Research paper

Combustion of amino guanidinium 5,5′-azobis-1H-tetrazolate/polyvinyl acetate-coated ammonium nitrate mixture

Kazuo Iwakuma* and Kazuo Hasue*[†]

*Department of Applied Chemistry, School of Applied Sciences, National Defense Academy, 1−10−20 Hashirimizu, Yokosuka-shi, Kanagawa, 239−8686 JAPAN
Phone: +81−46−841−3810
[†]Corresponding author: khasue@hotmail.com

Abstract

Tetrazoles are considered safe and easy-to-handle gas-generating agents. In this study, aminoguanidinium 5,5′-azobis-1H-tetrazolate (AGAT) was selected as a fuel owing to its high heat of formation and combustibility in an inert atmosphere. Ammonium nitrate (AN) is known as an environmentally friendly oxidizer. However, AN is hygroscopic, which is a problem during manufacturing and storage and when it is mixed with organic fuels that have low burning rates. Despite reports that the stoichiometric AGAT/AN mixture does not burn, this study shows that it is burnable when AN is coated with polyvinyl acetate (ANc). Adding Cu or CuO to the AGAT/ANc mixture increased the burning rate to values comparable to those of the guanidine nitrate/strontium nitrate/basic copper nitrate mixture, a gas-generating agent used in practical applications. A 4-L tank test was conducted to examine the gas-generating ability of these mixtures. The gas-generating ability was improved to practically relevant values by adding Cu or CuO. To understand the improved burnability of AGAT/ANc, thermogravimetry and differential thermal analyses were performed and the temperature profiles were obtained by means of an embedded K-type thermocouple. Coating with polyvinyl acetate, or adding Cu or CuO, enhanced the decomposition of the mixtures. As the effect of the coating on the thermal decomposition was small, polyvinyl acetate may work as a fuel, and the close contact between AN and the fuel improved the burnability. Using the temperature profiles, the temperature gradients above the burning surface were obtained, indicating that the heat feedback from the gas phase reaction zone controlled the burning rates of the mixtures.

Keywords: aminoguanidinium 5,5′-azobis-1H-tetrazolate (AGAT), coating ammonium nitrate, combustion, polyvinyl acetate

1. Introduction

Ammonium nitrate (AN) is known as an environmentally friendly oxidizer and a clean gas-generating compound because it does not produce harmful gases or solid burning residues. However, AN is hygroscopic, which becomes a problem during manufacturing and storage. Furthermore, it contracts and expands during phase transitions, which causes structural damage to the grains. It was suggested that the hygroscopicity of AN could be prevented or minimized by a polyvinyl acetate (PVAc) coating[1]. In this study, AN was coated with PVAc (ANc) to prevent moisture absorption during sample preparation.

AN-based gas-generating agents, prepared by mixing AN with organic fuels, have poor combustion reactivities compared to those of some metal perchlorates[2)−4)] or nitrate mixtures[5)−7)]. It has been reported that gas-generating agents comprising AN, with burning rates of less than $10 \, \text{mm·s}^{-1}$ at 7 MPa, do not ignite reliably and often result in "no-fires" in the inflator[8]. Further, to avoid over-pressurization of the inflator, a pressure exponent of approximately 0.30 or less is desirable[9]. The primary purpose of this study is to obtain a gas-generating agent comprising AN with an improved burning rate and a

lower pressure exponent than the recommended value.

Tetrazoles store energy in their N−N and N−H bonds and release it when they break up into N_2 and H_2 gases. Tetrazoles have very little carbon, hence, smaller amounts of oxidizers are needed and they are expected to be suitable as safe and easy-to-handle gas-generating agents[10]. Tetrazoles with a high heat of formation[11] can be used as a fuel component to improve the burnability of AN-based gas-generating agents. It has been reported that amino guanidinium 5,5′-azobis-1 H-tetrazolate (AGAT)[12)−14)] and 1H-tetrazole (1HT)[15), 16)] are well-suited for this purpose. However, the combustion of AGAT/AN mixtures has been reported only in fuel-rich compositions[10]. Although these fuel-rich compositions show high burning rates, they tend to produce H_2 and harmful CO gases. While mixtures closer to a stoichiometric composition may lower H_2 and CO gas production, they are difficult to burn.

This study shows that the stoichiometric AGAT and ANc mixture is burnable at low burning rates. The burning behavior is improved by the addition of copper-based additives.

2. Experimental
2.1 Samples
Figure 1 shows the structural formula of AGAT. AGAT (Toyo Kasei Kogyo Co., Ltd.) was used as the fuel and AN as the oxidizer. Both AGAT and AN were crushed in a ball mill and their particle diameters were controlled to within 45−75 μm using Japanese Industrial Standard (JIS) sieves. For the burning rate measurements, test samples of the stoichiometric AGAT/ANc (20.75 : 79.25 (w/w)) mixtures and other mixtures' compositions were decided on either fuel poor or rich arbitrarily, as shown in Table 1. ANc was prepared as follows: A 1 wt% PVAc-acetone solution was obtained by dissolving 1.0 g of 50 wt% PVAc-ethyl acetate solution in 49.5 g of acetone. The ANc (with 0.002 wt% of PVAc) material was obtained by adding 20 g of the 1.0 wt% PVAc-acetone solution to 100 g of the AN
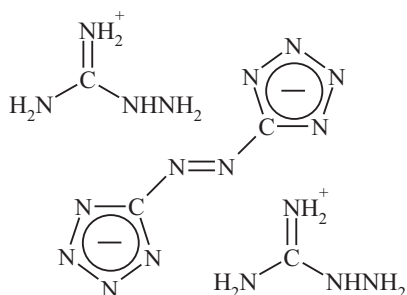

Figure 1　Structural formula of aminoguanidinium 5,5′-azobis-1H-tetrazolate (AGAT).

Table 1　Compositions of AGAT/ANc/additive mixtures[wt%].

AGAT	ANc	Additive
30	70	−
25	75	−
20.75	79.25	2 parts
18	82	−

powder. ANc was then dried using a vacuum dryer. The mixtures were compounded at the said mixing ratio for 30 min at 80 rpm in a rotary mixer. It has been reported that less than ~0.08 parts PVAc do not affect the burning rate of these mixtures[17]. As the part of PVAc used here was 0.002, we note that the burning rate may not be affected.

For the 4-L tank test, samples of the stoichiometric AGAT/ANc (20.75 : 79.25 (w/w)) mixtures were tested and, to enhance the burning rate, 2 parts of manganese(IV) oxide (MnO_2), copper(II) oxide (CuO), copper (Cu), iron(III) oxide (Fe_2O_3), and zinc oxide (ZnO) were added for every 100 parts of the stoichiometric AGAT/ANc mixtures. These additives were expected to enhance the decomposition of AN[18] and 2 parts of additives were determined on the basis of McLain's recommendation[19]. Commercial gas-generating agents, guanidine nitrate (GN), strontium nitrate (SrN), and basic copper nitrate (BCN), were mixed with the standard ratio of 56.05 : 19.45 : 24.50 by weight.

The thermal analyses were conducted on the stoichiometric AGAT/AN, AGAT/ANc, and the stoichiometric mixtures with 2-parts additives. The temperature profiles were obtained for the stoichiometric AGAT/ANc mixtures and the stoichiometric mixtures with 2-parts additives, i.e., Cu and CuO.

2.2 Burning rate
To measure the burning rate of mixtures with no additive, 4 g of the dried mixture was uniaxially pressed at a pressure of approximately 6 MPa for 5 min to form 5×5×70 mm strands. To measure the burning rate of mixtures with and without an additive, 4 g of the dried mixture was uniaxially pressed at a pressure of approximately 200 MPa for 3 min to form cylindrical pellets of diameter 14.7 mm and thickness 14 mm. With an additive, the strand quality was inferior to that of a pellet; therefore, the pellet type was chosen. The sides of the strands and pellets were coated with epoxy resin. Combustion tests were performed using a 4-L chimney-type strand burner with optical windows, under N_2 atmosphere in the range of 1−4 MPa at an initial temperature of 296 K. The sample was ignited using a heated nichrome wire (diameter 0.6 mm). The pressure in the chamber was measured using a strain-gauge pressure transducer. The transducer data after amplification through a signal amplifier were recorded using a digital data recorder. The burning rates (r) were deduced from the duration of the recorded pressure increase. The pressure increase continuously as the sample started to burn and stopped increasing when the combustion ceased. The average internal pressure (P) was calculated by averaging the pressures at the start and end of combustion.

2.3 Four-liter tank test
The gas-generation ability (generally measured by a 60-L tank test) was investigated using a 4-L tank test. As mentioned earlier, 4 g of the dried mixture was uniaxially pressed at approximately 200 MPa for 3 min to form cylindrical pellets (diameter 14.7 mm, thickness 14 mm).
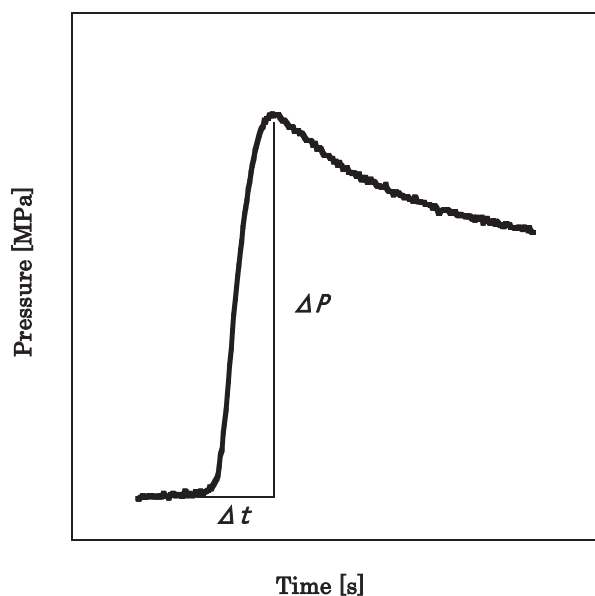
Figure 2   4-L tank test results.

Figure 3   Burning rates of mixtures. Dashed lines were obtained from Reference 14.

The sides of the pellets were uniformly coated with epoxy resin to achieve cigarette burning. The 4-L chimney-type strand burner, used for the burning rate measurements, was also used for these experiments with no modification other than closing the gas flow exit. The 4-L tank tests were conducted with a starting temperature and pressure of 296 K and 2 MPa, respectively. From the acquired pressure-time data (Figure 2), ΔP (the net pressure increase between the initial and maximum pressures) and Δt (the time from the onset of the pressure increase to when the pressure reached its maximum) were calculated to determine the rate of pressure increase ($\Delta P \cdot \Delta t^{-1}$).

2.4 Thermal analysis

Thermogravimetry (TG) and differential thermal analysis (DTA) were conducted at a heating rate of 5 K·min^{-1} in a He atmosphere (flow rate = 20 mL·min^{-1}) using an aluminum cell. The starting decomposition temperature (T_{TG}) was determined using the intersection point of a line extended from the baseline of the lower-temperature side and a tangent line drawn at the maximum inclination point of the weight loss line.

2.5 Temperature profile

To obtain the temperature profiles, cylindrical pellets (diameter 10.15 mm) were prepared by compressing ~1.5 g of the stoichiometric mixture at approximately 400 MPa for 5 min while embedding a K-type thermocouple (diameter: 50 μm). The output of the thermocouple was acquired with a transient recorder through a direct-current amplifier. A temperature-time curve was obtained, from which the temperature-distance curve was derived. The distance was obtained by multiplying the time by the burning rate.

3. Results and discussion
3.1 Burning rate
3.1.1 Effect of fuel content on the burning rate

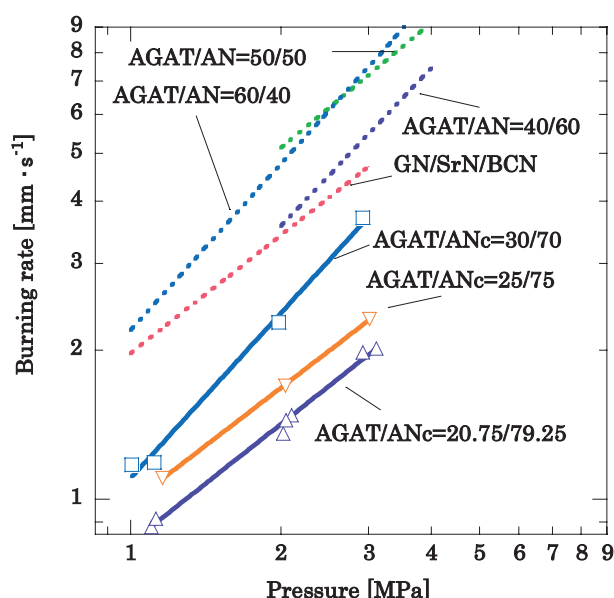The results of the burning rate test for each AGAT/

ANc mixture at 296 K are presented in Figure 3. It was confirmed that the burning rates of the mixtures followed Vieille's law, given by $r=aP^n$, where *a* is a constant that depends on the chemical composition and an initial propellant temperature and *n* is the pressure exponent of the burning rate.

For AGAT/AN mixtures, it has been reported that uniaxially pressed strands ignite, however, all of them displayed interrupted burning when the AGAT content was 30 wt%. When the weight content of AGAT was increased to 40 wt%, all the strands burned at over 2 MPa[14]. The reported burning rates of AGAT/AN with ratios of 40/60, 50/50, and 60/40 are shown as the dashed lines in Figure 3. At 7 MPa, their extrapolated burning rates were higher than the recommended 10 mm·s^{-1} [8], but the pressure exponents were larger than the recommended value of 0.3[9].

In this study, it was found that an AGAT/ANc mixture with a ratio of oxidizer rich 18/82 burned unstably and that AGAT/ANc mixtures burned only when the content of AGAT was ≥20.75 : 79.25 (Figure 3). However, the burning rate of the combustible mixture was lower than that of GN/SrN/BCN.

Figure 3 also shows that the burning rates of the AGAT/ANc mixtures decreased with increasing ANc content. It has been found that AGAT, alone, decomposes continuously in an inert atmosphere[12] through exothermic decomposition. Therefore, the reduction in the burning rate with ANc addition is likely due to the endothermic decomposition of ANc, which causes the absorption of heat.

3.1.2 Effect of additives on the burning rate

The effect of additives on the burning rate is shown in Figure 4. The burning rate approximately doubled by adding Cu or CuO and increased by 20% with MnO$_2$ addition. The burning rates of the AGAT/ANc mixtures with Cu or CuO were similar to that of the commercial GN

/SrN/BCN mixture. The constant a increased when Cu, CuO, or MnO$_2$ was added. The values of n, the pressure exponent of the mixtures, with no additive, Cu, CuO, and MnO$_2$ additives were 0.52, 0.74, 0.84, and 0.76, respectively. As these values are larger than the desirable value of 0.3[9], but comparable to that of GN/SrN/BCN (n = 0.79), we propose that the AGAT/ANc/Cu and AGAT/ANc/CuO mixtures can be used as practical gas-generating agents.

3.2 Four-liter tank test

The results of the 4-L tank tests are shown in Figure 5 and Table 2. The rate of pressure increase ($\Delta P \cdot \Delta t^{-1}$) for the AGAT/ANc mixtures was 0.08 MPa·s^{-1}, less than that of the GN/SrN/BCN mixture (0.16 MPa·s^{-1}). However, the net pressure increase (ΔP) of the AGAT/ANc mixture was larger than that of the GN/SrN/BCN mixture. However, the 4-L test results of the AGAT/ANc mixtures were not acceptable for a practical gas-generating agent, hence, the mixtures require improvements. The addition of some metallic oxides was tested to improve the $\Delta P \cdot \Delta t^{-1}$ value of the mixture. The addition of Cu or CuO increased the $\Delta P \cdot$ Δt^{-1} values to equal to or greater than that of the GN/SrN/BCN mixture. The addition of MnO$_2$, Fe$_2$O$_3$, and ZnO increased ΔP and the burning rate. However, they were less effective than Cu or CuO. The increases in $\Delta P \cdot \Delta t^{-1}$ were not as high as anticipated because, while the incorporation of these additives increased the value of ΔP, they did not decrease Δt sufficiently.

3.3 Thermal analysis

The TG and DTA curves of AN, ANc, AGAT, AGAT/ AN, and AGAT/ANc are shown in Figures 6(a) and 6(b), respectively.

The T_{TG} values of AN and ANc were 496 and 488 K, respectively. The decomposition of AN and ANc ended at approximately 550 and 540 K, respectively. The coating promoted the decomposition of AN, resulting in a lower decomposition temperature. We note that the close contact between the fuel (PVAc) and the oxidizer (AN) may improve the decomposition behavior of AN. In hydroxyl-terminated polybutadiene (HTPB) / AN propellants, AN particles are coated with HTPB, which
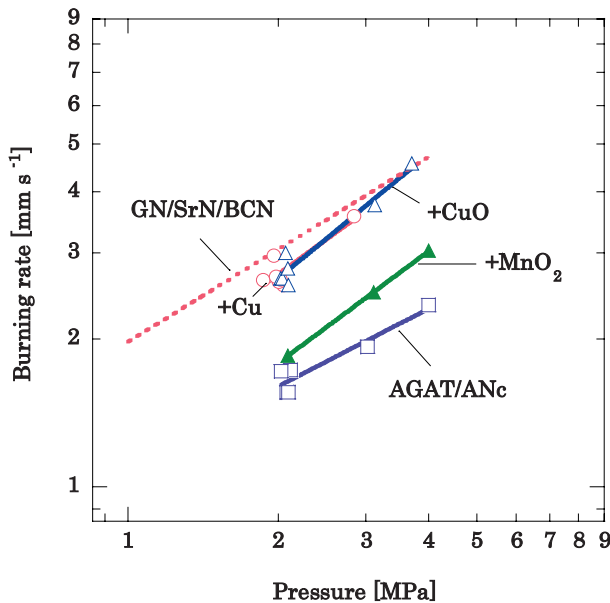

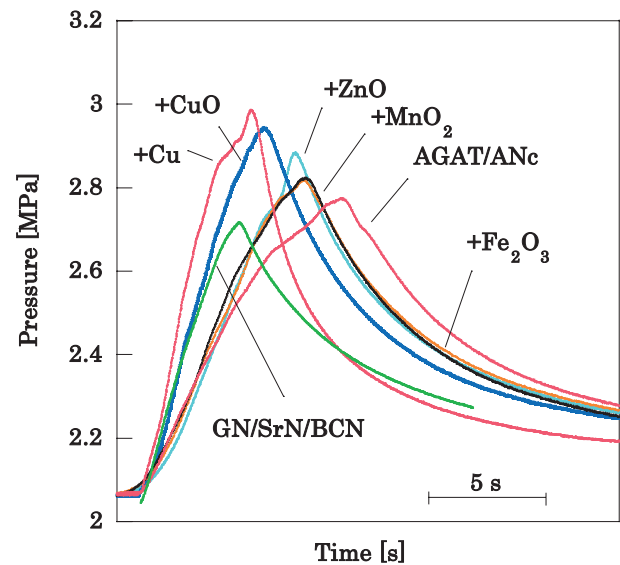**Figure 4** Burning rates of mixtures with and without additives.

Figure 5 Pressure–time histories of mixtures from the 4 L tank test.

Table 2 Gas generation ability of different additive mixtures.

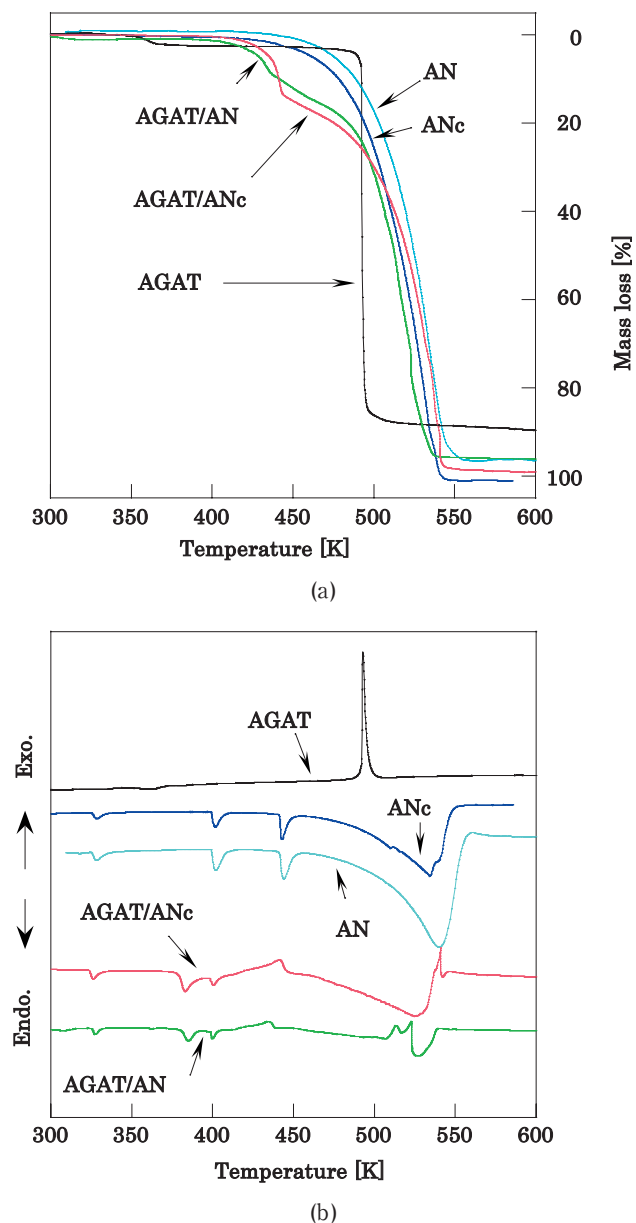|	Additive (2 parts)	Starting pressure [MPa]	Maximum pressure [MPa]	Pressure increase ΔP [MPa]	Elapsed time Δt [s]	Rate of pressure increase $\Delta P \cdot \Delta t^{-1}$ [MPa·s^{-1}]	r at 2 MPa [mm·s^{-1}]
AGAT/ ANc (20.75/79.25)	−	2.06	2.77	0.71	8.60	0.08	1.61
	MnO$_2$	2.06	2.82	0.76	7.24	0.10	1.80
	CuO	2.06	2.94	0.88	5.48	0.16	2.66
	Fe$_2$O$_3$	2.06	2.82	0.76	7.24	0.10	−
	ZnO	2.06	2.88	0.82	6.76	0.12	−
	Cu	2.07	2.98	0.91	4.56	0.20	2.71
GN/SrN/BCN	−	2.05	2.72	0.67	4.10	0.16	3.42

(a)

(b)

Figure 6   TG (a) and DTA curves (b) of AN, ANc, AGAT, AGAT/AN, and AGAT/ANc.

functions as a fuel. However, the involvement of the PVAc coating as a fuel is likely small and reductions in burning rates have been reported[17]. In contrast, in AGAT/AN, the AGAT and AN particles interact via point contacts, which are evidently smaller in area than the surface contacts between the AN particles and liquid PVAc.

The DTA curves of AN and ANc showed two endothermic phase-transition peaks at 326 and 400 K, respectively. Another endothermic peak, corresponding to the melting of these compounds was observed at 443 K. Higher-temperature endothermic peaks corresponding to the dissociation of AN and the subsequent vaporization of NH_3 and HNO_3[20] were observed at 540 and 533 K for AN and ANc, respectively.

The TG and DTA curves in Figure 6 also show that approximately 85 wt% of AGAT exothermically decomposed at 498 K.

The T_{TG} of the AGAT/AN mixture was 427 K and approximately 10 wt% of the mixture decomposed. The

decomposition of the mixture was completed at 535 K. The T_{TG} of the AGAT/ANc mixture was 435 K and approximately 15 wt% of the mixture decomposed. The decomposition of the AGAT/ANc mixture completed at 540 K.

As the decomposition of 85% AGAT causes a weight loss of approximately 17% of the stoichiometric mixture, the first weight loss may be predominantly caused by the decomposition of AGAT.

The DTA curves of the AGAT/AN and AGAT/ANc mixtures showed three endothermic peaks at 326, 385, and 400 K. The first and second peaks may correspond to the phase-transition peaks of AN and ANc at 326 and 400 K, respectively. The third peak may correspond to the melting point of AN and ANc at 443 K. In these mixtures, the melting points of both AN and ANc reduced owing to the addition of AGAT, and the liquefied AN and ANc molecules promoted the decomposition of AGAT.

The DTA curves of the AGAT/AN and AGAT/ANc mixtures showed small exothermic peaks at approximately 440 K and 435 K, respectively. These temperatures coincided with the first weight loss temperatures in the TG curves of AGAT/AN and AGAT/ANc. These small exothermic peaks may be due to the exothermic decomposition of AGAT and/or the reaction between liquefied AN and AGAT. Although the liquefied AN can react with AGAT, we observe from the large endothermic peak in the DTA curve that a significant part of AN did not react. The DTA curves of the AGAT/AN and AGAT/ANc mixtures showed exothermic peaks at approximately 520 K and 540 K, respectively. The DTA curves of the AGAT/AN and AGAT/ANc mixtures also showed exothermic peaks during AN decomposition. These could be due to the reaction between AN and the condensation products of AGAT.

Adding Cu or CuO increased the burning rate and improved the 4-L tank test results.

The effects of the Cu and CuO addition on the thermal decomposition of the AGAT, ANc, and AGAT/ANc mixtures are shown in Figures 7, 8, and 9, respectively.

The T_{TG} values of AGAT, AGAT/Cu, and AGAT/CuO are 494, 491, and 493 K, respectively, as shown in Figure 7 (a). The decomposition peak of AGAT (500 K) was slightly lowered by adding Cu (496 K) or CuO (498 K), as shown in Figure 7(b). Cu was slightly more effective than CuO in promoting the decomposition of AGAT.

The TG-DTA curves of ANc, ANc/Cu, and ANc/CuO are shown in Figure 8. The T_{TG} of ANc, ANc/Cu, and ANc/CuO are 488, 471, and 464 K, respectively. TG-DTA curves of AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc /CuO are shown in Figures 9(a) and (b). The T_{TG} values of AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO are 435, 416, and 413 K, respectively. CuO was marginally more effective than Cu in hastening the decomposition of both ANc and AGAT/ANc.

As ANc and AGAT may act separately in the condensed phase reaction zone, we believe that ANc, the main component, had a major effect on the decomposition behavior of the mixture. The enhanced contribution of
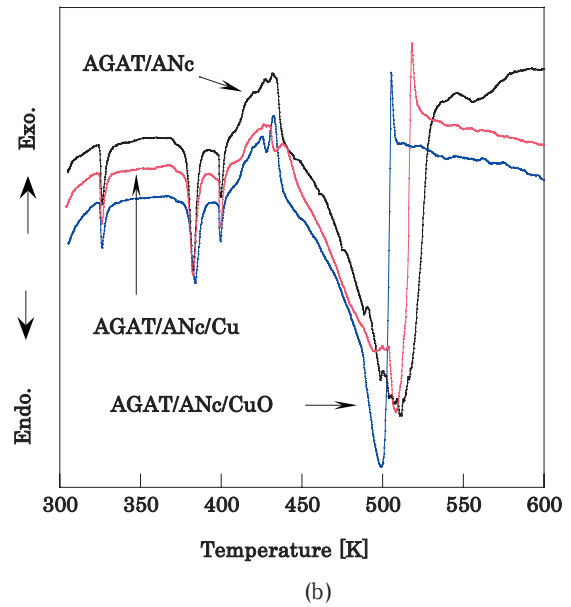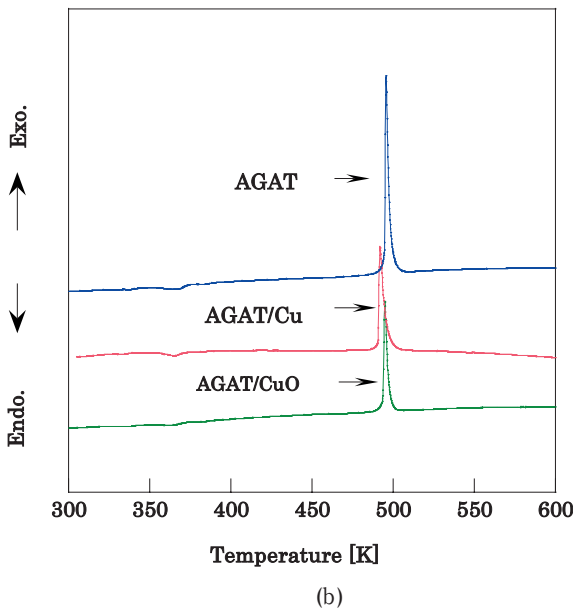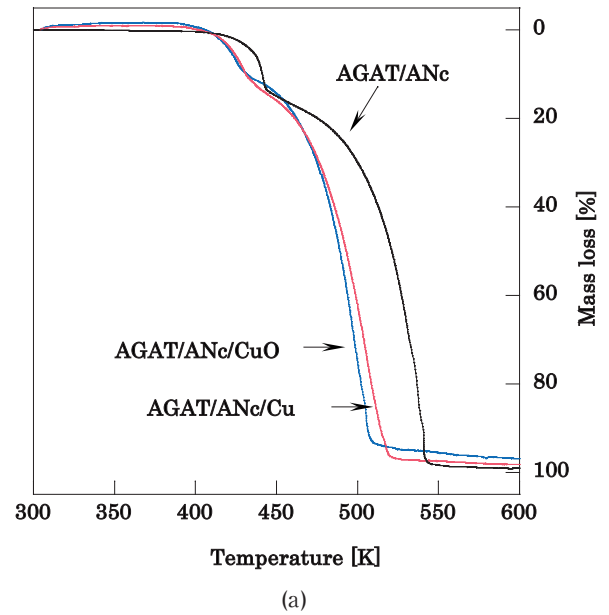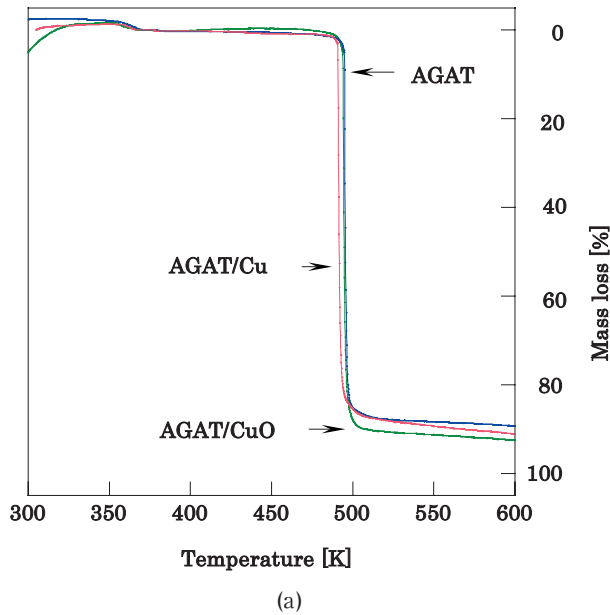
(a)

(b)

Figure 7 TG (a) and DTA curves (b) of AGAT, AGAT/Cu, and AGAT/CuO.

(a)

(b)

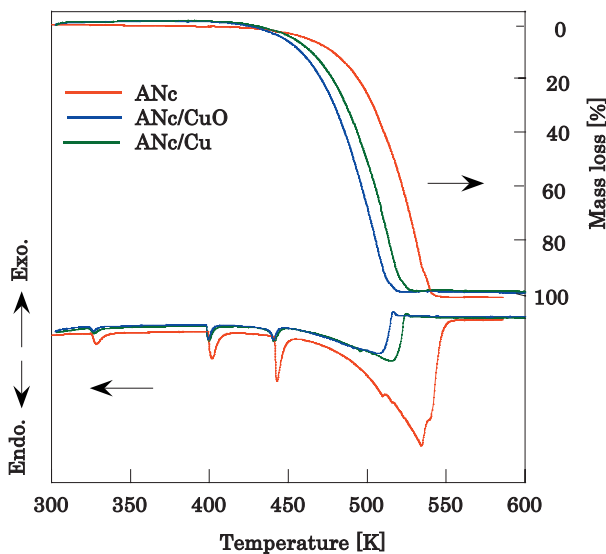**Figure 9** TG (a) and DTA curves (b) of AGAT/ANc, AGAT/ ANc/Cu, and AGAT/ANc/CuO.

Figure 8 TG and DTA curves of ANc, ANc/Cu, and ANc/ CuO.

CuO over Cu toward the decomposition of AGAT/ANc contradicts their respective contributions to the burning rate as their burning rates were relatively the same. This observation suggests that the condensed phase reaction zone may not control the burning rate.

3.4 Temperature profile

Figure 10 shows the temperature profiles at 2 MPa for the AGAT/ANc mixtures with and without additives. At this pressure, the burning rates of the AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO mixtures were 1.74 (2.03 MPa), 2.68 (1.98 MPa), and 2.58 mm·s^{-1} (2.10 MPa), respectively.

In the condensed phase zone, where the temperature increased from the initial temperature (To) to the decomposition temperature (Tu), no chemical reactions occurred. In the condensed phase reaction zone, where the temperature increased from Tu to the burning surface temperature (Ts), the phase change from solid to liquid
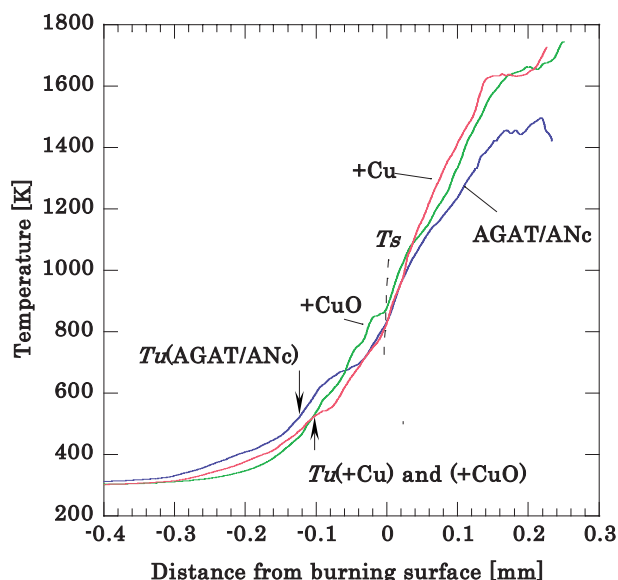
Figure10 Temperature profiles of AGAT/ANc mixtures with and without additives at 2 MPa.

and/or to gas occurred and reactive gaseous species formed, accompanied by endothermic or exothermic reactions[21].

The Tu of the three mixtures was approximately the same (~540 K). This is because no chemical reactions occurred in the condensed phase, and consequently, the effect of condensed phase reactions is negligible up to Tu.

Sabadel's method[22] cannot be used to obtain Ts of the AGAT/ANc mixtures. This is because the first deviations from linearity in their $\log(T\text{-}To)$ versus distance plots were closer to Tu than to Ts. In this study, Ts was measured using the change in the slope of the temperature profile. The Ts values of AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO were approximately 831, 831, and 879 K, respectively.

The distances between Tu and Ts for AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO were approximately 0.12, 0.10, and 0.10 mm, respectively. The temperature differences between Tu and Ts for the AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO mixtures were approximately 290, 290, and 340 K, respectively. In the region between Tu and Ts, the temperature of the AGAT/ANc/CuO mixture was, on average, higher than that of AGAT/ANc. This observation suggests that CuO might activate exothermic reactions in the Tu-Ts region.

It is reported that the condensed phase reaction zone controls the burning rate of fuel-rich AGAT/AN[12], and it is surmised that approximately 25% of the AGAT reacts with AN in the gas phase and that the remaining 75% possibly decomposes alone, producing heat in the condensed phase reaction zone, thereby controlling the burning rate.

The temperature gradients above the burning surface (dT/dx) of AGAT/ANc, AGAT/ANc/Cu, and AGAT/ANc/CuO were approximately 4.1, 7.3, and 5.8 K·m⁻¹, respectively.

From these results, we hypothesize that the heat feedback from the gas phase reaction zone controls the burning rate.

The burning rates of AGAT/ANc with Cu and CuO were almost similar, as shown in Figure 4. The addition of Cu and CuO increased the heat feedback from the gas phase reaction zone and increased the burning rate. Moreover, the addition of CuO might activate the exothermic reactions in the condensed phase reaction zone and compensates for the smaller heat feedback from the gas phase reaction zone than that of Cu.

4. Conclusions

From the combustion experiments on the mixtures of amino guanidinium 5,5′-azobis-1H-tetrazolate (AGAT) and ammonium nitrate coated with polyvinyl acetate (ANc), the following conclusions can be drawn.

1) The burnability of the mixture increased by coating ammonium nitrate with PVAc, and the stoichiometric AGAT/ANc mixture was observed to burn.

2) Close contact between a fuel (polyvinyl acetate) and an oxidizer (ammonium nitrate) may improve the burning behavior of ammonium nitrate.

3) Addition of Cu or CuO to AGAT/ANc mixtures increased the burning rate to values comparable to that of the guanidine nitrate/strontium nitrate/basic copper nitrate mixture, which is a gas-generating agent with practical applications.

4) The addition of Cu or CuO might increase the heat feedback from the gas phase reaction zone and increased the burning rate.

5) The addition of CuO might activate the exothermic reactions in the condensed phase reaction zone.

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