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Explosion strength of tri-n-butyl phosphate and fuming nitric acid (TBP/FNA) mixture evaluated by underwater explosion test

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Received: November 7, 2003 Accepted: January 20, 2004

Abstract

A mixture of tri-n-butyl phosphate (TBP) and nitric acid is used in the PUREX method for reprocessing nuclear fuel, and is performed safely. However, when fuming nitric acid (FNA) is employed, it behaves like a liquid explosive. In the present study, the explosion strength of TBP/FNA mixture was evaluated by underwater explosion tests. Shock wave energy, bubble energy, and total energy of detonation were determined. The explosion strength reaches a maximum and decreases as the mixture ratio deviates from stoichiometric. It was demonstrated that heat of detonation of TBP/FNA that determined from thermochemical calculation was consistent with total energy that determined from underwater explosion test.

1. Introduction

Plutonium and Uranium Recovery by Extraction (PUREX) is a technology for reprocessing spent nuclear fuel that is commonly used in commercial reprocessing plants. In the PUREX process, nitric acid is used to dissolve the spent fuel from cladding tubes, and tri-n-butyl phosphate (TBP) is used to extract uranium and plutonium from other fission products. When heated for a prolonged period, the mixture of TBP/nitric acid can form 'red oil', a highly dense, energetic, and organic-based material. Red oil is thought to have been the origin of the past accidental explosions in reprocessing plants, such at those having occurred at Tomsk-7 (Russia, 1993), and Savannah River (United States, 1953 and 1975). Scientists have investigated the hazards associated with red oil, and have performed some safety studies to examine the chemical behavior of the TBP/nitric acid [1-2]. However, the detonability of TBP/nitric acid mixture in the explosion worst-case scenario has not been sufficiently investigated.

As has been reported, when TBP is mixed with fuming nitric acid (FNA), the mixture can be detonated under sufficient ignition stimulus [3]. The mixture of TBP/FNA is detonable, even if red oil is not generated. Investigating the detonation performance of the TBP/FNA mixture would be of great value.

Some of the published data show the following conclusions. In a polyvinyl chloride (PVC) pipe with 20 mm in internal diameter of cylindrical charge, experiments have shown that the mixture detonates when the TBP/FNA mixture ratio falls within the range of 13/87 - 36/64 wt.%. The detonation velocity of the stoichiometric TBP/FNA (22/78 wt.%) mixture is 6.45 $km \cdot s^{-1}$ [3]. The critical thickness of detonation propagation of the mixture is very small, determined to be 0.5-0.6 mm, and independent of the mixture ratio [4]. Hence, previous studies, indicate that TBP/FNA exhibits behavior similar to that of liquid explosives.

In the present study we evaluate explosion strength of TBP/FNA mixture via underwater explosion test. The primary reason for using the underwater explosion test is to avoid the hazards associated with the acid during the experiment. In addition, this method is advantageous in that the dynamic effect (shock wave energy) and the static effect (bubble energy) of the energetic compositions can be determined simultaneously. The underwater explosion test is employed ordinarily with several hundred grams to several kilograms of sample. In this study, we employed underwater explosion test with smaller scale of several ten grams of the sample.

In our previous study [5], we attempted to evaluate the explosion strength of TBP/FNA via an underwater explo-

sion test. However, clear conclusions were not obtained, because of insufficient data.

The purpose of the present study is to determine the explosion strength of TBP/FNA mixtures, by determining shock energy, bubble energy, and total energy of detonation, and to evaluate their relation to the mixture ratio. Finally, the total energy determined in the experiments is compared with that determined by thermochemical calculation and with those of other energetic materials.

2. Experimental method

2.1. Materials

TBP ($(CH_3(CH_2)_3O)_3PO$, extra pure reagent), and FNA (nitric acid conc. 94 wt.%, guaranteed reagent) were used as samples. The materials were obtained from Nacalai Tesque Inc., Japan and were used as received. TBP/FNA with detonable composition (13/87 - 36/64 wt.%) was evaluated.

2.2. Measurement system

Figure 1 shows the experimental set-up used for the underwater explosion tests. The experiments were conducted in a cylindrical water tank of 5 m in depth and 8 m in diameter. The sample was set at a depth of 2.5 m. A stone weight was suspended to the sample assembly in order to suspend it in a vertical position.

Tourmaline gauges (PCB, 138A10) were used as pressure gauges. Two gauges were set at distances of 0.9 m or 1 m from the sample, at the same depth as the sample. The gauge was set in alignment with the horizontal or vertical direction of the sample (hereinafter called "horizontal gauge" and "vertical gauge," respectively). The gauge cable and the junction between the gauge and the cable were inserted into a steel pipe in order to protect the junction from shock and immersion in water. Therefore, only the tourmaline sensing element is exposed to the blast. A signal conditioner (PCB, 482A22, 4 mA) was provided between the gauges and the digitizer. The data were recorded by a digitizer (Gaugescope, CS1610) at a sampling rate of 10 $MS \cdot s^{-1}$.

The sample was ignited from the bottom by use of a booster and an Exploding Bridge Wire (EBW, Nippon Kayaku Co. Ltd.) detonator. A trigger was picked up by a current transformer (Pearson Electronic Co. Ltd, 411) from a high voltage firing unit (RISI, FS-62, 4 kV).

In order to avoid direct contact with FNA, the surface of the booster was covered with 80 μm-thick resin adhesive tape (Nitto Denko Co. Ltd., 903UL). The sample was inserted into the assembly by use of an injector, in order to avoid an inclusion of air bubble in the assembly.

The experiments can be classified into 2 methods, depending on the relative mass of the booster.

2.3. Method 1: using a booster of high relative mass

Previous studies have clarified that TBP/FNA can be completely detonated by a booster having a mass greater than 10 g [3-4]. The present method employs a 10 g booster, having a relative mass of 117%-129% with respect to the sample.

Figure 2a shows the assembly used in method 1. The volume of the sample is constant at 6.3 cm^3. Pentolite (Chugoku Kayaku Co. Ltd., ρ_0 = 1600 $kg \cdot m^{-3}$) is used as the booster.

2.4. Method 2: using a booster of low relative mass

In order to obtain more precise energy of the material,

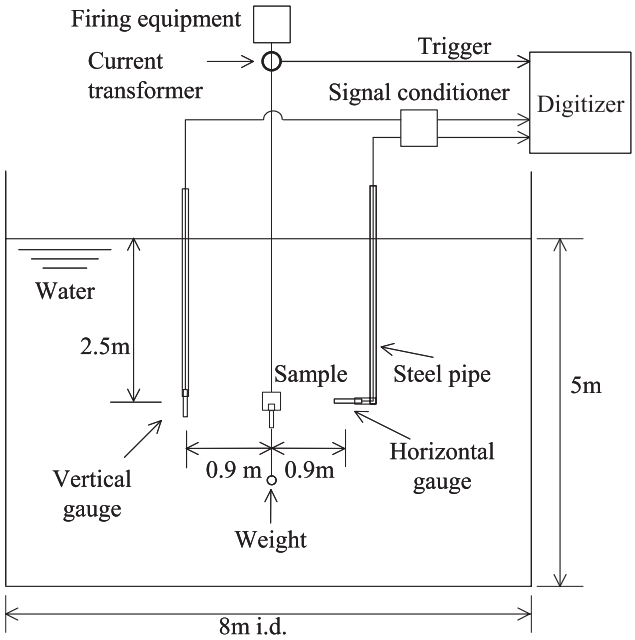

Fig. 1 Experimental set-up of the underwater explosion test.

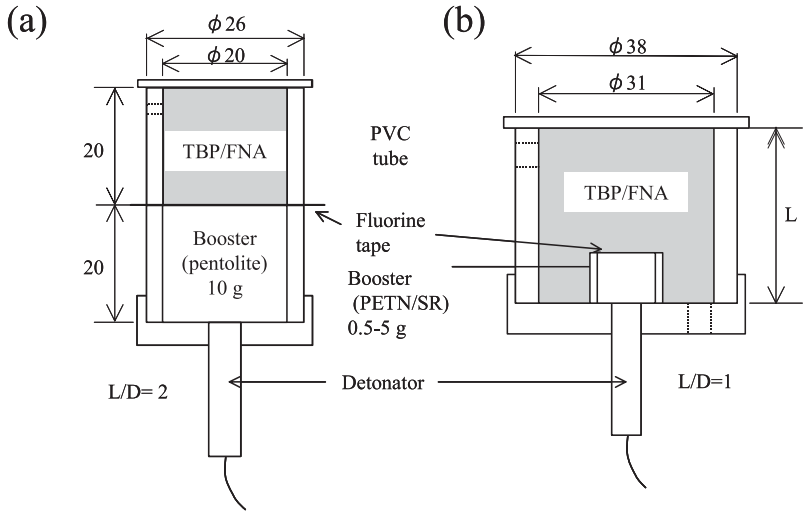

Fig. 2 Experimental assembly of (a) method 1 and (b) method 2.

use of a booster with low relative mass to the sample is preferable. However, some materials will not be detonated completely when a booster of low relative mass is used. In method 2, we attempted to perform experiments using boosters of 0.5 g, 1 g, 2 g, and 5 g, whose relative masses were 2%-20% with respect to the sample.

Figure 2b shows the assembly employed in method 2. The volume of the sample was constant at 21.4 cm^3. PETN/silicon rubber (70/30 wt.%, ρ_0 =1300 $kg \cdot m^{-3}$) was used as the booster.

3. Energy formula

The shock wave energy (E_s) was determined from the energy flux density at the gauge and under the assumption of spherical expansion [1] [6-9]:

$$Es = \frac{4\pi R^2}{\rho_w C_w W} \int_{\theta}^{6.7\theta} P^2(t) \cdot dt \qquad (MJ \cdot kg^{-1}) \qquad [1]$$

where R is the distance between the sample and the gauge (m), ρ_w is the density of water ($kg \cdot m^{-3}$), C_w is sound velocity in water ($km \cdot s^{-1}$), W is the mass of the sample (kg), and θ is the time from the start of shock to the time at which $P_{max} \cdot e^{-1}$ occurs. P_{max} is the maximum pressure value of the shock wave profile.

The bubble energy (E_b) was determined from the work performed in expanding the bubble to its first maximum with respect to the hydrostatic pressure and under the assumption of no boundary effects [2] [6-9]:

$$E_b = \frac{68.4 P_o^{5/2} T_b^3}{W} \qquad (MJ \cdot kg^{-1}) \qquad [2]$$

where P_0 is total hydrostatic pressure at sample depth (0.13 MPa), and T_b is bubble period (sec).

The total energy of detonation (E_{tot}) was determined from the sum of E_s and E_b, after consideration of the shock loss factor (μ) and charge shape factor (K_f) [7, 9].

$$E_{tot} = K_f (E_b + \mu E_s) \qquad (MJ \cdot kg^{-1}) \qquad [3]$$

K_f depends on L/D (Length/Diameter) of the assembly. K_f =1.00 for L/D=1, and K_f =1.02 for L/D=2.

4. Thermochemical Calculation

It was reported that E_{tot} could be assumed from thermochemical calculation of heat of detonation $-\Delta H_d$ [7]. In this study, Cheetah 2.0 thermochemical code [10] was used to determine $-\Delta H_d$. First a C-J condition calculation is performed, followed by an adiabatic expansion, and finally a calculation at 298 K, 1 atm is performed in order to determine $-\Delta H_d$. $-\Delta H_d$ is the energy that a calorimeter experiment would measure.

For calculation of TBP/FNA, BKWS product library, which contains phosphor substances, was adopted. For calculation of C, N, O, and H explosives, BKWC library was adopted.

5. Results

Figure 3 shows a typical shock profile obtained in the present experiment. The shockwave pulse and the 1st bubble pulse were observed. The inset graph is an enlargement of the shockwave pulse.

Figure 4 shows the relationship between the peak pressure of pentolite and scaled distance. This figure compares the experimental results from the vertical gauge, those from the horizontal gauge, and the reference data [6]. For both gauges, the measured peak pressure is consistent with the reference data, thereby confirming the validity of the peak pressure data.

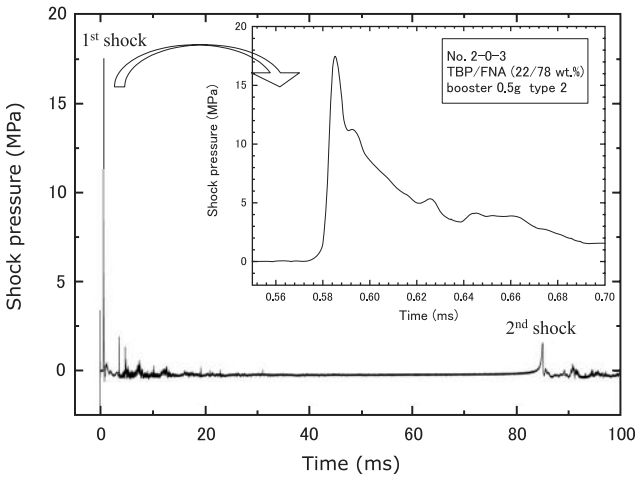

Fig. 3 Shock pressure profile of TBP/FNA with 0.5 g booster (method 2).

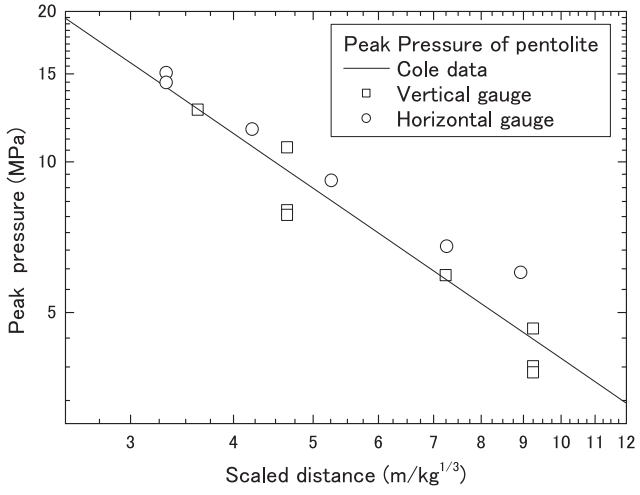

Fig. 4 Relationship between peak pressure of pentolit and scaled distance.

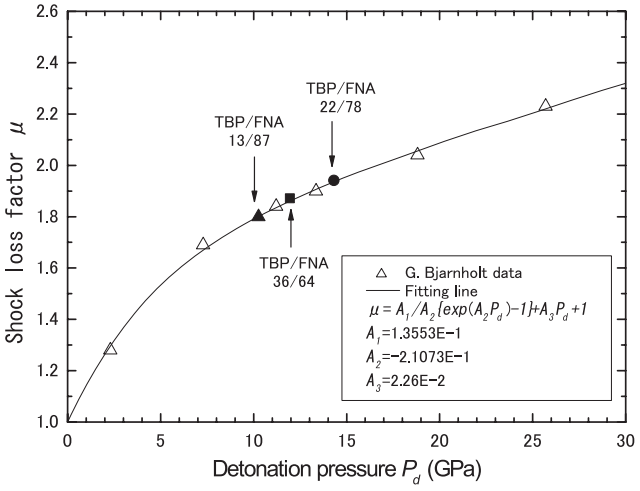

Fig. 5 Relationship between shock loss factor (μ) and detonation pressure (*Pd*).

Table 1 shows experimental results of method 1. The displayed values of E_S, E_b, and E_{tot} of TBP/FNA represent energy obtained after subtraction from E_S, E_b, and E_{tot} of detonator and booster that measured before.

Table 2 shows the experimental results of method 2. The detonation of TBP/FNA was confirmed at stoichiometry, and neither at the limit of detonation range, nor nitromethane. The reason is thought that the energy of booster was not enough for detonation of TBP/FNA at the limit of detonation range. For nitromethane, the previous study yielded the same finding; it could not be detonated with a booster less than 5 g which equivalent to energy less than 1.13 kJ [4]. Larger scale should be done for evaluation of the explosion strength of TBP/FNA at various mixtures by method 2.

6. Discussion

6.1. Fitting formula of shock energy loss factor (*μ*)

As is well known, μ can be determined as a function of detonation pressure (P_d) [7]. However, the function formula has not been determined. In order to obtain μ with accuracy, we attempted to fit formula [4] to the reference data.

$$\mu = \frac{A_1}{A_2}\{\exp(A_2 \cdot P_d) - 1\} + A_3 P_d + 1 \qquad [4]$$

where $A_{1\text{-}3}$ are interpolation parameters. Figure 5 shows the fitting result and μ of TBP/FNA. The result shows good fitting with R^2=0.99895. The values of parameters are A_1= 1.355E-1, A_2 =-2.107E-1, and A_3=2.260E-2. Since μ at the reference was determined at the scaled distance about 3 m·kg$^{-1/3}$, the experiments in the present study were conducted at a similar scaled distance.

P_d was estimated from calculation result yielded by Cheetah code. P_d can be estimated also from detonation velocity measured by experiment [5]. The difference of value of μ between that calculated from Cheetah, and that determined from detonation velocity was very small of ± 4 %.

6.2. Differences between vertical and horizontal gauges

Almost all the data of method 1 were measured by the vertical gauge. However, some noises were frequently found. In order to eliminate the influence of noise on the value of E_S, the noise was erased artificially. Subsequently, while referring to some previous underwater explosions studies [6-9], we applied the horizontal gauge. Because the data were obtained without any noise, all the data of method 2 was measured by the horizontal gauge. Rise times of the shock pulse, defined as the time from 10% to 90% of the peak, were 3 μs and 4 μs by horizontal gauge and vertical gauge, respectively. They were reasonable due to response time of the experimental apparatus of about 3 μs.

Table 1 Experimental result employing method 1 and calculation result.

Shot no.	Sample	Initiation method *)	Gauge direction	Sample mass (g)	P_{max} (MPa)	T_b (ms)	μ	E_s **) (MJ·kg^{-1})	E_b **) (MJ·kg^{-1})	E_{tot} **) (MJ·kg^{-1})	$-\Delta H_d$ Cheetah (MJ·kg^{-1})
1-0-1	Detonator (PETN)	–	vertical	0.8	3.51	25.25	2.35	1.27	2.45	5.54	5.93
1-0-2		–	vertical	0.8	2.72	25.29		1.13	2.47	5.22	
1-1-1	Pentolite	d	vertical	10	10.68	56.63	2.16	1.43	2.02	5.11	5.05
1-1-2		d	vertical	20	12.72	71.99	2.16	1.46	2.18	5.44	
1-1-3		d	horizontal	10	11.82	56.82	2.16	1.13	2.07	4.52	
1-2-1	TBP/FNA (13/87)	b + d	vertical	8.6	12.66	67.27	1.82	1.05	1.47	3.45	2.89
1-2-2		b + d	vertical	8.6	12.55	67.26		0.94	1.47	3.24	
1-3-1	TBP/FNA (17/83)	b + d	vertical	8.7	14.46	68.97	1.89	1.34	2.02	4.64	4.04
1-4-1	TBP/FNA (22/78)	b + d	vertical	8.3	11.91	70.31	1.94	1.40	2.17	4.99	5.32
1-4-2		b + d	vertical	8.3	13.14	71.41		1.70	2.42	5.84	
1-5-1	TBP/FNA (27/73)	b + d	vertical	8.4	14.45	69.40	1.90	1.42	2.19	4.99	4.76
1-6-1	TBP/FNA (31/69)	b + d	vertical	8.3	14.45	68.75	1.87	1.36	2.09	4.72	4.41
1-7-1	TBP/FNA (36/64)	b + d	vertical	7.8	12.45	68.81	1.82	1.15	1.97	4.14	4.36
1-7-2		b + d	vertical	7.8	12.42	69.12		1.31	2.03	4.51	
1-8-1	PETN powder	d	horizontal	28.11	17.30	90.84	2.35	1.29	3.09	6.19	5.96

*) d: detonator, and b: booster (pentolite: 10g)
**) The displayed energy values are values after subtraction of the energy of detonator and booster.

Table 2 Experimental result employing method 2.

Shot no.	Sample	Initiation method*)	Gauge direction	Sample mass (g)	Booster mass (g)	P_{max} (MPa)	T_b (ms)	E_s **) (MJ·kg^{-1})	E_b **) (MJ·kg^{-1})	E_{tot} **) (MJ·kg^{-1})
2-0-1	TBP/FNA (22/78)	b + d	horizontal	28.11	0.52	17.72	84.89	1.10	2.48	4.601
2-0-2		b + d	horizontal	28.11	0.52	19.05	84.84	1.16	2.47	4.722
2-0-3		b + d	horizontal	28.11	0.53	17.29	83.46	1.09	2.40	4.516
2-0-4		b + d	horizontal	28.11	0.53	18.21	83.48	1.10	2.40	4.537
2-1-1		b + d	horizontal	28.11	0.95	18.58	86.53	1.03	2.33	4.326
2-1-2		b + d	horizontal	28.11	0.95	19.41	86.52	1.07	2.33	4.401
2-1-3		b + d	horizontal	28.11	1.06	19.62	88.54	1.11	2.54	4.703
2-1-4		b + d	horizontal	28.11	1.06	19.52	88.55	1.25	2.54	4.961
2-2-1		b + d	horizontal	28.11	2.07	18.14	88.55	1.15	2.54	4.762
2-2-2		b + d	horizontal	28.11	2.07	18.32	88.55	1.20	2.54	4.864
2-2-3		b + d	horizontal	28.11	1.97	19.02	86.30	1.14	2.52	4.720
2-2-4		b + d	horizontal	28.11	1.97	19.21	86.30	1.16	2.52	4.768
2-3-1		b + d	horizontal	28.11	5.02	18.03	85.91	1.16	2.54	4.784
2-3-2		b + d	horizontal	28.11	5.02	19.12	85.91	1.17	2.54	4.807
2-3-3		b + d	horizontal	28.11	5.44	17.52	84.10	1.10	2.54	4.680
2-3-4		b + d	horizontal	28.11	5.44	18.04	84.11	1.13	2.54	4.731
2-4-1~4	TBP/FNA (13/87)	b + d	horizontal	29.28	0.5, 1, 2, 5	*no detonation* ***)				
2-5-1~4	TBP/FNA (36/64)	b + d	horizontal	26.46	0.5, 1, 2, 5	*no detonation* ***)				
2-5-1~4	Nitrome thane	b + d	horizontal	24.37	0.5, 1, 2, 5	*no detonation* ***)				

*) d: detonator, and b: booster (PETN/SR: 0.5, 1, 2, 5 g)
**) The displayed energy values are values after subtraction of the energy of detonator and booster.
***) Only detonations of the booster were observed; no detonation of TBP/FNA was observed.

However P_{max} measured by the horizontal gauge tends to be higher as shown in fig. 4, and θ tends to be shorter than that measured by the vertical gauge. As the result, E_S measured by the vertical gauge was higher than that measured by the horizontal gauge. Table 1 shows that for 10 g pentolite, E_S shows a 29% difference. But, for E_b, no difference is found.

The above-mentioned difference is thought to originate from the characteristics of the tourmaline sensing element. The disk shape of sensing element is considered having directionality. Further investigation should be conducted for clarification.

6.3. Explosion strength measured by method 1

Figure 6 shows the relation between E_S, E_b, and E_{tot} of TBP/FNA and mixture ratio measured by method 1. The explosion strength of TBP/FNA is found to depend on the mixture ratio. It reaches a maximum at stoichiometric composition, and decreases as the mixture ratio deviates from stoichiometric. At stoichiometric composition, the average explosion strength of TBP/FNA was E_S= 1.15 MJ·kg^{-1}, E_b= 2.30 MJ·kg^{-1}, and E_{tot}= 5.42 MJ·kg^{-1}.

The ratio of E_S and E_b to E_{tot} were around 28% and 44%, respectively. The remain ratio corresponds to loss of shock energy. Similar ratio was also founded in underwater explosion test of high explosives [7-9].

6.4. Explosion strength measured by method 2

Figure 7 shows E_S and E_b as functions of booster mass (g), respectively. A straight line was fitted to the energy (kJ). The y ordinate intercept, when the booster mass is 0 g, is thought to be the energy of net TBP/FNA. The energies for a booster mass of 0 g were divided by the net mass of the sample, thereby yielding energy per mass (MJ·kg^{-1}). E_S and E_b of net TBP/FNA were found to be 1.15 MJ·kg^{-1} and 2.58 MJ·kg^{-1}. Finally, using μ=1.94, it was obtained that E_{tot} = 4.81 MJ·kg^{-1}.

The results of E_S, E_b, E_{tot} with calculation in figure 7 were similar with the calculation in table 2, which was determined from simply subtraction of energy of detonator and booster. But, the results of E_S are on average 25% lower that the results from method 1, because of the employment of different direction gauge. The result of E_b is generally similar to that of method 1. The same result was identified also in case of pentolite that described before.

Same procedure also can be done to the pressure as a function of the cube root of booster mass (gr$^{1/3}$), which is a known empirical relation [6]. As a result, the pressure was calculated to be 17.35 MPa. This is one benefit of method 2; the peak pressure of the net TBP/FNA could be obtained.

Comparing to PETN powder (ρ_0 = 750 kg·m^3) which was determined by horizontal gauge, as shown in table 1, relative explosion strength of TBP/FNA at stoichiometry was found as E_S= 88%, E_b= 82%, and E_{tot}= 77% of PETN.

6.5. Comparison with other liquid explosives by thermochemical calculation

The Cheetah calculation result -ΔH_d of TBP/FNA is shown in table 1, and as a solid line in Fig.6. The result shows that -ΔH_d calculated by Cheetah was consistent with E_{tot} that determined from experiment. The error between -ΔH_d and E_{tot} was ± 6%. It means that E_{tot} of TBP/FNA can be estimated from -ΔH_d of the Cheetah calculation. From estimation of E_{tot}, then E_S, and E_b of TBP/FNA can be estimated from each ratio.

In comparison of the explosion strength of TBP/FNA to that of liquid explosives, Cheetah calculation -ΔH_d of liquid explosives was used. It can be estimated that the explosion strength of TBP/FNA at stoichiometry is higher than that of nitromethane of -ΔH_d = 4.86 MJ·kg^{-1}, and hydrazinium nitrate of -ΔH_d = 3.96 MJ·kg^{-1}. But it was estimated to be smaller than that of nitroglycerine of -ΔH_d = 6.30 MJ·kg^{-1} and methyl nitrate of -ΔH_d = 6.11 MJ·kg^{-1}.

7. Conclusions

The explosion strength of TBP/FNA has been successfully evaluated via underwater explosion. Shock energy (E_S), bubble energy (E_b), and total energy (E_{tot}) have been measured. Two methods were applied in the present study.

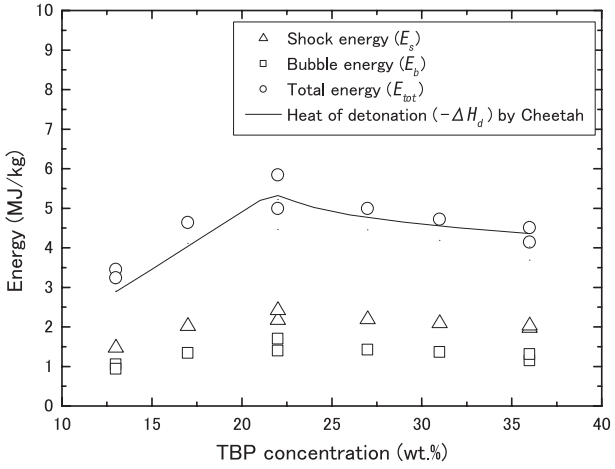

Fig. 6 Relationship between *Es, Eb, Etot,* -Δ*Hd*, and TBP concentration (method 1).

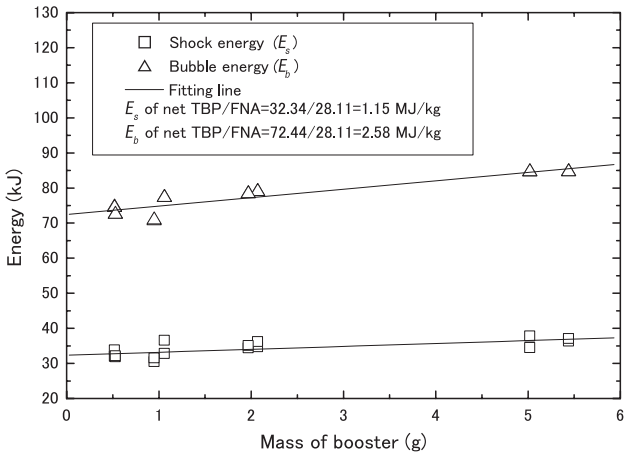

Fig. 7 *Es* and *Eb* of TBP/FNA (22/78 wt.%) with various small relative mass boosters (method 2).

Method 1 was an experiment using a booster of high relative mass, and the relation of the explosion strength of TBP/FNA with mixture ratio was determined. The explosion strength of TBP/FNA was found to be dependent on the mixture ratio. The explosion strength exhibits a maximum at stoichiometry and decreases as the mixture ratio deviates from the stoichiometry.

Method 2 was an experiment using a booster of low relative mass. By using various masses of booster, this method can determine peak pressure and the explosion strength of net TBP/FNA. However, this method can be used only for a composition that can be detonated by a booster of small relative mass.

The ratio of E_S and E_b to E_{tot} of TBP/FNA were similar to the ratio in the high explosives.

The heat of detonation $-\Delta H_d$ of TBP/FNA, which calculated by Cheetah thermochemical code shows consistency with E_{tot} of underwater explosion experimental result. Thus, E_{tot} of TBP/FNA can be estimated from $-\Delta H_d$ of Cheetah calculation. From the estimation of E_{tot}, then, E_S, and E_b of TBP/FNA can be estimated using relation of the ratio of E_S and E_b to E_{tot}.

The explosion strength of TBP/FNA at stoichiometry was found smaller than that of PETN. In the liquid explosives, the explosion strength of TBP/FNA was found higher than that of nitromethane and hydrazinium nitrate, but smaller than that of nitroglycerine and methyl nitrate.

Acknowledgments

This study was financially supported by the Budget for Nuclear Research of the Japan Ministry of Education, Culture, Sports, Science and Technology, based on screening and counseling by the Atomic Energy Commission.

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