

# Comparison of nitrocellulose stabilizing capability of lithium carbonate and conventional stabilizers

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## Abstract

At present, substances such as diphenylamine (DPA) and akardite II (AKII) are used in industrial applications to stabilize nitrocellulose (NC). Previous studies have shown that some inorganic salts with alkaline properties in aqueous solutions could also stabilize NC. In this study, we investigated the NC-stabilizing ability of  $\text{Li}_2\text{CO}_3$  on the basis of thermal stability,  $\text{NO}_x$  release, and impact sensitivity. In addition, a closed-vessel test was conducted to study the pressure release behavior during combustion. The results were compared with those for pure NC, NC/DPA, and NC/AKII. Thermal stability was measured in a heat-flux calorimeter at 120°C in an oxygen atmosphere. The induction period for heat release was longer for NC/ $\text{Li}_2\text{CO}_3$  than for pure NC. The induction periods for NC/ $\text{Li}_2\text{CO}_3$ , NC/DPA, and NC/AKII were comparable, although there was some variability depending on the type of NC. Release of  $\text{NO}_x$  from NC/ $\text{Li}_2\text{CO}_3$  was less than that from either pure NC or NC/AKII, but nearly equal to that from NC/DPA. Drop-hammer tests showed that NC/ $\text{Li}_2\text{CO}_3$  had almost equal or lower impact sensitivity than pure NC and NC/AKII. These results suggest that the stabilization effect of  $\text{Li}_2\text{CO}_3$  is comparable to those of conventional stabilizers. In the closed-vessel test, the maximum increase in pressure ( $P_{\max}$ ) and maximum rate of pressure increase ( $dP/dt_{\max}$ ) for NC/ $\text{Li}_2\text{CO}_3$  were slightly lower than those for pure NC, but greater than those for NC/AKII. This shows that  $\text{Li}_2\text{CO}_3$  degrades the combustion characteristics of NC to a smaller degree than AKII does.

**Keywords** : Self-ignition, stabilizer, thermal stability, impact sensitivity, combustion characteristics

## 1. Introduction

Nitrocellulose (NC) is widely used as an ingredient in propellants, explosives, and lacquers. However, it is unstable and can ignite spontaneously in the storage process. In recent years, Japan has witnessed several accidents

caused by the ignition of NC and other nitric acid esters<sup>1,2)</sup>. Thus, it is important to prevent the spontaneous ignition of NC for safe use in the related industries.

Extensive research has been carried out to clarify the mechanism underlying the spontaneous ignition of NC. It

has been proposed that this is caused by the reaction between NC and  $\text{NO}_2$  formed by the cleavage and hydrolysis of the O- $\text{NO}_2$  bond<sup>3</sup>. On the basis of this idea, several mechanisms have been proposed for the release of heat during the ignition of NC<sup>4-6</sup>. All of these mechanisms suggest that  $\text{NO}_2$  plays an important role in the spontaneous ignition of NC. Hence, substances such as diphenylamine (DPA) and akardite II (AKII), which can trap  $\text{NO}_2$  molecules, have been added to stabilize NC.

Previous studies have shown that NC is stabilized when inorganic salts yielding an alkaline solution are added in the solid state<sup>7,8</sup>. However, highly water-soluble inorganic salts cannot be used as NC stabilizers because the absorbed moisture may destabilize NC. Therefore, inorganic salts that are poorly soluble in water but yield highly alkaline aqueous solutions are suitable for use as NC stabilizers. On the basis of these considerations,  $\text{Li}_2\text{CO}_3$  is thought to be an ideal NC stabilizer, as it has relatively poor water solubility (1.3 g / 100 g water) and a highly alkaline aqueous solution (pH = 11–11.5)<sup>9</sup>.

In this study, we investigated the effect of  $\text{Li}_2\text{CO}_3$  on the thermal stability of several types of NC, and analyzed its impact sensitivity and pressure behavior during the combustion of NC. We evaluated the thermal stability of NC by performing a calorimetric analysis and measurement of the amount of  $\text{NO}_x$  released from NC. Furthermore, we analyzed its impact sensitivity and pressure behavior during combustion by carrying out drop-hammer and closed-vessel tests, respectively.

## 2. Experimental

Five types of NC (NC-A, NC-B, NC-C, NC-D, and NC-E) were used in our experiments. NC-A was purchased from the Sigma-Aldrich Corp. NC-B was obtained by storing NC-A at room temperature for a constant period. NC-C and NC-D were manufactured by the Asahi Kasei Chemicals Corp., while NC-E was imported from Germany. All the samples were first wetted with approximately 25 wt% 2-propanol or water. The wetted samples were then dried under a vacuum at room temperature for 3–4 days. NC-A and NC-B particles with diameters of 75–106  $\mu\text{m}$  were separated by sieving and used for further analysis. NC-C, NC-D, and NC-E were used as they were, without sieving. Therefore, the NC-A and NC-B samples were powdery and comprised fine particles, while NC-C, NC-D, and NC-E were fibriform.  $\text{Li}_2\text{CO}_3$  was purchased from Wako Pure Chemical Industries Ltd. (purity : 99%). The conventional stabilizers DPA and AKII were used as the control samples. DPA was purchased from Wako Pure Chemical Industries Ltd. (purity : 99.5%), and AKII was provided by the NOF Corp. These additives were used after being crushed to the desired particle size.

The thermal stability, impact sensitivity, and combustion characteristics of pure NC and the NC samples containing the additives were measured to evaluate the effectiveness of  $\text{Li}_2\text{CO}_3$  in NC. Thermal stability studies were carried out in a heat-flux calorimeter C80 (Setaram Instrumentation) and an  $\text{NO}_x$  meter (Anatech Yanaco Co., Ltd.). The impact sensitivity of NC was determined by the

drop-hammer test (Kuramochi Kagaku Co., Ltd.), and the combustion characteristics of NC were estimated by observing its pressure behavior during combustion using the closed-vessel test developed by the University of Tokyo<sup>10</sup>.

## 3. Results and discussion

### 3.1 Thermal analysis

Approximately 50 mg of pure NC (NC-A, B, C, D, or E) and the NC samples containing the  $\text{Li}_2\text{CO}_3$ , DPA, and AKII additives were placed in 4-mL steel ampoules. The samples were stored at 120°C in an oxygen atmosphere, during which time their thermal and pressure behaviors were investigated. The induction period, i.e., the time taken for the release of heat from each sample was used as the stability index. The induction period indicates the intersection of a tangent on the maximum inclination point of heat flow or pressure and the base line, as shown in Fig. 1. In addition, the induction period for pressure release was calculated, and this was also used as an index of stability.

Our results showed that the addition of  $\text{Li}_2\text{CO}_3$  resulted in an increase in the induction periods of the NC samples. Figures 2 and 3 show the thermal and pressure behaviors of pure NC-B and the NC-B samples containing the additives, respectively. These figures show that the induction periods of NC-B/ $\text{Li}_2\text{CO}_3$  were longer than those of pure NC-B, NC-B/DPA, and NC-B/AKII. In addition, as shown in Fig. 4, the induction periods of all the types of NC samples containing  $\text{Li}_2\text{CO}_3$  were longer than those of pure NC by at least 25 h. Furthermore, a comparison of the induction periods of the all types of NC samples containing  $\text{Li}_2\text{CO}_3$ , DPA, and AKII revealed that the ability of  $\text{Li}_2\text{CO}_3$  to prolong the induction period was comparable to those of DPA and AKII, although there was some variability depending on the type of NC. These observations indicated that  $\text{Li}_2\text{CO}_3$  had a stabilizing effect on NC.

Figure 4 also shows that the stabilizing efficiency of the additives depends on the type of NC sample used. The increase in the induction period is greater for NC-A/ $\text{Li}_2\text{CO}_3$

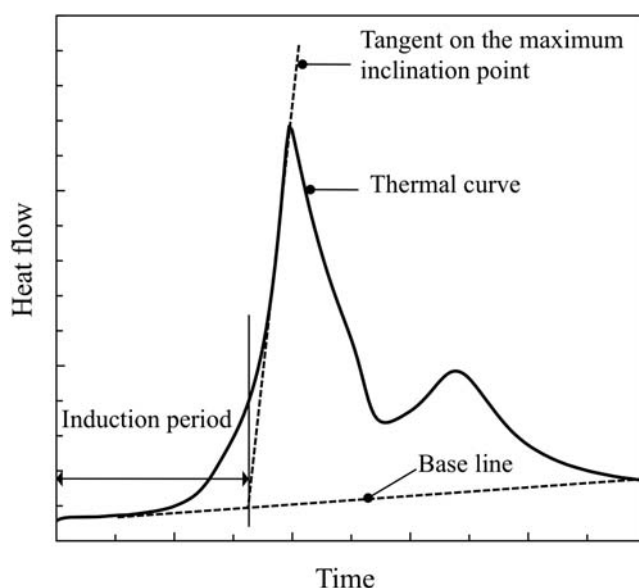
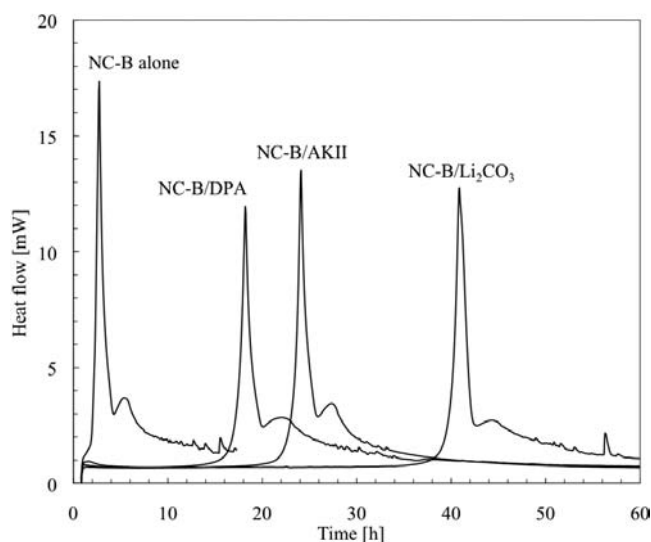
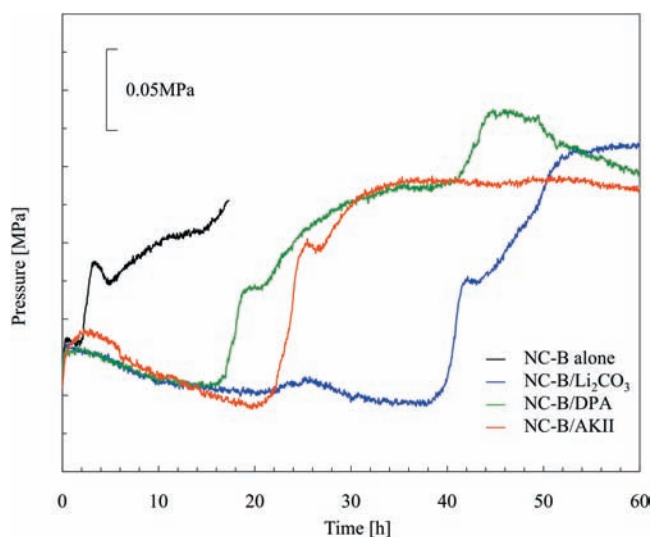


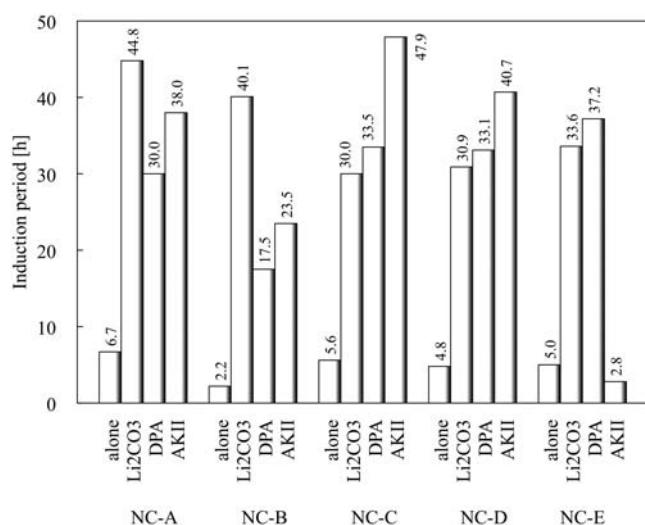
Fig. 1 Example of the thermal curve and the definition of the induction period in this study.



**Fig. 2** Thermal behavior of pure NC-B and NC-B containing the additives.  
 NC content : 50 mg, Additive content : 2 wt%, Atmosphere : O<sub>2</sub> (atmospheric pressure)



**Fig. 3** Pressure behavior of pure NC-B and NC-B containing the additives.  
 NC content : 50 mg, Additive content : 2 wt%, Atmosphere : O<sub>2</sub> (atmospheric pressure)



**Fig. 4** Induction period measured by thermal analysis.  
 NC content : 50 mg, Additive content : 2 wt%, Atmosphere : O<sub>2</sub> (atmospheric pressure)

and NC-B/Li<sub>2</sub>CO<sub>3</sub> than it is for NC/DPA and NC/AKII. On the other hand, the increase in the induction periods of the NC-C/Li<sub>2</sub>CO<sub>3</sub> and NC-D/Li<sub>2</sub>CO<sub>3</sub> samples is comparable to the increase in the induction periods of NC-C/DPA and NC-D/DPA, but is less than that of NC/AKII. This difference in the stabilizing effect of Li<sub>2</sub>CO<sub>3</sub> is thought to result from the difference in the particle diameters of the NC samples and the evaporability of the additive. Li<sub>2</sub>CO<sub>3</sub> is nonvolatile and does not blend well with NC-C or NC-D, as these samples contain large particles; however, Li<sub>2</sub>CO<sub>3</sub> is comparatively easy to blend with NC-A and NC-B because these samples comprise fine particles. On the other hand, since both DPA and AKII are volatile, they can be easily blended with NC samples, even those containing large particles. In particular, the distribution of DPA, which has a low melting point (53°C)<sup>11</sup>, is comparatively high in NC. In fact, the overall color of the NC/DPA sample changes from white to green or yellow after it is stored for a certain period; this is probably due to the formation of nitro or nitroso derivatives of DPA<sup>12</sup>. From these observations, we propose that the stabilization effect of Li<sub>2</sub>CO<sub>3</sub> is stronger in NC-A and NC-B than in NC-C or NC-D.

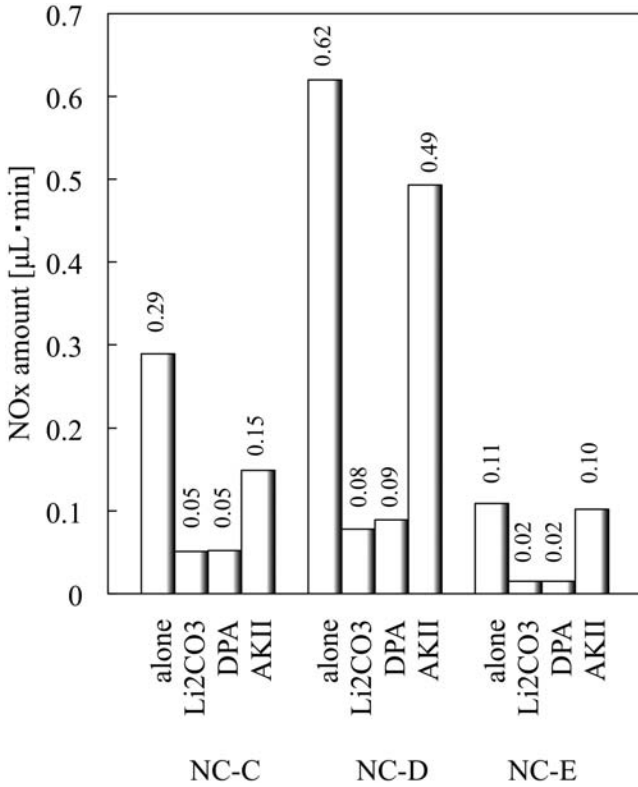
The induction period of NC-E/AKII hardly changes, while the induction periods of NC/Li<sub>2</sub>CO<sub>3</sub> and NC/DPA show an increase. The same trend was observed in the three cycles of reproducibility tests conducted on NC/AKII. The reason for this phenomenon cannot currently be explained. However, we report that this characteristic behavior is possibly seen in NC/AKII.

### 3.2 Measurement of the amount of NO<sub>x</sub> produced

The total amounts of NO and NO<sub>2</sub> released from each NC sample at 65°C were measured using an NO<sub>x</sub> meter. The experimental samples used were pure NC-C, NC-D, and NC-E (1.3 g each), and NC containing 2 wt% of Li<sub>2</sub>CO<sub>3</sub>, DPA, or AKII. A 34-mL glass test tube was used as the ampoule. The experiment was performed using the method prescribed by the National Defense Standard<sup>13</sup>.

As shown in Fig. 5, the amount of NO<sub>x</sub> released from NC/Li<sub>2</sub>CO<sub>3</sub> was less than that released from the pure NC sample. Furthermore, NO<sub>x</sub> release from NC/Li<sub>2</sub>CO<sub>3</sub> was less than that from NC/AKII, but nearly equal to that from NC/DPA. These results indicated that Li<sub>2</sub>CO<sub>3</sub> effectively stabilized NC in terms of suppressing NO<sub>x</sub> release. By contrast, the efficiency of AKII was comparatively low in all the NC samples analyzed in this study.

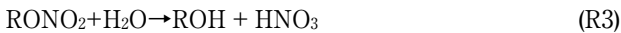
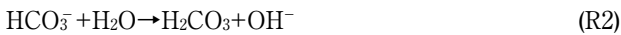
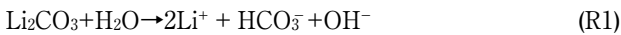
The amount of NO<sub>x</sub> released from NC is independent of the thermal behavior of NC (mentioned in Section 3.1). For example, the ability of Li<sub>2</sub>CO<sub>3</sub> to suppress the release of NO<sub>x</sub> is stronger than that of AKII; however, the effect of Li<sub>2</sub>CO<sub>3</sub> on the thermal behavior of NC is comparable to that of AKII. This may indicate that the mechanism underlying the reaction of NC with Li<sub>2</sub>CO<sub>3</sub> is different from that underlying the reaction of NC with AKII or DPA. The aqueous solution of Li<sub>2</sub>CO<sub>3</sub> is alkaline because Li<sub>2</sub>CO<sub>3</sub> dissociates in water in the manner shown in R1 and R2. From these dissociation reactions, it is assumed that Li<sub>2</sub>CO<sub>3</sub> suppresses the release of NO<sub>x</sub> from NC by neutralizing the acid that accelerates the hydrolysis of the O-NO<sub>2</sub> bond (as



**Fig. 5** Amount of NO<sub>x</sub> generated from NC. NC content : 1.3 g, Additive content : 2 wt%, Atmosphere : Air (atmospheric pressure)

shown in R3). On the other hand, AKII or DPA would trap NO<sub>x</sub> molecules or other radical species released from NC<sup>(12),14)–16)</sup>. Consequently, NO<sub>x</sub> release is prevented more effectively in NC/Li<sub>2</sub>CO<sub>3</sub> than in the other NC samples.

AKII had a greater ability to stabilize NC than did DPA; in contrast, DPA was more effective than AKII in suppressing NO<sub>x</sub> release. Kimura *et al.* suggested that volatile DPA affected the results obtained in the Abel heat test<sup>17)</sup>. Thus, it was assumed that the gas-phase reaction between DPA and NO<sub>x</sub> helped to suppress the release of NO<sub>x</sub> from NC.



### 3.3 Impact sensitivity test

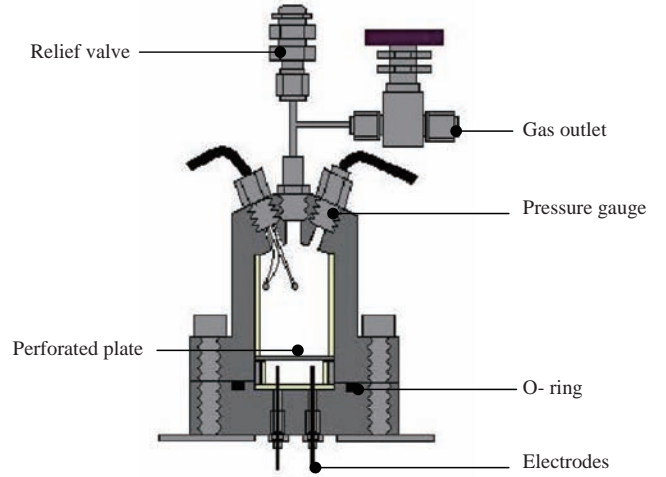
The impact sensitivities of pure NC–D and NC–D containing 2 wt% of the additive (Li<sub>2</sub>CO<sub>3</sub> or AKII) were investigated. The sample amount, test procedure, and method of judging the explosion were decided in accordance with the Japan Explosive Society standards<sup>18)</sup>. The test was repeated 20 times using the up-and-down method<sup>19)</sup>; the height corresponding to the 50% explosion point (H<sub>50</sub>) and the energy at H<sub>50</sub> (E<sub>50</sub>) were calculated. H<sub>50</sub> was calculated using the following formula :

$$\log H_{50} = C + d \left( \frac{A}{N_s} \pm \frac{1}{2} \right) \quad (1)$$

where C is the logarithm of the lowest drop height, and d is the logarithm of the interval of the drop heights. A and

**Table 1** Impact sensitivity obtained for the drop–hammer test.

Sample	H <sub>50</sub> [cm]	E <sub>50</sub> [J]
Pure NC	20.0	9.8
NC/Li <sub>2</sub> CO <sub>3</sub>	28.3	14
NC/AKII	23.8	12



**Fig. 6** Apparatus for the closed–vessel test.

N<sub>s</sub> are denoted by Σ(i · n) and Σ(n), respectively. Here, n is the number of explosions at each drop height, and i indicates the sequence number of the drop heights counted from the lowest drop height.

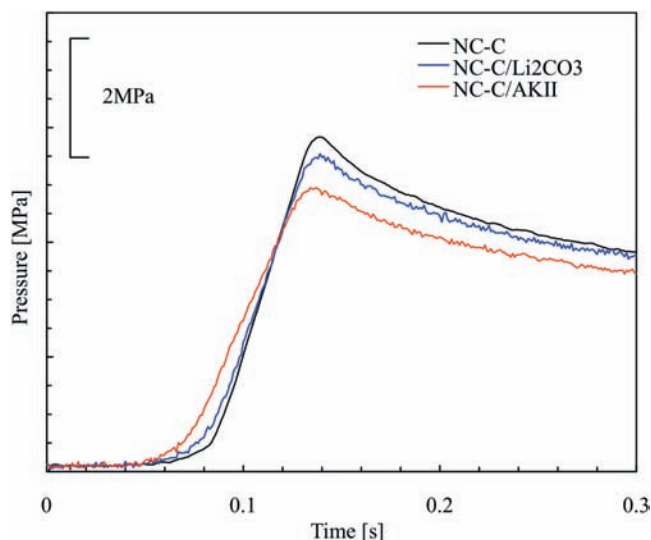
E<sub>50</sub> is the product of H<sub>50</sub>, acceleration due to gravity (0.098 cm · s<sup>-2</sup>), and the mass of the drop hammer (5kg) :

$$E_{50} = 0.098M \cdot H_{50} \quad (2)$$

The results summarized in Table 1 indicate that the impact sensitivity of NC/Li<sub>2</sub>CO<sub>3</sub> is lower than that of pure NC and NC/AKII. Further investigations must be carried out to clarify the reason for the small difference in the impact sensitivities of the samples; this is because the drop–hammer test is carried out only 20 times in this study, and the difference between the drop heights in each test cycle is comparatively large. However, it is apparent that the addition of Li<sub>2</sub>CO<sub>3</sub> does not result in any significant increase in the impact sensitivity of NC.

### 3.4 Closed–vessel test

For the closed–vessel tests<sup>10)</sup>, samples were prepared by casting NC–C/Li<sub>2</sub>CO<sub>3</sub> and NC–C/AKII into pellets. The resulting pellets were subjected to the closed–vessel test in a 52–mL combustion chamber to investigate the pressure behavior and combustion characteristics of NC. Figure 6 shows the deflagration test apparatus, which comprises a 52–mL closed vessel equipped with a pressure transducer (Kyowa Electronic Instruments Co., Ltd., PE–200KWS), a gas outlet, and a safety relief valve. The NC pellets were ignited using a mixture of Ti/KNO<sub>3</sub> powder (100 mg; primary igniter) and B/KNO<sub>3</sub> pellets (200 mg; secondary igniter). These samples were placed at the bottom of the combustion chamber and ignited by applying a voltage of 30 V to a coiled nichrome wire between the electrodes.



**Fig. 7** Pressure behavior during combustion. NC content: 1.3 g, Additive content: 2 wt%, Atmosphere: air (atmospheric pressure)

The results of the closed-vessel test (Fig. 7) show that the maximum pressure ( $P_{\max}$ ) and maximum rate of pressure increase ( $dP/dt_{\max}$ ) of NC/ $\text{Li}_2\text{CO}_3$  were higher than those of NC/AKII. However, the  $P_{\max}$  and  $dP/dt_{\max}$  of NC/ $\text{Li}_2\text{CO}_3$  were lower than those of pure NC. This indicated that  $\text{Li}_2\text{CO}_3$  did not degrade the combustion characteristics of NC as severely as the conventional stabilizers did. The reaction between AKII and the radical species generated during the combustion is thought to result in a decrease in the  $P_{\max}$  and  $dP/dt_{\max}$  of NC/AKII. In addition, it is assumed that the  $P_{\max}$  and  $dP/dt_{\max}$  of NC/ $\text{Li}_2\text{CO}_3$  hardly decreased because  $\text{Li}_2\text{CO}_3$  is not a radical scavenger like AKII.

#### 4. Conclusions

The efficiency of  $\text{Li}_2\text{CO}_3$  in NC was determined by measuring its thermal stability, impact sensitivity, and pressure behavior during combustion. The following conclusions can be drawn from the observed results:

- The thermal behaviors of pure NC, NC/ $\text{Li}_2\text{CO}_3$ , NC/DPA, and NC/AKII were investigated using a heat-flux calorimeter. The results showed that the induction periods of NC/ $\text{Li}_2\text{CO}_3$  were longer than that of pure NC, but comparable to that of NC/DPA and NC/AKII.
- The release of  $\text{NO}_x$  was suppressed in NC/ $\text{Li}_2\text{CO}_3$  in comparison to pure NC and NC/AKII, while nearly equal amounts of  $\text{NO}_x$  were released from NC/ $\text{Li}_2\text{CO}_3$  and NC/DPA. On the basis of these results, as well as the thermal analysis results, it appeared that the stabilizing effect of  $\text{Li}_2\text{CO}_3$  was similar to that of conventional stabilizers.
- According to the results of the drop-hammer test, the impact sensitivity of NC/ $\text{Li}_2\text{CO}_3$  was equal to or lower than that of pure NC and NC/AKII. Thus, it was concluded that the addition of  $\text{Li}_2\text{CO}_3$  did not cause any notable increase in the impact sensitivity of NC.
- The results of the closed-vessel test revealed that the  $P_{\max}$  and  $dP/dt_{\max}$  of NC/ $\text{Li}_2\text{CO}_3$  were higher than those of NC/AKII but lower than those of pure NC. This indi-

cated that  $\text{Li}_2\text{CO}_3$  did not degrade the combustion characteristics of NC as much as the conventional stabilizers did.

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# ニトロセルロース安定剤としての炭酸リチウムの性能と 従来安定剤との比較

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現在、ニトロセルロース (NC) の安定剤としてジフェニルアミン (DPA) やアカルダイトII (AKII) などが使用されている。一方、水溶液中で塩基性を示す無機塩を固体状態でNCに添加するとNCが安定化するという報告があり、新規安定剤として期待される。本研究では、炭酸リチウム ( $\text{Li}_2\text{CO}_3$ ) に着目し、 $\text{Li}_2\text{CO}_3$  を添加したNCの熱安定性、 $\text{NO}_x$  発生量、打撃感度、および、燃焼性に関して検討を行ない、NC単独、NC/DPA、NC/AKIIの場合と比較することにより、 $\text{Li}_2\text{CO}_3$  の安定剤としての性能を評価した。熱流束型熱量計を用いた $120^\circ\text{C}$ 、酸素雰囲気下における熱安定性試験では、NC/ $\text{Li}_2\text{CO}_3$  の発熱までの誘導期は、NC単独の場合よりも延長することが確認された。また、NCの種類によるばらつきはあるが、 $\text{Li}_2\text{CO}_3$  は、DPAおよびAKIIと同程度まで安定化することがわかった。また、NC/ $\text{Li}_2\text{CO}_3$  から発生する $\text{NO}_x$  量は、NC単独、NC/AKIIの場合より低く、NC/DPAと同程度まで抑制された。NC/ $\text{Li}_2\text{CO}_3$  の落つい打撃感度は、NC単独あるいはNC/AKIIよりも若干低く、 $\text{Li}_2\text{CO}_3$  添加により著しく感度が高くなることはないことが確認された。また、燃焼時のNC/ $\text{Li}_2\text{CO}_3$  の最大到達圧力および最大圧力上昇速度はNC単独よりは抑制されるが、NC/AKIIよりは高いことが確認された。この結果から、 $\text{Li}_2\text{CO}_3$  は、AKIIと比較してNCの燃焼性を低下させないことが示唆された。

**Keywords** : Self-ignition, stabilizer, thermal stability, impact sensitivity, combustion characteristics

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