Research paper

Fabrication and electro-explosive performance of carbon nanotube energetic igniter

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Abstract

A carbon nanotube energetic igniter was fabricated by integrating carbon nanotube composite energetic materials with a Cu layer realized onto a ceramics substrate. Carbon nanotube composite energetic materials were prepared by means of the wet chemical method, filling multiwalled carbon nanotubes with KNO_3. DSC curves show that the carbon nanotube composite energetic materials released chemical heat of 876.1 $J \cdot g^{-1}$ at the peak value of 386.8 ℃. The electro-explosive performances of the carbon nanotube energetic igniter were investigated using a capacitor-discharger apparatus. The electro-explosive time decreased exponentially with increasing the charging voltage and approached a certain value of 39.3 μs. The electric explosion process of the carbon nanotube energetic igniter recorded by high speed photography shows that chemical reaction of carbon nanotube composite energetic materials was involved in the electric explosion process, accompanied by more heat release.

Keywords : carbon nanotubes, KNO_3, nanoenergetic materials, electrophoretic deposition, electro-explosive performance

1. Introduction

A pyrotechnic igniter is the initial source of energy in numerous civilian and military energy-demanding systems, which is used to initiate an explosive, burning, electrical, or mechanical train. In recent years, microenergetics have drawn a growing interest in the ordnance systems and civil industry. However, traditional electropyrotechnic igniters activated by the electrical energy of a bridgewire do not satisfy demands from them for batch fabrication and high level of integration. Nanoenergetic materials (nEMs) particularly offer the promise of much higher energy densities, faster rate of energy release, greater stability, and more security[1]. They can be utilized to realize microscale energetic devices, such as nano igniters, which are fabricated with standard microsystem techniques allowing mass production and high level of integration and reliability. Moreover, the exothermic reaction of nEMs make the nano igniters be able to generate much more output energy than input energy. Therefore, the application of nEMs to microenergetics has received steadily growing interests. There are some studies in the literature on the application of nEMs to microenergetics[2]−[8].

As is well known, carbon nanotubes (CNTs) are one sort of particular one-dimensional nanomaterials, which are made of one layer or more than one layer of graphitic sheets rolled into a cylinder while the tube tip is closed by hemispherical or polyhedral graphitic domes. One fascinating aspect of CNTs is their cavities. The hollow nanotube cavities can be used to incorporate foreign materials in order to generate novel compounds or nanostructured materials[9]. It was predicted that CNTs filled with oxidants may find practical use as energetic materials, which can make use of graphitic sheets as fuel and inherit the remarkable thermal and mechanical robustness from CNTs.

In this study, carbon nanotube composite nEMs were prepared by means of the wet chemical method filling multiwalled carbon nanotubes with KNO_3. The fabricated carbon nanotube composite nEMs was then characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC) and thermogravimetry (TG). A carbon nanotube energetic igniter was developed by integrating carbon nanotube
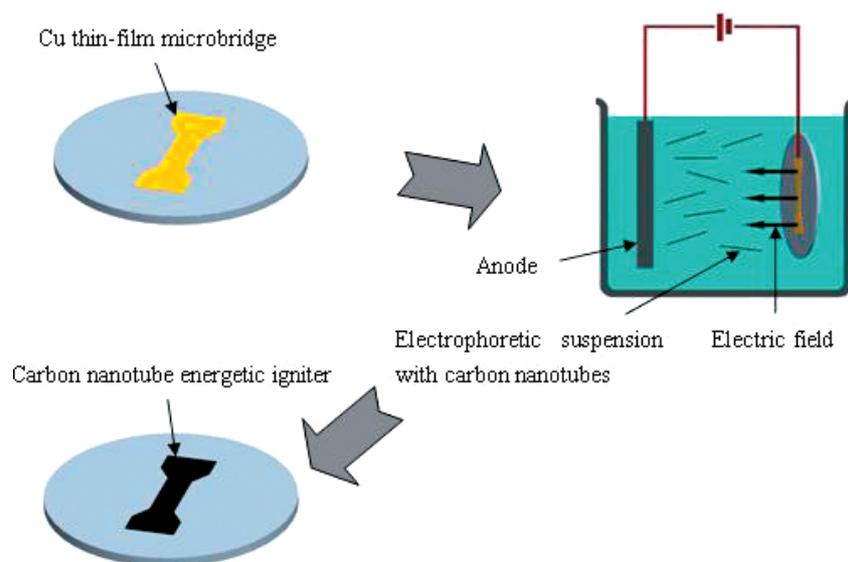
Figure 1   Fabrication process flow.

composite nEMs with a Cu layer realized onto a ceramics substrate. The electro-explosive performances of the carbon nanotube energetic igniter were investigated using a capacitor-discharger apparatus.

2. Experimental
2.1 Preparation of the carbon nanotube composite nEMs

Multiwalled aligned carbon nanotubes prepared by the catalytic decomposition of CH_4 were provided by Shenzhen Nanotech Port Ltd. Co. (Shengzhen, China) and they were used as received. The inner diameter, length and specific surface area of the carbon nanotubes were 5 nm-20 μm, 5 μm-15 μm and 40 $m^2 \cdot g^{-1}$-1300 $m^2 \cdot g^{-1}$, respectively.

A typical experimental procedure was described as follows. Samples of carbon nanotubes were suspended in concentrated HNO_3 and saturated solution of KNO_3, and stirred at 140°C for 24 h. After cooling to the ambient temperature, the black residue was completely rinsed with deionised water and then dried in vacuum at 60°C overnight.

2.2 Fabrication of the carbon nanotube energetic igniter

The process flow of the fabrication is shown in Figure 1. The carbon nanotube energetic igniter was fabricated by electrophoretic deposition. The ceramics substrate was cleaned and dried thoroughly. As a conducting layer, a Cu thin-film microbridge with 1 mm in width and 1 μm in thickness was deposited on the substrate by magnetron sputtering through a designed mask. The electrophoretic suspension was composed of carbon nanotube composite nEMs, $Al(NO_3)_3$, ethanol and acetone. The carbon nanotube composite nEMs were suspended by ultrasonication for 30 min. The distance between the Cu thin-film microbridge as the cathode and the Pt plate as the anode was adjusted to be 1 cm. Under constant voltage of 25 V for 20 min, carbon nanotube composite nEMs were evenly deposited on the graphical Cu thin-film microbridge.

2.3 Characterization

The carbon nanotube composite nEMs were characterized by TEM (JEM-1200, JEOL, Japan) and XRD (D8ADVANCE, Bruker, Germany). The exothermic reaction of the carbon nanotube composite nEMs was characterized by DSC/TG (STA449C, NETZSCH, Germany). The DSC experiments was carried out at a temperature range from 20°C to 700°C at a heating rate of 5°C·min^{-1} under a 99.999% N_2 flow. The surface morphology of the carbon nanotube energetic igniter was characterized by SEM (ULTRA plus, ZEISS, Germany).

2.4 Test of the electro-explosive performances

The electro-explosive performances were tested using a setup shown in Figure 2. The carbon nanotube energetic igniters were ignited electrically using a capacitor-discharger apparatus, whose voltage and current signals were analyzed by an oscillograph (LeCroy TM 44Xs). Under the different charging voltages, the electro-explosive times were measured by means of optical probe. The electric explosion process of the carbon nanotube energetic igniter was recorded by high speed photography (HG-100K, Redlake, USA).

3. Results and discussion
3.1 Characterization of the carbon nanotube composite nEMs

Typical TEM images of the carbon nanotube composite nEMs are shown in Figure 3. It can be seen that the hollow cavities of CNTs were filled with substances. Meanwhile, there was not a substance being attached to the surfaces of CNTs. The high-resolution TEM image shows the legible lattice stripe and micro-region electronic diffraction pattern, which indicated that the crystalline substance was KNO_3.

The carbon nanotube composite nEM was characterized by XRD as shown in Figure 4. Three peaks can be observed at 26.00°, 42.96°, and 54.26°, which are assigned to the hexagonal graphite planes (002), (100) and (004), respectively, indicating the well graphitized nature
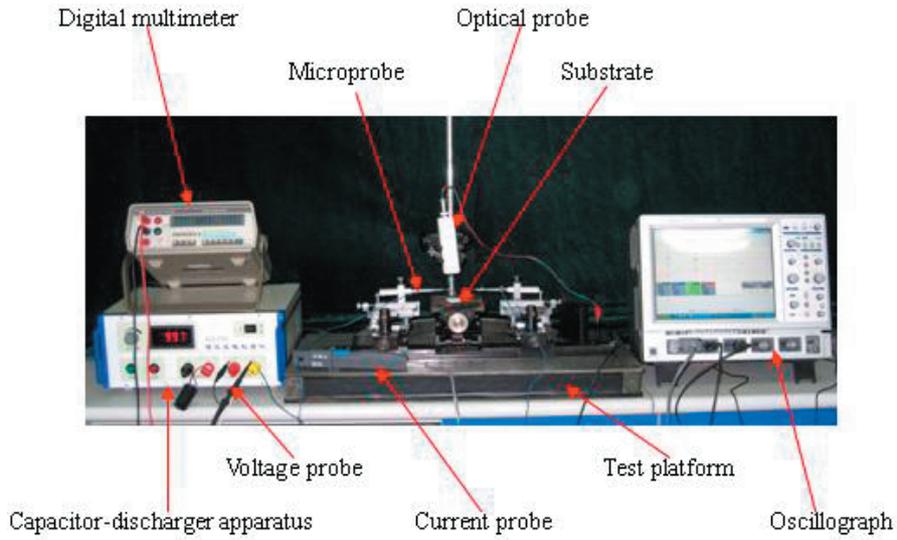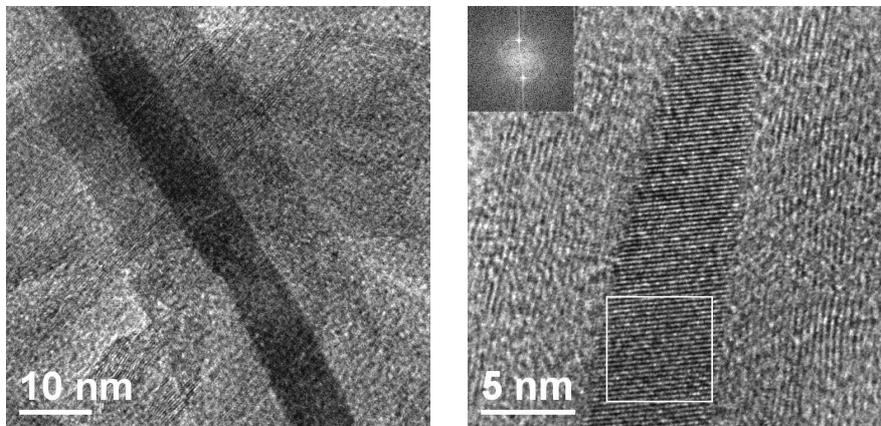
Figure 2   Measurement setup.

Figure 3   Typical TEM images of the carbon nanotube composite nEMs.
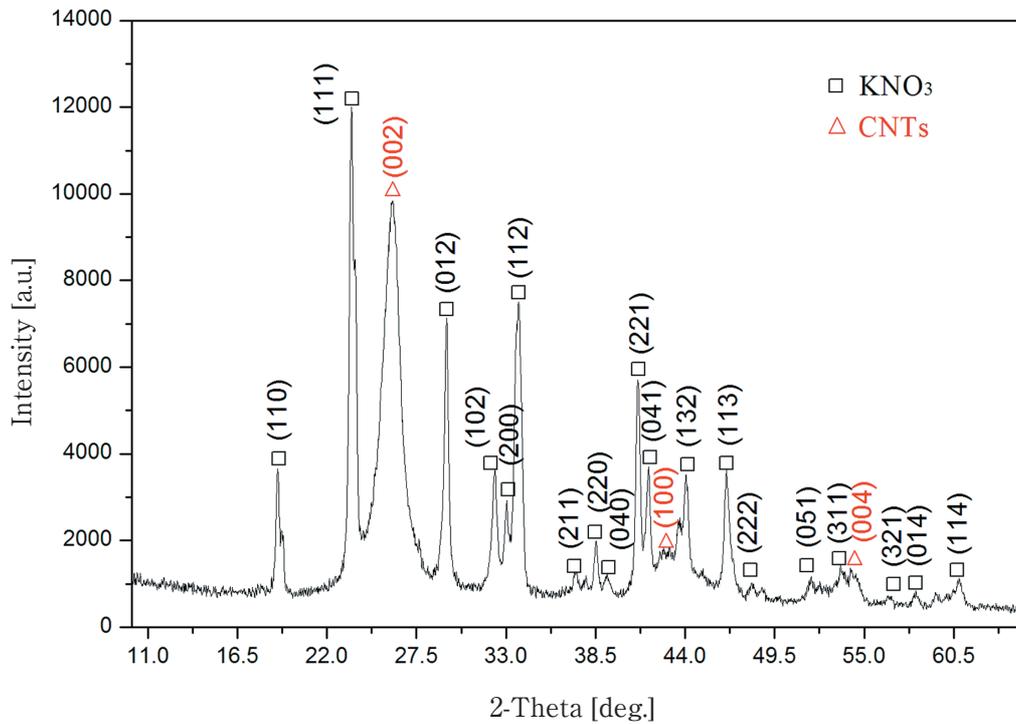

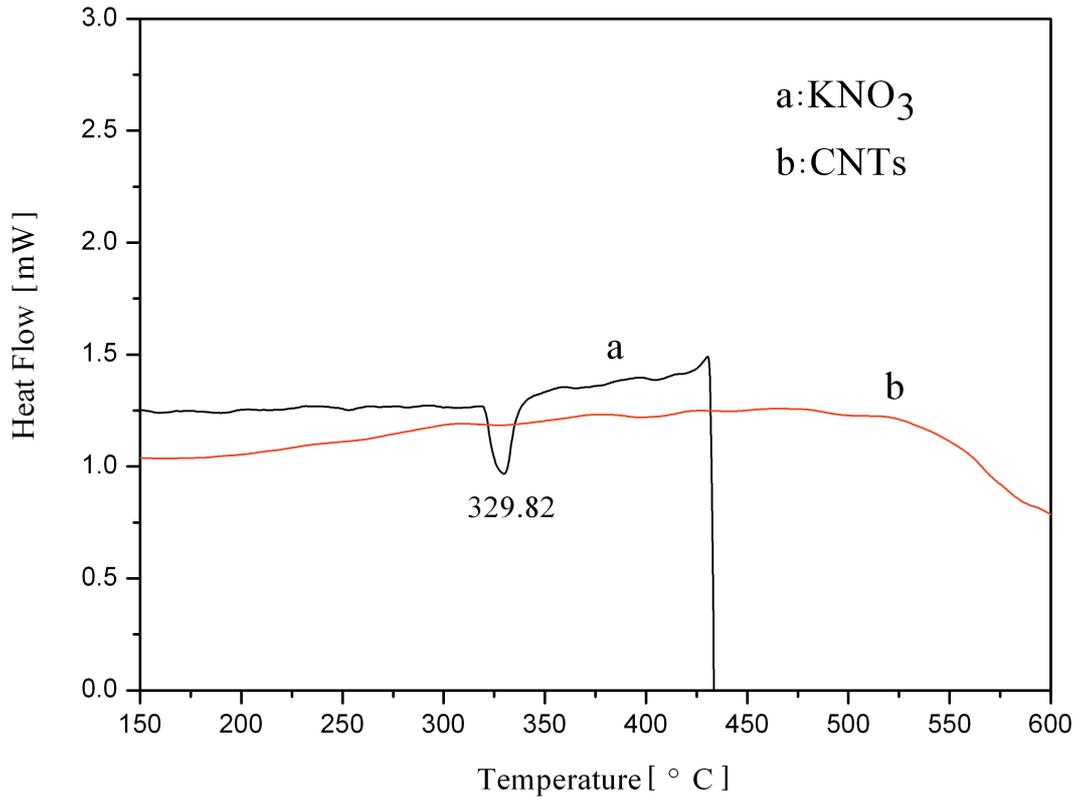**Figure 4**   The XRD pattern of the carbon nanotube composite nEMs.

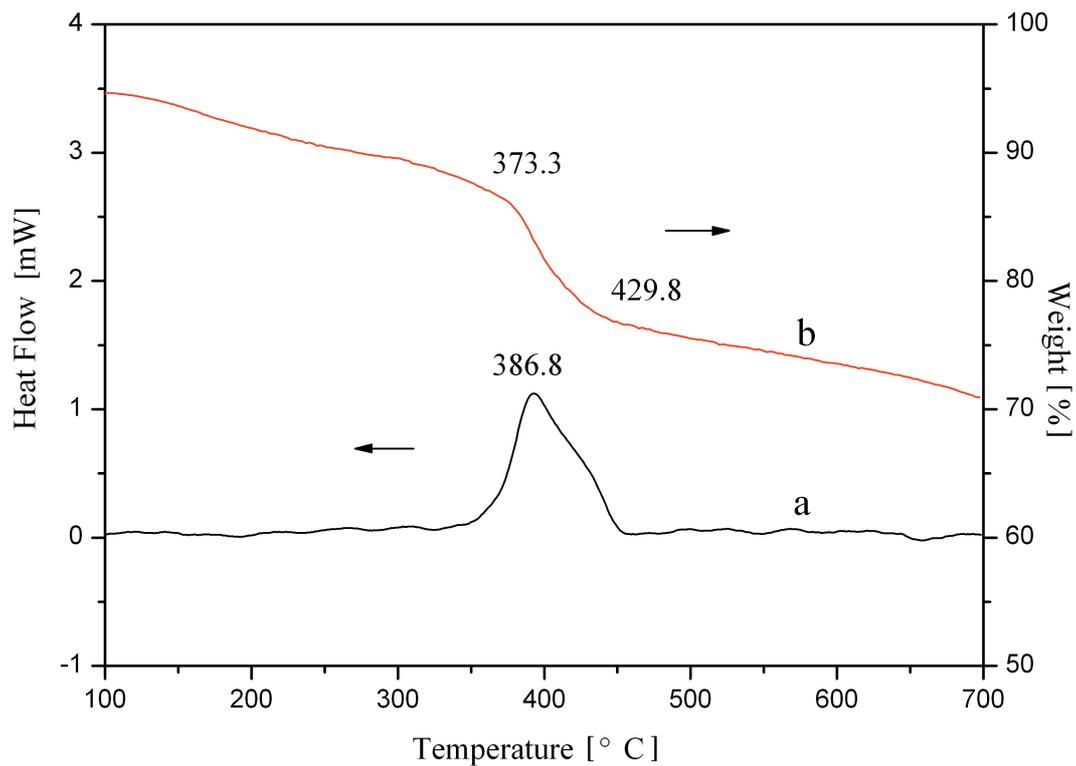**Figure 5**   DSC curves of KNO₃ and CNTs.

Figure 6   DSC-TG curves of the carbon nanotube composite nEMs.

of the CNTs. The diffraction peaks of KNO_3 can be clearly seen from the XRD pattern, which are ascribed to (110), (111), (012), (102), (200), (112), (211), (220), (040), (221), (041), (132), (113), (222), (051), (311), (321), (014), and (114) planes, respectively. The presence of the crystalline KNO_3 component in the hollow cavities of the CNTs was further supported by XRD measurement.

Figure 5 shows the DSC curves of KNO_3 and CNTs. The

DSC curve of KNO_3 shows an endothermic peak for the melting at about 329.82 °C. No endothermic and exothermic peaks were observed in the DSC curve of CNTs, which suggested that CNTs had no significant physical and chemical changes under the condition.

The exothermic reaction of the carbon nanotube composite nEMs was characterized by DSC-TG as shown in Figure 6. The DSC curve shows an exothermic peak.
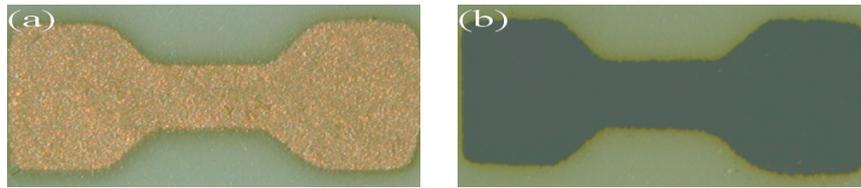
Figure 7 Microscopy images of (a) the Cu thin-film microbridge and (b) the carbon nanotube energetic igniter.
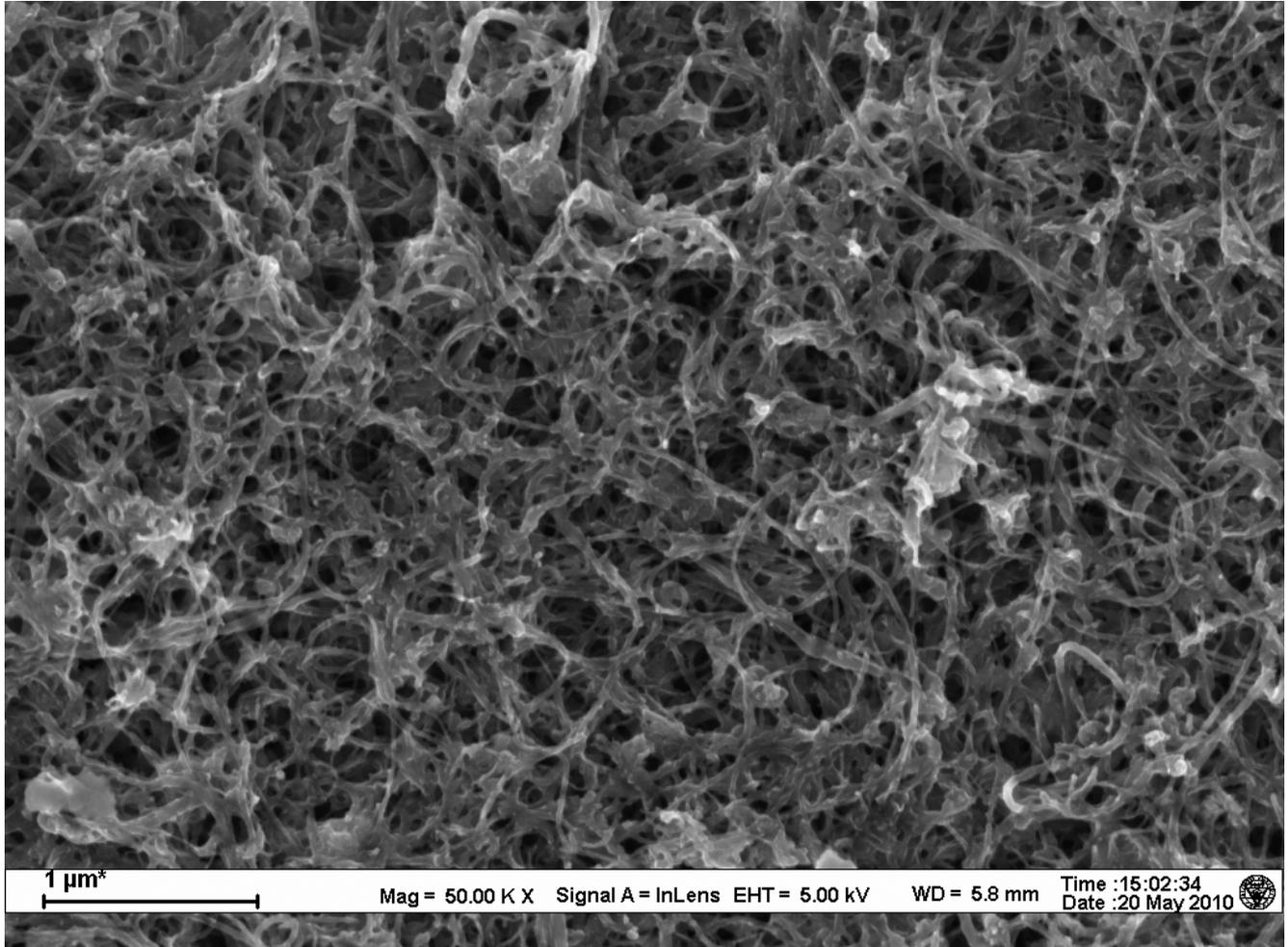

Figure 8 SEM image of the carbon nanotube energetic igniter.

The characteristic temperature of the exothermic peak was about 386.8 °C. The reaction enthalpy was calculated to be about 876.1 J·g^{-1}. The mass loss started at about 373.3 °C and ended at about 429.8 °C with the mass loss of about 25.3 %.
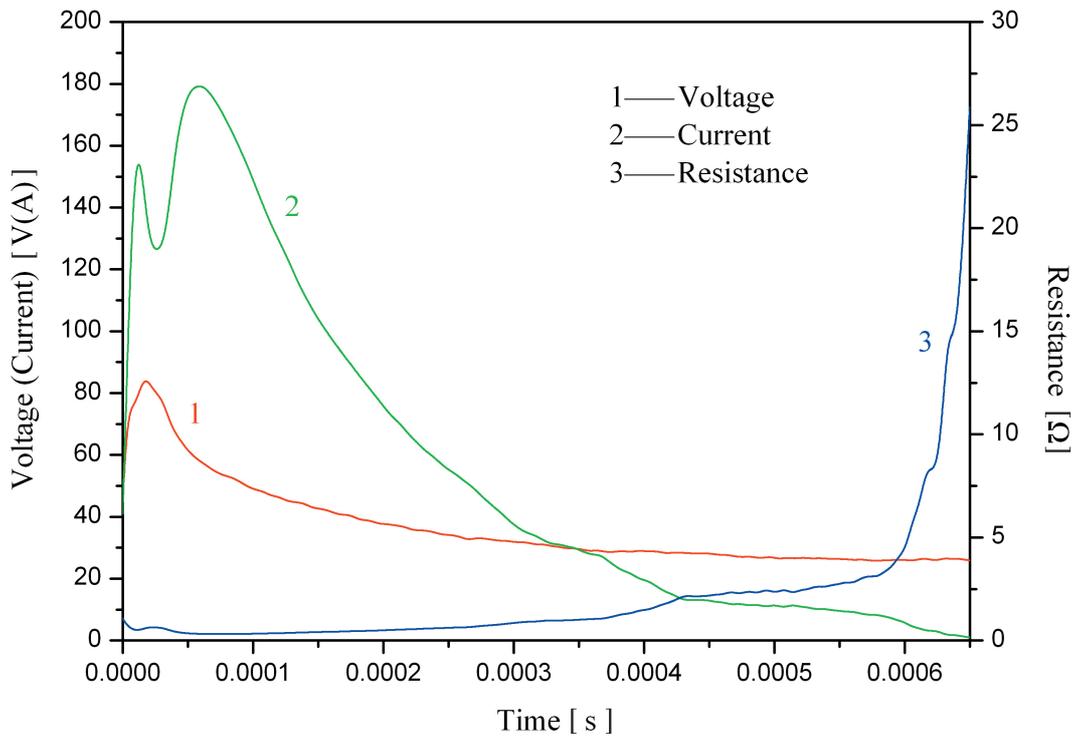
3.2 Characterization of the carbon nanotube energetic igniter

Figures 7a and b show the microscopy images of the Cu thin-film microbridge and the carbon nanotube energetic igniter, respectively. The width of the bridge area is 1 mm. Figure 8 shows the SEM image of the carbon nanotube energetic igniter. As can be seen in the figure, the carbon nanotube composite nEMs are distributed randomly and uniformly on the Cu thin-film microbridge without large aggregations. The surface of the carbon nanotube energetic igniter was porous.
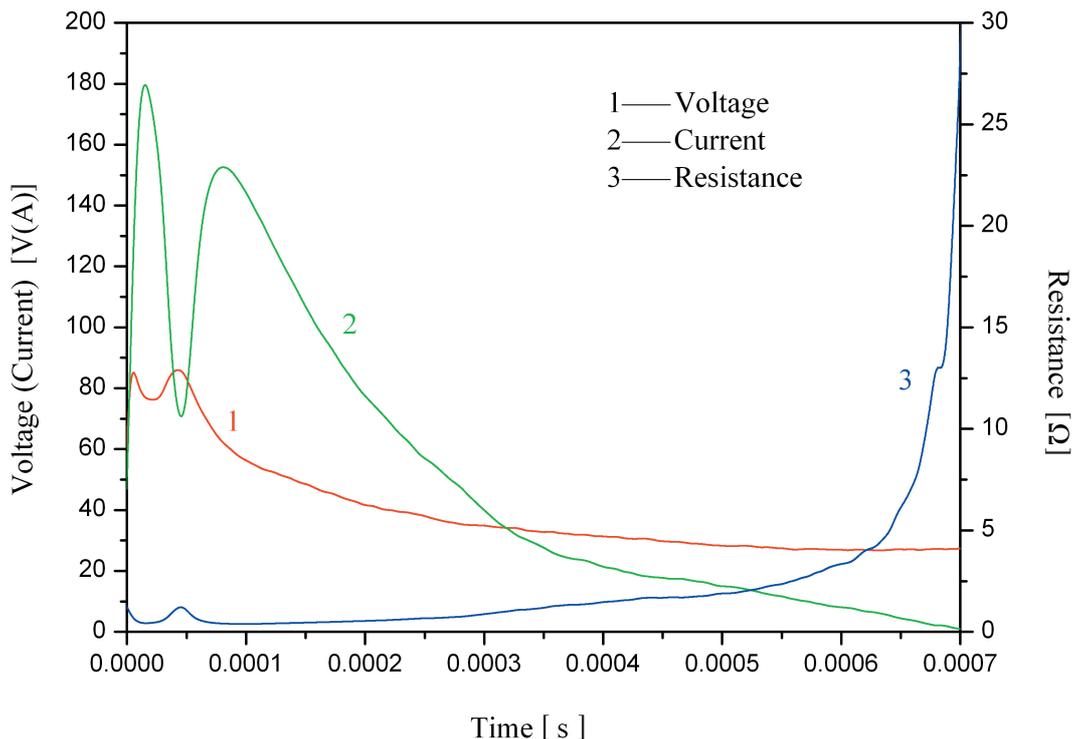
3.3 Electro-explosive performances of the carbon nanotube energetic igniter

Figures 9 a and b show the typical characteristic curves of the electric explosion processes for the Cu thin-film microbridge and the carbon nanotube energetic igniter. The charging voltage was 110 V and the capacitance was 470 μF. The curve of time vs voltage of the Cu thin-film microbridge showed only one peak, while that of the carbon nanotube energetic igniter showed two peaks, which suggested that the Cu thin-film microbridge and the carbon nanotube energetic igniter conducted different electro-explosive behaviours. Besides melting and vaporization of the Cu thin-film microbridge, it is suggested that the chemical reaction of the carbon nanotube composite nEMs was involved in the electric explosion process of the carbon nanotube energetic igniter.

The electro-explosive time was defined as a time interval from electrification to voltage signal transition of

(a)

(b)

Figure 9   Typical characteristic curves of the electric explosion processes for (a) the Cu thin-film microbridge and (b) the carbon nanotube energetic igniter.

optical probe. Figure 10 shows the relationships between charging voltages and the electro-explosive times for the Cu thin-film microbridge and the carbon nanotube energetic igniter. The capacitance was 470μF. Compared with the Cu thin-film microbridge, the carbon nanotube energetic igniter possessed longer electro-explosive time under the same charging voltage. The electro-explosive time exponentially decreased with increasing the charging

voltage. When the charging voltage was more than 105 V, the electro-explosive time of the carbon nanotube energetic igniter approached a certain value of 39.3 μs.

The electric explosion processes of the Cu thin-film microbridge and the carbon nanotube energetic igniter were recorded by high speed photography as shown in Figures 11 and 12. The charging voltage was 110 V, the capacitance was 470μF, and the frame rate was 20000 fps.
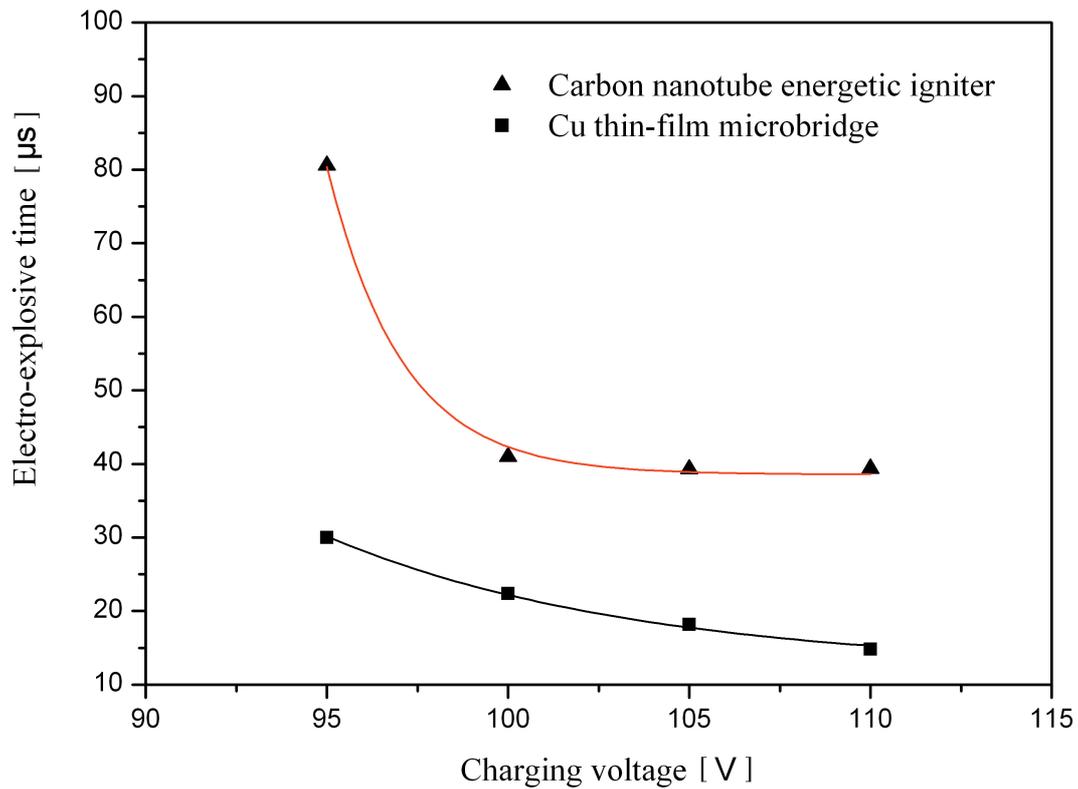
Figure 10   Relationship between charging voltage and the electro-explosive time.
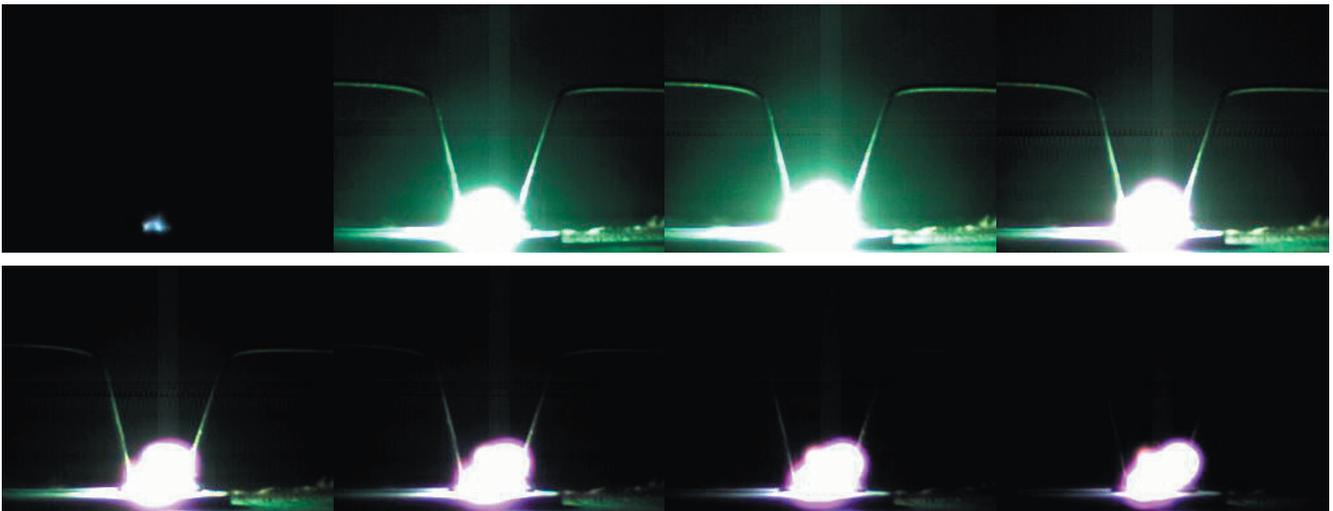

Figure 11   Electric explosion process of the Cu thin-film microbridge.

Compared with the Cu thin-film microbridge, the carbon nanotube energetic igniter possessed more vigorous electro-explosive behaviour and went through a longer duration of electric explosion. It indicated that chemical reaction was involved in the electric explosion process of the carbon nanotube energetic igniter, accompanied by more heat release.

4. Conclusion

A carbon nanotube energetic igniter was fabricated by integrating carbon nanotube composite nEMs with a Cu thin-film microbridge, which was deposited onto a ceramics substrate by magnetron sputtering through a designed mask. Carbon nanotube composite nEMs were prepared by mean of the wet chemical method filling multiwalled carbon nanotubes with KNO_3. DSC curves show that the carbon nanotube composite energetic materials released chemical heat of 876.1 $J·g^{-1}$ at the peak value of 386.8 $^{\circ}$C. The electro-explosive performances of the carbon nanotube energetic igniter were investigated using a capacitor-discharger apparatus. The electro-explosive characteristic curves suggested that the Cu thin-film microbridge and the carbon nanotube energetic igniter conducted different electro-explosive behaviours. The electro-explosive time exponentially decreased with increasing the charging voltage and approached a certain value of 39.3 μs. Compared with the Cu thin-film microbridge, the carbon nanotube energetic igniter possessed more vigorous electro-explosive behaviour and went through a longer duration of electric explosion. The
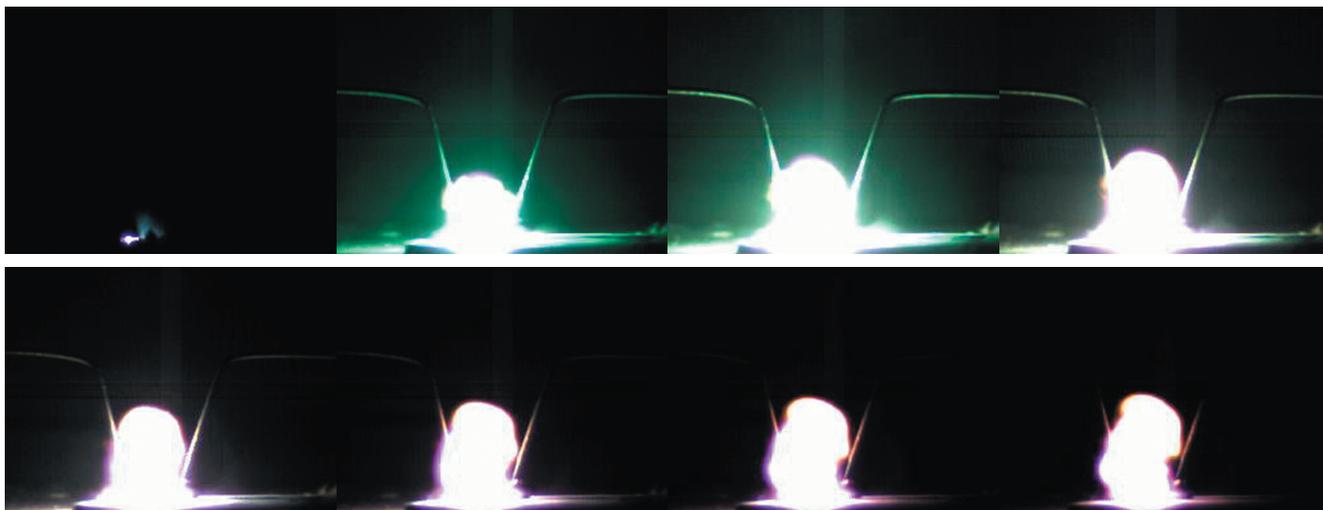
Figure 12   Electric explosion process of the carbon nanotube energetic igniter.

results show that chemical reaction of carbon nanotube composite nEMs was involved in the electric explosion process, accompanied by more heat release. This work will probably open the door to the application of the carbon nanotube composite nEMs to microenergetics.

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