研究論文

Screening test of gas-generating agents using closed strand burner

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In order to test the effectiveness of the gas-generating agents of airbag inflators to be used in motor vehicles, it is important to measure the gas generation behavior within a closed vessel that closely simulates the airbag. The 60L-tank test is one of the most widely used methods. However, 60L-tank test apparatus are not very common in institutes other than automobile companies and explosives manufacturers. In addition, there is a need for a relatively large amount (20g) of sample for each test.

In this study, we devised a new method of screening test of gas-generating agents, utilizing a closed chimney-type strand burner. Compared to the conventional methods, the new method have the merits in that: 1) there is no need to additionally purchase 60L-tank test equipments; 2) it requires less amount of samples compared to the 60L-tank test (total sample weight: 60L-tank test 20g; new method 4g); 3) it is closer to the 60L-tank test in volume than the small-scale deflagration test (volume: small scale deflagration test 52mL; new method 4L); and 4) it is easy to observe the burning behavior of the pellet.

To test the validity of the method, we compared the results of the rate of pressure rise inside the vessel with those of the 60L-tank test for compositions consisting of tetrazole/oxidizer mixtures.

In the screening test, the composition to be tested was prepared by sieving and drying powder samples of tetrazole and oxidizer, then mixing at a designated fuel/oxidizer-weight ratio by the use of a rotary mixer. The composition was then pressed into a 4g pellet. Unlike the linear burning rate measurement, no restricting agent was applied at the bottom and at the sides of the pellet. After mounting the pellet inside the vessel, N_2 gas was introduced. The vessel was closed, after checking that the operational pressure reached the designated level. The deflagration of the pellet was initiated by the use of a nichrome wire. The pressure change inside the strand burner after ignition was measured employing a strain-gauge pressure sensor. The deflagration behavior of the pellet was also monitored by a video camera.

Comparing the results between the rate of pressure rise inside the strand burner and the pressure rise after 50ms, together with the rate of pressure rise inside the tank of the 60L-tank for the tested compositions, there were linear relationships for gas-generating agents whose fuel are composed of different tetrazoles, indicating good correlation. There was also a linear relationship for the result of the rate of pressure rise between the strand burner and the combustor of the 60L-tank. The results suggested the possibility of the new method as a screening test of gas-generating agents.

Received : May 17, 2002
Accepted : August 30, 2002
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1. Introduction

In order to test the effectiveness of the gas-generating agents of airbag inflators to be used in motor vehicles, it is important to measure the gas generation behavior within a closed vessel that closely simulates the airbag. The 60L-tank test is one of the most widely used methods. However, 60L-tank test systems are not very common in institutes other than automobile companies and explosives manufacturers. In addition, there is a need for a relatively large amount (20g) of sample for each test.

In this study, we devised a new method of screening test of gas-generating agents, utilizing a chimney-type strand burner with its orifice closed. We also examined the validity of the new method by comparing the rate of pressure rise inside the strand burner and the total pressure rise and the rate of pressure rise inside the 60L-tank, together with the rate of pressure rise inside the combustor.

2. New method of evaluation

The new method of evaluating the performance of the gas-generating agent to be used for airbag inflator employs the chimney-type strand burner, which is commonly used for the measurement of the linear burning rate of strands consisting of propellants or gas-generating agents.

The new method has the following advantages:

1) There is no need to additionally purchase 60L-tank test equipments;
2) It requires smaller amount of samples (Total sample weight: 60L-tank test 20g; 1L-tank test 25g; new method 4g);
3) It is closer to the 60L-tank test in volume than the small-scale deflagration test[1] (volume: small scale deflagration test 52mL; new method 4L); and
4) It is easy to observe the burning behavior of the strand.

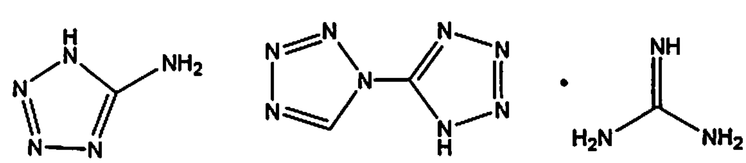
3. Experimental

3.1 Reagents used

5-Amino-1H-tetrazole (abbreviated as 5-ATZ from now on; Nippon Koki, Co. Ltd.) and guanidine salt of 1,5'-bi-1H-tetrazole (abbreviated as 15BG from now on; Toyo Kasei, Co. Ltd.) were chosen as the fuel of the gas-generating agent, as shown in Fig.1. The oxidizers were $KClO_4$(Kanto Chemical, Co. Ltd.), KNO_3(Wako Chemical, Co. Ltd.), $Sr(NO_3)_2$(Wako Chemical, Co. Ltd.), NH_4NO_3(Wako Chemical , Co. Ltd.) and CuO(Wako Chemical , Co. Ltd.). Each powder reagent was first ground employing a porcelain ball mill, sieved through 100 mesh (149μm pass) sieve, then dried *in vacuo* for 24hours at 45℃ and stored in a dessicator for at least 24hours.

3.2. Preparation of the sample

The reagents were mixed at a designated mixing ratio as shown in Table.1, by the use of a rotary mixer. After drying the mixture again in vacuo for 24hours at 45℃ and stored in a desiccator, it was then pressed into a cylindrical pellet (diameter



(a)5-amino-1H-tetrazole(5-ATZ) (b)Guanidine salt of 1,5'-bi-1H-tetrazole(15BG)

Fig.1 Schematic diagram of the 60L-tank test system

Table.1 Tetrazole/oxidizer mixtures tested (unit: wt%)

Sample number	Fuel		Oxidizer					Additive
	5-ATZ	15BG	NH_4NO_3	$Sr(NO_3)_2$	KNO_3	CuO	$KClO_4$	Urea
No.1	23. 3	-	57. 1	-	-	19. 6	-	-
No.2	33. 4	-	33. 6	-	-	-	33. 0	-
No.3	36. 5	-	-	63. 5	-	-	-	-
No.4	-	37. 5	-	-	62. 5	-	-	-
No.5	-	37. 5	-	-	-	-	62. 5	-
No.6	-	37. 5	-	-	-	-	62. 5	5. 0

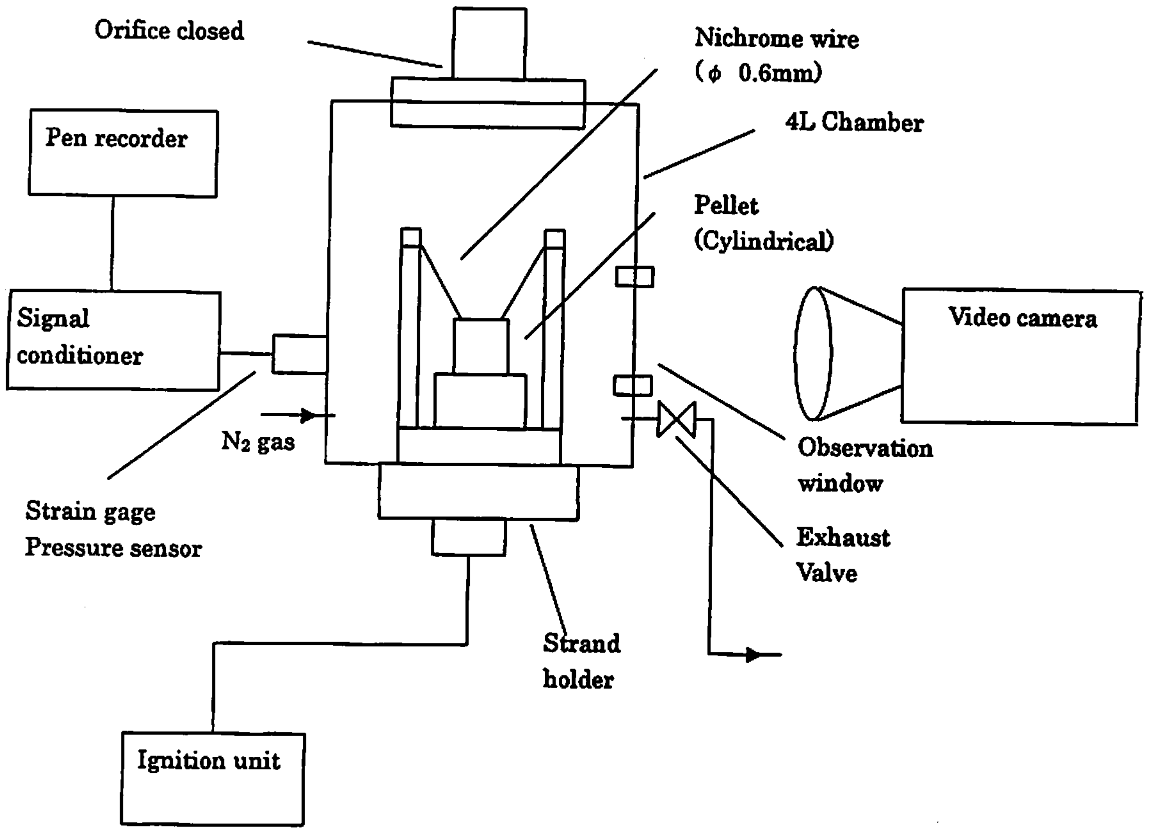


Fig.2 Schematic diagram of the screening test utilizing closed chimney-type strand burner

14.70 mm, height 12 ~15 mm each), employing a hydraulic press. The pressure applied was 60kgf cm^{-2} (5.88MPa) for 5-ATZ compositions and 100kgf cm^{-2} (9.8MPa) for 15BG compositions. Unlike the linear burning rate measurement, no restricting agent was applied at the bottom and at the side of the pellet.

3.3. Apparatus

3.3.1. Closed chimney-type strand burner

The schematic diagram of the measurement system utilizing a 4L chimney-type strand burner is shown in Fig.2. In this study, chimney-type strand burner SCTA-50 (Kyowa Giken, Co. Ltd.) was used. The orifice of the strand burner was closed by insertion of a plain steel disk. The pellet to be tested was fixed on the platform of the strand holder. After inserting the strand holder into the vessel, the atmosphere inside the strand burner was replaced with N_2 gas, repeating twice the process of introducing the gas and releasing it through an exhaust valve. Then, the N_2 gas was introduced into the vessel at 20kgf cm^{-2} (1.96MPa). The internal pressure of the vessel was detected by strain-gauge pressure sensor PG-100KU-F (Kyowa Dengyo, Co. Ltd), and after amplification through signal conditioner CDV-230C (Kyowa Dengyo, Co. Ltd), it was recorded on a pen recorder. After the internal pressure reached the designated level, the exhaust valve of the vessel was closed, and the pellet was ignited by the use of nichrome wire (diameter 0.6mm). Together with the change in pressure within the vessel, the burning behavior of the strand was also monitored by a VHS video camera through an observation window.

3.3.2. 60L-tank test

The schematic diagram of the 60L-tank test apparatus is shown in Fig.3. In this study, the facility of Nippon Koki, Co. Ltd was used. For each set of mixture, 10 of 2g pellets (diameter 14.7mm, height 6~7mm each) in case of HAT compositions, and 15 of 1g pellets (diameter 14.7mm, height 3~4mm each) in case of 15BG compositions, were inserted into the combustor. A spacer (diameter: 36mm (outside), 19mm (inside); height: 10mm) was inserted into the combustor in the case of 15BG compositions, since the total mass of the sample

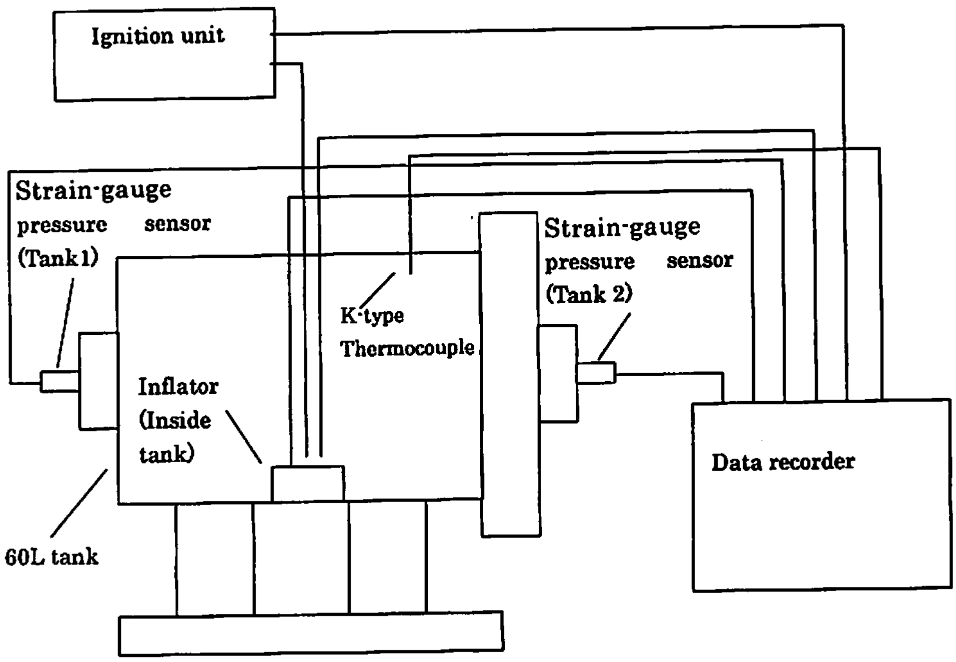


Fig.3 Chemical structures of tetrazole compounds considered in this study

cmparatively small. The combustor was then installed inside the 60L-tank. After the vessel is closed, the pellets inside the combustor were ignited by the use of B/KNO_3 igniter. Then, the change in pressure at two locations inside the tank, P_{tank1} and P_{tank2}, and the change in pressure inside the combustor P_{comb} were measured employing strain-gauge sensors (Kyowa Dengyo, Co. Ltd; PGM-10KC for the pressure inside the tank and PGM-500KE for the combustor). The data were recorded on a DL708E data recorder (Yokogawa Denki, Co. Ltd). The change in temperature inside the tank, T_{tank}, was also measured by K- thermocouple and the data was recorded on the same data recorder.

3.3.3. Analysis

3.3.3.1. Rate of pressure rise within the strand burner

The schematic diagram of the pressure-time characteristic within the chimney-type strand burner for a pellet of certain composition is given in Fig.4. The rise in pressure, $\Delta P_{strand\ burner}$, is given by

$$\Delta P_{strand\ burner} = P_{peak} - P_{initial} \quad [MPa] \qquad (1)$$

The time from the initial to the peak pressure, $\Delta t_{strand\ burner}$, is given by

$$\Delta t_{strand\ burner} = t_{peak} - t_{initial} \quad [s] \qquad (2)$$

From (1) and (2), the average rate of pressure rise, $R_{av.\ strand\ burner}$, is given by,

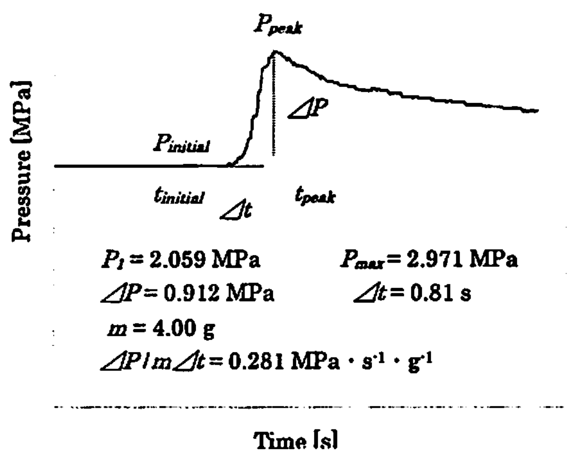


Fig.4 Pressure – time curve of 5-ATZ/$Sr(NO_3)_2$ mixture at stoichiometric ratio in chimney-type strand burner

$$R_{av.\ strand\ burner} = \Delta P_{strand\ burner} / (m_{pellet} \cdot E\ \Delta t_{strand\ burner}) \ [MPa\ s^{-1}\ g^{-1}] \qquad (3)$$

Where, m_{pellet} [g], is the mass of the pellet tested.

3.3.3.2. 60L-tank test

An example of typical pressure-time characteristics within the combustor and the tank for the 60L-tank test is given in Fig.5. The pressure within the combustor reaches its peak within 10 ms after ignition. Almost as soon as the internal pressure of the combustor reaches its peak, the

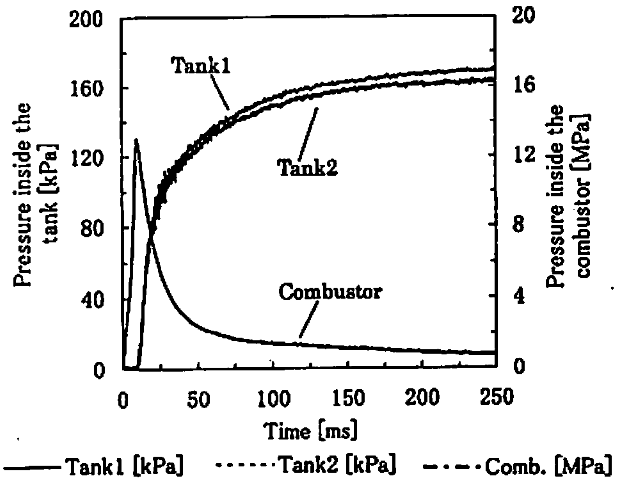


Fig.5 Time profile of the change in pressure inside the tank and the combustor for the 60L tank test (5-ATZ/$Sr(NO_3)_2$ mixture tested)

pressure within the tank begins to rise, and reaches the maximum after 40∼100ms. The average rate of pressure rise within the combustor, $R_{av\ combustor}$, was derived similarly through equations (1)', (2)' and (3)'.

$$\Delta P_{av\ combustor} = P_{peak} - P_{initial} \quad [MPa] \qquad (1)'$$

$$\Delta t_{av\ combustor} = t_{peak} - t_{initial} \quad [s] \qquad (2)'$$

$$R_{av\ combustor} = \Delta P_{av\ combustor} / (m_{pellet} \cdot \Delta t_{av\ combustor}) \quad [MPa\ s^{-1}\ g^{-1}] \qquad (3)'$$

The rise in pressure after 50ms, $\Delta P_{50ms,60L\text{-}tank}$, and the average rate of pressure rise within the 60L-tank after 50ms, $R_{50ms\ 60L\text{-}tank}$, were also derived from (4), (5) and (6). These values after 50ms are important since the airbag is required to achieve full-bag expansion within a time range of 40∼50ms after ignition.

$$\Delta P_{50ms,60L\text{-}tank} = P_{50ms} - P_{initial} \quad [MPa] \qquad (4)$$

$$\Delta t_{50ms} = 0.0050 - t_{initial}\ [s]\ (t_{initial} \text{ is not necessarily equal to } 0) \qquad (5)$$

$$R_{av\ 60L\text{-}tank} = \Delta P_{60L\text{-}tank} / m_{60L\text{-}tank}\ \Delta t_{50ms} \quad [MPa\ s^{-1}\ g^{-1}] \qquad (6)$$

Where, $m_{60L\text{-}tank}$ [g], is the total mass of pellets in a combustor.

3.3.3.3. Evaluation of the validity of the new method

In order to evaluate the validity of the new method, the correlations between the average rate of pressure rise within the 4L-strand burner and

1) the average rate of pressure rise within the combustor;
2) the total pressure rise within the 60L-tank after 50ms; and
3) the average rate of pressure rise within the tank after 50ms

were compared.

4. Results and discussions

The comparison between the average rate of pressure rise for the 4L-strand burner and that of the combustor of the 60L-tank, for each set of experiment, is given in Fig.6. Number displayed in the diagram corresponds to the sample number listed in Table 1. For both 5-ATZ mixtures and

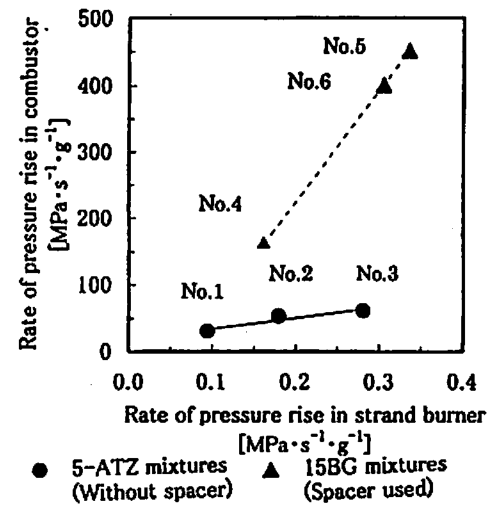


Fig.6 Relationship between average rates of pressure rise inside chimney-type strand burner and combustor of 60L-tank (Number corresponds to the sample number listed in Table.1)

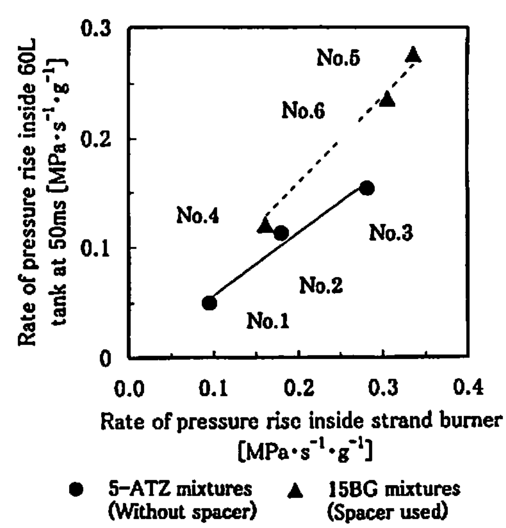


Fig.7 Relationship between average rate of pressure rise inside strand burner and average rate of pressure rise inside 60L-tank at 50ms (Number corresponds to the sample number listed in Table.1)

Table 2 Correlative expressions between the average rate of pressure rise inside the closed 4L - strand burner (SCTA-50, Kyowa Giken, Co. Ltd.) and the results of the 60L - tank test (Nippon Koki, Co. Ltd.)

Expression	Values of a[-], b[MPa s^{-1} g^{-1}], c[-], and d[s·g]	
	5-ATZ mixtures (Without spacer)	15BG mixtures (Spacer used)
$R_{av\ combustor} = a \cdot R_{av.\ strand\ burner} + b$	a=2.46 x 10^2 b=18.9	a=1.67 x 10^3 b=10.5
$R_{av\ 50ms,\ 60L\ tank} = c \cdot R_{av.\ strand\ burner}$	0.568	0.796
$\Delta P_{av\ 50ms,\ 60L\ tank} = d \cdot R_{av.\ strand\ burner}$	0.447	0.796

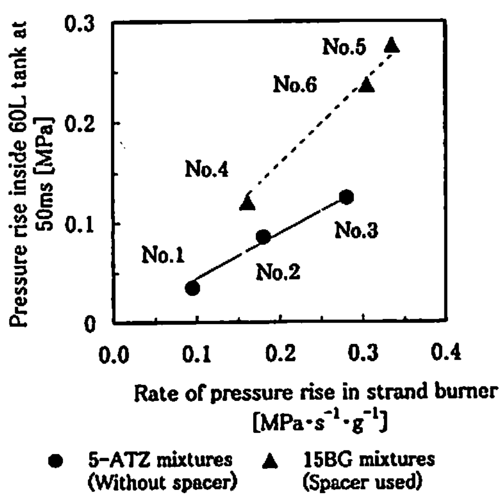


Fig.8 Relationship between average rate of pressure rise in chimney type strand burner and pressure rise inside 60L-tank at 50ms (Number corresponds to the sample number listed in Table.1)

15BG mixtures, there were linear relationships between the average rate of pressure rise within the strand burner and that of the combustor of the 60L-tank. There was a difference in the tendency in correlations for 5-ATZ mixtures and 15BG mixtures as shown in Fig.6 and Table.2, because a spacer was inserted into the combustor in the case of 15BG mixtures, whereas in the case of 5-ATZ mixtures, a spacer was not used.

The comparison between the average rate of pressure rise within the strand burner and the average rate of pressure rise of the 60L-tank at 50ms after ignition is given in Fig. 7. For both 5-ATZ mixtures and 15BG mixtures, there were linear relationships between the average rate of pressure rise within the strand burner and the average rate of pressure rise of the 60L-tank at 50ms.

The comparison between the average rate of pressure rise within the strand burner and the total pressure rise of the 60L-tank at 50ms after ignition is given in Fig. 8. Also in this case, there were linear relationships between the average rate of pressure rise within the strand burner and the pressure rise of the 60L-tank at 50ms after ignition, for both 5-ATZ mixtures and 15BG mixtures. Figs. 7 and 8 show that, despite the difference in internal volume and structure between the 4L-strand burner and the 60L-tank, both the total pressure rise and the average rate of pressure rise after 50ms for the 60L-tank test can be estimated from the result of the strand burner test.

In conclusion, there were linear relationships between the results for the closed 4L-strand burner and the 60L-tank, suggesting the possibility as the new screening test of gas-generating agents.

5. Conclusions

A new method of screening test of gas-generating agents was devised, which utilizes a 4L chimney-type strand burner. In order to examine the validity of the screening test, comparisons were made between the new screening method and the 60L-tank test for gas-generating agents whose fuel are composed of tetrazoles, namely 5-ATZ and 15BG. As a result, there was a linear relationship between the rate of pressure rise inside the strand burner and those inside the combustor of the 60L-tank for the tested compositions, indicating good correlation. There was also a linear relationship for the results of the rate of pressure rise between the strand burner and the 60L-tank after 50ms, and also between the rate of pressure rise of the strand burner and the pressure rise inside the 60L-tank after 50ms. The results suggested the possibility of the new method as a screening test of gas-generating agents.

References

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