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Generation and characterization of zirconium nitride nanoparticles by wire explosion process

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Abstract

In the present study, wire explosion process (WEP) has been used to produce zirconium nitride (ZrN) nanoparticles. The produced ZrN nanoparticles were characterized through X-ray diffraction (XRD) and by the selected area electron diffraction (SAED) studies. The size and shape of the particles were analyzed using Transmission Electron Microscope (TEM). An analysis based on Log-normal probability distribution was used to quantify the particle size distribution of the powder. High speed photography was used to observe the wire explosion process.

Keywords: Nanoparticle; Zirconium nitride; Wire explosion; Log-normal distribution; XRD; TEM

1. Introduction

Nanoparticles often have good electrical, thermal, mechanical, chemical, optical, magnetic properties, etc., and materials scientists are challenged to identify, build, control, and test the nanostructured object, and to demonstrate the potential of these nano-structures in scientific, industrial or medical applications. By engineering size, shape, morphology and composition of the nanoparticles, the required characteristics of the nanostructured material could be achieved [1,2]. Zirconium nitride (ZrN) has good chemical and physical properties with high thermal stability, hardness, abrasive resistance and electrical conductivity [3,4]. It has many applications such as refractory material [5], hard coating for cutting tools [6] and Josephson junction in electronics [7]. Zirconium nitride particles were generated by various processes, which include, ball milling process [8], and benzene-thermal method [9]. Fu et al. [5] produced cubic phase zirconium nitride particles (in the range 30–100 nm) by reduction–nitridation of zirconium oxide powder (at 1000 °C for 6 h) in ammonia gas with magnesium as the reducing agent. Recently, Jiang and Yatsui [10] have demonstrated that it is possible to produce nanoparticles by wire explosion process and shown that it is a simplest and a cost-effective process. In the wire explosion technique, by providing proper energy to the conductor for evaporation and by maintaining suitable medium for nitridation, it is possible to generate nitride nanoparticles.

Having known all this, in the present work, a methodical experimental study was carried out to produce zirconium nitride nanoparticles of much lower size, by wire explosion process, by exploding zirconium conductor in a nitrogen atmosphere. The synthesized powders were characterized by X-ray diffraction (XRD) and by selected area electron diffraction (SAED) analysis. Size and shape of the particle formed by wire explosion process were analyzed by using transmission electron microscope (TEM). Particle size distribution studies were performed by adopting log-normal probability distribution. The relationship between size of the nanoparticles generated by the wire explosion process and the ambient pressure of nitrogen were analyzed.

2. Experimental details

The process of nanoparticle formation by wire explosion process includes conversion of solid conducting material to a

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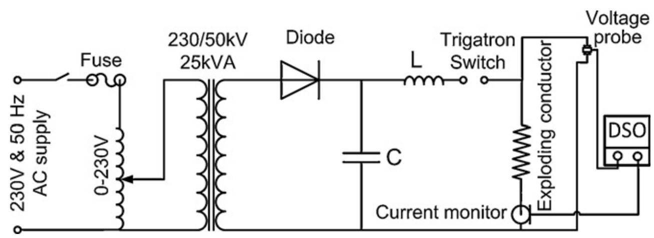

Fig. 1. Basic electrical circuit.

vapor state by injecting a high magnitude of current (of the order 10^8 A/cm^2) within a microsecond duration [11]. The vaporized metal is allowed to cause local reaction with the surrounding medium instantaneously and cooled to form nucleation of particle and then stabilized to retain its size and shape of nanosized particles. Thus, the wire explosion process is basically a one step process for the generation of nanoparticles.

In the present work, the zirconium nitride particles were generated by wire explosion process, in which zirconium conductor was exploded in the nitrogen ambience.

Fig. 1 shows a basic circuit used for exploding the conducting wires to form nanopowders. Table 1 provides the details of the parameters used in the present study. The switch S is a high voltage trigatron-gap, R is resistance of the exploding wire and L is the Inductance contributed by internal inductance of the capacitor and lead inductance. The basic circuit works like an under damped RLC circuit and maximum power it could deliver is VI. The capacitor is charged by the rectifier circuit and discharged through the wire. Energy stored in the capacitor is given by $W=(1/2)CV^2$ where, C is the capacitance of the capacitor and V is the charging voltage of the capacitor. By varying the charging voltage, it is possible to deposit the required energy. By closing the switch S, the voltage appears across the wire and the current (controlled by the RLC circuit) rises, causing Joule heating of the conductor.

The magnitude, shape and duration of current flow in the circuit depend on capacitance of the capacitor (C), resistance of the exploding conductor (R), and the circuit inductance (L) [12]. In present work, circuit parameters match the condition for an under-damped RLC circuit, where

$$\frac{R^2}{4L^2} < \frac{1}{LC} \tag{1}$$

Then, the magnitude of current flow in the circuit can be written as

$$i(t) = \frac{Ve^{(-R/2L)t}}{\sqrt{(L/C)-(R^2/4)}} \sin\left(\sqrt{\frac{1}{LC}-\frac{R^2}{4L^2}}t\right) \tag{2}$$

Table 1
Experimental details.

Capacitance	Charging voltage	Wire diameter	Length of wire
3 μF	27.5 kV	0.5 mm	39 mm

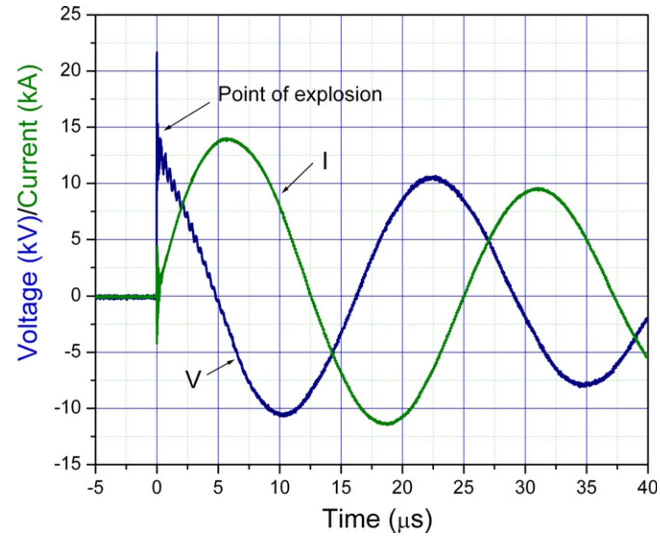

Fig. 2. Voltage and current waveforms during the explosion.

Fig. 2 is the applied voltage and the current waveforms measured during wire explosion process in nitrogen ambience. The applied voltage was measured at the input terminal of the exploding conductor using high voltage probe (EP-50K, PEEC.A, Japan). The current flow in the return conductor was measured using high bandwidth current transformer (Pearson Electronics, USA, CT Model No. 101), which is the current flow through the exploding conductor (up to the point of vaporization) and on vaporization forming plasma, thereby the plasma current continues to flow in the circuit.

At the point of explosion, a dip in magnitude of current followed with damping of current with oscillations is observed. The dip in magnitude of current at the point of explosion is due to increase in resistivity of the wire due to vaporization. The resistivity (φ) of the wire conductor vary with temperature (T) and density (γ) of the wire material and can be calculated as [13]

$$\varphi(\gamma, T) = \rho_0[1+\theta(T-T_0)]\left(\frac{\gamma}{\gamma_0}\right) \tag{3}$$

where $\gamma=\gamma_0[1-\alpha(T)(T-T_0)]$, γ_0 is the density at the melting temperature, θ is the temperature coefficient of resistivity and α is the thermal expansion coefficient. The feature of damping oscillation after current drop indicates that the resistance of the wire becomes low again, indicating that the wire is turned to a plasma state contributing continuous oscillatory current to flow even after physical absence of wire conductor [14,15]. In the present study, capacitance of the capacitor is 3 μF and summation of lead inductance in the circuit with internal inductance of capacitor is measured to be 5.3 μH, which is contributing to the frequency of oscillation of 40 kHz.

The amount of energy deposited to the wire conductor, up to the point of breakdown, has higher influence on the size of nanoparticles formed. Minimum Energy supplied to the conductor for vaporization will be equal to the sublimation energy (W_S) of that conductor. The sublimation/vaporization