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PERFORMANCE OF PRESSURE VESSEL TEST CONCERNED WITH HEATING RATE OF PRESSURE VESSEL AND BURSTING PRESSURE OF RUPTURE DISK

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Summary

Heating rate characteristics of samples were examined for Dutch and US pressure vessels and sample cups with round and flat bottoms. The heating rate is fairly constant between 100 and 200°C and can be controlled by adjusting the voltage of the electric furnace. The heating rate is greater in the US pressure vessel than in the Dutch one and is also greater for the flat bottom cup than for the round one.

The 'bursting pressure' of rupture disks were measured by the N_2 pressure method and the ADCA (azodiacarbonamide) decomposition method which is more practical. Overall the bursting pressure increased with the pressure rising rate, though the data were scattered. This dispersion seems to be characteristic of the rupture disk itself.

The maximum pressure by ADCA decomposition was examined by means of pressure sensors, strain gauges and a Bourdon gauge in order to minimize the dispersion in the bursting pressure values measured of the rupture disk.

The results can best be summarized as follows: The maximum pressure attainable increases with sample mass; the dispersion in the maximum pressure data is rather large. It is concluded that this dispersion is due to the fluctuation in the pressure pattern of the thermal decomposition in the pressure vessel. The strain gauge is more suitable than the Bourdon gauge as pressure sensor, though the latter may be used if a correction is made for the effect of the needle for indicating the maximum pressure. The pressure at the top of a pressure vessel is slightly higher than that measured at the side walls by about 0.3 kg/cm^2, so the effect of dynamic pressure is small.

The round bottom cup gives smaller maximum pressure, albeit with larger data dispersion, than the flat one.

1. Introduction

The pressure vessel test (PVT) was developed at TNO (Applied Scientific Research) in the Netherlands and introduced by Siemens [1] and Noller et al.

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[2] as a tool for evaluating the violence of thermal decomposition of organic peroxides. Then, the pressure vessel tests have been standardized and used by the Society of the Plastics Industry OPPSD [3] and the European Economic Commission ADR [4] in the USA and Europe, respectively. Recently, the Organisation of Economic Cooperation and Development (OECD-IGUS) and the United Nations Committee of Experts on Transport of Dangerous Goods have jointly reexamined the PVT data [5,6] and PVT methods have been established by the UN Recommendation on the Transport of Dangerous Goods [7].

On the other hand, in Japan, a part of the Fire Protection Law (FPL) has been amended [8] and the hazardous materials which should be regulated by the law were decided to be classified by suitable tests. The class 5 hazardous materials, which are also called self-reactive materials, were agreed to be classified by a thermal analysis and a PVT for the ability to propagate explosions and the violence of thermal decomposition, respectively [9,10].

So far, the results of PVT have been evaluated by using PVLD (Pressure Vessel Limiting Diameter) [3,4,11]. However, this procedure requires many trials and the test results are not necessarily reliable. The self-reactive materials by the FPL are divided into 3 categories based on the PVT. For this classification, it is not necessary to use the traditional PVT procedure. In the new FPL, only two orifices of 1 mm and 9 mm diameter are required and 10 trials are carried out for each orifice. A self-reactive material which bursts the rupture disk with 1 mm or 9 mm orifice more than 5 times is classified hazardous or highly hazardous material, respectively. This procedure looks more reasonable than the conventional one in that the total number of trials is less as only two kinds of orifices are used, and data reliability is higher since 10 trials are conducted against only 3 trials of PVLD per orifice.

But, at the moment, there are very few applications of this new procedure, and the effects of heating rate, cup geometry, rupture disk, orifice diameter, type of pressure vessel and so on have not been known that well. We earlier examined the properties of conventional PVT [12], but not enough for using the new PVT procedure properly. Here, we describe the results of the examinations on the heating rate characteristic of PVT and bursting pressure of rupture disk.

2. Experimental

2.1 Materials

Silicone oil for determining heating rate was WF-30, which was used in an open system oil bath supplied by Wako Pure Chemicals Co., Ltd. and the azodicarbonamide (ADCA) used was a commercial product from Otsuka Chemical Co., Ltd.

2.2 Apparatus

The electric furnace used was a single furnace which was shown in a previous paper [12]. The resistance of the nichrome heating wire was 8.4 Ω. The Dutch and US pressure vessels were used and are shown in Figs. 1 and 2, respectively. A Bourdon pressure gauge with a needle indication for the maximum pressure was attached to the side of the Dutch pressure vessel and a strain gauge pressure sensor was also used instead of the Bourdon gauge, if necessary. A rupture disk for the Dutch pressure vessel is made of aluminium (JIS A-1050-P-H24), 59 mm in diameter and 0.1 mm in thickness. Another one is made of brass

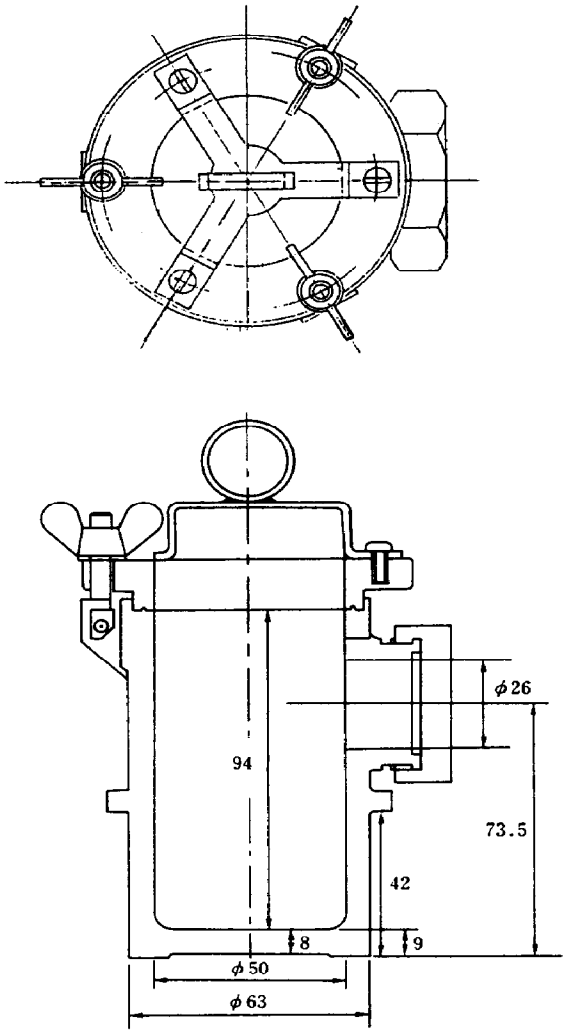


Fig. 1. Schematic of Dutch pressure vessel.

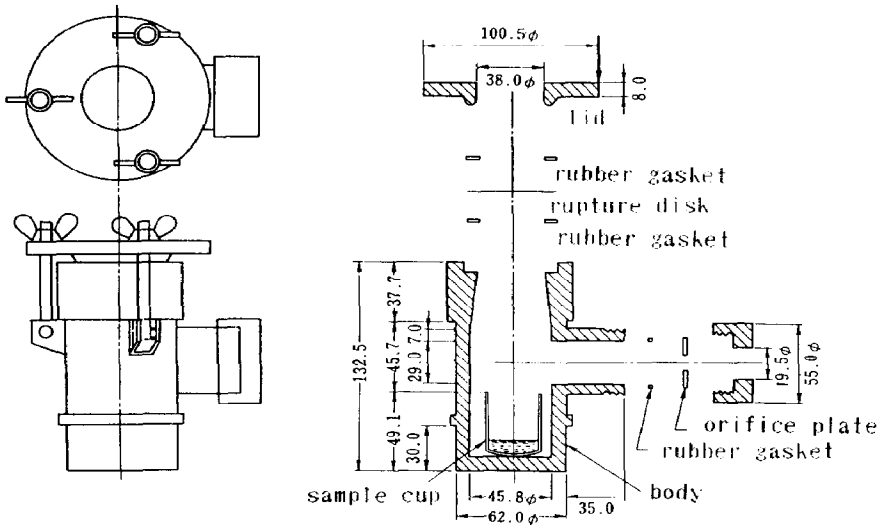


Fig. 2. Schematic of US pressure vessel.

(JIS C-2801-P), 59 mm in diameter and 0.05 mm in thickness. The rupture disk for the US pressure vessel is made of aluminium (JIS A-1050-P-H24), 53 mm in diameter and 0.1 mm in thickness. The M-8 aluminium cup [12] with round bottom made by Maru-emu Co., Ltd. and AC-5 aluminium cup with flat bottom by Iuchi Seieido Co., Ltd. were used as sample container in the Dutch and US pressure vessels in order to make for easy cleaning inside of the vessels, though the standard Dutch pressure vessel test did not contain the sample cup. The sizes of the former and the latter are 33 mm $^{\phi}\times$41 mm$^{H}\times$1 mm^{t} (30 cm^3) and 32 mm $^{\phi}\times$39.5 mm$^{H}\times$0.5 mm^{t} (29 cm^3), respectively. Heating was supplied through above-mentioned electric furnace, though the standard Dutch pressure vessel test heating was supplied through a gas flame. Temperatures were measured by Alumel–Chromel thermocouples with stainless protection sheath. Voltage and electric current supplied to the heater for the pressure vessel were controlled by a transformer with volt and current meters. The strain gauge pressure sensor used was a TP-BP 20K made by TEAC Co., Ltd. The output of the strain gauge was amplified by a DC amplifier (TEAC, Model SA-58), recorded on a data recorder (TEAC, Model MR-10), introduced into a personal computer (NEC, Model PC-9801VX21) through an AD converter (AUTNICS Co., Ltd., Model S210), and then analyzed.

3. Experimental procedures

3.1 Measurement of heating rate characteristics

The following procedure was followed to measure the heating rates

(1) 84 volt was applied to the electric furnace for more than 1 hour.
(2) 5 g silicone oil was poured into a sample cup and the cup was placed in a pressure vessel with an orifice, 1 mm in diameter and 3 mm in thickness.
(3) A rupture disk with two holes of 2 mm diameter was subsequently attached.
(4) Two thermocouples were placed in the silicone oil, one inside of the cup and the other at the bottom of the pressure vessel, outside of the cup, respectively.
(5) The vessel was then put on the furnace.
(6) The temperatures were simultaneously recorded on the strip flow chart.
(7) Pressure vessel, applied voltage and sample cup were changed and the experiments repeated.

3.2 Determination of bursting pressure of rupture disks

3.2.1 N_2 pressure method

(A1) The pressure vessel was connected to a N_2 cylinder at the vessel orifice nozzle.
(A2) A strain gauge was attached at the opposite side of the vessel.
(A3) A rupture disk was put in place and tightened firmly with the flange bolts.
(A4) As soon as the data recorder was switched on, N_2 pressure was applied to the vessel.
(A5) The bursting pressure of the disk and the pressure rising rate were determined from the pressure recording.
(A6) Pressure vessel, rupture disk and pressure rising rate were changed and the experiments repeated.

3.2.2 ADCA decomposition method

(B1) 7 g ADCA was weighed into a sample cup and placed in the center on the bottom of a pressure vessel with a 1-mm diameter orifice.
(B2) An aluminium rupture disk was put in place and tightened. A water layer was then put over the rupture disk to keep its temperature low during the test.
(B3) The vessel was placed on the heater, which was kept before for more than 1 hour in the condition of being able to supply heat at a rate of 40°C/min between 100 and 200°C to the pressure vessel, and at the same time, a stop watch was started.
(B4) The data recorder was switched on about 30 s before the start of decomposition of sample was expected and the pressure rise was recorded.
(B5) The time when the sample began decomposing and fume left the orifice was recorded.
(B6) The bursting pressure value was determined from the pressure recording.

3.3 Determination of maximum decomposition pressure

(1) ADCA was weighed into a sample cup and placed in a pressure vessel with 1-mm diameter orifice.
(2) Instead of a rupture disk, a lid-mounted strain gauge was put in place and tightened firmly with the flange bolts.
(3) A Bourdon gauge was attached to the side of the vessel and the maximum pressure indicating needle of the Bourdon gauge was set to 0 kg/cm^2 G.
(4) Same procedure as (B3)
(5) Same procedure as (B4)
(6) Same procedure as (B5)
(7) After the decomposition fume completely exhausted, the maximum pressure by the Bourdon and strain gauges were determined.

3.4 Examination of position of pressure sensors

(1) Two strain gauges were attached to the top and side of the same pressure vessel.
(2) The pressure of the decomposition fume was measured as above by using two strain gauges.

4. Results and discussion

4.1 Heating rate characteristics

Heating rate characteristics inside and outside of sample cups were examined using the Dutch and US pressure vessels and sample cups with round and flat bottoms. Typical heating rate curves are shown in Figs. 3 and 4. The relationships of the average heating rates between 100 ~ 200 °C and 200 ~ 300 °C and voltage on the electric heater are listed in Tables 1 and 2 and shown in Figs. 5 and 6. The following observations can be made:

(1) The heating rate is faster outside than inside the cup, showing that heat is conducted from the outside to the inside of the cup.
(2) The heating rate is greater for the flat bottom cup than for the round bottom one. This may be attributable to the better conductivity of the flat bottom of the former.
(3) The heating rate is greater in the US pressure vessel than in the Dutch one, even though the former has more mass.

4.2 Bursting pressure of rupture disks

4.2.1 N_2 pressure method

The bursting pressures were measured by the N_2 pressure method for both

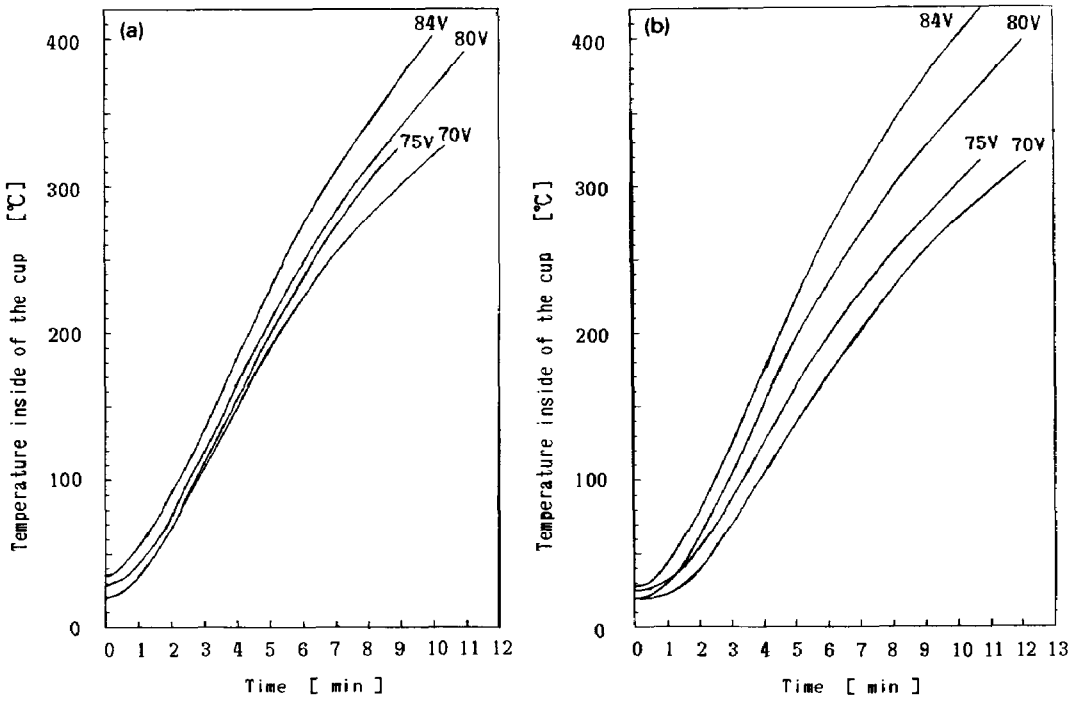
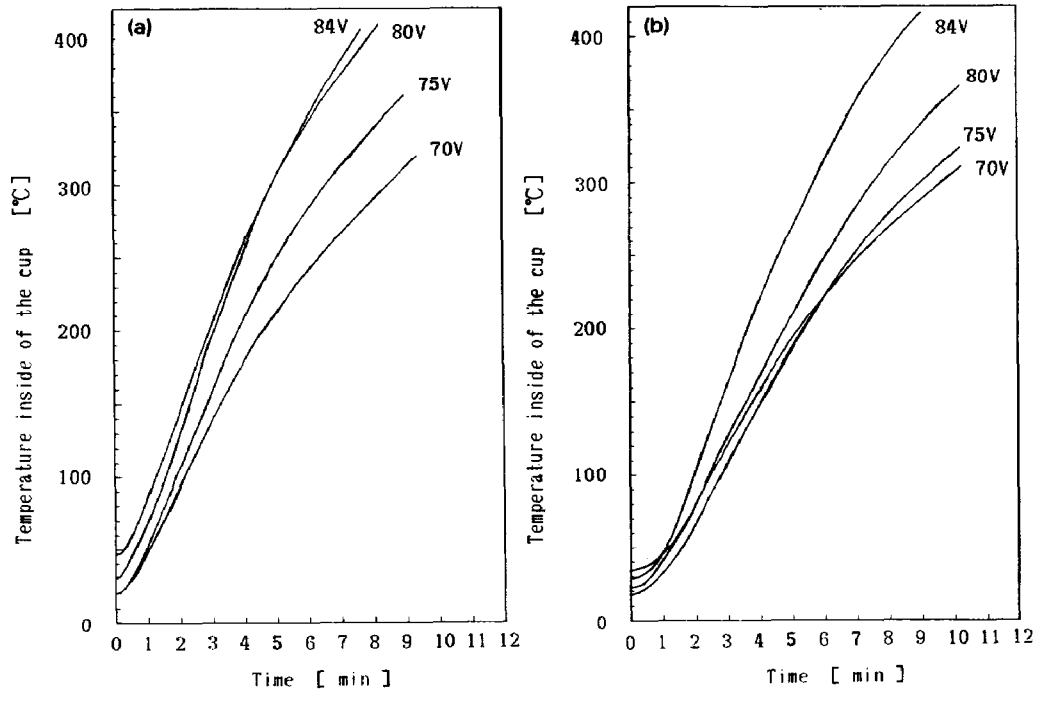


Fig. 3. Heating rate curves of Dutch pressure vessel: (a) flat bottom cup, and (b) round bottom cup. Voltage was varied to 70, 75, 80 and 84 V, respectively.



(a) flat bottom cup (b) round bottom cup

Fig. 4. Heating rate curves of US pressure vessel: (a) flat bottom cup, and (b) round bottom cup. Voltage was varied to 70, 75, 80 and 84 V, respectively.

TABLE 1

Heating rate characteristic using Dutch pressure vessel

Voltage (V)	Current (A)	Cup type	Position of thermocouple (inside or outside of cup)	Heating rate °C/min	
				100–200°C	200–300°C
84	10.1	round	inside	49.8	44.4
			outside	60.6	48.8
		flat	inside	46.6	43.9
			outside	55.6	48.8
80	9.8	round	inside	42.6	31.7
			outside	42.6	38.5
		flat	inside	44.2	39.2
			outside	58.8	46.5
75	9.1	round	inside	35.1	27.4
			outside	37.0	32.0
		flat	inside	43.5	35.7
			outside	57.6	43.0
70	8.5	round	inside	32.8	23.8
			outside	45.5	31.6
		flat	inside	39.9	28.2
			outside	55.5	33.6
60	7.4	round	inside	–	–
			outside	–	–
		flat	inside	19.5	9.2
			outside	25.5	14.2

US and Dutch pressure vessels. An example of pressure rate patterns, which was observed at the side wall of the Dutch pressure vessel, is shown in Fig. 7.

The bursting pressures of rupture disks of the Dutch and US pressure vessels are listed in Tables 3 and 4, respectively. The bursting pressure data were rather scattered. Comparing the aluminium rupture disks, Al(I) and Al(II) in Table 3, we assume that the average bursting pressure is higher for the former.

In order to confirm this assumption, the relationship between bursting pressure and N_2 pressure rate was examined. The results are listed in Fig. 8. Overall, the bursting pressure seems to increase with pressure rate, however, the data are scattered. This dispersion seems to be characteristic of the rupture disk itself.

TABLE 2

Heating rate characteristic using US pressure vessel

Voltage (V)	Current (A)	Cup type	Position of thermocouple (inside or outside of cup)	Heating rate °C/min	
				100–200°C	200–300°C
84	10.1	round	inside	60.6	50.0
			outside	102.6	82.3
		flat	inside	66.7	54.0
			outside	78.4	66.7
80	9.8	round	inside	45.2	35.7
			outside	100.0	69.0
		flat	inside	60.2	50.9
			outside	105.3	71.4
75	9.1	round	inside	39.2	28.2
			outside	71.9	71.4
		flat	inside	51.0	39.4
			outside	73.3	46.5
70	8.5	round	inside	36.4	11.5
			outside	39.4	16.9
		flat	inside	40.4	26.2
			outside	58.0	33.9
60	7.4	round	inside	18.8	6.9
			outside	25.0	9.4
		flat	inside	19.4	5.3
			outside	22.9	10.0

4.2.2 ADCA decomposition method

The practical bursting pressure of rupture disks were determined from the decomposition of 7 g ADCA. The results are listed in Table 3. The bursting pressures were measured both when the rupture disk was covered and not covered by a layer of water. The bursting pressure of the former was higher. ADCA begins to thermally decompose at 210°C in the sealed cell-differential scanning calorimeter (SC-DSC). Above result suggests that the aluminium rupture disk looses its strength at this temperature changing its bursting pressure. The bursting pressure using ADCA and water was similar to that by the N_2 pressure method under high pressure rate conditions. In determining the bursting pres-

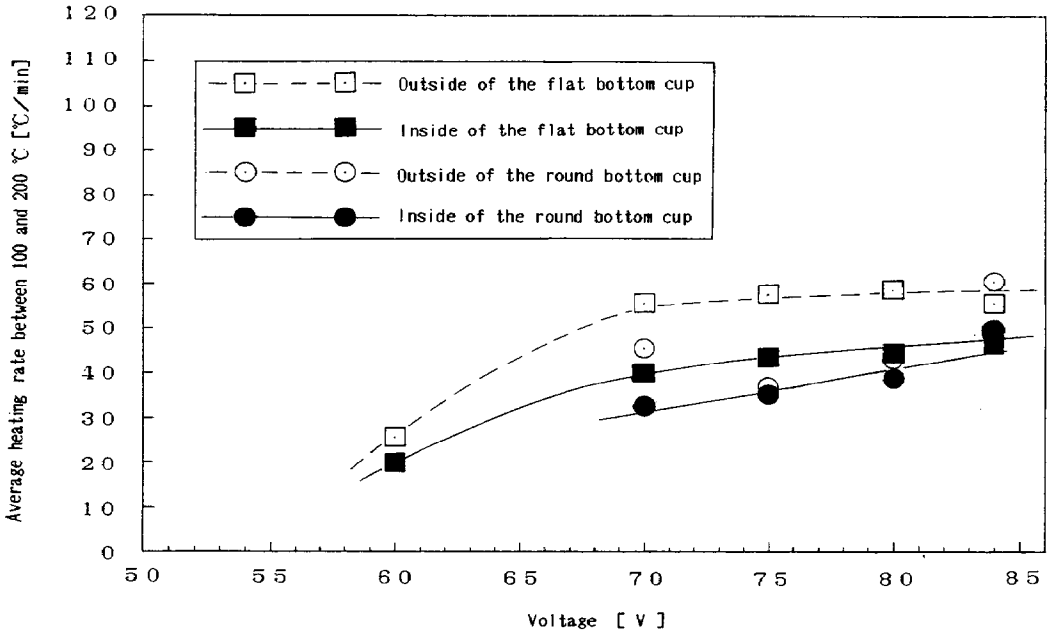


Fig. 5. Heating rate characteristic of Dutch pressure vessel.

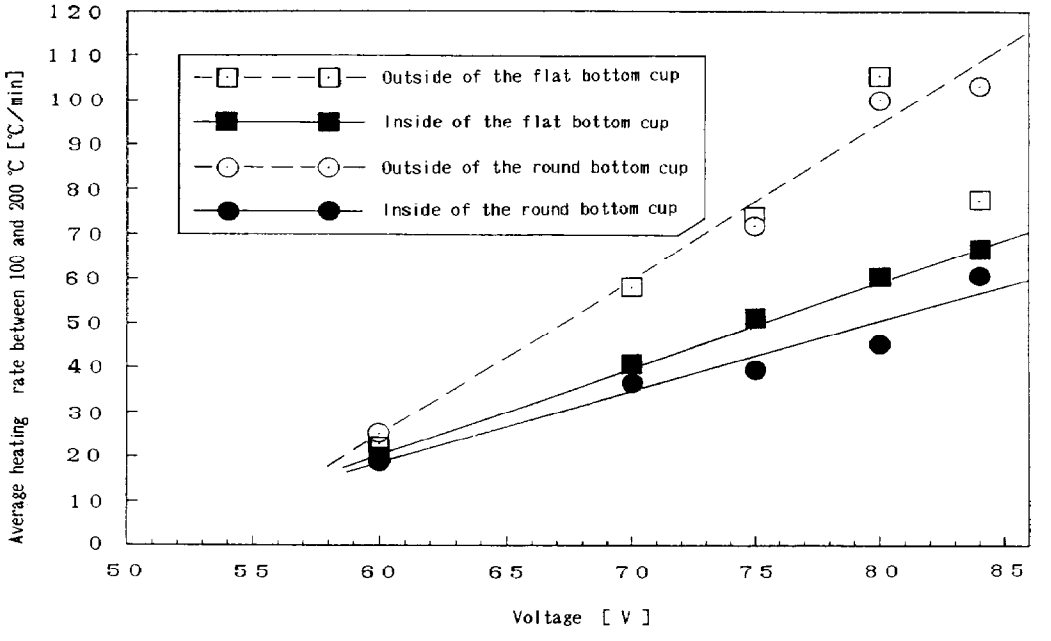


Fig. 6. Heating rate characteristic of US pressure vessel.

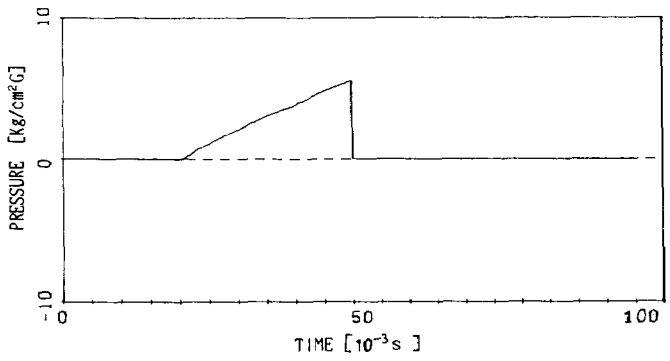


Fig. 7. Example of pressure rate pattern by N_2 method (Dutch pressure vessel, 0.1 mm^t Al rupture disk, sampling time 100 ms, P_{max} 5.6 kg/cm^2, $(dP/dt)_{max}=0.17$ kg/cm^2/s).

TABLE 3

Bursting pressure of rupture disks in the Dutch pressure vessel

Materials		N_2			ADCA 7 g (flat bottom cup)	ADCA 7 g (flat bottom cup)
type of rupture disks		Al (I)	Al (II)	brass	Al (not covered by water)	Al (covered by water)
Bursting pressure (kg/cm^2 G)	1	7.18	4.79	3.26	4.17	6.15
	2	7.11	5.59	6.96	3.36	6.77
	3	5.82	5.94	4.88	4.38	5.51
	4	5.66	5.54	5.63	2.51	6.83
	5	4.94	5.94	4.51	4.61	5.40
	average	6.14	5.56	5.05	3.81	6.13
	dispersion	0.87	0.42	1.23	0.77	0.60
N_2 pressure rate kg/cm^2/s		3~6	0.1-0.3	0.2-0.3	–	–

sure by the N_2 pressure method, it is recommended to use a high pressure rate in order to yield similar results as with the real pressure vessel test. The dispersion of the bursting pressure was also encountered in the ADCA decomposition method.

4.3 Determination of maximum pressure

As the dispersion of bursting pressure of the rupture disk was observed, a method for determining the maximum pressure was examined by using two pressure sensors, a strain gauge and a Bourdon gauge equipped with a needle for indicating the maximum pressure.

TABLE 4

Bursting pressure of rupture disks in the US pressure vessel

Material		N_2
Type of rupture disk		Al
Bursting pressure (kg/cm^2 G)	1	6.21
	2	5.14
	3	5.63
	4	3.96
	5	7.16
	6	7.05
	7	6.98
	8	4.60
	9	5.70
	10	6.63
	average	5.90
	dispersion	1.04
N_2 pressure rate $kg/cm^2/s$		0.1–0.3

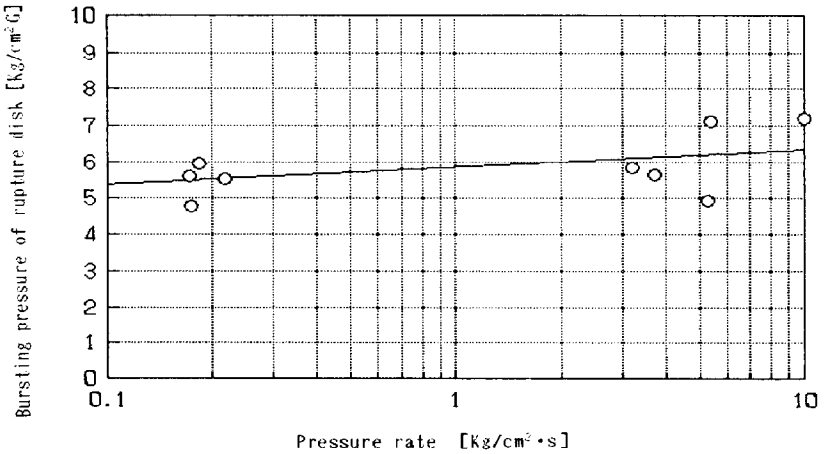


Fig. 8. Relationship between bursting pressure and N_2 pressure rate (Dutch vessel, 0.1 mm^t Al rupture disk).

4.3.1 Calibration of the Bourdon gauge

The indication of the Bourdon gauge by static N_2 pressure equalled that of the strain gauge.

4.3.2 Relationship between sample mass and maximum pressure

The maximum pressure of decomposition products of ADCA in the pressure vessel was determined with various initial sample masses and the results are

listed and shown in Table 5 and Fig. 9, respectively. These results show that: (1) the maximum pressure increases with sample mass, (2) the dispersion in the maximum pressure data is large, (3) the maximum pressure as measured by the Bourdon gauge is smaller than that by the strain gauge, and (4) the difference in the maximum pressure near the position of attachment is not clear due to large dispersion in the observed pressure data.

TABLE 5

The maximum pressure of ADCA decomposition products*

ADCA	Case 1**			Case 2**	
	Maximum pressure (kg/cm^2 G)		Time to decomposition fume (min. s)	Maximum pressure	Time to decomposition fume (min. s)
	Top of PV (strain gauge)	Side of PV (Bourdon gauge)		Side of PV (strain gauge)	
3 g	1.92	1.0	4.36	1.37	4.25
	3.19	2.2	4.42	1.46	4.17
	2.08	1.1	4.32		
4 g	3.04	2.2	6.11	3.22	4.42
	3.11	–	4.10	3.35	4.35
	4.93	–	3.40		
	4.30	2.7	4.00		
	2.08	–			
5 g	2.76	2.0	4.58	7.20	4.30
	2.80	1.8	4.54	6.01	4.35
	3.63	2.7	5.02	2.60	4.45
	6.51	5.0	4.43	5.43	4.39
	5.27	3.75	4.38	5.93	4.45
	6.10	4.5	5.05		
6 g	6.42	4.7	6.34	6.31	4.42
	5.91	4.9	4.15	9.02	4.42
	7.49	6.9	5.05	6.35	4.45
7 g	10.16	6.5	4.52	7.71	4.25
	5.30	4.25	4.45	6.87	4.34
	7.88	6.2	4.45	11.19	4.46
				7.71	4.42

*Dutch pressure vessel, flat bottom cup, voltage of electric furnace 70 V, 8.4 A, orifice $1^{\phi} \times 3^{t}$.

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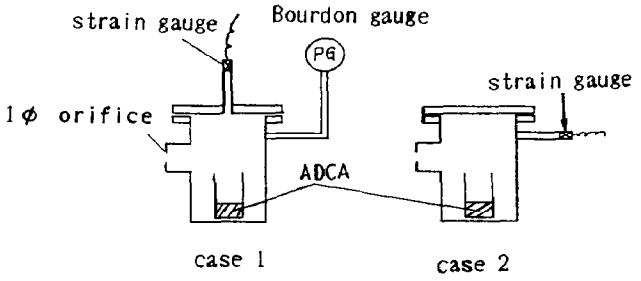

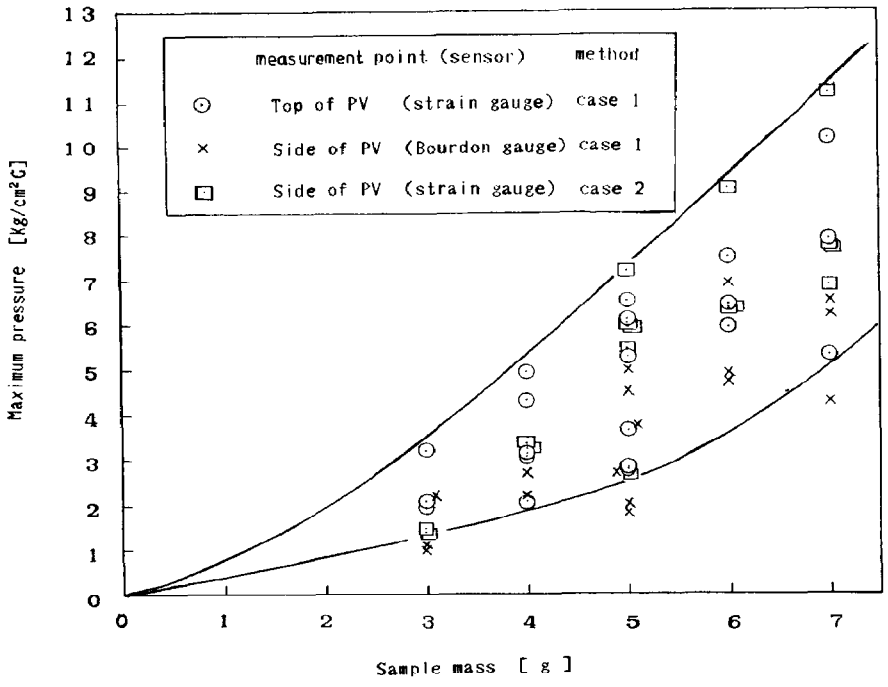

Fig. 9. Relationship between ADCA mass and maximum pressure.

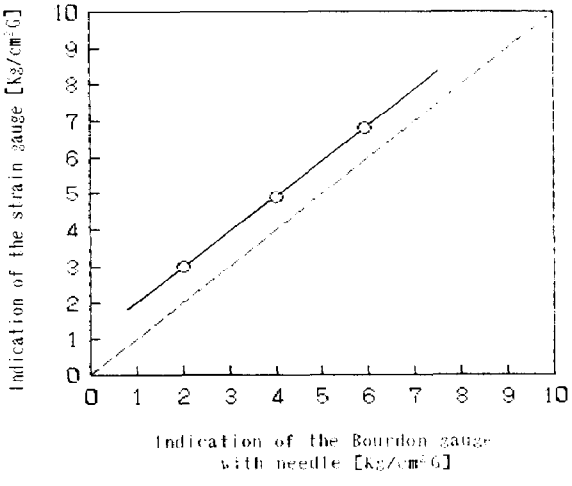

Fig. 10. Maximum pressure measurement: strain gauge vs. Bourdon gauge. Needle effect.

4.3.3 Maximum pressure by the Bourdon gauge

Reasons for the lower maximum pressure indication of the Bourdon gauge may be attributable to: (1) the effect of the needle (coulomb friction), (2) the effect of difference in dynamic pressure at sensor positions, and (3) response delay to pressure rate due to rapid decomposition.

The effect of the needle for indicating the maximum pressure was examined by using static N_2 pressure. The results are shown in Fig. 10. The figure shows that the needle indication of the Bourdon gauge is lower by 0.9 to 1.0 kg/cm^2. This effect was corrected for and the corrected Bourdon gauge pressure is plotted against the strain gauge pressure in Fig. 11. When the maximum pressure increases, the difference between these two pressures becomes larger. This may be attributable to reasons (2) and (3) above.

4.3.4 Dispersion of the maximum pressure

The reason for the dispersion in the maximum pressure readings in Fig. 9 may be due to the fluctuation in the pressure pattern of the decomposition

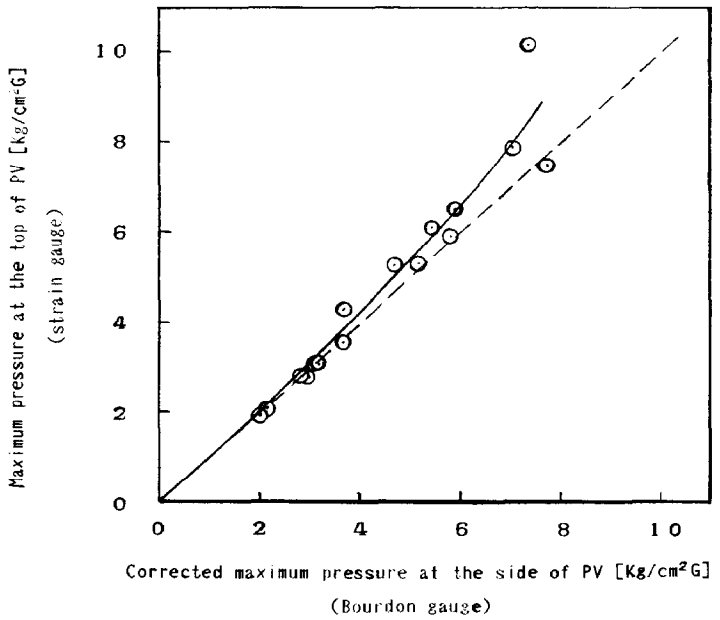

Fig. 11. Relationship between maximum pressure of strain gauge measured at the top of the pressure vessel and the corrected maximum pressure measured by the Bourdon gauge at the vessel side. (Dutch vessel, flat bottom cup, ADCA mass 3-7 g, voltage of electric furnace 70 V, 1 mm diameter orifice.)

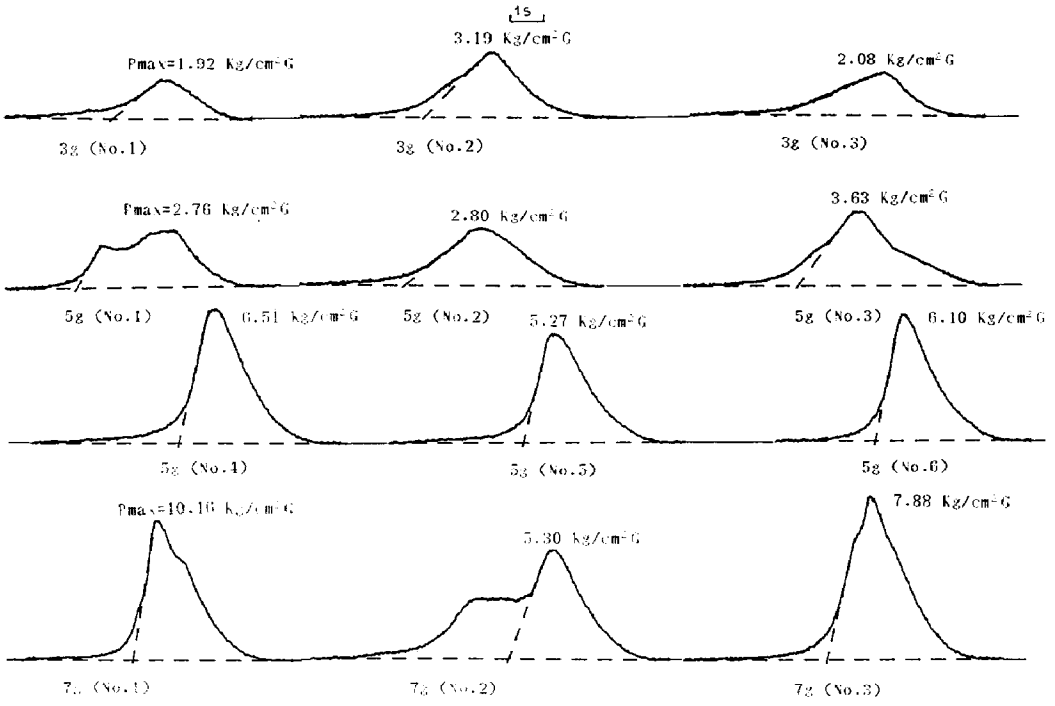

Fig. 12. Examples of pressure pattern of the decomposition reaction of ADCA. (Dutch vessel, flat bottom cup, ADCA mass 3–7 g, 70 V, 1 mm orifice).

reaction of ADCA. Examples of pressure patterns are shown in Fig. 12. The sample compound gives a different pressure pattern in the thermal decomposition in the pressure vessel.

4.3.5 Differences in pressure at different sensor positions

The two strain gauges were attached to both top and side of the pressure vessel and the pressures were observed simultaneously. The results are listed in Table 6. Clearly the two pressures are different, the pressure at the top being higher by about 0.3 kg/cm^2 on average. Therefore, the actual bursting pressure of a rupture disk is different from the pressure observed at side of the vessel. However, this pressure difference is smaller than the data scatter in bursting pressure measurement of the rupture disk.

The pressure readings at the top have been plotted against those taken at the side in Fig. 13. It is shown that the higher the maximum pressure is, the larger the difference between both pressures is, i.e. the more violent the decom-

TABLE 6

Difference of maximum pressure between the top and the side of pressure vessel (PV)*

Position of sensor (kind of sensor)	Type of cup					
	Flat bottom cup			Round bottom cup		
	Top of PV (strain gauge, kg/cm^2 G)	Side of PV (strain gauge, kg/cm^2 G)	Time to decomposition fume (min. s)	Top of PV (strain gauge, kg/cm^2 G)	Side of PV (strain gauge, kg/cm^2 G)	Time to decomposition fume (min. s)
1	3.29	3.16	4.49	2.47	1.89	4.23
2	3.65	3.57	4.43	1.47	1.00	4.23
3	6.14	5.66	4.30	5.56	5.26	4.04
4	4.58	4.29	5.02	3.40	3.27	4.13
5	3.92	3.71	4.56	5.14	4.91	4.00
average	4.32	4.08	4.48	3.61	3.27	4.13
dispersion	1.00	0.87	0.11	1.55	1.66	0.09

*Dutch pressure vessel, voltage of electric furnace 70 V (flat bottom cup), 79 V (round bottom cup), ADCA 5 g, orifice $1^{\phi} \times 3^{t}$.

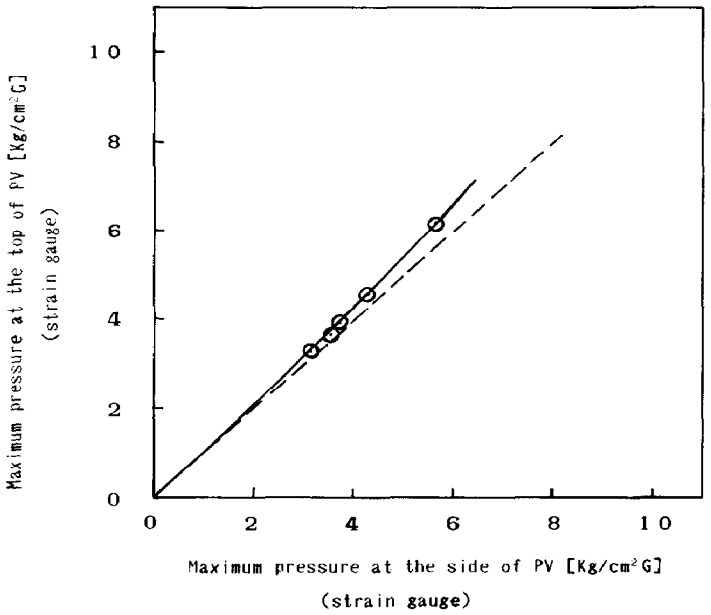

Fig. 13. Relationship between the maximum pressure measured by strain gauges at the top and the side of the pressure vessel. (Vessel details, see Fig. 12).

position is. The curves in Figs. 11 and 13 are similar. This means that the corrected Bourdon gauge pressure is correct and, therefore, we can neglect the response delay of the Bourdon gauge.

4.3.6 Effect of cup type

The experimental results of the maximum pressure using flat and round bottom cups are listed in Table 6. The round bottom cup gave a smaller maximum pressure but with larger dispersion in the pressure readings than the flat one.

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