

Thermal decomposition mechanism of ammonium nitrate and copper (II) oxide mixtures

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Abstract

The purpose of this study is to develop an improved understanding of the thermal decomposition mechanism of ammonium nitrate (AN) and copper(II) oxide (CuO) mixtures. Analysis using differential scanning calorimetry (DSC) and evolved gas analysis based on thermogravimetry-differential thermal analysis-mass spectrometry (TG-DTA-MS) were carried out and decomposition behavior was observed using a hot stage with a CCD camera. DSC results showed that AN/CuO mixture had a higher exothermic onset temperature than pure AN and that CuO does not promote the decomposition of AN. From TG-DTA-MS results, the endothermic reaction of AN/CuO mixture has its onset after the AN melting point of 442 K and results in the evolution of H₂O. At approximately 500 K or above, AN/CuO mixture generates significant quantities of N₂ and H₂O and an exothermic reaction begins to offset the endothermic processes. Above 510 K, AN/CuO mixture began to evolve NO₂. Observations with a CCD camera have found that AN/CuO mixture transforms from a mixture of colorless liquid AN and black CuO powder into a blue-green liquid which is presumably a solution of copper (II) nitrate. Around 493 K, this liquid changes to a deep blue color due to the presence of a copper (II) amine complex. Finally, above 510 K, the deep blue liquid briefly turns blue-green, then changes into a black powder with the evolution of gas. Based on these results, a decomposition mechanism for AN/CuO mixture is proposed, in which the mixture reacts to form copper (II) nitrate and a copper (II) amine complex following melting and these species then undergo exothermic decomposition to generate N₂, H₂O, NO₂ and CuO.

Keywords : ammonium nitrate, copper (II) oxide, thermal decomposition, thermal analysis, evolved gas analysis

1. Introduction

Ammonium nitrate (AN) is widely used as a fertilizer and as an ingredient in industrial explosives or oxidizing chemical mixtures, because it is relatively cheap, it releases almost 100% gaseous products when it reacts, and it has a positive oxygen balance (+20.0 g g⁻¹). Recently, mixtures of AN with suitable combustibles have been proposed as gas generating agents in automotive air-bag systems or next-generation halogen-free propellants¹.

Unfortunately, tragic accidental explosions involving AN have occurred in the past^{2,3}, including recent incidents at the West Fertilizer Company storage facility in West Texas⁴, and at an AZF plant in Toulouse⁵.

For the safe development and use of AN-containing devices, it is important to understand the physical and

chemical properties as well as reaction mechanisms of such mixtures. Previously, the authors have studied the thermal properties of AN mixtures with various carbons and with copper (II) oxide (CuO) as well as the thermal decomposition mechanism of AN/carbon combinations, which serve as a simple example of solid reducing agent and metallic oxide catalyst mixtures⁶⁻⁹.

Improving the combustion performance of AN is a major challenge associated with the development of AN-containing devices, propellants, and gas generating agents, so efforts have been directed towards increasing performance by the addition of catalysts or reducing agents¹⁰⁻¹³. Metallic oxides are well known as combustion catalysts, and CuO has been reported to accelerate the combustion of AN compositions¹¹. However, the associated

Table 1 Physical properties of AN and CuO.

	Ave. particle diameter [μm]	Assay [%]	Density [kg m^{-3}]
AN	140	>99	260
CuO	5	>99.9	–

mechanism is not well understood. Thus, an improved understanding of the effects of metallic oxides, including CuO on the combustion and thermal decomposition of AN mixtures and the associated reaction mechanism are needed to improve the combustion performance of AN mixtures.

The aim of this study was to evaluate the effects of CuO on the thermal decomposition of AN and to determine the thermal decomposition mechanism of AN and CuO mixtures (AN/CuO mixture). The thermal decomposition properties of AN/CuO mixture were investigated using differential scanning calorimetry (DSC) and the effects of the CuO on the evolved gases were explored using thermogravimetry-differential thermal analysis-mass spectrometry (TG-DTA-MS). Furthermore, the thermal decomposition behaviors were observed using a hot stage and a CCD camera.

2. Experimental

2.1 Materials

Commercially available powdered explosive grade AN (Mitsubishi Chemical Corporation Co., Ltd.) and powdered CuO (Kojundo Chemical Lab. Co., Ltd.) were used in this study. Table 1 gives the physical properties of AN and CuO. AN/CuO mixture was carefully prepared by hand-mixing of these two ingredients at a 90/10 mass ratio between AN and CuO.

2.2 Thermal analysis

Mettler Toledo HP DSC827e differential scanning calorimeter (DSC) was used to study thermal behaviors of AN and AN/CuO mixture samples. Samples of approximately 1.5 mg were placed in stainless steel sealed pans under 0.1 MPa atmospheric air pressure and heated from 303 to 623 K at a heating rate of 5 K min^{-1} . The onset temperature of the exothermic or endothermic peaks and the heat of reaction were determined.

2.2 Evolved gas analysis

Rigaku TG8120 thermogravimetry-differential thermal analysis (TG-DTA) unit was connected to a Shimadzu GC-2010 mass spectrometer (MS) for non-isothermal and isothermal gas analysis studies. The TG-DTA-MS data were acquired simultaneously to study thermal behavior of the AN and AN/CuO mixture samples and to identify the gases evolved while the samples were heated. Samples with masses of approximately 3.0 mg were placed in open aluminum pans and heated from 303 to 623 K at a heating rate of 10 K min^{-1} in helium. The system was simultaneously purged with 200 mL min^{-1} of helium. The

mass spectrometer was operated in electron impact ionization mode, with selected ion monitoring at $m/z = 16, 17, 18, 28, 30, 32, 44,$ and 46 .

2.3 Visual observations of decomposition behavior

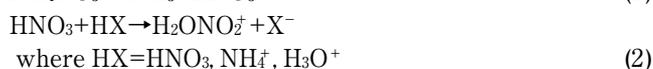
Apparatus consisting of a Mettler Toledo FP84HT hot stage connected to a FP90 heat controller and a CCD camera was used in this study. Approximately 3.0 mg samples were placed in open aluminum pans and heated from 303 to 623 K at a heating rate of 10 K min^{-1} . The decomposition behavior of each sample was visually observed using the CCD camera.

3. Results and discussion

3.1 Thermal analysis

The sealed cell DSC profiles obtained for AN and AN/CuO mixture are shown in Figure 1. The onset temperature, maximum heat flow, and heat of reaction are summarized in Table 2. AN/CuO mixture had approximately the same heat of reaction and a slightly higher value of maximum heat flow compared to pure AN, in addition to a higher onset temperature.

AN melts at 442 K and begins to decompose just after it melts¹⁾. Oxley et al. showed that the first stage of AN decomposition is a dissociation into NH_3 and HNO_3 according to Equation (1), and the subsequent step is oxidation of NH_3 by the HNO_3 decomposition products according to Equations (2)–(4)¹³⁾.



Because CuO is a basic oxide, it readily reacts with a strong acid HNO_3 to form copper nitrate and this

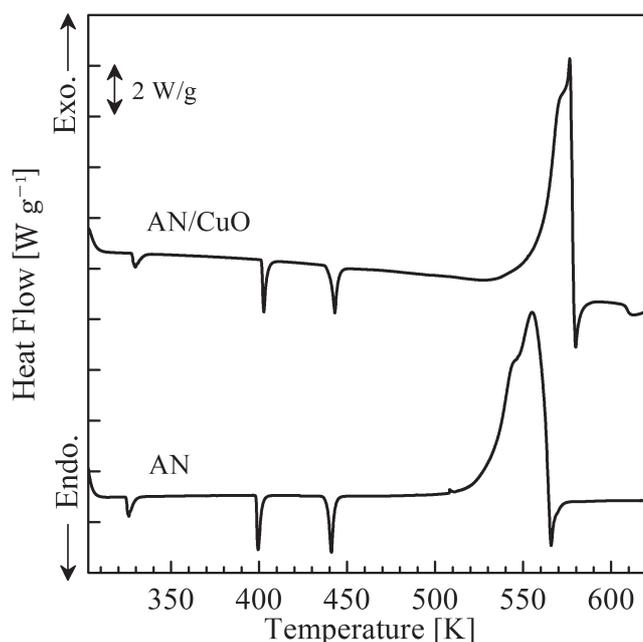


Figure 1 SC-DSC profiles of AN and AN/CuO mixture at 5 K min^{-1} .

Table 2 Thermal properties of AN/CuO mixture from SC-DSC results.

	Onset temp. [K]	Heat of reaction [kJ g ⁻¹]	Max heat flow [W g ⁻¹]
AN	533	1.6	6.8
AN/CuO	551	1.5	8.2

neutralization decreases the quantity of free HNO₃. Decreasing the free HNO₃ slows the initiation of AN decomposition according to Equation (2) and this is believed to be the reason in which AN/CuO mixture exhibited a higher onset temperature than AN.

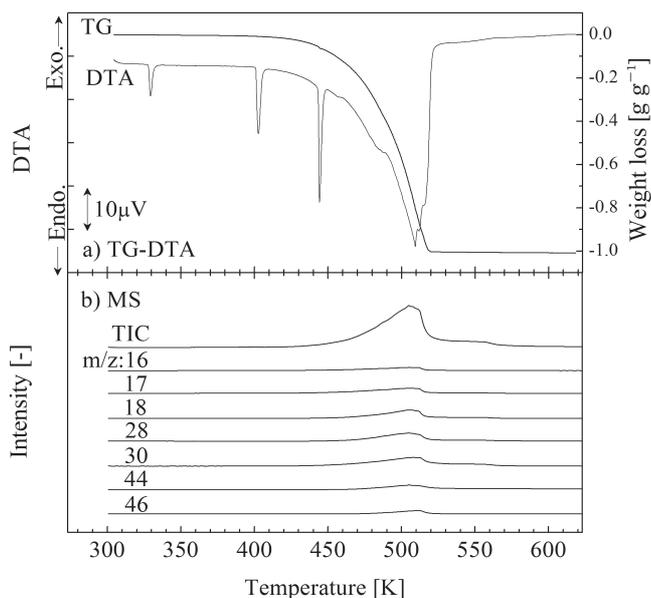
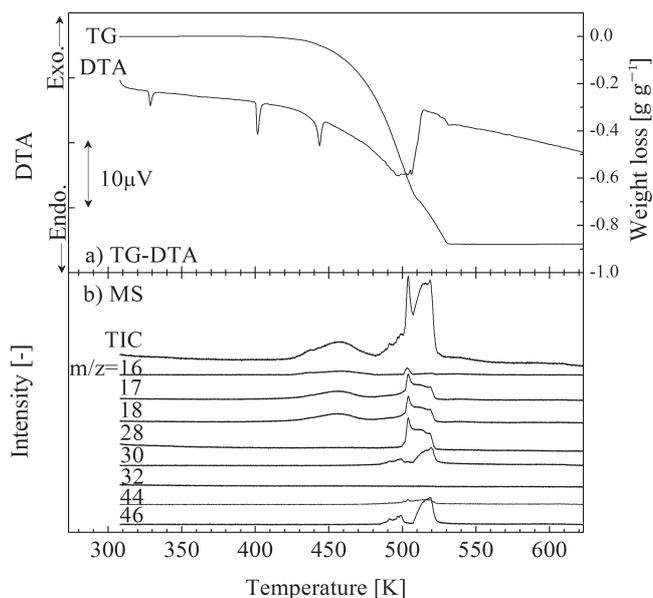
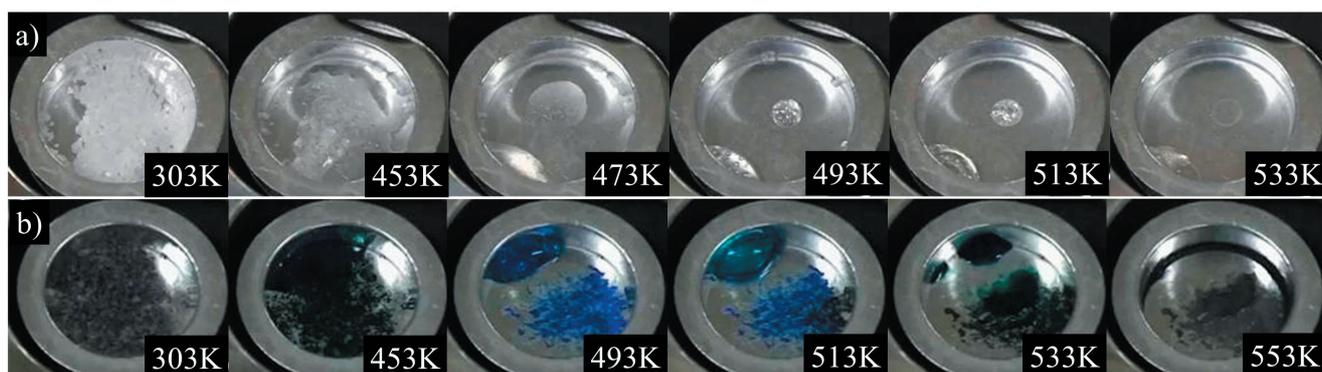
3.2 Evolved gas analysis and visual observation

The TG-DTA-MS profiles obtained for pure AN are shown in Figure 2. During the thermal decomposition of AN, an endothermic reaction was observed to continue after melting. The endothermic peak and accompanying weight loss were due to the dissociation of AN and the subsequent vaporization of NH₃ and HNO₃. Because AN

weight loss ratio reaches the final value of -1.0 as shown in Figure 2 a), it is assumed to have gasified completely. With regard to evolved gases, main products were NH₃ ($m/z = 17$ and 16), HNO₃ ($m/z = 46, 30, 18, 17, 16,$ and 28)¹⁴, N₂O ($m/z = 44, 30,$ and 28), H₂O ($m/z = 18$ and 17) and N₂ ($m/z = 28$). NH₃ and HNO₃ were generated from the dissociation of AN according to Equation (1) and subsequently vaporized, and N₂O, H₂O, and N₂ have been reported as the main AN decomposition gases¹. CCD camera images of the thermal decomposition behavior of AN as shown in Figure 3 a), in which the AN was observed to melt into a colorless liquid before undergoing gasification starting at 442 K. The gasification continued up to approximately 530 K, at which point no residue was observed on the sample pan.

Meanwhile, AN/CuO mixture exhibited different decomposition and gasification behaviors from the pure AN. The TG-DTA-MS profiles obtained with AN/CuO mixture are shown in Figures 4 and 5, and the CCD camera images of its thermal decomposition are presented in Figure 3 b).

The TG-DTA-MS results for AN/CuO mixture shows an endothermic reaction that continued above the melting

**Figure 2** a) TG-DTA and b) MS results for AN heated in helium at 10 K min⁻¹.**Figure 4** a) TG-DTA and b) MS results for AN/CuO mixture heated in helium at 10 K min⁻¹.**Figure 3** CCD camera images of the thermal decomposition behaviors of a) AN and b) AN/CuO mixture heated on a hot stage at 10 K min⁻¹.

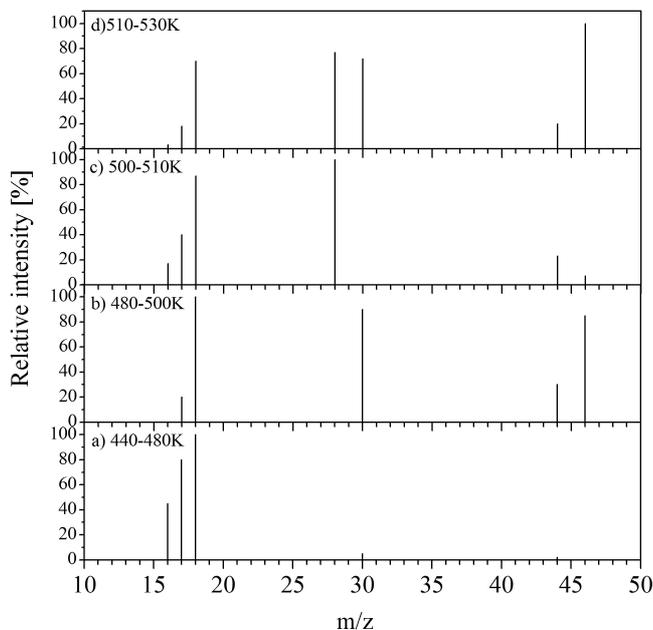


Figure 5 Averaged mass spectra of evolved gas from AN/CuO mixture over the temperature ranges of a) 440–480 K, b) 480–500 K, c) 500–510 K, and d) 510–530 K.

point of 442 K, just as it was observed with pure AN. Between 440 and 480 K, AN/CuO mixture evolved H_2O ($m/z = 18, 17$) and NH_3 ($m/z = 17, 16$) but not HNO_3 , as can be seen by comparing results as shown in Figure 5 a). We suggest that this result is due to a reaction which evolved H_2O and consumed HNO_3 occurred in sample pan. These results also demonstrate that NH_3 was vaporized similarly as AN, as was the case for pure AN. The image acquired at 456 K (Figure 3 b)) shows that AN/CuO mixture transformed from a mixture of colorless liquid AN and black CuO powder to a blue-green liquid, due to gradual melting of CuO. This is not unexpected, since it is well known that basic oxide CuO will react with a strong acid HNO_3 to form copper (II) nitrate $[\text{Cu}(\text{NO}_3)_2]$ according to Equation (5) and that anhydrous $\text{Cu}(\text{NO}_3)_2$ is deep blue-green in color¹⁵. Based on these results, AN and CuO are thought to have reacted to form $\text{Cu}(\text{NO}_3)_2$ and H_2O as shown in Equation (5).



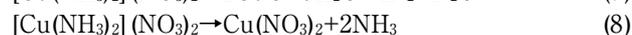
In the TG-DTA-MS results shown in Figures 4 and 5 b), NH_3 is not found in the gaseous products generated between 480 and 500 K and thus the concentration of evolved NH_3 gas has decreased within this temperature range. In the image obtained at 493 K (Figure 3 b)), the blue-green liquid is seen to transform into a deep blue color, which matches the color of copper (II) amine complex^{16,17}. Morozov et al. reported that $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ was produced from a mixture of $\text{Cu}(\text{NO}_3)_2$ and AN upon heating in the range of 433 to 493 K¹⁸. Thus, we conclude that $\text{Cu}(\text{NO}_3)_2$ reacted with NH_3 to form the copper (II) amine complex $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ according to Equation (6).



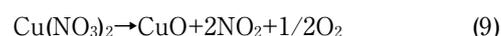
Basic copper (II) nitrate ($\text{Cu}_2(\text{OH})_3\text{NO}_3$) has also been reported as an intermediate formed during AN/CuO mixture decomposition¹⁹. Dyukarev et al found that $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ hydrolyzed to basic copper (II) nitrate and AN¹⁷. We therefore believe that the presence of H_2O derived from the decomposition of AN or impurity in the samples accelerated the formation of basic copper (II) nitrate.

At temperatures over approximately 500 K, AN/CuO mixture evolved a significant quantity of gaseous products, as seen in the MS results shown in Figures 4 and 5 c). N_2 ($m/z = 28$) and H_2O ($m/z = 18, 17$) were evolved along with minor amounts of N_2O ($m/z = 44, 28$) and NH_3 ($m/z = 17, 16$). According to the image obtained at 513 K (Figure 3 b)), gasification is seen to be more active, and according to the TG-DTA result shown in Figure 4 a), an exothermic reaction began to offset the endothermic reactions associated with volatilization and dissociation of AN.

Southern et al indicated that $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ exothermically decomposed to form CuO, H_2O , N_2 , N_2O , and minor amount of NO and NO_2 according to Equation (8)²⁰. Dyukarev et al. reported that $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ also dissociated to $\text{Cu}(\text{NO}_3)_2$ and NH_3 under high vacuum (10^{-4} Pa) according to Equation (9)¹⁷. These results suggest that the $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ in AN/CuO mixture has undergone exothermic decomposition according to Equations (7) and (8). We suggested that $[\text{Cu}(\text{NH}_3)_2](\text{NO}_3)_2$ was subsequently regenerated from CuO and $\text{Cu}(\text{NO}_3)_2$ according to Equations (5) and (6), and such reactions according to Equations (5), (6), (7), and (8) proceed cyclically.



At temperatures over 510 K, AN/CuO mixture is seen to evolve NO_2 ($m/z = 46, 30$) according to the TG-DTA-MS results presented in Figures 4 and 5 d) and the images obtained at 513 and 553 K (Figure 3 b)), AN/CuO mixture transforming from a deep-blue liquid through an intermediate blue-green liquid and finally into a black powder. The blue-green liquid in the intermediate stage is believed to be $\text{Cu}(\text{NO}_3)_2$. It is well known that metallic nitrates decompose into the corresponding metallic oxides along with NO_2 and O_2 ²¹). In this study, however, O_2 ($m/z = 32$ and 16) was not detected as a product and thus we suggest that any O_2 that was generated has reacted with reducing gases, such as NH_3 , to form NO_2 , N_2O , NO, and H_2O . Therefore, $\text{Cu}(\text{NO}_3)_2$ would have decomposed into NO_2 and CuO according to Equation (9).



In the TG-DTA results as shown in Figure 4 a), the weight loss ratio of AN/CuO mixture plateaus at a final value of approximately 0.9. The remaining residue was approximately 10 % by weight of the initial weight, which is equal to the initial weight of CuO. Based on these results, it was considered that the added CuO worked catalytically and thus is eventually recovered as a solid residue CuO.

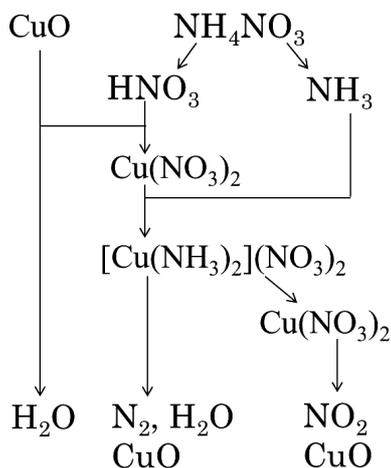


Figure 6 Proposed thermal decomposition mechanism for AN/CuO mixture.

3.3 Proposed thermal decomposition mechanism of AN/CuO mixture

Figure 6 shows a reaction scheme for the thermal decomposition of AN/CuO mixture, based on this study. In the first step, HNO₃ and NH₃ are formed by the dissociation of AN above its melting point of 442 K and according to Equation (1)¹⁾, followed by CuO reacting with HNO₃ to form Cu(NO₃)₂ and H₂O, as summarized in Equation (5). The TG-DTA-MS results indicated the evolution of H₂O but not HNO₃, and hence a reaction which consumes HNO₃ and evolves H₂O must have occurred. From the CCD camera images, AN/CuO mixture was found to have transformed into a blue-green liquid that matches the color of anhydrous Cu(NO₃)₂¹⁵⁾. We therefore conclude that CuO reacted with HNO₃ to form Cu(NO₃)₂, H₂O, and NH₃ according to Equation (5) in the initial stage. This reaction decreases the concentration of free HNO₃ in the molten AN and hence interferes with the initiation of AN decomposition according to Equation (2). For this reason, it is suggested that AN/CuO mixture had a higher onset temperature than pure AN in SC-DSC results.

In the second stage, Cu(NO₃)₂ and NH₃ react to form copper (II) amine complex, [Cu(NH₃)₂](NO₃)₂. The TG-DTA-MS results indicated that NH₃ was not produced at the temperature range between 480 and 500 K, and thus we propose that NH₃ reacted with Cu(NO₃)₂ to form copper (II) amine complex in the liquid phase. The CCD camera images indicated the transition of a blue-green liquid into a deep blue liquid at approximately 493 K, which is in good agreement with the color of the copper (II) amine complex^{16),17)}. In addition, Morozov *et al.* reported that [Cu(NH₃)₂](NO₃)₂ was produced by heating a mixture of Cu(NO₃)₂ and AN to between 433 and 493 K¹⁸⁾. From these results, we propose that Cu(NO₃)₂ reacted with NH₃ to form [Cu(NH₃)₂](NO₃)₂ according to Equation (6) in the second stage of the mechanism.

In the third stage, [Cu(NH₃)₂](NO₃)₂ exothermically decomposes into Cu(NO₃)₂, CuO, and various gases, *i.e.*, N₂, H₂O, N₂O and NH₃. The TG-DTA-MS results indicate that AN/CuO mixture evolves a significant quantity of gaseous at above approximately 500 K. From the MS result, major

part of evolved gases were N₂ and H₂O. The TG-DTA results also demonstrate that an exothermic reaction begins to offset the endothermic processes consisting of volatilization and dissociation of AN. Southern *et al.* reported that N₂ and H₂O are the primary gases evolved from [Cu(NH₃)₂](NO₃)₂ and that the decomposition of this compound proceeds by an exothermic reaction under atmospheric pressure¹⁹⁾. CCD camera images show that gasification occurs at this stage as the deep blue liquid gradually transforms into a blue-green liquid and a black powder mixture, which are believed to represent Cu(NO₃)₂ and CuO, respectively. We thus conclude that [Cu(NH₃)₂](NO₃)₂ exothermically decomposes into Cu(NO₃)₂, CuO, and various gases (N₂, H₂O, N₂O and NH₃) according to Equation (7) in the third stage.

Finally, Cu(NO₃)₂ decomposes to produce CuO and NO₂. From the TG-DTA-MS results, AN/CuO mixture begins to evolve NO₂ at temperature over 510 K and this is not unexpected since it is well known that Cu(NO₃)₂ decomposes to CuO, NO₂ and O₂ according to Equation (9)²¹⁾. Photographic images show that the blue-green liquid changes into a black powder which is presumably CuO, and thus we propose that Cu(NO₃)₂ decomposes to CuO and NO₂.

4. Conclusions

The thermal decomposition characteristics of AN and AN/CuO mixture were examined using DSC, the evolved gases were analyzed with TG-DTA-MS, and the decomposition behavior was visually observed using a hot stage and a CCD camera. The following conclusions can be drawn :

- 1) In a closed pan, pure AN and AN/CuO mixture exhibited approximately equivalent heats of reaction during SC-DSC analysis, although AN/CuO mixture had a higher onset temperature than AN.
- 2) In an open pan, AN showed only endothermic peaks during TG-DTA analysis, whereas AN/CuO mixture had an exothermic peak following an endothermic peak, with onset at approximately 500 K.
- 3) AN completely gasified, but AN/CuO mixture left a residue representing approximately 10 % by weight of the original mixture. This weight was equal to the initial weight of CuO in the sample.
- 4) AN/CuO mixture showed different gas evolution behavior than pure AN. AN/CuO mixture evolved H₂O after its melting point, and also N₂ and H₂O with small amount of N₂O and NH₃ at 500 K, and NO₂ after 510 K.
- 5) AN/CuO mixture transformed from a mixture of a colorless liquid and a black powder into a blue-green liquid after AN melting point of 442 K. This liquid generated significant gaseous products at approximately 500 K and transformed into a black powder residue via a blue-green liquid above approximately 510 K.
- 6) CuO reacts with AN to form Cu(NO₃)₂ and [Cu(NH₃)₂](NO₃)₂ as intermediate species.

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硝安／酸化銅 (II) 混合系の熱分解機構解析

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本研究は硝酸アンモニウム/酸化銅 (II) 混合系 (AN/CuO) の熱分解機構を解明することが目的である。DSCを用いた熱分解特性試験, TG-DTA-MSを用いた熱重量—生成ガス分析および加熱ホットステージとCCDカメラを用いた熱分解挙動の観察を行った。DSCの結果より, AN/CuO混合物の発熱開始温度はAN単体よりも高く, CuOはANの初期分解を促進しなかった。TG-DTA-MS結果より, AN/CuO混合物は融点442 Kから吸熱を開始し, 水を生成した。続いて, 約500 K以上で発熱を伴い窒素と水を多量に生成し, 510 Kからは二酸化窒素が生成した。CCDカメラによる観察結果より, 融点後に無色の液体ANと黒色粉末CuOの混合物は硝酸銅(II)と考えられる青緑色の液体に変化した。続いて, 約493 KにおいてAN/CuO混合物はアンミン銅錯体と考えられる深青色の液体に変化した。510 K以上でAN/CuO混合物はガス発生を伴い青緑色液体を経由し, 最終的に黒色粉末に変化した。これらの結果より, AN/CuO混合物はAN融解後に硝酸銅(II)およびアンミン銅(II)錯体を形成し, それらが発熱的に窒素, 水, 二酸化窒素およびCuOに分解生成する機構を提案した。

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