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Preparation of nanocomposite thoriated tungsten cathode by swaging technique ¹

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[Abstract] By using the high energy ball milling method, the nanosized ThO₂ powders were obtained. Through mixing powders, sintering and hot swaging processing, a nanocomposite thoriated tungsten cathode was fabricated. The relative density of the nanocomposite material is near 100%. The microstructure of nanocomposite cathode is quite different from that of conventional thoriated tungsten cathode. Most of thoria particles are less than 100 nm in diameter, and distribute on the boundaries of tungsten grains. The nanocomposite cathode shows a much lower arc starting field than that of conventional cathode, which will improve the performance of the cathode significantly.

[Key words] W-ThO2 cathode; nanosized powders; nanocomposite; swaging

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1 INTRODUCTION

Recent arc plasma technology has been widely used in many fields, such as robotic welding, plasma spraying, thermal plasma chemistry, cutting, metallurgy, as well as plasma thruster in space etc, due to its higher temperature, energy concentration and highly heating efficiency. The cathode as a key part of arc plasma device plays an important role in plasma application. In general, thermionic cathode materials are widely used in conventional W-2% ThO2 electrodes owing to their superior comprehensive capabilities including easy arc starting ability, lower erosion rate and better arc stability [1~4]. The major function of the thoria is to reduce the work function of the cathode so that the cathode spot is able to provide enough electrons at a lower temperature^[5,6]. Usually, the Coolidge method is widely used today for producing tungsten cathodes with little modification^[7]. After swaging, however, the morphologies of thoria in tungsten cathode are rod-like or needle-like, which deteriorate the properties of the cathode because of the coarse thoria morphologies. The previous experimental results showed that the properties of the cathodes rely not only on their composition but also on their microstructure [8~10], and with decreasing of particle size of thoria, the electron emission ability is improved greatly^[11]. For this reason, many efforts to decrease the particle size of thoria have been made.

Recently, a new kind of tungsten cathode activated with nanoscale ThO₂ particles has been developed by high energy ball milling and hot-pressing sintering technique^[9,11]. This cathode in which the size of thoria is about 100 nm demonstrates much better

performances than those of the commercial cathode in arc starting field and arc erosion. But the relative density of this kind of cathode is about 94.5%, lower than that of commercial cathode because the maximum pressing stress is less than 50 MPa, which retards the further improvement of the performance of the cathode. Also, manufacturing process is not suitable for industrial producting and greatly limites the application of this new type of cathode. The purpose of this investigation is to use the swaging method to produce tungsten cathode activated with nano ThO₂, and investigate the effect of microstructure on performance of the nanocomposite cathode.

2 EXPERIMENTAL

2. 1 Experimental materials

The materials used for this work were commercial W-2% ThO_2 material and tungsten cathode activated with nanoscale ThO_2 , respectively. The commercial cathode was selected as a reference. The processing steps for the nanocomposite cathode are outlined as follows.

Firstly, by using high energy milling technique, the nanosized ThO₂ powders were obtained. Secondly, the nanoscale ThO₂ powders were predispersed for 0.5 h by ultrasonic vibration in acetone. The average grain size of tungsten powders was about $2\,\mu m$. The mixtures of tungsten and nanosized ThO₂ powders with a mass ratio of W-2% ThO₂ were mixed for 1 h by ultrasonic vibration in acetone. The mixed powders were pressed at 60 MPa at room temperature. The green compact was presintered in dry hydrogen furnace at 1150~ 1200 °C for 1 h. This increases the strength of the compact sufficiently so that it could be

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given the final sintering treatment by passing current through it. The compact bar was sintered in a hydrogen atmosphere. The maximum temperature exceeded 3 000 °C, the total sintering time was 15~ 18 min. The final step was through continuous hand hot swaging to reach the required size.

2. 2 Microstructure analyses

By means of the Archimedes method, the density of nanocomposite thoriated tungsten cathode was measured. The microstructures of experimental cathodes were observed by transmission electron microscope (TEM), scanning electron microscope (SEM) and X-ray diffractometer (XRD), respectively. X-ray diffraction pattern was taken with a Rigaku D/ max-3A diffractometer using Cu K_{α} radiation. The thoria distribution in selected area of the nanocomposite cathode was tested by energy-dispersive X-ray spectrometer (EDX).

2. 3 Arc starting ability survey

In order to investigate the effect of microstructure and oxide particle size on the arc starting characteristic of the cathode materials, the prepared materials were machined to a disc. These samples were mechanically polished to a mirror finish and put into the vacuum chamber as cathodes. The anode made of pure tungsten was fastened. The plane of the anode facing the cathode was parallel to the cathode plane. An 8kV DC voltage was applied across the cathode and anode, and the cathode was driven upwards to the anode at a constant speed of 0.2 mm/min until arc starting occurred. After every arcing, the cathode was driven downwards to prepare for the next arc starting test. The procedure was repeated 100 times without any cathode transverse movement. macroscopic arc starting field was then calculated by dividing the applied voltage by the arc starting distance. All samples were taken out of the vacuum chamber after the 100 times are starting, and the morphology of cathode spots was observed by SEM.

3 RESULTS AND DISCUSSION

3. 1 Nanosized particles and phase composition

Fig. 1 gives the TEM photograph and electron diffraction pattern of nanoscale ThO₂ particles after high energy ball milling, showing that ThO₂ particle size is very fine and about 20~ 30 nm in diameter. The particle shape is nearly round. After swaging, a d 10 mm × 400 mm and a d 4 mm × 1000 mm rods are obtained. The density of the rods is 18. 34 g/cm³, which is about the theoretical density of W-2% ThO₂ (mass fraction) cathode, and is about 6% greater than that of cathode prepared by hot-pressing sintering method^[9]. This result shows that the swaging method is superior to hot-pressing sintering method,

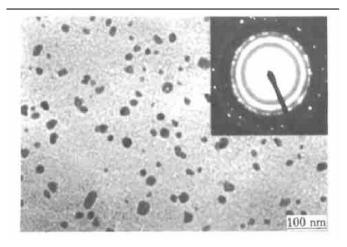


Fig. 1 TEM photograph and diffraction pattern of nanoscale ThO₂ powders after high energy ball milling in petrol

and is suitable for industrial process for nanocomposite cathode.

3. 2 Microstructure of nanocomposite cathode

The X-ray diffraction pattern displays that the two samples are of the same phase composition which consisted of W and ThO₂, as shown in Fig. 2. Fig. 3 is the SEM photographs of commercial and nanocomposite cathode samples. From Fig. 3, it can be seen that the microstructure features of the two samples are quite different. The ThO₂ in the commercial cathode is of the size of several micrometers up to 25 µm, and shows a needle like shape which is formed during swaging process. But for nanocomposite cathode, SEM observation shows that for most observation areas, it can be hardly found any thoria particles. EDX analyses show that there are still thorias in the tungsten matrix, as shown in Fig. 4. In Fig. 3(b), a few black particles about 1 \(\mu\)m in diameter can be found. The main reason for the formation of those big thoria particles is that the nanoparticles of thoria did not be dispersed evenly, and the assembled nanoparticles grew to a big particle during sintering. In order to avoid these big thoria particles, the dispersion process of nanothoria must be controlled carefully. For example, by high energy ball milling the nanothoria particles and tungsten powders could be mixed. The normal size and distribution of thoria in nanocomposite cathode are shown in Fig. 5(a). The black round particles are thoria. In Fig. 5(a), most of particles are smaller than 100 nm, and many particles are distributed on the grain boundaries of tungsten. From Fig. 5(a), it can be also noticed that some particles grew up to about 200 nm and distributed on grain boundaries during sintering and swaging.

Fig. 5(b) is the electron diffraction pattern of thoria particles in Fig. 5(a), and its indexing proves that the particles in Fig. 5(a) are thoria. From Fig. 5(a), it can be seen that the distribution of tho-

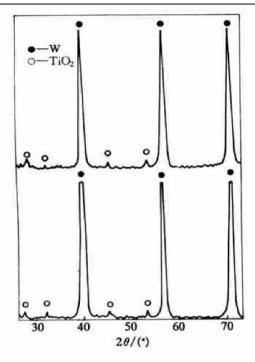


Fig. 2 X-ray diffraction patterns of nanocomposite cathode



Fig. 3 Microstructures of commercial cathode and nanocomposite cathode

(a) — Commercial cathode; (b) — Nanocomposite cathode

ria particles in tungsten matrix is not homogeneous. This distribution of thoria particles in tungsten matrix, specially, the rather bigger thoria particles on the grain boundaries are not good enough for the performance, which may include the electron emission

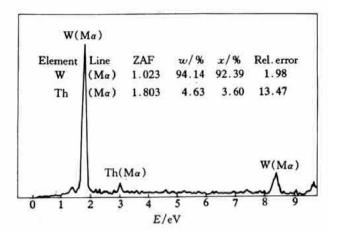


Fig. 4 EDX scanning results of selected zone of nanocomposite W-ThO₂ cathode

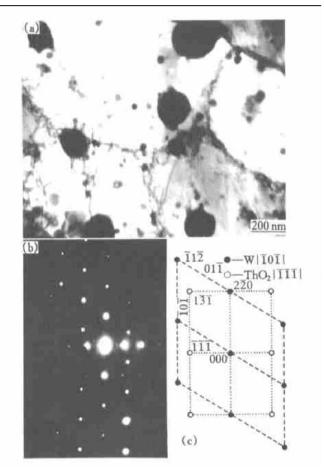


Fig. 5 TEM photograph, Electron diffraction pattern and its indexing of nanosized thoria particles distributed in tungsten matrix

(a) —TEM photograph;
(b) —Electron diffraction pattern;
(c) —Indexing of electron diffraction pattern

property and erosion rate of the cathode. If high energy ball milling is applied to the mixing of nanoparticles of thoria and tungsten powders, the nanoparticles will be dispersed better and pressed into tungsten particles. As a result, most thoria particles may distribute in tungsten matrix after sintering and swaging. Also, the big thoria particles about 200 nm in dir

ameter will be avoided because of the much better dis-

persion of nanoparticles and much lower diffusion rate of thoria in tungsten grain.

3.3 Properties

Fig. 6 presents are starting field of W-2% ThO₂ and nanocomposite W-2% ThO₂ cathode, as a function of are starting number. Fig. 7 shows the morphologies of the cathode spots after are burning of 100

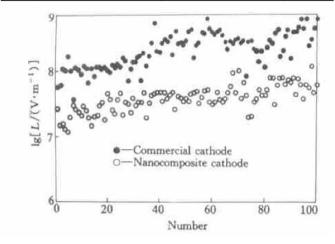


Fig. 6 Arc starting field (L) of cathode vs arc starting number



Fig. 7 Morphologies of cathode spot after 100 times arcing

(a) —Commercial cathode; (b) —Nanocomposite cathode

times. The average field of the arc starting for the nanocomposite cathode is approximately 1.43 × 10^8V/m and for the commercial cathode is 2.56 × 10⁸V/m, decreasing by about 44% (Fig. 6). A lower arc starting field implies lower work function, or stronger electron emission ability^[12]. As the size of thoria particle decreases into nano-scale, the work function of cathode materials decreases greatly, and the performance of nanocomposite cathode improves significantly. The obvious change of the surface morphology can be observed by using SEM (Fig. 7). The cathode spot of the nanocomposite sample is much more weakly molten compared to that of the commercial cathode. The reason for this is that the work function is reduced so that the cathode spot provides enough electrons at a lower temperature.

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