Research paper

Development of the testing method for mixing explosion hazards of pyrophoric substances

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Abstract

The conditions in which a steel tube burst were investigated experimentally by mixing triethylaluminum (TEA), water, and aluminum hydroxide ($Al(OH)_3$) in a steel tube. The purpose of this investigation was to develop a new testing method for estimating the hazards relating to explosions from mixing pyrophoric substances. The experimental results proved that a steel tube broke owing to the mixing reaction between TEA, water, and $Al(OH)_3$. The estimated pressure from the gases generated from the reaction of the samples were compared with the estimated resisting pressure of the steel tube in each test, and it was found that increasing the amount of samples per unit volume of the steel tube and reducing the obstacles in it through designing the arrangement of samples appropriately are important for this testing method.

Keywords : mixing explosion hazards, testing method, pyrophoric substances, triethylaluminum, water

1. Introduction

Aluminum alkyls are widely employed as components of Ziegler-Natta catalysts, which are used in industries for the polymerization of olefins such as ethylene and propylene[1]. Aluminum alkyls are known to be very reactive. Compounds with alkyl groups of C_4 and below ignite immediately on exposure to air. Moreover, they also react violently with water, alcohols, and halogenated hydrocarbons[2].

Although aluminum alkyls are treated carefully, accidents have occurred[2], the cause generally being their exothermic autoxidation or their contact with halogenated hydrocarbons. However, some accidents with unknown causes have also been reported. As a recent example, an accident occurred at an aluminum alkyls producing plant in 2007[3] in which the piping carrying triethylaluminum (TEA), one of the aluminum alkyls, burst and fired. The reaction hazards of TEA under closed conditions were subsequently investigated in order to determine the cause of the accident. It was found that the TEA and water system mixture in closed conditions decomposed into lower molecular weight compounds than those produced through well-known hydrolysis mechanisms. Moreover, it was found that aluminum hydroxide ($Al(OH)_3$) could be the source of water at high temperatures, and could be contribute to the mixed reaction between TEA and water[4].

However, it was difficult to prove that the burst was caused by the reaction between TEA, water, and $Al(OH)_3$ using existing explosibility testing methods. To understand the power of energetic materials, the strength of combustion is estimated using the burning rate test, the pressure vessel test, and the time/pressure test. Furthermore, the strength of explosion is estimated using the blast pressure measurement and the underwater explosion test[5]. Recently, many evaluations of the effects of explosives have been conducted using the blast pressure measurement[6)-8]. Moreover, dynamic effects of the detonation wave are generally estimated using the Hess brisance test, the Kast brisance test, and the detonation velocity measurement. The static effects of explosions are generally estimated using the Trauzl test, the ballistic pendulum test, the ballistic mortar test, and the underwater explosion test[5]. Samples for the above tests must be static at room temperature. Thus, using the above testing methods, it is difficult to estimate the explosion effects of pyrophoric substances such as TEA. Though testing methods for pyrophoric properties and reactivity with water have been described by the UN[9], it is difficult to evaluate the strength of explosion caused by the TEA-water reaction using these tests.

It was, therefore, desirable to develop a new testing method for estimating the strength of explosion caused by the TEA-water reaction. It was considered that such a testing method could be investigated by the development of a proving method for the bursting phenomenon in steel tubes caused by the TEA-water reaction. In this study, steel tube bursting was investigated using an experimental set-up of a TEA, water, and $Al(OH)_3$ mixture in a steel tube. Based on these experimental results, the possibility of a testing method for estimating the hazards relating to explosions from mixing pyrophoric substances was investigated.

2. Experiments

2.1 Materials

TEA from Tosoh Finechem Corporation was used without further purification. Deionized water was also used. Blasting caps and explosives were used to mix the TEA, water, and $Al(OH)_3$ remotely. Black powder and a handmade explosive were used as explosive substances. The handmade explosive was a mixture of potassium perchlorate (70 wt%, Japan Carlit) and aluminum flake (30 wt%, Nakatsuka), and it is referred to as an explosive of report composition hereafter. The scale that adhered to the steel piping in the TEA production plant in which the explosion accident occurred, hereafter referred to as the $Al(OH)_3$ scale, was used. The major component of the $Al(OH)_3$ scale was estimated to be $Al(OH)_3$ by analysis using thermogravimetric-differential thermal analyzer-mass spectrometry (TG-DTA-MS) and inductively coupled plasma-optical emission spectroscopy (ICP-OES)[4].

2.2 TEA mixing tests in the steel tube using blasting caps

Figure 1 shows a block diagram of the mixing tests. A remote mixing technique for a steel tube using blasting caps was designed. A steel tube (30 mm in diameter and 200 mm long) was used, which was enclosed at each end by steel screw caps and polytetrafluoroethylene (PTFE) tape. A hole (2 mm in diameter) was made in one or both the screw caps to insert blasting caps. TEA was packed into glass bottles in a glove box under a nitrogen atmosphere, and water was inserted into glass bottles under air. These samples and blasting caps (one or two, depending on the experiment) were inserted into the steel tube. A view of the steel tube was recorded using a video camera during the mixing tests. In test Nos.1-2 and 1-3, the surface of the steel tube was measured using a K-type sheath thermocouple (1.6 mm in diameter).

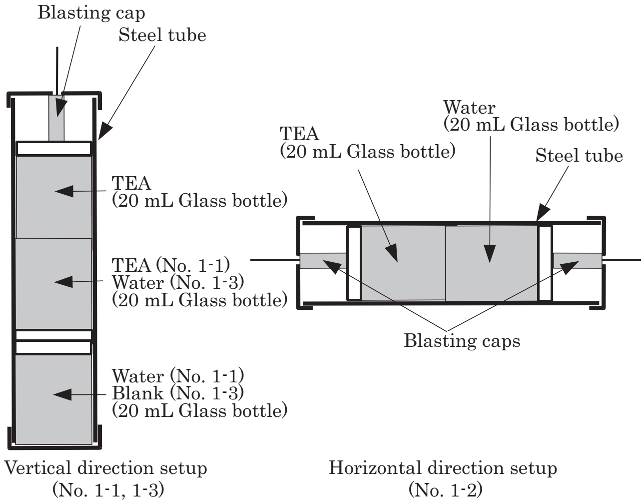

Figure 1 Block diagram of the TEA mixing tests in the steel tube using blasting caps.

Table 1 shows the experimental conditions. In test No.1-1, a steel tube was positioned vertically. TEA was packed in two glass bottles, the top and bottom bottles (Figure 1), and water was filled in the middle glass bottle. A blasting cap was fitted on the top of each sample. The glass bottles were broken by the impact of the blasting cap. In test No.1-2, a steel tube was positioned horizontally. The left glass bottle was filled with TEA and the right one with water. Two blasting caps were fitted on both ends of the samples, allowing impacts from blasting caps to be applied to these ends. In test No.1-3, a steel tube was positioned along a vertical direction. The sequence of samples was as follows: water filled in the top glass bottle, TEA in the middle bottle, and an empty glass bottle at the bottom of the tube. The blank glass bottle was used as a spacer. A blasting cap was fitted on the top of the samples.

2.3 TEA mixing tests in the steel tube using black powder

Figure 2 shows a block diagram of the mixing tests in which black powder was used in place of blasting caps. A steel tube (30 mm in diameter and 200 mm long) was used, closed by steel screw caps at each end. A hole of 2 mm diameter was made halfway along the length of the steel tube to insert a fuse head in order to ignite the black powder. TEA was packed into glass bottles in a glove box under a nitrogen atmosphere, and water and/or $Al(OH)_3$ scale was filled in plastic bags under air. The plastic bags were sealed with a sealer. Black powder was packed with a fuse head (Nippon Kayaku) in a sheet of plastic wrap. These samples and the black powder were inserted into a steel tube. The steel tube was closed by two screw caps and a PTFE tape. A view of the steel tube was recorded using a video camera during the mixing tests. The surface of the steel tube was measured using a K-type sheath thermocouple (1.6 mm in diameter).

Table 1 shows the experimental conditions. The steel tube was positioned along a horizontal direction in these

Table 1 Experimental conditions and results of the mixing test in the steel tube.

No.	Tube size	TEA [g]	Water [g]	$Al(OH)_3$ [g]	Mixing method	Result	Remarks
1-1	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (vertical direction)	40	20	0	One blasting cap	No explosion	Samples were not mixed completely
1-2	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (horizontal direction)	20	10	0	Two blasting caps	No explosion	Samples blew out and leaked out
1-3	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (vertical direction)	20	10	0	One blasting cap	No explosion	Samples blew out
2-1	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (horizontal direction)	80	28	0	Black powder 2 g	No explosion	Samples blew out and leaked out
2-2	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (horizontal direction)	80	26	5	Black powder 2 g	No explosion	Samples and flames blew out.
2-3	$^{\phi}30 \times {}^{L}200 \times {}^{t}5$mm (horizontal direction)	80	27	6	Black powder 2 g	Explosion	
3-1	$^{\phi}25 \times {}^{L}212 \times {}^{t}0.5$mm (horizontal direction)	42	26	5	Explosive of report composition 2 g	No explosion	Sealant melted
3-2	$^{\phi}25 \times {}^{L}212 \times {}^{t}0.5$mm (horizontal direction)	42	26	5	Explosive of report composition 2 g	No explosion	Samples were not mixed completely
3-3	$^{\phi}32 \times {}^{L}220 \times {}^{t}5$mm (notching part : $^{t}0.5$mm) (horizontal direction)	80	26	5	Explosive of report composition 2 g	Explosion	
3-4	$^{\phi}32 \times {}^{L}220 \times {}^{t}5$mm (notching part : $^{t}0.5$mm) (horizontal direction)	80	26	5	Explosive of report composition 2 g	Explosion	

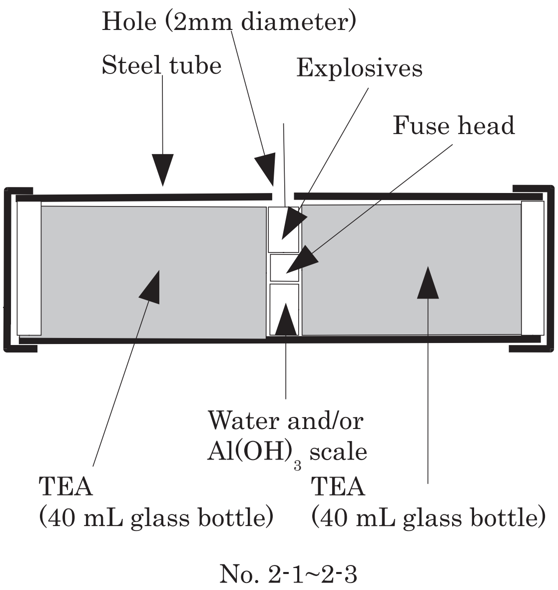

Figure 2 Block diagram of the TEA mixing tests in the steel tube using black powder.

mixing tests. In test No.2-1, TEA was packed in two glass bottles. Black powder and water were placed between the two glass bottles. The glass bottles and plastic bags were broken by the pressure arising from the generation of gas from the black powder. In test No.2-2, TEA was packed in two glass bottles, and black powder, water, and $Al(OH)_3$ scale were placed between the two glass bottles in separate bags. In test No.2-3, water and $Al(OH)_3$ scale were packed in the same bag for better mixing of samples, with all other conditions being the same as those for test No.2-2.

2.4 TEA mixing tests in the steel tube with a blank flange

Figure 3 shows a block diagram of the mixing tests. The steel tube was enclosed by a blank flange in place of steel screw caps. The design pressure of the flange was 14 MPa. Copper electrodes were inserted using a pressure sealing gland (Conax Technologies). Moreover, an explosive of report composition was used in place of black powder in this test. The electrical lead from the fuse head was clipped to the copper electrodes. A view of the steel tube was recorded using a video camera during the mixing tests.

Table 1 shows the experimental conditions. The steel tube was positioned along a horizontal direction. In test No.3-1, a steel tube (25 mm in diameter and 0.5 mm in thickness) was used ; a PTFE sealant was used for the pressure sealing gland. TEA was packed in three glass bottles. An explosive of report composition was placed between the glass bottles, as shown in Figure 3. Water and $Al(OH)_3$ scale were packed into a plastic bag, and the glass bottles and the explosive of report composition were wrapped in the plastic bag containing the water and $Al(OH)_3$ scale. In test No.3-2, a sealant made of magnesium silicate was used in place of the PTFE sealant, with all other conditions being the same as those for test No.3-1. In test No.3-3, a steel tube (32 mm in diameter and 5 mm in thickness) with a notching section (0.5 mm thickness) halfway along its length was used. Figure 4 shows a sketch drawing of the steel tube. TEA was packed in two

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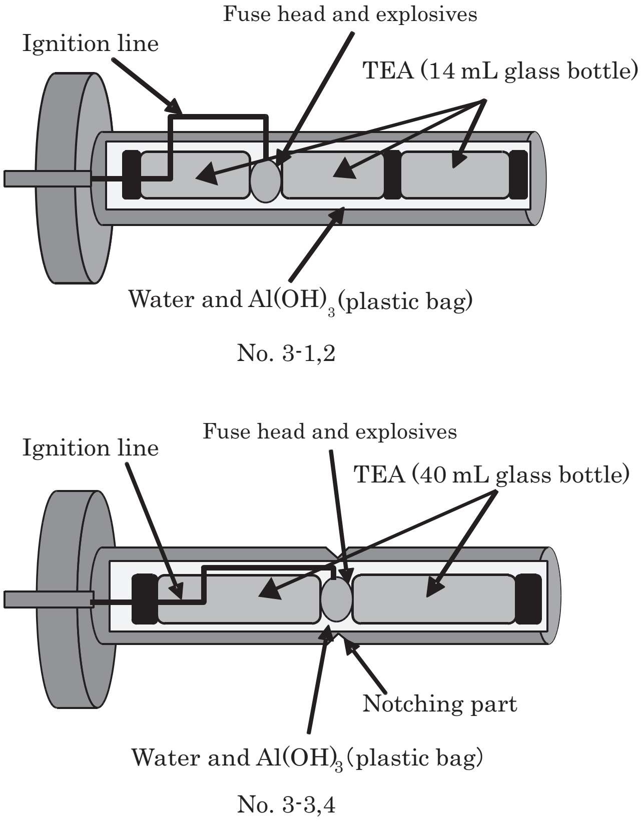


Figure 3 Block diagram of the TEA mixing tests in the steel tube with a blank flange.

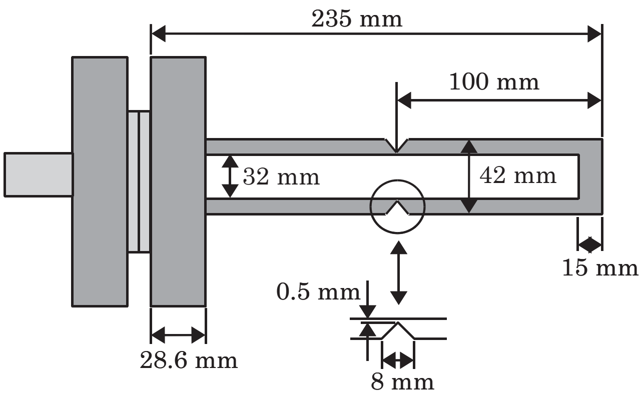


Figure 4 Sketch drawing of the steel tube in test Nos.3-3 and 3-4.

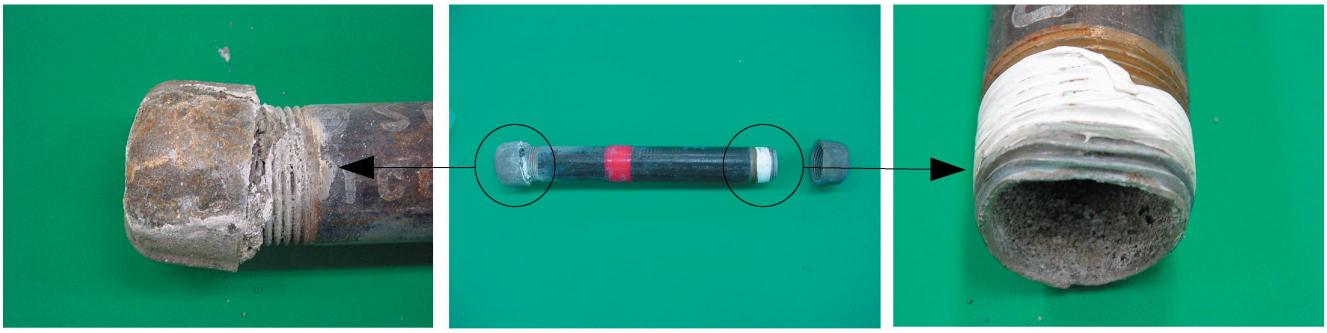
glass bottles and the explosive of report composition was placed between them. Water and $Al(OH)_3$ scale were packed into a plastic bag, and the glass bottles and the explosive of report composition were wrapped in the plastic bag containing the water and $Al(OH)_3$ scale. A sealant made of graphite was used in place of PTFE and magnesium silicate sealants. Moreover, a pressure gage was equipped to the steel tube in order to measure the pressure in the steel tube. The test conditions in test No.3-4 were the same as those in test No.3-3 so that the reproducibility of the mixing tests could be investigated. Furthermore, the progress of the phenomenon was measured using a high-speed camera (MEMRECAM Fx-K 4, NAC Corporation). The ignition signal was used as the starting pre-trigger for the camera. The measuring speed was 5000 frames per second. The camera was fixed at the exterior of an explosion pit in which the steel tube was arranged. A mirror was attached over the steel tube, and the reflected image was recorded from a window in the explosion pit.

3. Results and Discussion

Table 1 shows the experimental results and remarks. The details of the experimental results of each test are described as follows.

In test No.1-1, a small amount of sample blew out of the hole of the screw cap after ignition. The steel tube did not break or change shape. When the interior of the steel tube was observed, it was found that a portion of the TEA was left at the bottom of the steel tube. Before the mixing test, a blank test was performed and it was confirmed that the glass bottles were broken by the impact of the blasting caps, but the cap (made of polyethylene) of the bottle was not broken. The cap of the bottle was also not broken in this test. Thus, it can be inferred that the cap of the bottle may prevent the complete mixing of TEA and water. In test No.1-2, flames blew out of the hole and joint of the screw cap after ignition. The steel tube did not break or change shape. The caps of the bottle were left after the test. It is therefore safe to say that the flames were generated by combustion of TEA. Thus, it was TEA that blew out of the hole and joint of the screw cap, propelled by the increasing pressure in the steel tube. The TEA then burned in air. In test No.1-3, samples blew vigorously out of the hole of the screw cap after ignition. The steel tube did not break or change shape. No TEA was left in the steel tube after the test. The temperature of the surface of the steel tube increased from room temperature to 65 °C after ignition. In the blank test, the temperature of the surface of the steel tube increased to 26 °C. It can thus be concluded that the temperature increase in test No.1-3 was due to the reaction between TEA and water.

In test No.2-1, samples blew vigorously out of the hole of the steel tube after ignition. Moreover, TEA leaked from the joint of the screw cap and ignited spontaneously in the air. The steel tube did not break or change shape. No TEA was left in the steel tube after the test. The temperature of the surface of the steel tube increased to 80 °C at 60 s after ignition. This temperature increase was due to the reaction between TEA and water, because no TEA was left after the test. In test No.2-2, samples blew vigorously out of the hole of the steel tube after ignition. About the same time, a blue jet flame was observed. The steel tube did not break or change shape. A small amount of TEA was left in the steel tube after the test, and ignited spontaneously in the air. The temperature of the surface of the steel tube increased to 124 °C at 56 s after ignition. This temperature increase might have been greater than that observed in test No.2-1, because the $Al(OH)_3$ might have raised the temperature of the source of water and contributed to the mixed reaction between TEA and water[4]. In test No.2-3, the steel tube blew out after ignition and the samples caught fire. Figure 5 shows the view of the steel tube after the test. A screw cap blew off from the steel tube. The screw cap and a part of the screw cutting

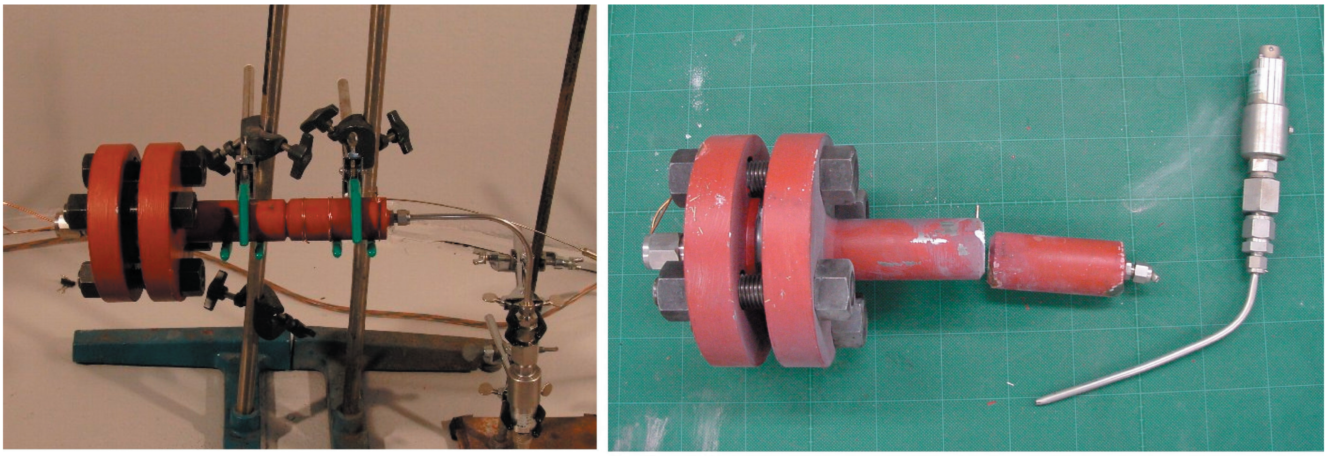


Partial view
(a part of screw cap)

Whole view of the steel tube

Partial view
(a part of screw cutting)

Figure 5 View of the steel tube after test No.2-3.



Steel tube before the test

Steel tube after the test

Figure 6 View of the steel tube before and after test No.3-3.

of the steel tube changed shape. The other screw cap remained attached to the steel tube, although it came off the screw thread. The temperature of the surface of the steel tube increased to 82 °C at 1s after ignition. This phenomenon was obviously faster and more violent than those of test Nos.2-1 and 2-2. It seems probable that the sample mixing was improved by the arrangement of samples.

In test No.3-1, gases blew out of a part of the pressure sealing gland after ignition. It was observed that the PTFE sealant melted after this test. This fact suggests a high temperature in the steel tube due to the reaction between TEA, water, and $Al(OH)_3$ scale. In test No.3-2, the pressure sealing gland did not break. The color of the steel tube changed but the steel tube did not break or change shape. When the interior of the steel tube was observed, it was found that some TEA was left at the bottom of the steel tube, which ignited spontaneously after contact with air. Thus, TEA, water, and $Al(OH)_3$ scale seem not to have been mixed completely in this test. In test No.3-3, the steel tube broke at the notch. Figure 6 shows the view of the steel tube before and after the test. When the interior of the steel tube was observed, it was found that no samples were left inside. Moreover, the explosion seems not to have been a detonation because the steel tube did not break into pieces. The pressure in the steel tube could not be measured because the stainless tube for pressure measurements also broke.

In test No.3-4, the steel tube broke at the notching at 5.8 ms after ignition. This demonstrated that the experimental results under the testing conditions were reproducible, because the result of test No.3-4 was equal to that of test No.3-3. Figure 7 shows the snapshots from the high-speed camera between 5.6 ms and 6.6 ms after triggering. After the steel tube split at the notch, smoke blew out of the split, and the smoke spread over the whole view by 6.6 ms after triggering. As shown in Figure 6, the speed of reaction was very fast: only one shot was unobscured even when the reaction was recorded at 5000 shots per second.

It was proved that the steel tube was broken by the mixing reaction between TEA, water, and $Al(OH)_3$ scale under the testing conditions of test Nos.3-3 and 3-4. The underlying reason for this result was investigated by comparing the pressure arising from the generated gases with the resisting pressure of the steel tube estimated using the evaluation equation for breaking strength by an internal pressure of a hollow cylinder and a three-dimensional finite element analysis for the elastic deformation.

In our previous study, the TEA and water system mixture in closed conditions is known to decompose into lower molecular weight compounds than those produced by well-known mechanisms of hydrolysis of TEA. Using

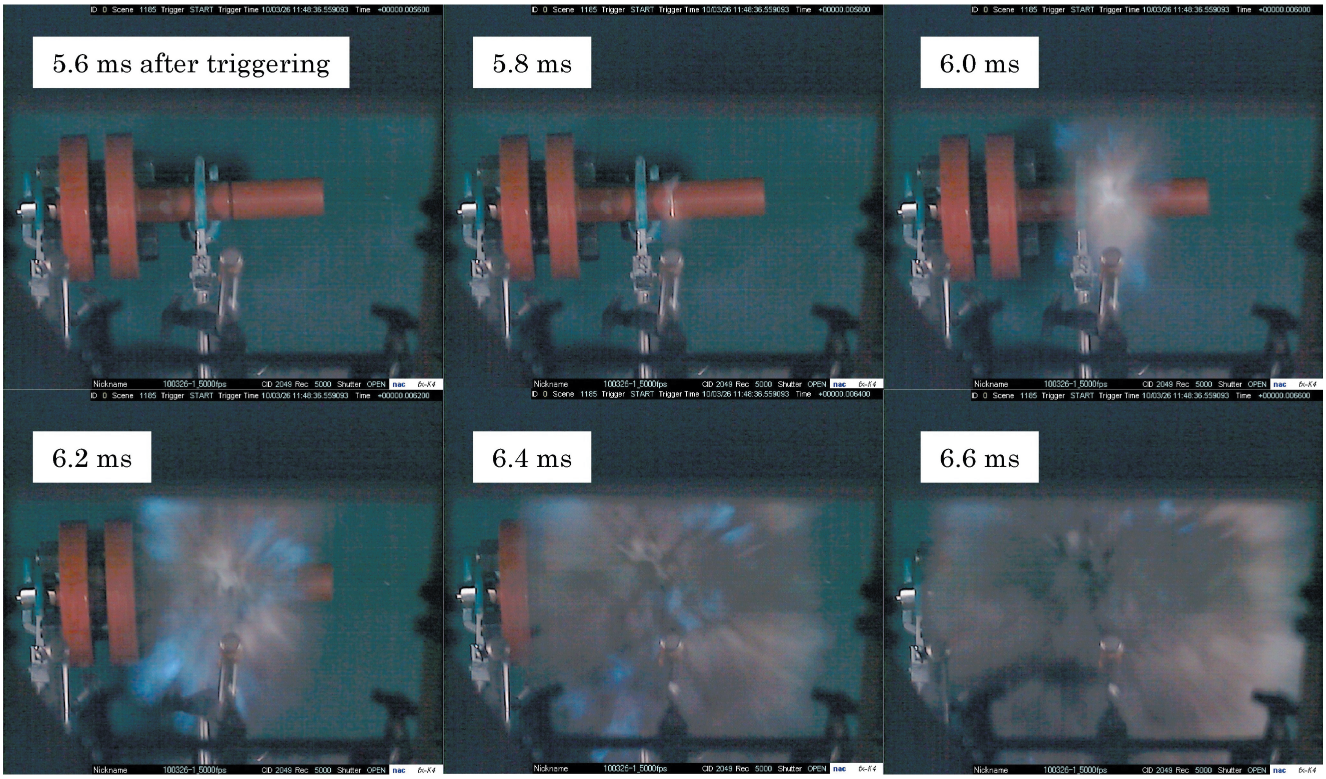


Figure 7 Snapshots from 5.6 ms to 6.6 ms after triggering using a high-speed camera for test No.3-4.

equilibrium calculations, the reaction between TEA and water has been estimated as follows[4] :

$$(C_2H_5)_3Al+H_2O \rightarrow 1.6CH_4+5.1H_2+0.3Al_2O_3+0.1Al_4C_3+4.0C$$

On the basis of the above reaction formula, it was assumed that 6.7 mol of non-condensable gases were generated from 1 mol of TEA, and this ratio was used to estimate volumes and pressures at room temperature (using the ideal gas law) for each test.

The resisting pressure of the steel tube was estimated as follows. For the tubular-type steel tube in test Nos. 1, 2, 3-1, and 3-2, the stress in the radial direction was calculated using the evaluation equation for breaking strength by an internal pressure of a thick hollow cylinder shell[10]. For the notched steel tube in test Nos. 3-3 and 3-4, the stress in the radial direction was analyzed using a three-dimensional finite element method based on the theory of elasticity. The three-dimensional finite element analysis software (Sansei-kai, Japan) was used for this analysis[11]. The resisting pressure of the steel tube was assumed to be the analyzed pressure if the stress in the radial direction exceeded the tensile stress of a JIS SGP steel tube (290 MPa)[12] and that of a JIS STPG370 steel tube (370 MPa)[13].

Table 2 shows the estimated pressures in the steel tube and the corresponding estimated resisting pressures. The resisting pressures of the steel tube in test Nos. 1 and 2 were estimated to be 60 MPa. On the other hand, the pressure in the steel tube at test No.1 was estimated to be 21-42 MPa, while that for test No.2 was estimated to be 80 MPa. The packing density of test No.2 (7.7-8.0 × 10^{-1}g-sample cm^{-3}) was larger than that of test No.1 (2.1-4.3 × 10^{-1}g-sample cm^{-3}) because the amount of TEA in test No.2 was larger than that in test No.1. Thus, the pressure in the steel tube in test No.2 was larger than that in test No.1. These theoretical results support the experimental result : the steel tube blew out only (the screw cap being blown off) in test No.2-3. However, the steel tube did not break or change shape. This fact demonstrates that the resisting pressure of the screw cap part of the steel tube is lower than that of the body part of the steel tube. Thus, the experimental results depend on the resisting pressure of the screw cap of the steel tube under this experimental condition. However, the reproducibility of the testing seems not to be satisfactory because of the difficulty in sufficiently estimating and defining the resisting pressure of a screw cap part compared to the body part.

Table 2 The estimated pressures in the steel tube and the estimated resisting pressure of it.

No.	Pressures in the steel tube [MPa]	Resisting pressure of the steel tube [MPa]
1-1	42	60
1-2	21	60
1-3	21	60
2-1	80	60
2-2	80	60
2-3	80	60
3-1	50	15
3-2	50	15
3-3	51	15
3-4	51	15

The resisting pressures of the steel tube in test Nos. 3-1 and 3-2 were similar to those in test Nos. 3-3 and 3-4. The pressures in the steel tube in test Nos. 3-1 and 3-2 were also similar to those in test Nos. 3-3 and 3-4. The differences in the test conditions were owing to the differences in the arrangement of samples, in turn because of the differences in the volumes in the steel tubes. The fact that some TEA has been left after test No.3-2 shows that reducing obstacles in a steel tube through designing the arrangement of samples appropriately is important in mixing the samples completely.

Consequently, this investigation determined that the hazards relating to explosions from mixing pyrophoric substances can be evaluated by the testing method employed in test Nos. 3-3 and 3-4. A foreseeable extension of this study would be to examine the testing method for its validity and to improve it by the accumulation of investigation results for other pyrophoric substances.

4. Conclusions

In this study, as part of the evaluation of the explosion hazards of aluminum alkyls, the conditions in which a steel tube burst were investigated experimentally by mixing TEA, water, and $Al(OH)_3$ in a steel tube. The purpose of this investigation was to develop a new testing method for estimating the hazards relating to explosions from mixing pyrophoric substances. As a result, it was proved that a steel tube broke owing to the mixing reaction between TEA, water, and $Al(OH)_3$ scale. The results from that particular testing method were shown to be reproducible. By comparing the estimated pressure from the gases generated from the reaction of the samples with the estimated resisting pressure of the steel tube in each test, it was also found that increasing the amount of samples per unit volume of a steel tube and reducing the obstacles in it through designing the arrangement of samples appropriately is important for this testing method. A foreseeable extension of this study would be to examine the testing method for its validity and to improve it by the accumulation of investigation results for other pyrophoric substances.

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自然発火性物質の混合危険性評価手法の開発

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自然発火性物質の混合危険性の新たな評価方法を開発するために，トリエチルアルミニウム，水および水酸化アルミニウムを鋼管内で混合し，鋼管が破壊する条件を実験的に検討した。その結果，トリエチルアルミニウム，水および水酸化アルミニウムの混合反応により，鋼管が破壊されることを実証した。反応により発生するガスによる鋼管内の圧力と鋼管の耐圧をそれぞれ推算し，比較した結果，鋼管内での自然発火性物質の混合危険性の評価方法においては，鋼管内の単位容積における試料量を多くすることと，鋼管内の障害物により試料の混合が阻害されないように試料を配置することが必要であることがわかった。

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