Nanopowder production by gas-embedded electrical explosion of wire*

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A small electrical explosion of wire (EEW) setup for nanopowder production is constructed. It consists of a low inductance capacitor bank of 2 μF–4 μF typically charged to 8 kV–30 kV, a triggered gas switch, and a production chamber housing the exploding wire load and ambient gas. With the EEW device, nanosize powders of titanium oxides, titanium nitrides, copper oxides, and zinc oxides are successfully synthesized. The average particle size of synthesized powders under different experimental conditions is in a range of 20 nm–80 nm. The pressure of ambient gas or wire vapor can strongly affect the average particle size. The lower the pressure, the smaller the particle size is. For wire material with relatively high resistivity, such as titanium, whose deposited energy W_d is often less than sublimation energy W_s due to the flashover breakdown along the wire prematurely ending the Joule heating process, the synthesized particle size of titanium oxides or titanium nitrides increases with overheat coefficient k ($k = W_d/W_s$) increasing.

Keywords: electrical explosion of wire, nanosized powders, particle size

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1. Introduction

The increasing surface area to volume as the size diminishes can make the properties of nanosize materials with size in 1 nm–100 nm different from their bulk properties. Nanosize materials have been applied in a variety of areas ranging from electronics to telecommunication, from material science to biotechnology and medicine.[1–5]

Electrical explosion of wire (EEW) in ambient gas is a new technique for producing nanosized powders.[6,7] It is often carried out on a small pulsed power device. Within a very short time, a pulsed current is delivered into a thin wire load. The energy deposited in the wire by Joule heating melts, evaporates, and ionizes the wire material, resulting in plasma that expands into the ambient gas. The high temperature plasma then experiences fast cooling by radial thermal conduction to the high-density ambient gas as well as thermal radiation. Condensation and nucleation of wire vapor take place, which gives rise to the formation of nanosized particles with well-defined structure and morphology.[8–11] Studies have shown that the synthesized particle size is strongly dependent on experimental conditions, such as the pressure of ambient gas, the overheat coefficient k, etc.[12–14] k is defined as the ratio of deposition energy W_d ($W_d = \int_0^{T_d} V_R(t) \cdot i(t) \cdot dt$, where $V_R(t)$ and $i(t)$ are wire voltage and current, respectively) to the sublimation energy W_s.

In this article, with a small EEW setup, nanosized powders of titanium oxides, titanium nitrides, copper oxides, and zinc oxides are successfully synthesized, and the relationships between powder size and experimental conditions (the pressure of surrounding gas as well as wire vapor, the overheat coefficient k) are investigated. According to experiments with different wire materials, we also find that the voltage waveform and overheat coefficient k of wire explosion are highly dependent on the property of wire material. For materials with relatively low resistivity, such as copper, a higher burst peak always appears in the voltage waveform, and k could easily exceed 1.0 even up to 2.0 or much more. While for materials with relatively high resistivity, such as titanium, a much lower burst peak appears in the voltage waveform, and k is often less or only a little more than 1.0.

The rest of this article is organized as follows. In Section 2 the EEW experimental setup is generally described. In Section 3 the experimental results are given and they are discussed. Finally, some conclusions are drawn from the present study in Section 4.

2. Experimental setup

The experimental setup for electrical explosion of wire is shown in Fig. 1. It consists of a low inductance capacitor bank of 2 μF–4 μF typically charged to 8 kV–30 kV, a triggered gas switch, and two high voltage electrodes to deliver the pulse to the thin wire loads. Eight pieces of wire were installed in a

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production chamber filled with 10 kPa–80 kPa ambient gas, so the setup was able to explode eight pieces of wire one by one by rotating the wire holder without opening the chamber. A membrane filter was installed between the chamber and a vacuum pump. The floating powders were collected on the surface of the filter by evacuating the chamber with a pump. As the chamber was filled with oxygen or nitrogen, metal oxides or metal nitrides could be synthesized respectively.

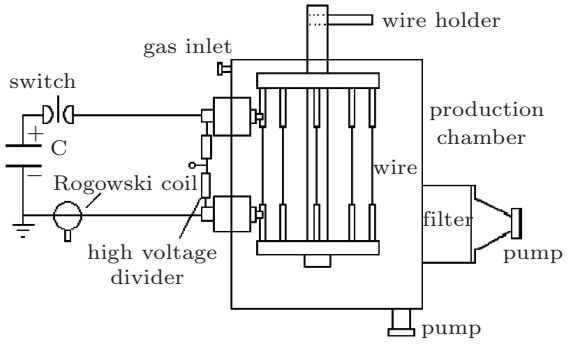


Fig. 1. Schematic diagram of the experimental setup.

3. Results and discussion

3.1. Waveforms of explosive current and voltage

Figure 2 shows the typical explosive current and voltage waveforms obtained with a) titanium wire and b) copper wire explosion in air of 50 kPa.

The current and voltage waveforms have the typical features of wire explosion, a sharp peak in voltage and a sudden drop in current. The voltage peak and current drop are all caused by the rapid increase in wire resistance due to the vaporization. After the time of burst peak (regarded as the flashover moment), the peak voltage falls quickly, which is attributed to flashover breakdown along the wire, and current is shunted to the discharge plasma around the wire, which indicates that the Joule heating process nearly ceases.

From Fig. 2, we can see that the voltage waveform of titanium wire is much different from that of copper wire. A higher voltage plateau and a lower burst peak appear in the titanium voltage waveform. It is due to the much different properties of wire materials. The values of melting beginning resistivity ρ_1 of titanium and copper are 156 μΩ·cm and 9.9 μΩ·cm,[15] respectively, and the values of vaporizing beginning resistivity ρ_2 of titanium and copper are 158 μΩ·cm and 26.3 μΩ·cm, respectively. Titanium wire with a higher and almost unchangeable resistivity within the liquid heating process results in the higher voltage plateau. And as is well known, insulation breakdown under pulsed voltage is dependent on the product of applied voltage and duration time. The higher voltage plateau of titanium wire would result in flashover breakdown at a lower burst peak voltage and prematurely end the vaporization process as well as the Joule heating process, which would lead to the deposition energy much lower than the energy of the whole wire vaporization.

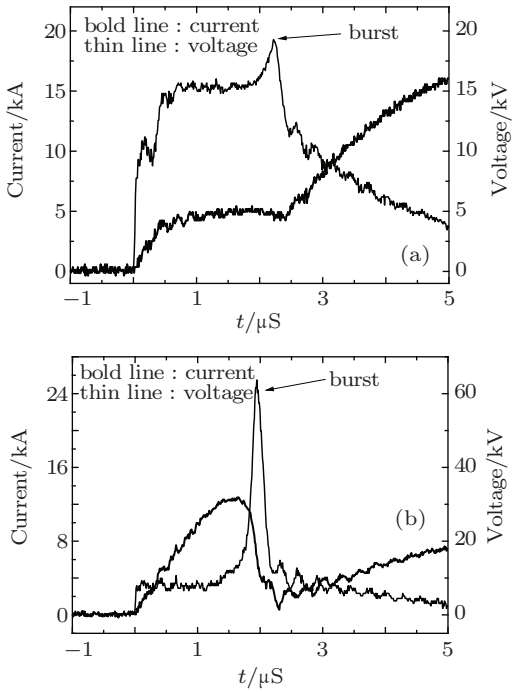


Fig. 2. Current and voltage waveforms with capacitor charging voltage at 20 kV: (a) titanium wire explosion in air, the wire with 0.23 mm in diameter and 85 mm in length; (b) copper wire explosion in air, the wire with 0.20 mm in diameter and 85 mm in length.

Based on Fig. 2, the deposition energy up to the burst peak is calculated to be 127 J for titanium wire (correspondingly, $k = 0.88$) and 174 J for copper wire ($k = 1.40$).

3.2. Explosion products and conditions

Several species of synthesized powders and typical experimental parameters are shown in Table 1.

Table 1. Several species of synthesized powders and typical experimental parameters.

Wire	Diameter/mm	Length/mm	Storage capacitance/μF	Charging voltage/kV	Ambient gas (at 50 kPa)	Explosion products	Average particle size/nm
Ti	0.18	85	4	18	air	TiO_2	44.75
Ti	0.23	85	4	18	air	TiO_2	59.81
Ti	0.23	85	4	18	N_2	TiN	29.69
Cu	0.20	85	2	20	air	$CuO+Cu_2O$	50.31
Zn	0.51	85	4	24	air	ZnO	61.76

All the above experiments are carried out with wires of 85 mm in length and at an ambient gas pressure of 50 kPa. Phases of explosion products are analyzed by X-ray diffraction (XRD) method (D8 ADVANCE, BRUKER). Specific surface area is determined by Brunauer–Emmet–Teller (BET) method (Autosorb-1-C, Quantachrome) using N_2 adsorption, and the average particle size is estimated from specific surface area on the assumption that the particles are all in spherical shape.

In order to study the dependence of the average particle size on the ambient gas pressure, titanium wire explosions are also carried out under different gas pressures. Figure 3 shows the estimated average particle size for titanium wire explosion in air and in nitrogen. As the gas pressure increases from 10 kPa to 80 kPa, the average particle sizes of titanium oxides and titanium nitrides change from 23 nm to 69 nm and 21 nm to 34 nm, respectively.

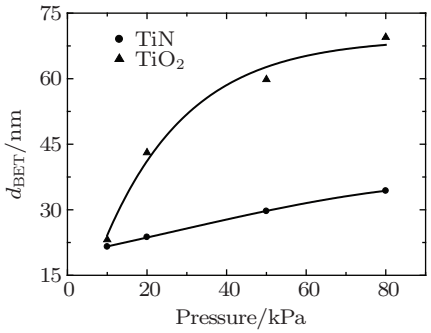


Fig. 3. Curves of average particle size versus ambient gas pressure for samples obtained with titanium wire explosion in air and in nitrogen respectively.

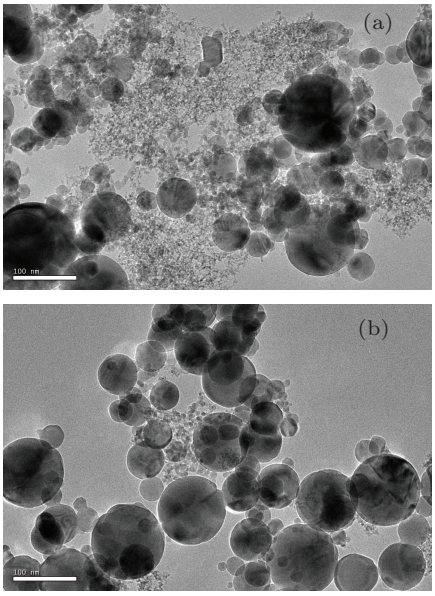


Fig. 4. TEM images for samples obtained with titanium wire explosion in air at (a) 10 kPa, (b) 50 kPa.

Figure 4 shows transmission electron microscopy (TEM) images of nanosize powders obtained with titanium wire explosion in air. Synthesized particles were observed to be almost spherical. At a pressure of 10 kPa, the sizes of most particles are less than 50 nm. In contrast, at a pressure of 50 kPa, the number of larger particles (more than 50 nm) increases obviously.

As an example, the XRD patterns for samples obtained with titanium wire explosion in air are shown in Fig. 5. All the particles are identified as pure TiO_2 (including anatase and rutile) by the XRD analysis. The content of anatase increases with gas pressure increasing.

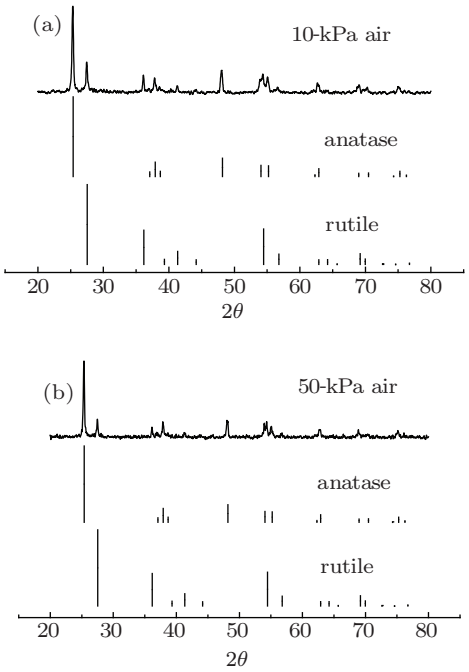


Fig. 5. XRD patterns for samples obtained with titanium wire explosion in air at (a) 10 kPa, (b) 50 kPa.

For the EEW method, the synthesized particles rapidly grow along with the thermalizing collisions and chemical reaction between metal atoms and ambient gas molecules. A decrease of ambient gas pressure would weaken the collisions and reaction, which goes against the particle growth. Consequently, lower ambient gas pressure corresponds to smaller sized particles. Similarly, the initial concentration of metal vapor that is dependent on the wire diameter affects the particle size in the same way. As shown in Table 1, the average particle size obtained with 0.18-mm titanium wire explosion is smaller than that obtained with 0.23-mm titanium wire explosion. As for 0.51-mm zinc wire explosion, the average particle size of zinc oxides is even up to 62 nm (shown in Table 1).

The dependence of average particle size on overheat coefficient k is also investigated. Generally, overheat coefficient k within specific range increases with storage capacitor voltage or ambient gas pressure increasing. Figure 6 shows the current and voltage waveforms for titanium explosion in nitrogen of 20 kPa with different values of capacitor charging voltage

u_{charge}. As u_{charge} increases, the burst peak voltage as well as the current profile become higher and earlier. Based on Fig. 6, overheat coefficient k, the ratio of deposition energy W_{d} to the sublimation energy W_{s} can be calculated. Figure 7 shows the average particle size versus overheat coefficient k.

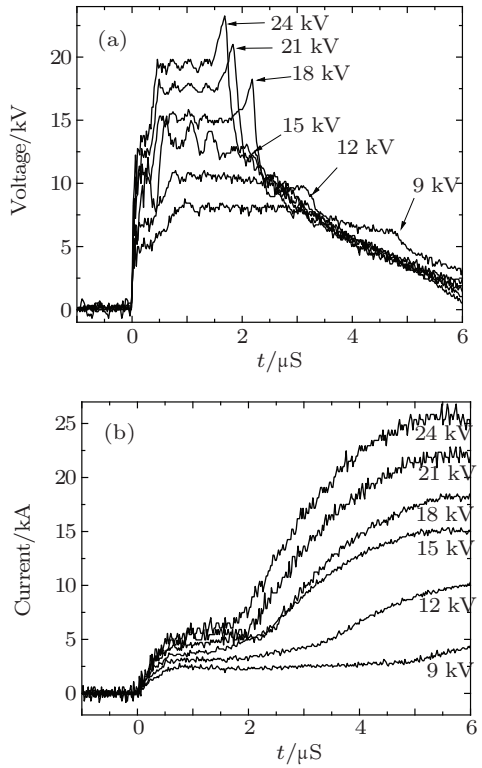


Fig. 6. (color online) Waveforms for titanium explosion in nitrogen with different capacitor charging voltages, showing (a) voltage waveforms and (b) current waveforms.

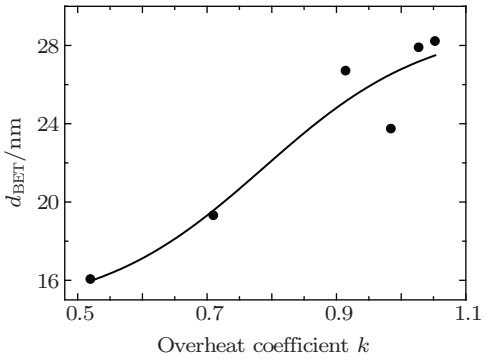


Fig. 7. Average particle size versus overheat coefficient k.

Generally, increasing the energy injected into the wire load will enhance the expansion rate of explosion products, which favors the reduction of average particle size for most metals at $k > 1.0$. As mentioned above, k for titanium metal is less or a little more than 1.0 due to the earlier flashover discharge along the wire. $k < 1$ indicates that the wire cannot be evaporated completely, so the explosion products include metal vapor and droplets in liquid state. The scattering droplets react with the ambient nitrogen (or oxygen) and burn into vapor by the released nitridation energy (or oxidation energy). The formation of nitride nanopowders is attributed to the explosion vapor as well as the sequent burning vapor. As the energy deposited in the wire rises, a larger fraction of the wire to be evaporated will increase the concentration of the explosion vapor. As a result, the average particle size increases correspondingly. Owing to the nitridation energy, titanium nitrides can be synthesized efficiently at $k < 1$.

4. Conclusions

In this article, an EEW setup for nanopowder production is constructed, and several species of nanopowder (titanium oxides, titanium nitrides, copper oxides, and zinc oxides) are successfully synthesized. Moreover, the dependence of average particle size on experimental conditions is also investigated. With suitable experimental conditions, the average particle size of synthesized powders can be easily controlled in a range of 20 nm–80 nm. The particle size, the pressure of ambient gas, and the energy coefficient k are closely related to each other. The average particle size increases with ambient gas pressure increasing. For titanium wire explosion, of which the maximum limit of overheat coefficient k is about 1.0, the average particle size increases with k increasing.

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