb filled with argon produces solid carbonaceous products and metal oxides. In the RDXph/TiH₂ detonation products, unreacted titanium hydride is also present.

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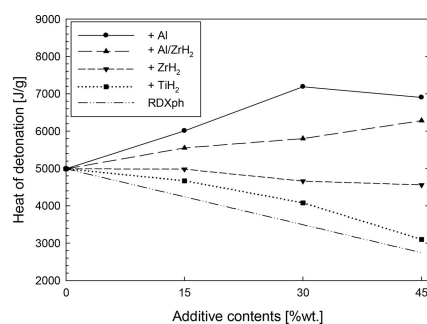
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