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# An experimental approach to an optimized method for producing $Al_2O_3$-coated aluminum nanopowder as a core-shell superconductor by electro explosion of wire

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The most common way to produce aluminum nanopowders is electro explosion of wire. In this procedure we just need to have available source of electrical energy. Aluminum nanopowders have a wide range of use like metallurgical or chemical industries. $Al_2O_3$ coated Aluminum nanopowders are also used as high temperature superconductors. This research is based on electrical explosion of wire by using low voltage DC current with suitable amperage. Effect of using various voltages was studied and the best parameter for it was achieved at 36 V. At the same time, ethanol as a suitable atmosphere is suggested and a very cost effective way of producing aluminum nanopowders offered which can produce 20 to 150 nanometer aluminum nanopowders. Transmission electron microscopy and dynamic light scattering were employed to analyze and measuring respectively. Also X-ray diffraction method was used to demonstrate quality of $Al_2O_3$-coated aluminum nanopowders.

**Key words**: aluminium nanopowder, electro explosion of wire, eew, core-shell superconductor.

## INTRODUCTION

Annual sales of aluminum powders (ANP) and granules (<1 mm) worldwide is estimated at 200 k tones each year primarily comprising sales to the metallurgical, chemical and paint and pigment industries. Specialist end uses include rocketry, explosives, powder metallurgy, etc. [1]. Production of high temperature superconductors from core-shell $Al_2O_3$ coated aluminum nanoparticles which its critical temperature is three times that of pure aluminum is one of the recent usage of it [2].

The most common way to produce aluminium nanopowders is Electro Explosion of Wire (EEW) [3].

High productivity (100 - 200 g of ANPs/h for each machine) and low cost of the powders, because electrical energy is consumed for the metal wire destruction only (no additional plasma sources, no vacuum and cooling system), are the reasons of popularity of this procedure [4].

The most important advantage of this procedure is to straight transition of electrical energy to thermal energy. EEW explosion could force materials to transit to liquid or vapor or plasma condition. The velocity of expansion of atoms could be reach to 1000 m/s; and temperature also could reach 10000K [5]. Mechanical milling is not good to produce aluminum nanopowders. Usually EEW and PC are used for producing this product [6].

EEW is a phenomenon of explosive destruction of a metallic wire when passing a high density current (more than $10^{10}$ A/m$^2$) through the wire with use of LC-circuit. The main information about the explosion process is obtained from waveforms of voltage and current (Fig. 1). Initially, the wire is heated-up by resistance heating and partly melted (slight increase in the voltage waveform). Further heating until t1 makes the wire liquid state. From this moment the wire begins to expand intensively and thereby the electrical resistance increases abruptly by several orders. At t2 the current in the circuit ceases and a pause phase is built. At t3 the breakdown of the explosion products occurs and the arc stage (secondary discharge) begins. If the residual voltage across the capacitor is small or equal to zero, the arc stage does not exist [4].

**Table 1.** Conventional methods for production of ANP's:

| Method | Quoted size (nm) | Production Rate |
|---|---|---|
| Electro-explosion of wire | 50–500 | 100 g/h |
| DC Plasma torch | 50–150 | 2 kg/h short run |
| Chemical: alane adducts | 65–500 | Low |
| Inert gas atomisation | 100–5000 | 0.5 kg/h |

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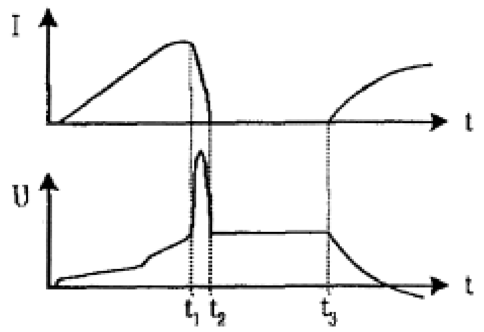


**Fig. 1.** Typical waveform of I and U vs. Time during EEW process.

Particle size can be controlled by controlling pressure in the reactor. Reduced pressure in flame pyrolysis leads to rapid quenching and fine particles, but at normal pressure there is an opportunity for coarsening by coalescence [5].

Fig. 2 shows a schematic of the cooling rate versus particle size. Rapid solidification in finer powders confers desirable microstructural refinement that is exploited in some powder metallurgical products to achieve, e.g. optimum dispersion strengthening or superplastic forming properties in consolidated parts [7].

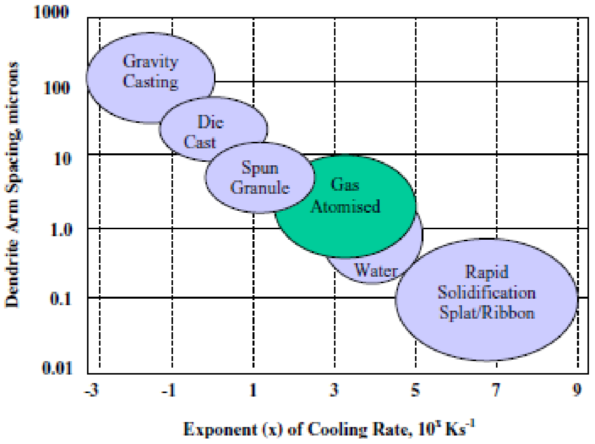


**Fig. 2**. Effect of cooling rate on dendritic arm Spacing.

Because traces of these substances are present almost everywhere: during the period after production and before application, the powders often come into contact with such oxidizing media. Hence, a lot of studies, devoted to this problem, were published, but the problem of the protection of the surface of the metal nanoparticles seems to be one of the most important for the development of applications for industrial metallic nanopowders [8]. Surface passivation is one of the most important problems about production of aluminum nanopowders [9] but to produce core-shell $Al_2O_3$ coated aluminium nanoparticle for the superconducting usage, there should be a thin layer of oxide layer around the nanopowder [2].

The coating of particles by organic reagents leads to a considerable reduction of the specific metal content because the particles hold a lot of organic substances on their surface [4].

The passivation may comprise exposing the powder to a passivating atmosphere containing argon and oxygen while periodically or continuously mixing such powder to maintain exposure to such atmosphere while maintaining a temperature of such powder at or below 20$^{o}$C. The passivation may comprise coating the powder to be passivated with a coat to retards penetration of oxygen and exposing the coated powder to an atmosphere containing oxygen concentration high enough so that the powder would initially combust absent the coat. The exposure is for a period of time effective to allow the atmosphere to form a passivating oxide layer on the powder. The coat may contain a long chain aliphatic carboxylic acid. The coat may be removed when the oxide layer has a thickness effective to prevent spontaneous combustion in air. The passivation may be performed while cooling the powder and the time may be 10-30 hours. The wire may be exploded in length of between 15 and 30 cm and a diameter of between 0.3 and 0.6 mm. in common procedure the explosion may be performed in an atmosphere consisting essentially of argon or an argon/hydrogen mixture [10].

## MATERIALS AND METHODS

In this research we used DC power supplies with suitable amperage and variable voltage. We used 12V batteries to produce 12 to 36V power supplies. As the explosion take place in a moment we should have a power supply which could maintain useful amperage in this procedure. It is common to use transformer and capacitors in one circuit to achieve this amount of amperage. If we the power supply be unable to produce this amount, welding situation would take place and there would be no nano particle size produced. In this research we used 12V batteries as an easy way to achieve suitable amperage.

Based on Mr. Perasenjit, et Al. patent [11], a homemade EEW apparatus was made. Fig. 3 shows the schematic view of this apparatus. There would be two electrodes from the same material we want to make nanopowders from them. We used aluminium wire with the diameter of 0.4 mm; and also a plate of aluminium as anode. Each of them was connected to the power supply with copper wire. In this procedure both electrodes are used to make nano particles. So in this research we used aluminium foils to rate the production of nano particles. In commercial way of production about 150 g of ANP's produces in each hour [6] and in this research the rate of 30 g per hour achieved that is reasonable; because of the low cost of investment.

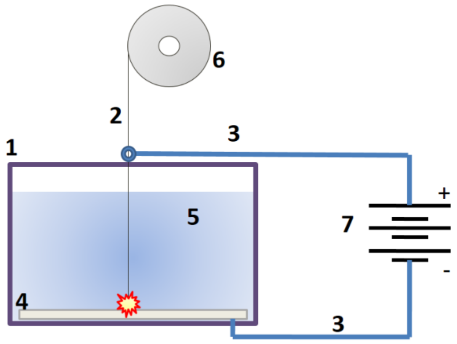


**Fig. 3.** Schematic view of homemade EEW apparatus: 1- Explosion Vessel, 2- Aluminium wire, 3- Copper wires, 4- Aluminium foil, 5- ethanol, 6- wire feeder, and 7- Batteries.

In commercial procedure it is common to use Argon atmosphere which is cycled by turbo-compressors in the explosion vessel [10]. In this research we used pure Ethanol as a liquid atmosphere which obtains suitable cooling and has a good viscosity that allows production of nanopowders.

It is very important to be conscious about combustion of Ethanol. So the anode should be place in the down part of vessel, because there would be no oxygen to start burning there. It is also important to have closed door vessel as shown in Fig. 3. There is just a tiny hole on the upper part of vessel.

Finally, DLS test were done by a ZEN 3600 apparatus were made by McLaren Company England to find out how is the size distribution of powders; Electron Microscope Photographs were taken by an 80Kev EM 900 electron microscope that confirmed the results; and also XRD test were done to confirm $Al_2O_3$ aluminium nanopowders were produced.

## RESULTS AND DISCUSSION

The test took place under the situation of using 12, 24, and 36 volt power supplies. As the voltage increases the size distribution of powders get smaller and smaller. By using 36 volt power supply the nanopowders produced that their size distribution were about the commercial ANP's.

By using 12V power Supply the size distribution were about 450 to 1480 nanometers which are unacceptable (Fig. 4).

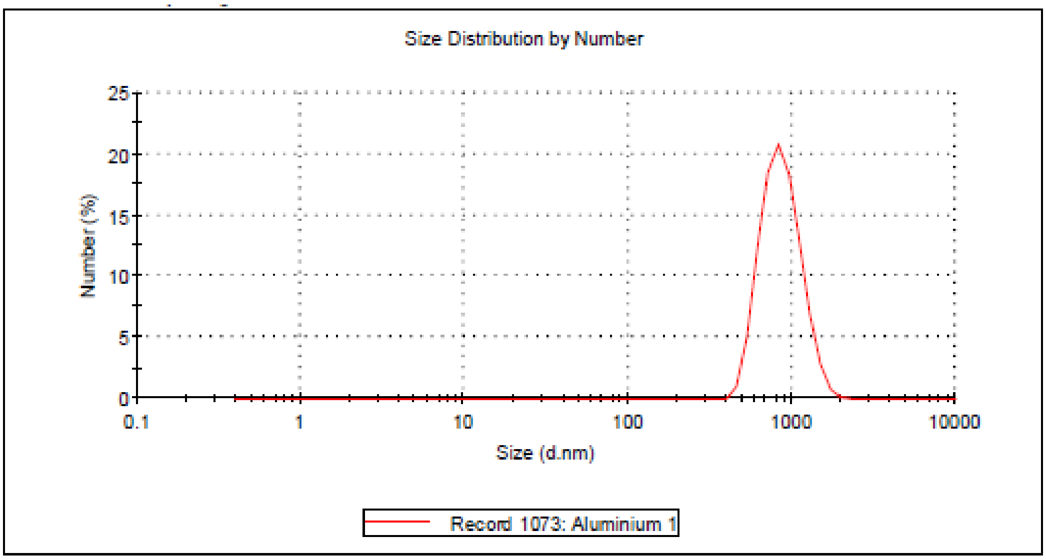


**Fig. 4.** Size distribution of powders produced by 12V power supply

In the Fig. 5 and Fig. 6 there would be shown two picks which may be as the result of variable diameter of wire in various parts [12].

By using 24V power Supply the size distribution were about 105 to 255 and 400 to 1400 nanometers which are bigger than commercial product.

As can be seen in fig. 5 the size distribution is getting smaller by increasing the voltage which causes the increase of supplied energy for explosion of wire. It is easily predictable that by one another increase in voltage would lead to production of smaller nanopowders

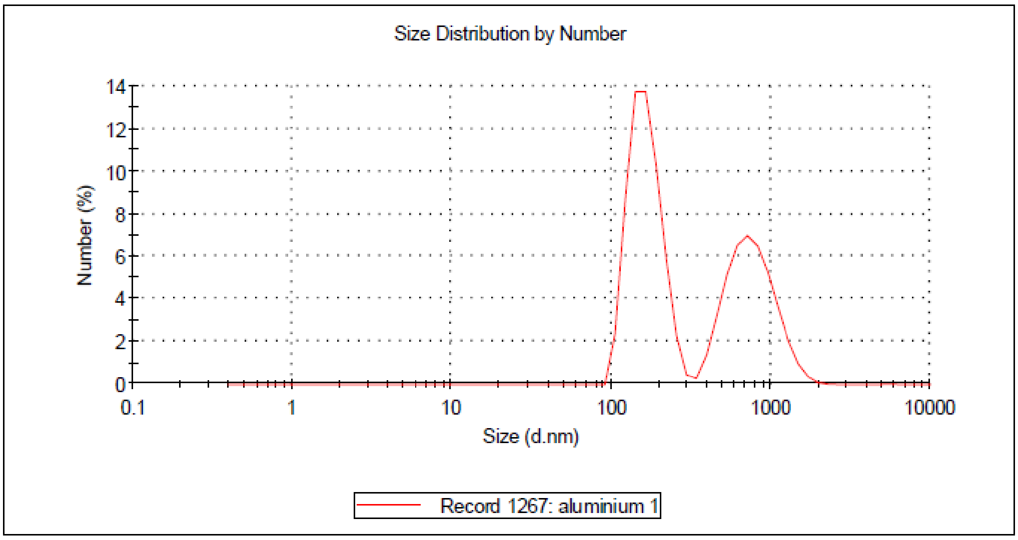


**Fig. 5.** Size distribution of powders produced by 24V power supply

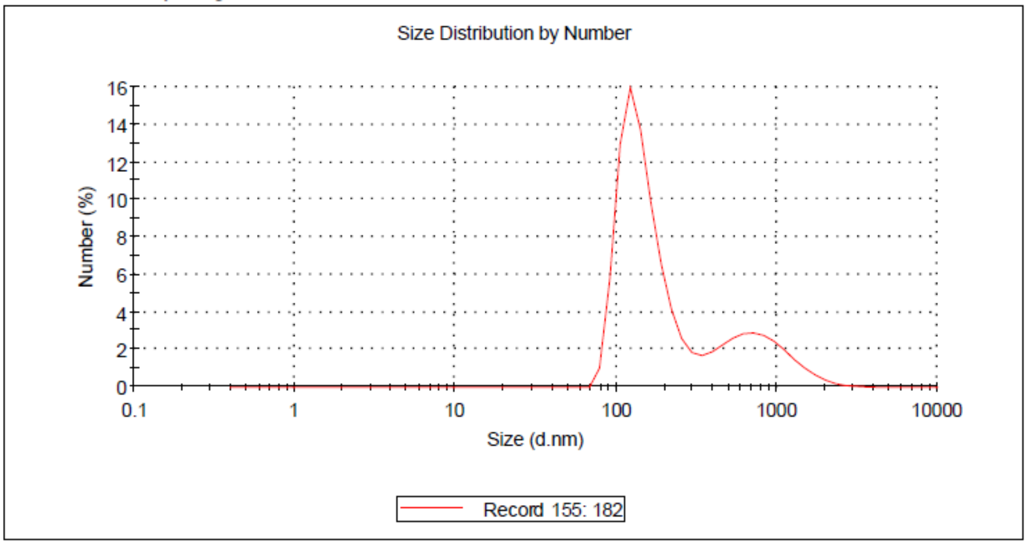


**Fig. 6.** Size distribution of powders produced by 36V power supply

By using 36V power Supply the size distribution were about 78 to 220 nanometers which are as same as commercially produced nano particles.

By taking electron microscope photographs, we found out that the size distribution of nanopowders are between 20 to 150 nanometer. The reason of difference of the result of DLS test and Electron Microscope Photography is cheaper accuracy of DLS test. Table 2 shows the complete rusults of DLS tests.

Refer to table 1, in comparison with Mr. Kearns' results [1], it is intelligible that produced nanopowders are smaller and their size distribution is about the results of DC Plasma torch method. It could be as a result of stable energy providence, suitable viscosity, acceptable cooling power, and oxidation preventing environment. So vaporized atoms would be propagated in long distances and then they would be cooled down and make nanoparticles before being completely oxidized.

**Table 2.** Size distribution of powders produced by homemade eew apparatus:

| **Size d. nm** | **Mean Number %** | | |
|---|---|---|---|
| | 36 V | 24 V | 12 V |
| **78.82** | 0.1 | 0.0 | 0.0 |
| **91.28** | 5.8 | 0.0 | 0.0 |
| **105.7** | 12.9 | 2.4 | 0.0 |
| **122.4** | 15.9 | 8.6 | 0.0 |
| **141.8** | 13.7 | 13.7 | 0.0 |
| **164.2** | 9.9 | 13.7 | 0.0 |
| **190.1** | 6.6 | 10.3 | 0.0 |
| **220.2** | 4.2 | 5.9 | 0.0 |
| **255.0** | 2.6 | 2.3 | 0.0 |
| **295.3** | 1.9 | 0.5 | 0.0 |
| **342.0** | 1.7 | 0.3 | 0.0 |
| **396.1** | 1.9 | 1.4 | 0.0 |
| **458.7** | 2.3 | 3.2 | 1.0 |
| **531.2** | 2.6 | 5.2 | 0.0 |
| **615.1** | 2.8 | 6.5 | 12.3 |
| **712.4** | 2.9 | 7.0 | 18.5 |
| **825.0** | 2.8 | 6.5 | 20.8 |
| **955.4** | 2.5 | 5.3 | 18.4 |
| **1106** | 2.0 | 3.6 | 12.9 |
| **1281** | 1.5 | 2.1 | 7.1 |
| **1484** | 1.0 | 1.0 | 2.9 |
| **1718** | 0.7 | 0.4 | 0.8 |
| **1990** | 0.4 | 0.1 | 0.1 |
| **2305** | 0.2 | 0.0 | 0.0 |
| **2669** | 0.1 | 0.0 | 0.0 |
| **3091** | 0.1 | 0.0 | 0.0 |
| **3580** | 0.0 | 0.0 | 0.0 |

By using EEW procedure, Argonide produces aluminium nanopowders that their size distribution is about 50 to 500 nanometer, and the rate of production is about 100 g per hour. And also Tetronics produces 50 to 150 nanometer powders by using DC plasma torch procedure. By comparison of the results we would find out that by using this low cost way, smaller and better aluminium nanopowders are produced [1].

Because of lower need to investment the lower rate of production is completely ignorable. At the same time by using 36 V power supply the range of produced ANPs is got smaller than conventional procedure and it could be compared by plasma assisted procedure which make this new procedure completely cost effective.

Doing the passivation during the cooling is another profit of this low cost way of producing ANPs. As can be seen in Fig. 7 there is a thin layer of $Al_2O_3$ around the spherical Aluminum nanopowders. This thin coat of $Al_2O_3$ improves the superconducting properties of aluminum nanoparticles in comparison whith pure aluminium.

As can be seen in the Fig. 7 the morphology of nanopowders are spherical which is suitable, because in the next steps we need to compose it by other products to make a final product.

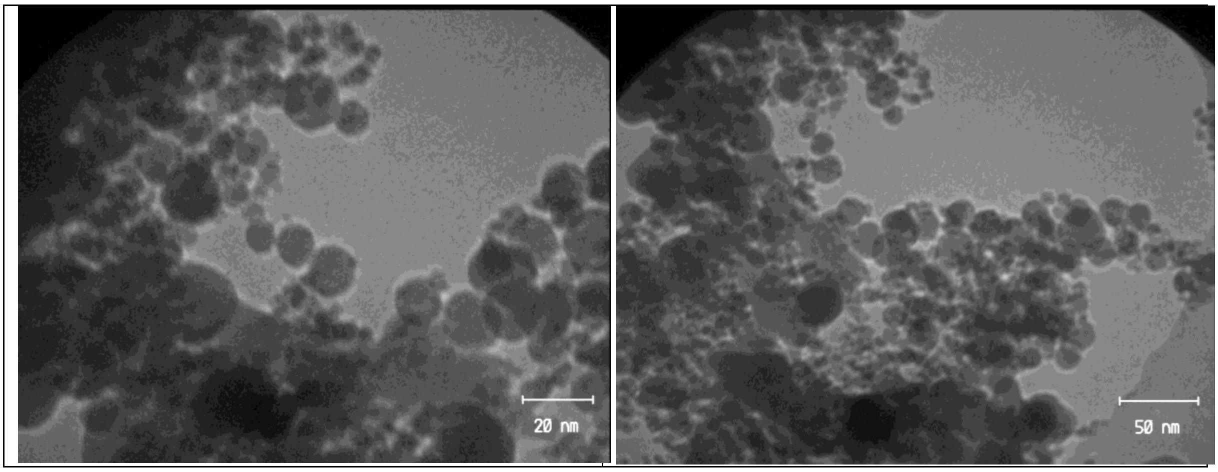


**Fig. 7**. Nanopowders produced by 36V power Supply.

XRD test showed that the nanopowders were purely aluminium with FCC substructure (Fig. 8). So Ethanol can prevent the oxidation of nano particles. Because of the nanometric particle size of nanopowders XRD could not show the presence of thin $Al_2O_3$ coat but its presence were proven by electron microscopy.

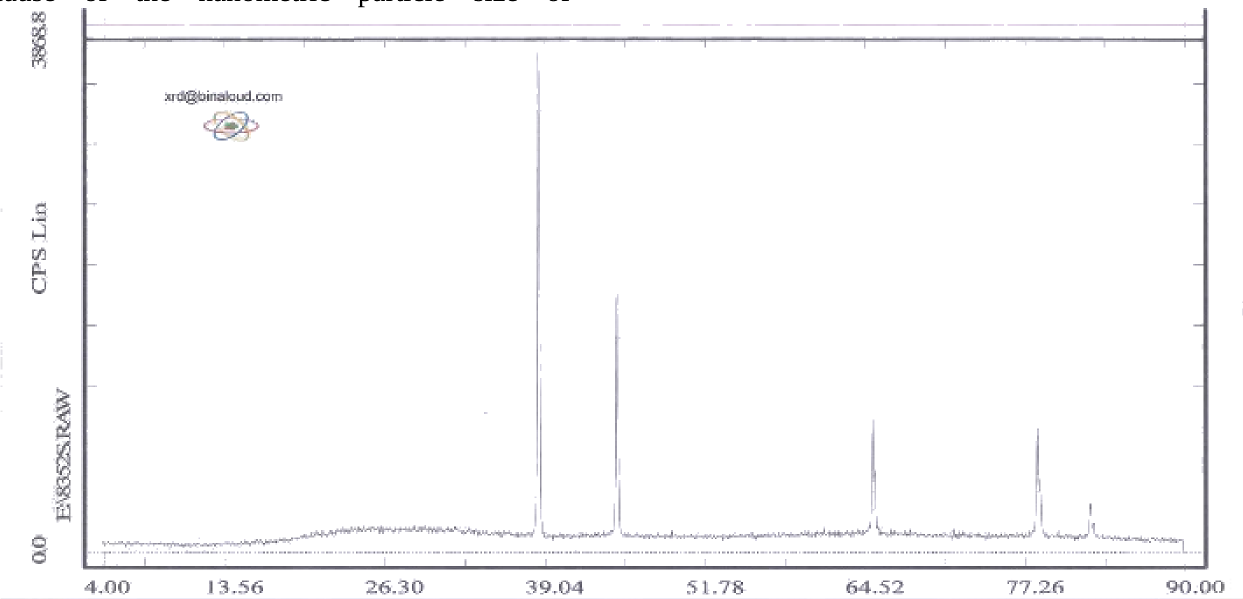


**Fig. 8.** The result of XRD test which prove synthesis of pure aluminum with FCC structure.

Since there is no reported oxidation by XRD, it is visible that Ethanol is well selected. Good velocity, cooling power, and preventing from completely oxidation of nanopowders were achieved by using Ethanol.

As mentioned before by increasing voltage the size distribution got better and better and finally the suitable rang produced by using 36 volt DC power supply. A low cost way of producing $Al_2O_3$ coated ANP's suggested and a good and easily available atmosphere found in this study.

## CONCLUSION

A low cost way of producing $Al_2O_3$ coated ANP's which can be used as core-shell high temperature metamaterial superconductor suggested that could produce nanopowders with size distribution between 20 to 150 nm that are smaller than common EEW method. Effect of various voltages on the size distribution of produced ANP's studied and 36volt as the optimum voltage for producing acceptable ANP's suggested.

Ethanol as a good liquid environment which prevent powders from complete oxidation and perform a useful rate of cooling offered. A thin layer of oxide layer would be present to produce and improve the superconductivity properties of powders. And finally a very low cost apparatus for making ANP's presented.

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