Production of Nano powder of aluminum nitride by Pulsed Power Method

M. M. DAEMI ATTARAN; A. ERFANIAN; P. GHASSEMI KIAN
PULSENIRU Ind. Co.
34 Ayazi, Morghab St., Khoramshahr St., Tehran
IRAN

www.pulseniru.com

Abstract: - After revision of applications, physical and chemical properties and various methods for production of nano powder of AlN, Pulsed Electrical Exploded Wire Discharge (EEW) method for production of various kinds of these nano powders has been studied in detail. In this research, the theory of Electrical Pulsed Wire Discharge and also optimum parameters of wire explosion has been investigated. The pulsed power system that used in this method included a 6μF-50kV capacitor bank, 20kV Spark gap switch with current peak of 150kA and driver circuit, control and related charger and voltage and current diagnostic instruments.
Capacitor will be charge up to 9kV and will be discharge by spark gap switch into the wire. Current pulse has been measured by a high frequency Rogowski coil. After every discharge, the wire will be exploded, melted, Sublimated and condense in collision with ambient nitrogen gas particles. Collection of nano particle produced by this method will be done by a 50nm membrane disc filter, by circulation of nitrogen gas with a dry pump.
SEM observation representative of high percent of AlN nano powder in achieved powder with average less than 50nm dimension. AlN nano powder uses for cooling applications in electronic industries and for RF windows coating in microwave industries.

Key – Words: - AlN, Nano Powder, EEW, Low Inductance Pulse Capacitor, Pulsed Power, and Spark Gap

1 Introduction

The existence of AlN was discovered in 1862 by F. Briegler and A. Geuther and the first synthesis was realized by J.W. Mallets in 1877[1]. The possibility of producing Al nitrides by EEW method was demonstrated in 1966 by Joncich[4] . Since AlN have high thermal conductivity therefore it can be used as a substrate for high density high power integrated circuits [6].
Various methods for production of AlN nano powder are as carbothermal reduction (reaction among Al_2O_3, C and N_2) and direct nitridation (Nitridation of Al powder by N_2) that are commercial methods, CVD, RF plasma, Arc plasma and EEW method [2].Among these methods EEW seems to be very hopeful for the mass production due to high conversion efficiency using simple and compact devices [3].
Very fine powders of average size on the order of 10 nm have been obtained by this method. Electrical Exploded Wire (EEW) is a unique process where high-density plasma can be easily produced by driving a pulsed high current through a thin wire. It has been proved that the plasma produced by pulsed wire discharge can be used in synthesis of nanosize powders [5].
In the current paper we described our experiments and achieved results in production of nano powder of AlN by EEW method.

2 Theory

As shown in fig.2 When a high-density (10^4–10^6 A/mm^2) current pulse, (which is usually produced by the discharge of a capacitor bank), passes through a wire, the density of the energy in the wire may considerably exceed the binding energy because of a high rate of the energy injection and an expansion lag of the heated material. As a result, the material boils up in a burst, a bright light flashes, and a mixture of superheated vapor(plasma) and boiling droplets of the exploding wire material and a shockwave scatter to the ambient atmosphere [4].
The generated plasma expands naturally into the ambient gas that will be cool slowly and uniformly in the process of particle collisions [5]. These collisions caused reaction between Al and N_2 so AlN Nano powder will be produce.
We define critical length ℓ_c of the exploding wire, when the current pause is zero and the exploding metal is overheated mostly without the arc discharge, which

accompanies the explosion that is describe by following expression:

$$\ell_c[\text{mm}] = \text{B}(\text{W}_0\text{DLZ})^{0.36} \qquad (1)$$

That B=27.7 for Al, $W_0 = \frac{1}{2}CV^2$, D =Wire diameter ,L= circuit inductance and Z= circuit impedance that equal to $\sqrt{\frac{L}{C}}$ [4].

When the wire length $\ell < \ell_c$ or ambient-gas pressure is too low, explosions are accompanied by a surface discharge and electrical breakdown may occur along the wire surface or within the gas, especially at the moment when the wire is vaporized.
Therefore unexpected breakdown may cause inefficient wire heating and, as a result, metal droplets; (Figure 1d). When $\ell > \ell_c$, explosions occur with a current pause (Figure 1b) [4].

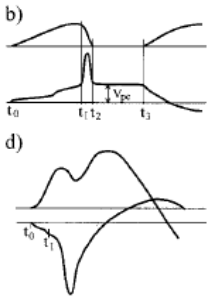

Figure1:discharge current(top) and voltage(down) oscillograms: current-pause explosion (b);explosion with surface arc discharge (d)

3 Experimental procedure

Typical Experimental conditions are as following table:

Parameter	Value
Ambient Gas	N_2
Wire Diameter(D)	0.2mm
Wire Material	Aluminum
Heat resistance h_b	0.9-1.09A^2s/mm^4
Sublimation Energy w_s	33J/mm^3
ℓ Wire length	75mm
Charge	9kV
Voltage V	
Current Peak	22KA
Capacitance C	6μF
Circuit Inductance L	0.4μH
Circuit Resistance R	0.15Ω
Circuit Impedance Z	0.2582Ω
Capacitor Energy W_0	243J
Dimensional Constant B	27.7

Table 1: Experimental Setup Properties

Our Experimental setup is as figure 2.

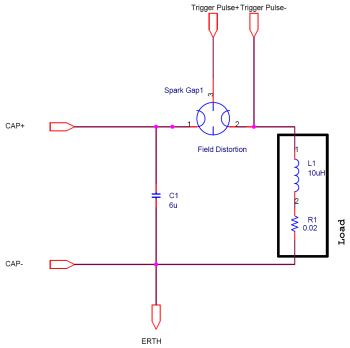

Figure 2: Exprimental Setup For Production Of Nano powder by EEW method

A low inductance(14nH) Capacitor bank will be charge up to 9KV through HVDC power supply and then discharge in to the explosive wire that be stood between two springy clamps by a field distortion mid-plane spark gap switch model FD20-100 that made by PULSENIRU Co.
Ambient gas pressure of Reaction chamber decrease up to 0.1 Torr through a circulation vacuum pump and then fill by N_2 gas up to atmospheric pressure. The produced Nano particle will be collected by a 50nm membrane disk filter that placed through circulation pump. After every discharge, the wire will be exploded, melted, Sublimated and condense in collision with ambient nitrogen gas particles.

4 Results

Discharge current of explosion of wire in ambient gas has been measured by Rogowski coil as be shown in fig.3

As be seen when switch close completely the current raises (point 1) so wire start to be exploded in (point 2) and will exploded completely in (point 3) that circuit will be open in this time because increasing in plasma resistance.

After explosion, current will be continuing with reminded voltage across capacitor through plasma (point 4). Average particle size that be obtained by SEM analysis has been showed diameter between 26-44nm for collected Nano particles (fig.4).

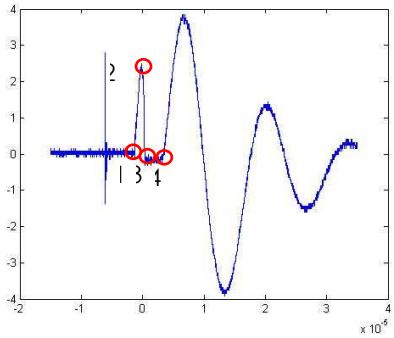

Figure 3: Discharge Current

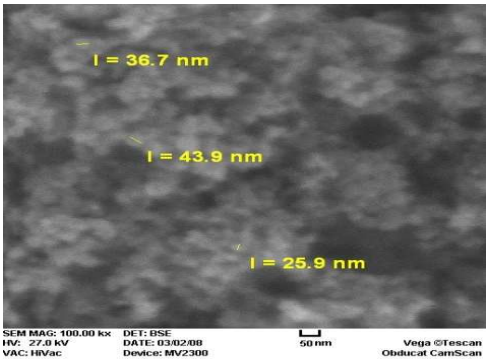

Figure 4: SEM Photograph of Nano powder

5 Conclusion

EEW method has been used to synthesis of AlN nano powder and the following notice has been concluded:

1. When length of exploded wire is more than critical length we have current paused explosion.
2. AlN nano particle with size of 26nm has been obtained by EEW method

References:

[1] Bernhard H. Mussler, Aluminum Nitride (AlN), *Am. Ceram.Soc.Bull.79(6)*,2000,http://www.anceram.com/pdf/aln_artikel_bull.pdf

[2] M. M. Daemi Attaran,"Production of AlN nano powder by pulsed wire discharge", *Malek Ashtar University, master thesis for electrical engineering*

[3] Weihua Jiang and Kiyoshi Yatsui, "Synthesis of nanosize powders by pulsed wire discharge, " *11th IEEE International Pulsed Power Conference, Vol. 1, Page. 214 – 219, 1997.*

[4] Yu A. Kotov,"Electric explosion of wires as a method for preparation of nanopowders ", *Journal of Nanoparticle Research: 539–550, 2003, Kluwer Academic Publishers. Printed in the Netherlands*

[5] W. Jiang, T. Suzuki, H. Suematsu, and K.Yatsui, "INDUSTRIAL APPLICATIONS OF PULSED WIRE DISCHARGE*", 14th IEEE International Pulsed Power Conference. Vol. 1, Page. 433 – 436, 2003.*

[6] Y.kinemuchi, C.Sangurai, T.Suzuki, H.Suematsu, W.Jiang and K.Yatsui, "Synthesis of nanosize powders of aluminum nitride by pulsed wire discharge", *Pulsed Power Plasma Science, Vol 2, page. 1830- 1833. , 2001.*