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Safety Evaluation on Compound by Thermal Analysis

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Abstract: By the using of TGA and DTA technique, the thermal decomposition of a kind of ignition powder, which contains $KClO_3$, C and DDNP, was investigated. The results show that the whole reaction consists of three parts, that this reaction mechanism belongs to uneven distributed solid phase reaction. In order to control this kind of reaction completely, first of all is to avoid the initiating decomposition reaction.

Keywords: TGA; DTA; Thermal decomposition; Mechanism

1 Introduction

Under the function of outside energy, ignition powder can be easily ignited. Its function is to accept outside energy and transit it into other kind of energy combustion heat energy or shock wave energy, then transmit this kind of energy to initiating charge or explosive and initiate them.

In the process of production, storage, transportation or using of energetic material, there must be some kinds of stimulation by outside energy. Of all these kinds of energies, heat energy can be seen everywhere and most of the explosive accidents are arisen by it. Of all kinds of energetic materials, the initiating charge is the most sensitive one to heat.

In recent years, thermal analysis was often applied to the study of the decomposition characteristics of energetic materials. In this study, we combined the TGA and DTA to investigate the thermal decomposition of the mixture that contains $KClO_3$, C and DDNP. The results can be useful in the usual use of this kind of energetic material.

2 Experiments

2.1 Materials

The sample used in this work consists of $KClO_3$, C and DDNP. This mixture, which was dissolved in a kind of solvent, first was provided by local correlative factory. Then, we dried it into powder under a relative low temperature. The dosage used in this experiment is 6.3933 mg.

2.2 Thermal Analysis

The experiment was carried out on a kind of thermal analysis equipment SDT2960 Simultaneous DSC/TGA that was produced by American TA Company with heating rate of 10℃/min. In the experiment, the powder to be analysed was put in the environment that only contains nitrogen. The comparative material used in the thermal analysis experiment is Al_2O_3.

3 Results and Discussion

The result is shown in Fig. 1. From this figure, we can see that the thermal decomposition of the powder mainly consists of two parts. During the first part, the peak temperature in the DTA curve is 158.50℃ and the weight loss is 5.541%; during the second part, the peak temperature in the DTA curve is 316.35℃ and the weight loss is 61.26%. When the temperature of this system is below 100℃, the weight loss of the powder is very small, so we can consider that there is no chemical reaction. With the temperature rising to 140 ℃ or so, we can evidently find that the reaction starts.

Corresponding to both the TG and the DTA curve, there appears an inflexion in the TG curve and a small heat release apex in the DTA curve. As the temperature still rises, the reaction speed of the powder also rises quickly up to a maximum when the temperature is about 310℃.

After the chemical reaction, the weight of the powder is 0.4437 mg, so the whole weight decrement during this progress is 5.9496 mg. Judging from the TG curve, we know that the weight reduces 4.0943 mg from 307.88℃ to 316.35℃ and the weight loss during this time holds 68.82% of the whole weight loss. Furthermore, we find that there is a large heat release apex in the DTA curve when the temperature is about 313℃. This phenomenon shows that the powder reacts very quickly at that time and releases a great amount of heat.

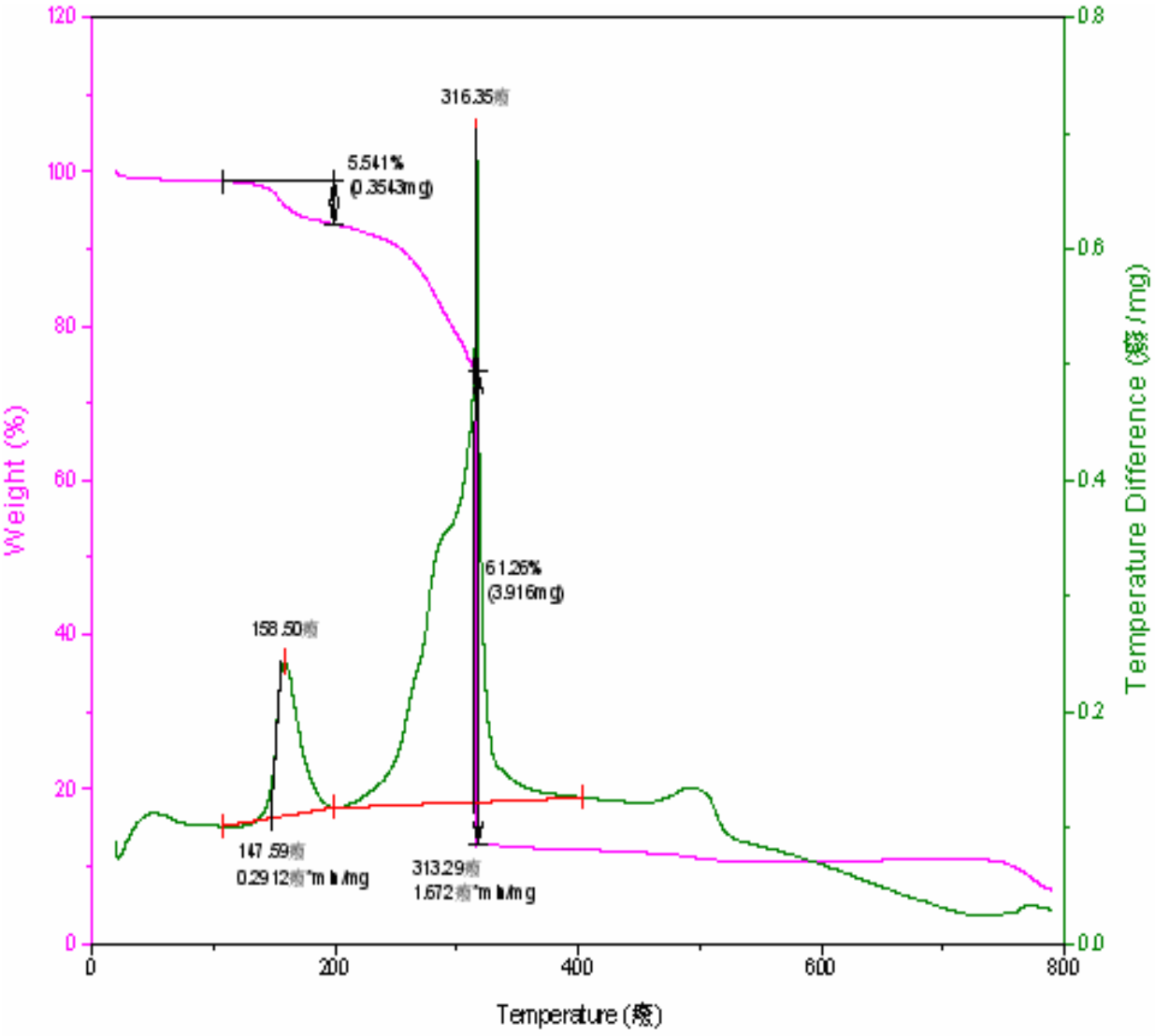

Fig. 1 TGA-DTA curves of the thermal analysis

By obtaining data from TG curve and basing on the formula $\alpha = (M_0 - M)/(M_0 - M_f)$, we can transfer the weight of the powder to the decomposition ratio of the powder. The results are listed in Table 1. Then we can gain the $\alpha \sim T$ curve of this whole chemical reaction progress. This figure is shown in Fig. 2.

The α in the upper formula represents the decomposition ratio of the powder, M_0 represents the initiating weight of sample, M_f is the sample weight after the testing and M is the weight during the progress.

Table 1 The decomposition ratio at each temperature

Temperature/℃	19.81	39.21	140.82	158.56	167.23	198.55	262.59	276.76
Decomposition ratio	0	0.0091	0.0203	0.0465	0.0577	0.0727	0.1213	0.1550
Temperature /℃	300.00	307.88	313.71	315.34	315.93	316.32	316.35	401.56
Decomposition ratio	0.2260	0.2522	0.2783	0.3083	0.6150	0.9253	0.9404	0.9440
Temperature /℃	451.88	496.61	750.52	756.73	767.95	773.87	780.04	788.20
Decomposition ratio	0.9478	0.9553	0.9627	0.9665	0.9778	0.9852	0.9927	0.9998

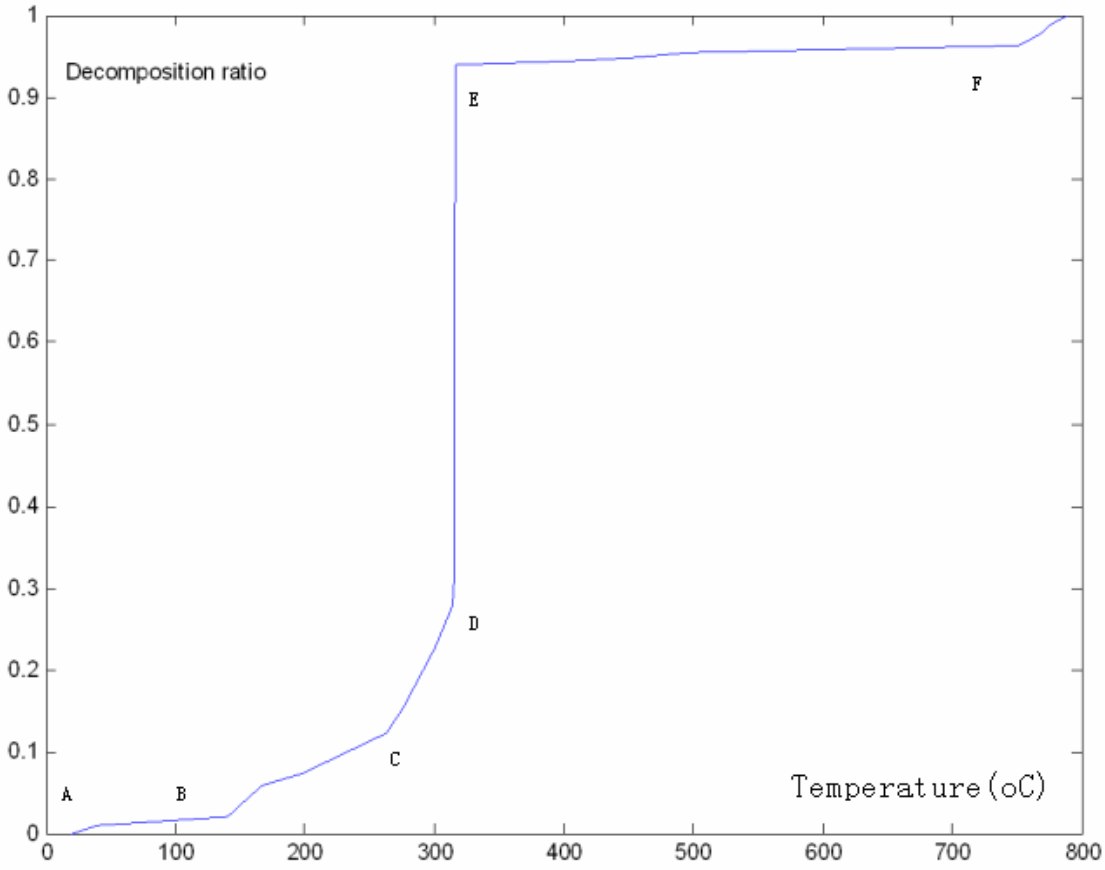

Fig. 2 The decomposition versus temperature

Judging from the above figure, we know that the thermal decomposition of this kind of powder is very complicated. This reaction mechanism belongs to uneven distributed solid phase reaction. Usually, this kind of reaction consists of three parts. The first step includes absorption and resolution. The second step is the reaction on the interface or inside the solid phase, but this reaction only takes place in several points. The three steps are the accumulation of hot reaction points and the formation of reaction nucleus. According to the reaction course of thermal decomposition, the reaction kinetics of solid thermal decomposition is decided by the speed of the formation, development and diffusion of nucleus. The active energy needed when the nucleus is formed is larger than that needed when the

nucleus is growing up. Once the nucleus is formed, the nucleus will can grow up and diffuse immediately.

In Fig. 2, the segment AB represents the dissolution of the gas that is absorbed by solid powder. Of all the ingredients in this kind of solid powder, DDNP is the most sensitive one to heat. Under the circumstance of 100℃ and after 48 hours, the reduction of DDNP is only 1.57%. When the conditional temperature is 155℃, this powder will be ignited in one minute. This temperature corresponds to the point B in Fig. 2. So the reaction speed at point B rises. BC segment represents the abduction progress and gas is slowly released during this period. Under normal circumstance, the ingredient $KClO_3$ begins to decompose into $KClO_4$ and KCl only when the temperature reaches 370℃. But the deduction period advances this decomposition. At point C, the reaction begins to accelerate until point D where the reaction speed is the peak speed. Owning to the high reaction speed, most of powder is consumed during DE. After point E, the reaction speed become very slow and we can approximately consider that the chemical reaction has completed at point E.

4 Conclusions

The thermal decomposition of this kind of powder accord with common that of the common pyrotechnics. This progress must undergo two processes. The first one is the initiating decomposition reaction and corresponds to the AC curve in Fig. 2. During this process, the reaction speed is decided by the minimum speed of solid powder at that temperature. The second one is the auto-catalyzed reaction and corresponds to the CE curve in Fig. 2. During this process, the reaction is accelerating all the time.

Of all the ingredients of this solid powder, DDNP reacts first. In order to control the chemical reaction of this kind of solid powder completely, the initiating decomposition reaction must be checked. According to the results obtained from this experiment, the environmental temperature must be lower than 140℃ lest the chemical reaction happens.

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