Article

DDT test for smokeless powders

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A 50/60 steel tube deflagration-to-detonation transition (DDT) test was conducted for 58 types of smokeless powder manufactured by the NOF Corporation in order to determine appropriate criteria for predicting detonation. Of the products tested, 26 were deemed to detonate under the test conditions, and the mass distribution of fragments recovered from the tests of these propellants exhibited two distinct regions with slope 0.6 (small fragments) and 1.6 (large fragments). From an analysis of the detonation criterion used in Japan, by which detonation is defined to occur when less than half of the original mass of the tubing is recoverable in fragments larger than 200 g, the quickness and surface area-to-volume ratio were found to be reliable indicators of the possibility of detonation.

1. Introduction

On August 1, 2000, an explosion occurred at the Taketoyo plant of the NOF Corporation in Aichi prefecture, Japan. The explosion was attributed to 7.1 t of smokeless powder and 0.6 t of gas generants (mainly composed of perchlorate) that had been stored in a temporary explosive storage facility. The accident resulted in injuries of 79 people and damage of 888 houses in the area [1]. Based on the report, which detailed the creation of a large crater on the concrete floor of storage facility, smokeless powders are considered to have detonated rather than undergone combustion and deflagration.

Numerous DDT studies concerning smokeless powder have been reported. Cruysberg et al. [2] determined that an explosion accident in 1972 during the manufacture of a porous propellant in the Netherlands involved the detonation of 350 kg of porous single-base propellants. This report was based on field trials involving the same quantity of propellants, and peak pressure measurements and blast effects were found to correspond to a TNT mass equivalence of between 20 and 60%.

Bernecker [3,4] reported a detailed DDT process for porous propellant systems, and discovered that the DDT behavior is closely related to the theoretical maximum density and surface-to-volume ratio.

Recently, Yang et al. [5] reported a DDT test of three types of smokeless powder with data obtained using a strain gauge and ionization probe. A rapid pressure increase near the igniter was found to be a necessary condition for the formation of the compressive wave, which may explain the differences in DDT characteristics.

The deflagration-to-detonation transition (DDT) behavior is the most important property of explosives. Although, there is a large body of research on the DDT of smokeless powders, there has been no research involving the quantitative recovery of fragments after tests as in the present

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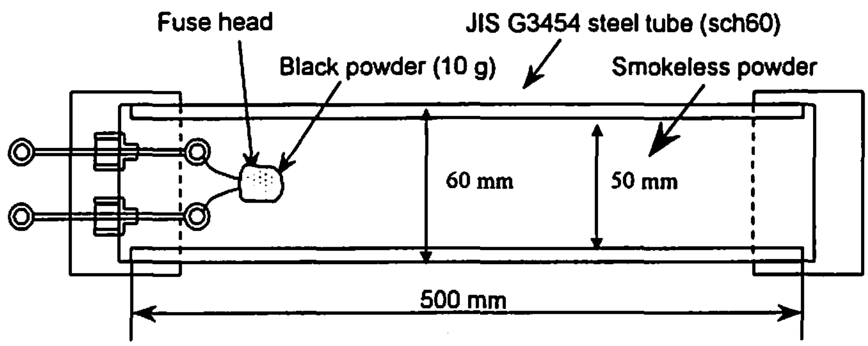

Fig. 1 50/60 DDT test showing ignition method

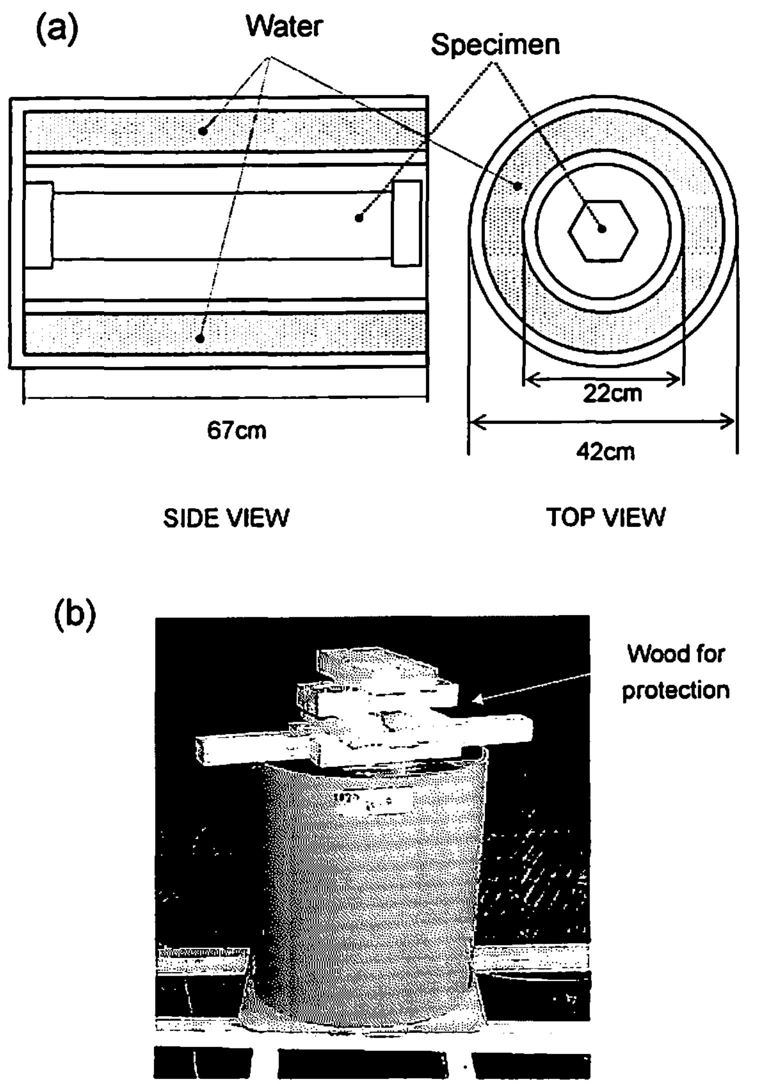

Fig. 2 Water protection system. (a) Schematic, and (b) photograph of actual system.

study. Therefore, there remains a need to report research on the DDT of smokeless powders. The present authors conducted DDT tests on 58 types of smokeless powders, and examined the relationships between quickness, surface area/volume (*S/V*) ratio and DDT behavior.

2. Experimental

2.1 DDT test

Propellants were encased in carbon-steel tubing

Table 1 Comparison between 50/60 DDT test and modified USA DDT test

Name		50/60 DDT test	Modified USA DDT test
Steel tube size	Diameter	49.5mm	74mm
	Thickness	5.5mm	7.6mm
	Length	500mm	457mm
	Volume	962cm^3	1965cm^3
Ignition point		End point	Middle point
Ignitor		10 g of black	20 g of black powder
Go/nogo criteria		Mass distribution of fragments	Hole formed in witness plate*

* Witness plate : 8 mm-thick mild steel plate welded to the tube

(G3454, pressure-resistance type) as specified in the United Nations (UN) BAM 50/60 steel tube test [6]. The inner and outer diameter of the steel tubing was 50 mm and 60 mm, respectively, and each tube was 500 mm in length. The cap was S25C steel, fitted with a gasket and fitting suitable for an ignition system. The total weight of the DDT assembly (Fig. 1) was about 5300 g. The charge volume of smokeless powder was 962 cm^3, and a polyethylene bag containing 10 g of black powder and a fuse head (lead thiocyanate + $KClO_3$) was used as the igniter. Experiments were conducted for 58 types of smokeless powders manufactured by the NOF corporation, including 24 types of single-base propellants, 30 types of double-base propellants and 4 types of triple-base propellants.

After ignition, the fragments were recovered and data collected using a personal computer and sorted according to mass. Fragments smaller than 1 g were not recorded in this study because it would take an unreasonable amount of time. However, the results are considered not to be affected by this omission as will be seen later. The results were plotted as a cumulative number with respect to mass, also as shown later.

Table 1 shows the conditions of the 50/60 DDT test and the Modified USA DDT test (test 5(b, ii) with 20 g igniter) [6]. The diameter of the tube used in the USA test is 1.5 times larger than for the 50/60 DDT test, corresponding to twice the tube volume. However, the facilities available to the present authors were unsuitable for the larger test. Therefore, the 50/60 test was conducted and the transition length taken as twice that of the standard USA test. In the USA test, the go/no-go criterion is defined as whether a hole is created in a witness plate. In this work, the go/no-go criterion is decided by the mass distribution of fragments. Specifically, the ratio of the total mass of recovered fragments heavier than 200 g (W_{200}) to the original mass of the tube (W_0) is employed as the index for the detonation criterion.

Figure 2 shows the double-shell water protection and fragment-recovery system acting as a momentum trap. The outer and inner shells are constructed from VU400 and VU200 polyvinyl chloride (Japan Industrial Standard K6741). The shell is 67 cm in length, and the total volume of water contained is 58.7 L. The top was covered with wood. This system effectively limits damage to the detonation pit and facilitates the recovery of fragments.

2.2 Quickness and *S/V* measurement

Figure 3 shows the method of quickness measurement employed in this study, as developed by the NOF corporation. Quickness [7] is a measure of the burning rate of a smokeless powder for a given geometrical form and pressure, and is measured as a relative value because at present there is no international standard of quickness measurement. In this method, a 150 cc vessel is filled with 30 g of sample with 0.5 g of black powder as the igniter. The vessel pressure during a test is monitored using a pressure gauge and recorded on a personal computer. The 20% and 80% points with respect to maximum vessel pressure (P_{100}) are denoted P_{20} and P_{80}, and 20% and 80% of the elapsed time to the maximum vessel pressure are denoted t_{20} and t_{80}. Then, quickness is defined as the

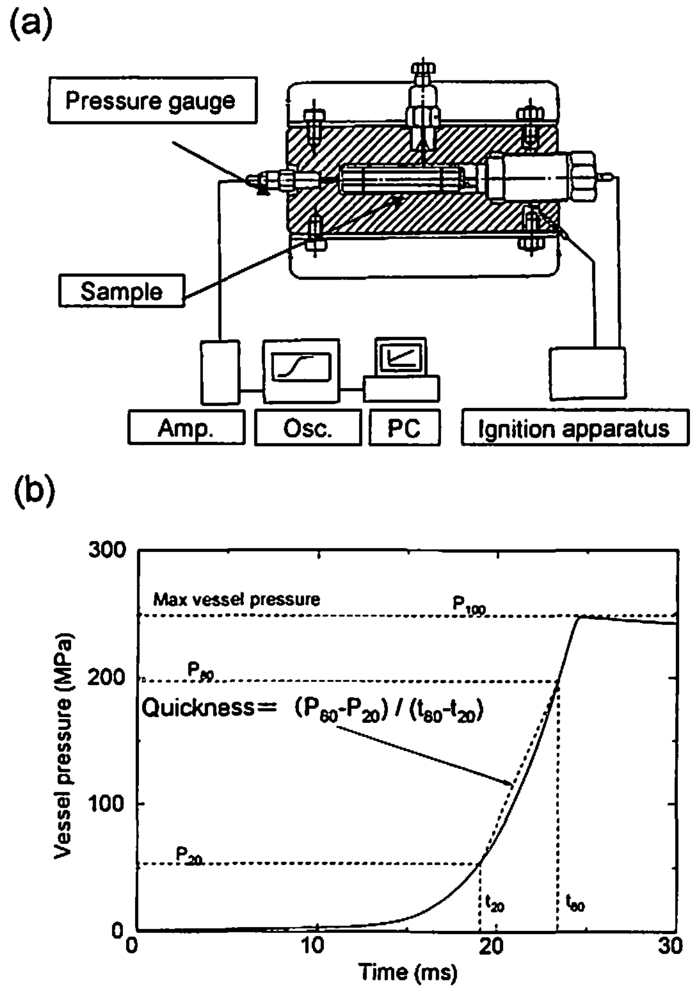

Fig. 3 Measurement of quickness. (a) Schematic of apparatus and (b) variation in bore pressure over time.

following equation.

$$\text{Quickness} = \frac{P_{80} - P_{20}}{t_{80} - t_{20}} \qquad (1)$$

The density of propellants was measured by the Archimedes method, and the specific surface area was measured by Bruanuer-Emmet-Teller (BET) absorption. The theoretical maximum density (TMD) was calculated using computational code according to the chemical properties. The S/V ratio was determined by measuring the surface area and volume of each pellet of explosive.

3. Results and discussion

3.1 DDT test

Table 2 lists the properties of the smokeless powders tested and the results of DDT tests. The list is sorted in order of increasing detonation criterion (W_{200}/W_0) in each of the powder types; single-base (SB), double-base (DB), and triple-base (TB) propellants. The criterion for SB and DB propellants varied from 0 to 100%, whereas all the values for the TB propellants were close to 100%, indicating non-detonation (no-go).

3.2 Test criteria

After the 50/60 DDT tests, the steel tube fragments are collected and weighed. In this study, detonation was defined to have occurred if less than half of the recovered tube pieces were heavier than 200 g, that is

$$\frac{W_{200}}{W_0} < 0.5 \qquad (2)$$

Table 2 also shows the UN test class division results for a range of single- and double-based propellants. Except for DB-13, the detonation thresholds obtained in the present study are in

Table 2 DDT test results

No.	Category	Name	Code Name	Properties of smokeless powders: Composition(wt.%)	TMD (g/cm^3)	Speecitic surface area (m^2/kg)	S/V (1/mm)	Quickness (MPa/ms)	Amount of load(g)	50/60 DDT results: Charge density (g/cm^3)	W_{200}/W_0(%)	Recovery ratio of fragments (%)	UN DDT test class division
1	SB	SB-1	PSB	NC99.0/Other1.0	1.67	3.59	6.0	60	477	0.48	4.0	68.0	—
2	SB	SB-2	—	NC/Other	1.66	7.59	12.58	30	961	0.96	5.5	—	—
3	SB	SB-3	35I	NC97.5/Other1.0	1.67	6.4	10.67	53	502	0.50	8.3	81.4	1.1
4	SB	SB-4	—	NC95.0/Other5.0	1.74	7.86	13.33	35	533	0.53	9.3	79.1	1.1
5	SB	SB-5	—	NC97/Other3	1.67	3.5	5.85	23	533	0.53	20.7	88.7	—
6	SB	SB-6	—	NC97/Other3	1.67	4.79	8.01	23	635	0.63	28.5	—	—
7	SB	SB-7	—	NC97.5/Other2.5	1.67	6.4	10.67	53	551	0.55	28.9	—	1.1
8	SB	SB-8	84I	NC99.0/Other1.0	1.67	5.6	9.33	30	480	0.48	29.9	—	—
9	SB	SB-9	—	NC/Other	—	—	3.73	—	528	0.53	36.3	—	—
10	SB	SB-10	—	NC97/Other3	—	—	—	—	835	0.83	41.2	—	—
11	SB	SB-11	—	NC81.3/Other18.7	1.84	4.08	7.5	50	572	0.57	42.0	96.9	—
12	SB	SB-12	—	NC80.0/Other20.0	—	—	—	—	317	0.32	51.4	—	—
13	SB	SB-13	—	NC98.7/Other1.3	—	—	9.43	—	718	0.72	52.6	—	—
14	SB	SB-14	—	NC98.4/Other1.6	1.67	8.99	15.00	30	1008	1.01	54.4	—	1.1
15	SB	SB-15	—	NC91/DNT5.0/Other3.5	1.63	5.18	8.44	30	805	0.80	64.2	—	—
16	SB	SB-16	NY-500	NC91.35/Other8.65	1.63	4.23	6.91	9	966	0.96	69.7	—	—
17	SB	SB-17	—	NC32.0/Other68.0	1.94	2.37	4.62	15	830	0.83	73.7	—	—
18	SB	SB-18	—	NC92/Other8	1.63	3.60	5.87	4	993	0.99	75.4	—	—
19	SB	SB-19	—	NC/Other	1.65	2.90	4.80	50	909	0.91	80.1	—	—
20	SB	SB-20	—	NC95.5/Other4.5	1.82	65.79	120	—	749	0.75	93.8	—	—
21	SB	SB-21	—	NC93.5/Other6.5	1.68	2.37	3.98	4	1068	1.07	95.7	—	1.3
22	SB	SB-22	—	NC90/Other10	1.63	2.36	3.86	2.5	1029	1.03	96.3	—	1.3
23	SB	SB-23	—	NC97.3/Other2.7	1.63	1.87	3.04	3	1026	1.02	96.9	—	1.3
24	SB	SB-24	—	NC88.7/Other11.3	1.64	2.04	3.33	2.5	1003	1.00	99.7	—	1.3
25	DB	DB-1	SS	NC71.8/NG26.5/Other1.7	1.65	6.59	10.86	30	613	0.61	0.0	64.5	—
26	DB	DB-2	—	NC57.75/NG40.0/Other2.25	1.64	4.56	7.50	12	888	0.89	4.6	—	—
27	DB	DB-3	M9	NC57.75/NG40.0/Other2.25	1.64	6.50	10.67	64	718	0.72	4.9	70.3	—
28	DB	DB-4	NY-100	NC58.75/NG40.0/Other1.25	1.63	15.35	25.00	75	705	0.70	5.0	78.2	—
29	DB	DB-5	—	NC55.0/NG40.6/Other4.2	1.64	7.74	12.67	32	751	0.75	5.7	—	1.1

No.	Category	Name	Code Name	Properties of smokeless powders: Composition(wt.%)	TMD (g/cm^3)	Spcecitic surface area (m^2/kg)	*S/V* (1/mm)	Quickness (MPa/ms)	50/60 DDT results: Amount of load(g)	Charge density (g/cm^3)	W_{200}/ W_0(%)	Recovery ratio of fragments (%)	UN DDT test class division
30	DB	DB-6	MJ-B	NC65.7/NG33.1/Other1.2	1.64	7.82	12.86	40	592	0.59	10.5	–	–
31	DB	DB-7	NY-300	NC81.5/NG15.5/Other3.0	1.66	8.48	14.09	75	997	0.99	13.3	–	–
32	DB	DB-8	9P	NC57.7/NG40.0/Other2.3	1.63	15.37	25.00	76	628	0.63	13.8	–	–
33	DB	DB-9	–	NC57.75/NG40.0/Other2.25	1.64	6.49	10.67	64	700	0.70	16.0	–	–
34	DB	DB-10	–	NC66.5/NG20.0/Other13.5	1.66	7.59	12.58	30	792	0.79	17.1	–	–
35	DB	DB-11	NP	NC82.8/NG14.8/Other2.4	1.66	15.10	25.00	75	601	0.60	21.3	–	–
36	DB	DB-12	–	NC57.75/NG40.0/Other2.25	1.64	17.95	29.49	64	536	0.54	25.7	–	–
37	DB	DB-13	–	NC/NG44.0/Other	1.63	5.88	9.60	35	762	0.76	26.1	–	1.3
38	DB	DB-14	–	NC78.05/NG20.0/Other1.95					882	0.88	37.1	–	–
39	DB	DB-15	–	NC59.6/NG39.7/Other0.7	1.63	7.43	12.15	30	510	0.51	47.5	92.2	1.1
40	DB	DB-16	–	NC77.45/NG19.5/Other3.05	1.68	3.21	5.40	13	874	0.87	53.9	–	–
41	DB	DB-17	–	NC57.1/NG41.6/Other1.3	1.63	1.84	3.00	15	748	0.75	55.3	–	1.3
42	DB	DB-18	–	NC58.3/NG39.2/Other2.5	1.63	3.41	5.56	18	717	0.72	58.7		1.3
43	DB	DB-19	–	NC77.45/NG19.5/Other3.05	1.39	0.85	5.51	18	602	0.60	67.1	–	–
44	DB	DB-20	–	NC81.95/NG15.00/Other3.05	1.68	0.86	1.44	5	847	0.85	68.4	–	–
45	DB	DB-21	–	NC60.1/NG38.2/Other1.7	1.63	3.16	5.15		751	0.75	73.2	–	1.3
46	DB	DB-22	–	NC55.4/NG44.1/Other3.5	1.63	1.23	2.00	6.5	803	0.80	82.9	92.8	1.3
47	DB	DB-23	–	NC50.7/NG45.0/Other4.3	1.63	0.56	0.91	5	740	0.74	97.7	–	–
48	DB	DB-24	–	NC62.5/DEGDN36.7/Other1.0					811	0.81	99.1	–	–
49	DB	DB-25	–	NC62/DEGDN38/Other1	1.56	0.45	0.71	5	533	0.53	99.6	–	–
50	DB	DB-26	–	NC50.7/NG45.0/Other4.3					866	0.86	99.8	–	–
51	DB	DB-27	–	NC62.5/DEGDN36.7/Other1.0	1.56	0.6	0.94	5	815	0.81	99.9	–	–
52	DB	DB-28	–	NC53.3/NG35.1/Other11.6					728	0.73	100.0	–	–
53	DB	DB-29	–	NC62.5/DEGDN36.7/Other1.0	1.56	0.61	0.95	5	817	0.82	100.2	–	–
54	DB	DB-30	–	NC51.5/NG35.0/Other13.5	1.57	0.68	1.07	5	660	0.66	100.5	–	–
55	TB	TB-1	–	NC20.4/NG19.0/NQ54.7/Other6.3	1.68	0.43	0.72	5	869	0.87	98.6	–	–
56	TB	TB-2	–	NC20/NG19/NQ54/Other7	1.64	0.85	1.39	18	741	0.74	100.0	–	–
57	TB	TB-3	–	NC/NG/NQ/Other					809	0.81	100.0	–	–
58	TB	TB-4	–	NC20/NG19/NQ54.7/Other6.3	1.64	1.10	1.81	10	964	0.96	100.4	–	–

good agreement with the UN test class division results. The reason for the discrepancy for DB-13 is unknown. Division 1.1 represents detonation, and Division 1.3 corresponds to non-detonation. We can therefore consider the present go/no-go criteria to be appropriate.

3.3 Distribution of fragment mass

Shock fragmentation of solid objects has been studied extensively in the laboratory and in geological planetary systems. For a wide variety of fragmented objects, it has been found that the cumulative distribution of sizes and masses can be represented by a power law for some non-negligible range. The differential mass distribution is written as follows.

$$n(m) \propto m^{-\beta} \qquad (3)$$

where $n(m)$ is the cumulative mass, m is the fragment mass, and β is the coefficient of the power law.

Equation (3) was introduced to characterize the shock fragmentation of solid objects. For many brittle materials, including soap, paraffin and frozen potato, $\beta = 1.5\sim2$ [8]. For thin glass rods, $\beta=$ 0.6 (small mass) and $\beta = 1.5$ (large mass) [9]. These values for glass represent the transition from one-dimensional fragmentation to three-dimensional fracturing.

Martho et al. [10] examined three models for the fragment size distribution of an exploded iron cylinder; the Rosin-Ramler-Bennet (R-R-B) model, Gates-Gaudin-Scumann (G-G-S) model and logarithmic-probability (L-P) model. These models were discussed with respect to four types of explosives; dynamite, slurry, emulsion, and pentaerythrite tetranitrate (PETN). The G-G-S model was found to fit the experimental results most accurately, and is equivalent to the differential mass distribution given by equation (3). Thus, this definition of the differential mass distribution is adopted in the present study.

Figure 4 shows the mass distribution of fragments and photograph of the recovered tubing for single-base propellants SB-4 and SB-11, and double-base propellants DB-1 and DB-22. The fragments recovered for DB-1 are the smallest in this study. The slope value (β) for this propellant changed from 0.6 to 1.6, with a transition at approximately $m_t = 60$ g. There are no fragments larger than 200 g in this case.

The ratios for the detonation criterion indicate that the first three of these four propellants detonated, with DB-22 being the exception. The transition point m_t increases from 60 g, to 100 g to 200 g, in the first three cases. No transition point is seen in the data for DB-22, and the slope value (β) is 0.6 over the entire weight range. The recovery of fragments smaller than 10 g is difficult, as the figures indicate.

Two slopes can be seen in the present mass distributions; $\beta = 0.6$ in the small mass region indicative of one-dimensional breakage, and $\beta = 1.8$ in the large mass region, reflecting three-dimensional breakage.

3.4 Relationship between DDT and S/V

The quickness and S/V values (Figure 5) clearly separate the detonation and non-detonation results. The dotted region in the figure represents the region in which detonation occurred (GO result) for the present experimental configuration. The threshold for quickness is 20 $MPa \cdot ms^{-1}$, and for the S/V ratio is 6 mm^{-1}. Apart from 3 exceptions (SB-14,15 and 19), larger values of both quickness and S/V correlate directly with detonation. Therefore, the detonation condition can be predicted in most cases from this data. In the case SB-14 and SB-15, the ratio for the detonation criterion is 50%, at which detonation may still occur. For SB-19, it is considered that the quickness was overestimated.

The use of quickness and the S/V ratio for estimating the detonation condition is reasonable because quickness is defined from the variation in vessel pressure with time, and directly represents physical properties.

4. Conclusion

Of 58 types of smokeless powder, including 24 types of SB, 30 types of DB, and 4 types of TB, 26 types of smokeless powder (11 types of SB, 15 types of DB) were found to detonate under the present DDT test conditions. The mass distribution of fragments recovered from detonated tests had two

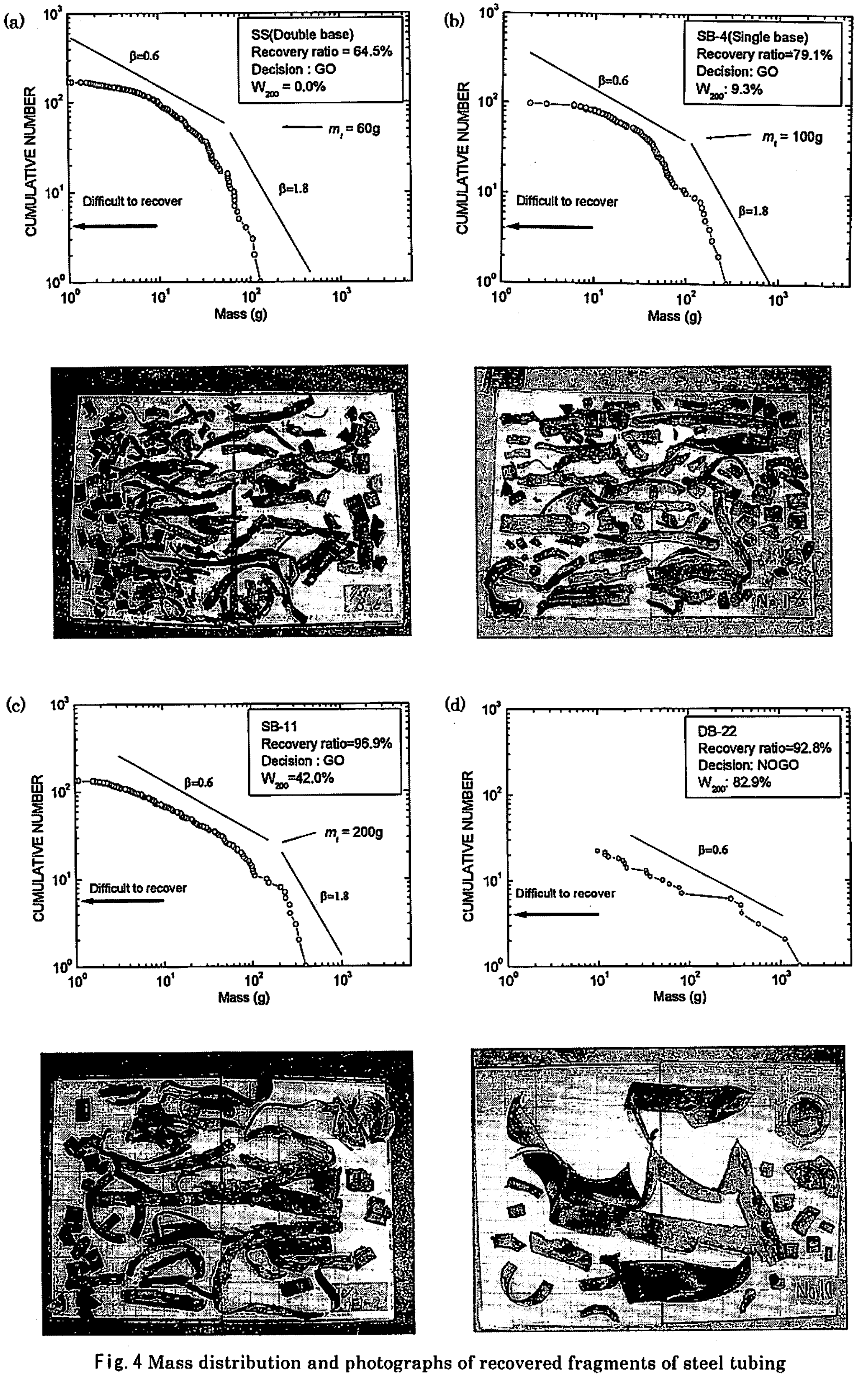

Fig. 4 Mass distribution and photographs of recovered fragments of steel tubing

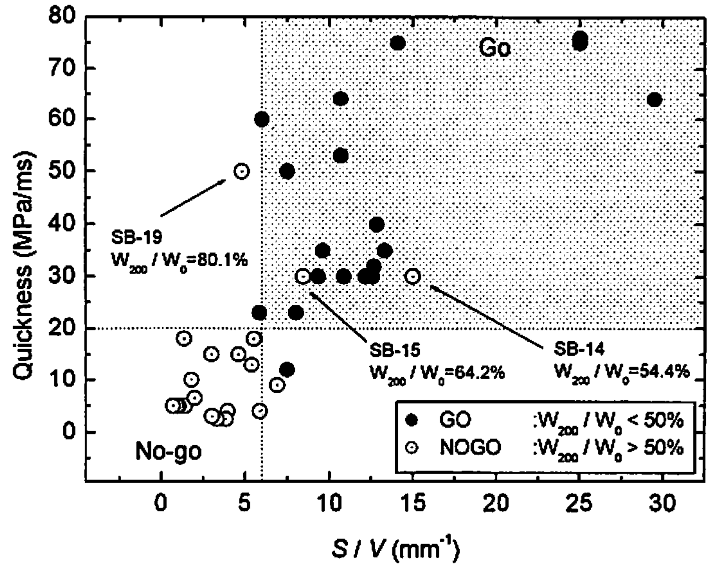

Fig. 5 Relationship between quickness and S/V with respect to the detonation criterion

distinct regions with slope $\beta= 0.6$ (small fragments) and 1.6 (large fragments). From an analysis of the relationship between the detonation criterion and the quickness and *S/V* ratio of the propellants, it was found that propellants with high possibility of detonation can be identified with reasonable confidence based on the quickness and *S/V*. Therefore, DDT behavior can be estimated based on quickness and *S/V* data without the need to conduct expensive and time-consuming DDT tests. This result is expected to contribute significantly to safety research.

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