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Thermal behavior and combustion performance of Al/Bi$_2$O$_3$ nano thermites with the effect of potassium perchlorate

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Abstract

To study the effect of the addition of potassium perchlorate (KClO$_4$) on the performance of nano thermites, Al/Bi$_2$O$_3$ nano thermites with different mass fraction (wt%) of KClO$_4$ was made by ultrasonic dispersion method. Then, all samples were tested by SEM, DSC and rapid excitation ignition. DSC results showed Al/Bi$_2$O$_3$/10 wt%KClO$_4$ had the highest heat release. Compared with Al/Bi$_2$O$_3$, the heat release increased by 28.32%. The results of activation energy calculations showed Al/Bi$_2$O$_3$/30 wt%KClO$_4$ had the lowest activation energy (Ea). Its activation energy was reduced by 30%, mere about 149.17 KJ mol$^{-1}$. Finally, it can be seen from the combustion situation that the addition of KClO$_4$ reduced the intensity of burning. This work found a way to increase the heat release or reduce the activation energy of nano thermites by controlling the amount of KClO$_4$ added.

1. Introduction

The application of nanotechnology has led to the extensive development of metastable intermixed composites [1, 2]. All aspects of its performance have been greatly improved [3, 4]. As a typical representative, nano thermite has high energy density and good safety performance. It plays a key role in welding [5], gas generating agent [6], explosives [7] and propellants [8, 9].

The traditional thermite is composed of micron Al powder and a single oxide. After excitation, a violent oxidation-reduction reaction will take place and release huge energy. By reducing the particle size of the metal fuel and adopting advanced assembly technology, the combustion performance of the thermite has been greatly improved [10]. The traditional thermite has a single performance and can no longer meet the requirements of actual projects. In recent years, additives have been added to the thermite to improve certain aspects of performance. It has attracted the attention of many scholars [11–13]. Kim [14] and co-authors added paraffin wax to the Al/CuO system. The results showed that it is beneficial to increase the energy release during combustion and the long-term storage of the thermite under high humidity conditions. An important engineering application of thermite is the recycling of waste steel. Sapchenko [15] and co-authors controls the combustion temperature of the thermite reaction by adding inert additives such as KCl to the thermite. This improves the recycling efficiency of waste steel.

As a strong oxidant, KClO$_4$ is often used as a component of pyrotechnics and propellants [16, 17]. Yang [18] and co-authors used the solvent/ non-solvent method to coat a layer of KClO$_4$ on the surface of the Al/CuO nano thermite. The combustion experiment showed that after adding KClO$_4$, the combustion rate of the nano thermite was significantly improved. Ishitha [8] and collaborators added ammonium perchlorate (NH$_4$ClO$_4$) to the solid propellant. The results showed that greatly increased the burning rate and pressure. At the same time, it needs to be pointed out that both NH$_4$ClO$_4$ and KClO$_4$ can decompose and release oxygen under high temperature conditions [19].

Bismuth oxide (Bi$_2$O$_3$) can react with Al powder and has a higher theoretical calorific value [20]. Wang [21] and co-authors prepared seven different doped δ- Bi$_2$O$_3$. They studied the role of oxygen ion migration in Bi$_2$O$_3$.
in the control mechanism of the thermite ignition reaction. Yi [22] and co-authors assembled carbon fiber with Al/Bi₂O₃ nano thermites. The high energy of Al/Bi₂O₃ combustion and the conductivity of carbon fiber are combined. It explores a way to detonate RDX without detonator and bridge membrane. It provides a basis for the research of nano thermites based on Al/Bi₂O₃.

The common methods for assembling nano thermites include ultrasonic mixing method, reaction inhibition ball milling method (ARM), sol-gel method and so on [10, 11, 23, 24]. The particle size distribution of nano thermites prepared by the ARM is uneven [25]. The sol-gel method cannot be applied to large-scale production. In contrast, the ultrasonic dispersion method is simple in operation and mature in technology and can be used for large-scale production [26].

In this article, Al/Bi₂O₃ nano-thermite with different mass fractions of potassium perchlorate was prepared by ultrasonic dispersion method. The samples were tested by Field Emission Scanning Electron Microscope (FE-SEM) to characterize their distribution. Differential Scanning Calorimetry (DSC) was carried out at different heating rates to study its activation energy and heat release. Finally, Ni-Cr wire ignition experiment was implemented to study its combustion performance.

2. Experiments

2.1. Materials

All chemicals were analytical reagent grade and could be used without further treatment or purification. Among them, Al nanoparticles (about 100 nm) were purchased from Aladdin Industrial Co., Ltd. (Shanghai, China). The high-energy additive, KCIO₄ was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Nano-Bi₂O₃ powder was purchased from Chengdu Kelong Chemical Co., Ltd. (Sichuan, China). In the experiment, both absolute ethanol and deionized water were used as solvent and dispersant.

In figure 1, it is the SEM of the nano-Bi₂O₃. The particle size histogram is produced by using the Sturges method [27]. The sum of N = 490 particle sizes is obtained by using Image-J software to take a point measurement on the SEM image. According to the Sturges formula, the grouping number k = 1 + 3.322 log

Figure 1. Electron microscopy and particle size distribution of Bi₂O₃. (a)-(c) SEM of Bi₂O₃ samples at different multiples, (d) Particle size distribution of Bi₂O₃.
(N) of the histogram can be obtained. The fitted curve approximately satisfies $D_0 = D \exp(\sigma^2/2)$, where the median size ($D$) is approximately 0.2257 µm, and the polydispersity parameter ($\sigma$) is approximately 0.0234 µm. The particles' surface is smooth and doesn’t have apparent damage marks. The agglomeration is not apparent.

2.2. Precursor preparation
The mass of each group was controlled at 200 mg. The Al powder contained in it should be excessive. For example, there was the preparation process of sample B. 45 mg nano Al powder and 135 mg nano Bi$_2$O$_3$ were dispersed in 5 ml deionized water and 20 mg KClO$_4$ was dissolved in 3 ml absolute ethanol. After stirring, they were mixed in the same beaker. Subsequently, the mixture was ultrasonically dispersed for 10 min to obtain a uniformly dispersed precursor suspension. The remaining groups were prepared separately according to the data in table 1, referring to the process of sample B.

2.3. Ultrasonic dispersion process
As shown in figure 2, the mass fraction of KClO$_4$ contained in each group was different. The four groups of samples were divided into four beakers and dispersed by ultrasound for ten minutes.

The reactant particles collided and mixed under the action of ultrasonic waves and finally reached the level of uniform mixing. Then, the sample was transferred to the drying box and dried at 60 °C for 12 h. Finally, the thermite samples with different mass fractions of KClO$_4$ were obtained.

2.4. Material characterizations and tests
The particle size and morphology analysis of the Bi$_2$O$_3$ and thermite were tested by FE-SEM technology (HITACHI High-Technologies corporation, S-4800 II Japan).

The DSC analysis (NETZSCH STA, 449F3, Germany) was implemented to investigate the thermal behavior of the nano thermites. All tests were carried out at heating rates of 5, 10, 15 and 20 °C min$^{-1}$ under the condition of argon gas. The temperature range was from 25 °C to 800 °C. Based on the data obtained from the test, the Kissinger method and Ozawa method were used to calculate the activation energy.

The ignition device was built using Ni-Cr wire, DC power, and high-speed camera. The sample was ignited and the whole process of burning was recorded.

2.5. Theoretical backgrounds
The typical method of thermal analysis kinetics is the Kissinger method [28], which is a kind of differential method.

The typical integration method is Ozawa method [29]. Compared with the traditional differentiation method, it avoids the approximate treatment of conversion rate and the selection of reaction mechanism function. Therefore, it can directly obtain the activation energy value with higher accuracy. The calculation results of the two methods confirm each other.
The basic formula of the Kissinger method is:

\[
\ln \left( \frac{\beta}{T_p^2} \right) = \ln \left( \frac{AR}{E} \right) - \frac{E}{RT_p} \tag{1}
\]

The basic formula of the Ozawa method is:

\[
\log \beta = \log \left( \frac{AR}{RG(\alpha)} \right) - 2.315 - 0.4567 \frac{E}{RT_p} \tag{2}
\]

Where \( \alpha \) is conversion rate, \( \beta \) is the linear heating rate in differential scanning calorimetry (\( ^\circ \text{C min}^{-1} \)), \( T_p \) is the absolute temperature (K), \( R \) is the general gas constant, \( A \) is the pre-factor, \( E \) is the activation energy of the mixture ignition reaction (J mol\(^{-1}\)).

3. Results and discussion

3.1. SEM of nano-thermites

Figure 3 is the SEM of the nano-thermites sample. Figure 3(a) is a mixed sample without KClO\(_4\). The Al powder and Bi\(_2\)O\(_3\) are both spherical, and the particle size of the Al powder is smaller than that of Bi\(_2\)O\(_3\). There is still a certain agglomeration in the thermite sample assembled by the ultrasonic dispersion method. This may be due to the high humidity in the storage environment of the sample, which leads to an increase in the adhesion between the particles and a certain agglomeration phenomenon. However, the distribution of Al powder and Bi\(_2\)O\(_3\) is relatively uniform, which is conducive to the continued progress of the reaction after ignition.

In figures 3(b)–(d), the massive particles are noticeable. As the mass fraction increases, it can be seen the distribution range of KClO\(_4\) has expanded. It shows that after ultrasonic dispersion KClO\(_4\) is evenly distributed in the thermite, instead of being distributed in one area in clusters. A large amount of Al powder and Bi\(_2\)O\(_3\) adhere to the surface of KClO\(_4\), which is conducive to reaction and combustion.
3.2. Thermal properties analysis

Figure 4 shows the DSC curves of Al/Bi₂O₃ nano thermites with different mass fractions of KClO₄ at a heating rate of 10 °C min⁻¹.

Figure 4(a) shows the DSC curve of Al/Bi₂O₃. There are two large exothermic peaks in the picture. Peak A corresponds to the first stage of the thermite reaction, whose onset temperature is 524.11 °C. The Al powder and Bi₂O₃ are both solid. The solid-solid phase reaction occurs at this stage with heat release 920.8 J g⁻¹. Subsequently, Peak B occurs at the temperature range of 649.92 °C-673.74 °C, releasing an amount of heat 621.1 J g⁻¹. At this stage, the Al powder gradually melted. The state of thermite reaction also changed from solid-solid phase gradually transition to liquid-solid phase.

Figure 4(b) shows the DSC curves of Al/Bi₂O₃/10 wt%KClO₄. There are three peaks in the picture. It can be seen that at 304 °C, there is an endothermic peak. At this time, KClO₄ undergoes a phase change, transforming...
from an orthorhombic crystal system to a cubic crystal system. The thermite reaction in the whole process is also divided into two stages. Peak C corresponds to the first stage and its ignition temperature is 516.74 °C, with heat release 765.6 J g⁻¹. The state is a solid-solid phase reaction. Peak D corresponds to the liquid-solid phase thermite reaction between molten Al and Bi₂O₃. Its ignition temperature is 655.20 °C, releasing an amount of heat 1213 J g⁻¹.

Then, the mass fraction of KClO₄ in the nano thermites was changed. The samples are tested by DSC, as shown in the figures 4(c)–(d). All curves have similar shapes. KClO₄ has a phase change at 304 °C. The thermite reaction is divided into two stages: solid-solid phase thermite reaction and liquid-solid phase thermite reaction. This indicates that Al powder and Bi₂O₃ will not affect the phase change of KClO₄. KClO₄ also did not change the phase state when the thermite reaction occurred. However, the interesting thing is that the heat release, ignition temperature and activation energy have all changed. As shown in the figure 4, the ignition temperature of four samples are 524.11 °C, 516.74 °C, 512.68 °C and 517.62 °C. It means that after KClO₄ is added, the ignition temperature of the nano thermite become lower. The heat release of the solid-liquid thermite reaction increases significantly. But with the increase of KClO₄ content, the enhancement effect gradually weakened.

In figure 5, the TG curve begins to decrease slowly, which is caused by the volatilization of residual water and alcohol in the sample. The rate of mass loss is significantly accelerated from 420 °C to 580 °C due to the thermal decomposition of KClO₄. However, its weight has only dropped by 8.14%, which means that KClO₄ has not completely decomposed. The thermal decomposition of KClO₄ is earlier than the thermite reaction and it overlaps with the solid-solid phase thermite reaction in the later stage.

The decomposition of KClO₄ will release heat, which makes the thermite reaction occur earlier. The released oxygen makes the internal component distribution of the thermite system more uniform [30], which makes the liquid-solid phase thermite reaction and combustion more complete. At the same time, the released oxygen may react with the excess Al powder and release some heat. As shown in table 2, Al/Bi₂O₃/10 wt% KClO₄ has the highest heat release. But with the increase of KClO₄ content, it will cause too much KCl produced by decomposition and a relative decrease of Al fuel. As an inert additive, KCl will inhibits the thermite reaction [15]. This results in a decrease in heat release. As shown in table 2, Al/Bi₂O₃/30 wt% KClO₄ has the least heat release.

| Sample | Ingredient | Heat release (J g⁻¹) (10 °C min⁻¹) |
|--------|------------|----------------------------------|
| A      | Al/Bi₂O₃  | 1541.9                           |
| B      | Al/Bi₂O₃/10 wt% KClO₄ | 1978.6                          |
| C      | Al/Bi₂O₃/20 wt% KClO₄ | 1786.2                          |
| D      | Al/Bi₂O₃/30 wt% KClO₄ | 1189.1                          |
Table 3. Peak temperature at different heating rates.

| Sample | Ingredient                  | Heating rate [°C min⁻¹] | Peak temperature [°C] |
|--------|-----------------------------|--------------------------|-----------------------|
| A      | Al/Bi₂O₃                    | 5                        | 564.50                |
|        |                             | 10                       | 583.79                |
|        |                             | 15                       | 589.98                |
|        |                             | 20                       | 603.39                |
| B      | Al/Bi₂O₃/10 wt%KClO₄        | 5                        | 542.43                |
|        |                             | 10                       | 561.98                |
|        |                             | 15                       | 571.12                |
|        |                             | 20                       | 578.03                |
| C      | Al/Bi₂O₃/20 wt%KClO₄        | 5                        | 544.37                |
|        |                             | 10                       | 559.71                |
|        |                             | 15                       | 571.05                |
|        |                             | 20                       | 579.65                |
| D      | Al/Bi₂O₃/30 wt%KClO₄        | 5                        | 534.79                |
|        |                             | 10                       | 565.19                |
|        |                             | 15                       | 576.91                |
|        |                             | 20                       | 584.24                |

Figure 7. Kissinger method linear fitting graph. (a) Al/Bi₂O₃, (b) Al/Bi₂O₃/10 wt%KClO₄, (c) Al/Bi₂O₃/20 wt%KClO₄, (d) Al/Bi₂O₃/30 wt%KClO₄.

3.3. Activation energy

Table 3 shows the peak temperature of the first exothermic peak of the thermite sample at different heating rates. It can be clearly seen that after KClO₄ is added, the peak temperature of the thermite reaction arrives earlier.

According to the data in Table 3, the Ozawa method is used to calculate and fit. The results are shown in figure 6. The Kissinger method is used to calculate and fit to verify the accuracy of the calculations. The results are shown in figure 7.

As shown in Table 4, the activation energy results obtained by the two calculation methods are similar and the absolute values of correlation coefficient are all greater than 0.98. This shows that the calculated activation energy has high accuracy.

The activation energy of Al/Bi₂O₃ is 213.21 KJ mol⁻¹. When 10 wt% and 20 wt% KClO₄ are added, the activation energies are 212.82 KJ mol⁻¹ and 216.66 KJ mol⁻¹, respectively. There is no obvious change in activation energy. However, the activation energy of Al/Bi₂O₃/30 wt%KClO₄ is 149.17 KJ mol⁻¹, which is 30% lower than the Al/Bi₂O₃.
Figure 8 is a schematic diagram of the thermite reaction process mechanism after KClO₄ is added. The thermal decomposition of KClO₄ occurs first and will overlap with the solid-solid thermite reaction in the later stage. KClO₄ decomposes to release heat, which promotes the activation of the thermite reaction. Therefore, the ignition temperature of the thermite sample containing KClO₄ decreases. In addition, the O₂ released by the decomposition of KClO₄ reduces the agglomeration of the thermite sample. On the other hand, it may react with the excess Al powder. This makes the exothermic heat of the liquid-solid thermite reaction significantly increased. When the content of KClO₄ is small, the effect of reducing the activation energy of the thermite reaction is not obvious. But Al/Bi₂O₃/10 wt%KClO₄ has the highest heat release. When the content of KClO₄ is high, the activation energy of the thermite reaction is significantly reduced. But at the same time, too much KCl produced by decomposition will inhibit the heat release and reaction degree of the thermite reaction. So, the Al/Bi₂O₃/30 wt%KClO₄ has the lowest heat release.

### 3.4. Ignition and combustion

As shown in figure 9, the DC Power was used to adjust the output voltage. A sample of 10 mg was deposited in the center of the Ni-Cr wire, whose length is 5 cm. The high-speed camera could take 15,000 photos per second and record the combustion process.

| Sample | Ingredient | Calculation method | Activation energy [KJ mol⁻¹] | Fitting coefficient |
|--------|------------|--------------------|-----------------------------|--------------------|
| A      | Al/Bi₂O₃  | Kissinger method   | 209.90                      | -0.9877            |
|        |            | Ozawa method       | 213.21                      | -0.9891            |
| B      | Al/Bi₂O₃/10 wt%KClO₄ | Kissinger method   | 209.80                      | -0.9974            |
|        |            | Ozawa method       | 212.82                      | -0.9979            |
| C      | Al/Bi₂O₃/20 wt%KClO₄ | Kissinger method   | 213.81                      | -0.9978            |
|        |            | Ozawa method       | 216.66                      | -0.9982            |
| D      | Al/Bi₂O₃/30 wt%KClO₄ | Kissinger method   | 143.05                      | -0.9883            |
|        |            | Ozawa method       | 149.17                      | -0.9902            |
As showed in figure 10, the ignition excitation time was the 0 ms starting point, and then the combustion conditions of 0.5 ms, 1 ms, 2 ms, 3 ms, 3.5 ms, 4 ms, and 6 ms were selected for comparative analysis.

It can be seen that within 0.5–1 ms after ignition, the sample burns most violently and sparks are concentrated. This means that the energy of the thermite combustion is released in a concentrated way. As the mass fraction of KClO4 increases, the intensity of combustion and the burning time gradually decrease. At 0.5 ms after ignition, Al/Bi2O3 burns with large and bright sparks, while Al/Bi2O3/KClO4 almost has no sparks. On the one hand, the Al powder content is reduced, which reduces the combustion intensity. On the other hand, excessive potassium perchlorate may have the effect of hindering the spread of combustion during combustion, which is called the heat sink effect.

Interestingly, the samples with KClO4 added a huge explosion sound during the combustion process. This is related to the strong oxidizing properties of KClO4.

4. Conclusions

In this paper, the effect of KClO4 on the Al/Bi2O3 nano thermites was explored. Al/Bi2O3 nano thermites with different mass fraction of KClO4 (from 10 wt% to 30 wt%) was made by ultrasonic dispersion method.

The DSC results showed that Al/Bi2O3/KClO4 nano thermites have two obvious exothermic areas. The first stage thermite reaction is a solid-solid phase thermite reaction and the second stage thermite reaction is a solid-liquid phase thermite reaction. The thermal decomposition of KClO4 occurs first and will overlap with the solid-solid phase thermite reaction in the later stage. After KClO4 is added, the ignition temperature of the thermite is advanced.
Different additions of KC\textsubscript{4}O\textsubscript{4} lead to different effects. The change in heat release can be seen from the DSC results. Al\textsubscript{4}/Bi\textsubscript{2}O\textsubscript{3}/10 wt\%KC\textsubscript{4}O\textsubscript{4} has the largest heat release, which is 1978.6 J g\textsuperscript{-1}. Compared with Al\textsubscript{4}/Bi\textsubscript{2}O\textsubscript{3}, its heat release increased by 28.32\%. The activation energy is calculated using the Kissinger method and Ozawa method. Al\textsubscript{4}/Bi\textsubscript{2}O\textsubscript{3}/30 wt\%KC\textsubscript{4}O\textsubscript{4} has the lowest activation energy, which is 149.17 KJ mol\textsuperscript{-1}. Compared with Al\textsubscript{4}/Bi\textsubscript{2}O\textsubscript{3}, its activation energy decreased by 30\%. The addition of KC\textsubscript{4}O\textsubscript{4} obviously reduces the intensity of combustion and duration of the burning of the nano thermites.

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**Data availability statement**

All data that support the findings of this study are included within the article (and any supplementary files).

**Conflicts of Interest**

The authors declare that there is no conflict of interest regarding the publication of this paper.

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