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Thermal behavior of aluminum powder and potassium perchlorate mixtures by DTA and TG

Short communication

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Abstract

In this work the thermal decomposition characteristics of micron sized aluminum powder + potassium perchlorate pyrotechnic systems were studied with thermal analytical techniques. The results show that the reactivity of aluminum powder in air increases as the particle size decreases. Pure aluminum with 5 μ m particle size has a fusion temperature about 647 °C, but this temperature for 18 μ m powder is 660 °C. Pure potassium perchlorate has an endothermic peak at 300 °C corresponding to a rhombic–cubic transition, a fusion temperature around 590 °C and decomposes at 592 °C. DTA curves for Al₅/KClO₄ (30:70) mixture show a maximum peak temperature for thermal decomposition at 400 °C. Increasing the particle size of aluminum powder increases the ignition temperature of the mixture. The oxidation temperature increased by enhance in the aluminum content of the mixture.

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1. Introduction

Potassium chlorate and potassium perchlorate are the main oxidizer in many compositions of propellants and pyrotechnics [1–5].

This paper presents the results of a study on potassium perchlorate/aluminum powder mixtures by simultaneous thermogravimetry–differential thermal analysis (TG/DTA). The aim of this study was investigation of the effect of aluminum content and particle size on the thermal behavior of different compositions of aluminum+potassium perchlorate mixtures. Thermal behaviour of pure micro-sized aluminum and its composition with explosives has been reported previously [6,7], but to the best our knowledge nothing has been published on the thermal behaviour of aluminum+potassium perchlorate.

2. Experimental

2.1. Materials

Potassium perchlorate (mesh 300) was purchased from Merck (Tehran, Iran). Highly pure aluminum powders (5 and 18 µm particles size) were prepared by metals laboratory of Malek Ashtar University of Technology (Tehran, Iran).

2.2. Procedure

A thermobalance (Stanton, model TR-01, sensitivity 0.1 mg) with a differential thermal analysis attachment (STA 1500) was used for DTA/TG studies. In this study, the heating rate was 10 °C/min in air at 1 bar and the sample mass was 4.0 mg. Four mixtures (30/70 and 50/50 mass %Al/KClO₄) as well as pure potassium perchlorate and two samples of pure aluminum powders with different particle sizes (5 and 18 μ m) were examined. Before testing, all samples were stored in a vacuum oven at 65 °C for at least 3 h.

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3. Results

3.1. Thermal behaviour of the individual components

3.1.1. Aluminum powders

Aluminum powders (with 5 and 18 μ m particle size) were examined in air to assess oxidation characteristics (Fig. 1). The powders exhibit an exothermic peak below the melting point of Al (~660 °C). The first mass gain for the 18 μ m sized Al was observed at 490–610 °C, and for aluminum with 5 μ m particle size at 484–603 °C. The 5 μ m particle size aluminum has a lower melting point (647 °C) than 18 μ m aluminum (660 °C).

3.1.2. Potassium perchlorate

DTA and TG curves of pure potassium perchlorate are shown in Fig. 2. An endothermic peak at $300 \,^{\circ}\text{C}$ corresponds to a rhombic–cubic transition. The melting point is at 590 $\,^{\circ}\text{C}$ with decomposition [8,9], oxygen and potassium chloride (KCl) are produced [10–12].

3.2. Binary mixtures

3.2.1. Aluminum (5 µm particle size)/potassium perchlorate mixture

The TG and DTA curves for $Al_5/KClO_4$ mixture (30:70) are shown in Fig. 3a. An endothermic peak at 300 °C corresponds to the rhombic–cubic transition. The DTA curve shows



Fig. 1. TG and DTA curves for pure aluminum powder: (a) $5 \,\mu$ m particle size and (b) $18 \,\mu$ m particle size (sample mass, 4.0 mg; heating rate, $10 \,^{\circ}$ C/min; air atmosphere).



Fig. 2. TG and DTA curves for potassium perchlorate (sample mass, 4.0 mg; heating rate, $10 \,^{\circ}\text{C/min}$; air atmosphere and potassium perchlorate with 50 μ m particle size).

an endothermic peak at about 587 $^{\circ}$ C, which corresponded with the temperature of fusion of potassium perchlorate. A sharp exotherm occurs near 600 $^{\circ}$ C with a mild reduction (about 4.9%) in the mass of sample that corresponds to the reaction of aluminum powder with potassium perchlorate according to:

 $8A1 + 3KClO_4 \rightarrow 4Al_2O_3 + 3KCl$

Fig. 3b show the TG–DTA results for $Al_5/KClO_4$ with mass ratio of 50:50. After the endothermic peak at 300 °C corresponding to the rhombic–cubic transition, an endotherm occurs at 596 °C



Fig. 3. TG and DTA curves for mixture of aluminum powder with 5 μ m particle size and potassium perchlorate with mass ratio (a) 30:70 and (b) 50:50 (sample mass, 4.0 mg; heating rate, 10 °C/min; air atmosphere and potassium perchlorate with 50 μ m particle size).



Fig. 4. TG and DTA curves for mixture of aluminum powder with 18 μ m particle size and potassium perchlorate with mass ratio (a) 30:70 and (b) 50:50 (sample mass, 4.0 mg; heating rate, 10 °C/min; air atmosphere and potassium perchlorate with 50 μ m particle size).

due to the fusion of potassium perchlorate. An exotherm with a limited decrease in the mass (about 3.4%) occurs at 615 °C, which corresponds with the reaction between aluminum and potassium perchlorate. The results show ignition temperature depends on aluminum content. A higher content of aluminum powder increases the decomposition temperature.

3.2.2. Aluminum (18 μm particle size)/potassium

perchlorate mixture

TG–DTA data for Al₁₈/KClO₄ mixtures are presented in Fig. 4. As can be seen in Fig. 4a, there are three thermal transitions, two are endothermic and another is exothermic. The first endotherm at 300 °C is the rhombic–cubic transition of KClO₄. Another endotherm at 605 °C corresponds to fusion of potassium perchlorate. The exothermic peak at 608 °C, where the mass decreases is related to the reaction between aluminum powder and potassium perchlorate. Fig. 4b shows TG and DTA curves for Al₁₈/KClO₄ (50:50) mixture. These results show the ignition temperature of the mixtures also increased when a higher content of aluminum powder was used.

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