# Synthesis of Nanosized Powders Using Wire Explosion[1]

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**Abstract – This paper presents new approaches to the producing powders using high-power Joule heating and high-energy plasmochemistry synthesis. The production of ultra-fine powders based on such metals as Al, W, Zr, Cu, Fe, Ag, Co, In, Pt, Ta, Ti, Sn, C has been investigated.**

**The influence of initial conditions on properties of powders is discussed. Basic factors are as follows: level and uniformity of heat, power of heating, density and chemical activity of surroundings, initial radius of wire, and initial crystallite's size of the wire metal. Investigations performed have shown that the electrical explosion allows synthesizing powders of different chemical compositions.**

**New technology was developed for manufacturing of novel carbon nanomaterials (fullerenes, nanotubes, carbonic nanoclusters) based on the idea of high-energy plasmochemistry synthesis with the use of explosion of metals in organic medium. A wide spectrum of fullerene-like materials including the highest one ($C_{70}$ and higher) was discovered. Ferromagnetic properties of carbon nanomaterials were detected and studied.**

## 1. Introduction

An interest now displayed in ultra-fine powders is determined by the promise of producing materials with new, improved properties. The full potential is not disclosed completely. According to the rising capabilities of various applications, various methods of the ultra-fine powders production and large-scale manufacture are developed and transferred to a technology. Among them, the exploding wire method is widely investigated by the researchers and companies of Russia [1–3], Ukraine [4], Germany [5], Japan [6], India [7], Korea [8], and others. First, systematic investigations have had a good start in Russia in the 70's [9, 10].

The major questions are how to produce a powder with specified characteristics and how to use the powder produced.

A choice of conditions for synthesis of powder with given properties is a serious question. Major properties of the produced powders, for instance, the median size, are considered at least in six-dimensional space of initial conditions. The introduced energy density $w$, the rate of heating or the current density $j$, the rate of cooling of expanding medium (i.e., the diameter of a wire $d$ and the density of environment $\gamma$), reactivity of environment, and initial microstructure of wire metal are also important factors [1–3].

With that, metals differ in such thermophysical properties as sublimation energy, ionization potential $w_i$, resistivity, density, etc. They behave differently under the Joule heating and electrical explosion. Besides, the resistance of wire influences on the current in the electric circuit, and an explosion can occur in widely varying modes.

Therefore, the goal of the present work is to systemize investigations on exploding wire method of controlled synthesis of nanosized powders and on the role of initial conditions in particles formation.

Next section describes the energy consumption and conditions of the matched explosion mode with the given specific energy introduced into the wire. Section 3 is dedicated to the principal regulations of particles formation; our description rests on evidence obtained experimentally. A description of synthesis of carbon nanomaterials is made in final Section 4.

## 2. The energy consumption

The electrical circuit used for the exploding wire is shown in Fig. 1, *a*. In Fig. 1, *b* a typical oscillogram for the discharge current is shown.

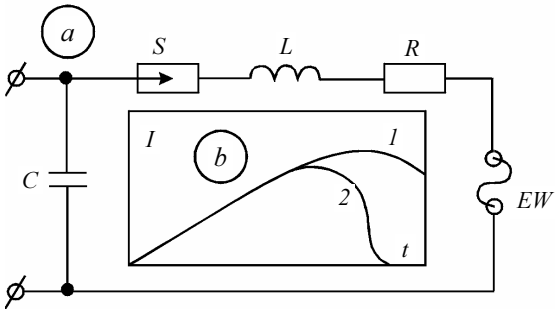


Fig. 1. The electric circuit of the set (*a*) and the current oscillograms (*b*) for shorting (*1*) and during the electrical explosion (*2*)

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To perform an explosion, the capacitor, $C$, is discharged through the wire to be exploded ($EW$) having the cross section $S$ and the length $l$.

The greatest advantage of the exploding wire method over other evaporation techniques lies in the fact that the electric energy is transferred directly into the heat.

The specific energy released in a wire due to Joule heating is given by the equation

$$w = \int j^2 \rho dt \approx \overline{\rho} \int j^2 dt, \qquad (1)$$

where $j$ is the current density in the wire; $\overline{\rho}$ is the specific resistance, and $t$ is the time.

Based on the general notions about the wire material state, the process can be classified in two stages: the heating stage and the stage of the explosion as such. At a certain point in time corresponding to the end of the heating stage the explosive-like expansion and abrupt increase in the specific resistance begins. At the heating stage, the material resistance is mainly determined by the introduced energy, or by the temperature $T$, and here one can use the notion of "the integral of specific current action" $h$:

$$h = \int_{T_0}^{T} \gamma \frac{cdT}{\rho(T)} = \int_{w_0}^{w} \frac{dw}{\rho(w)} = \int_0^t j^2 dt, \qquad (2)$$

where $c$ is the specific heat; $\gamma$ is the density of the given metal.

There exists an unambiguous relation between the energy density introduced and the integral of specific current action $h_e$, corresponding to the whole explosion. Using physical modeling and numerous experimental data of author's work, simple dependencies on the similarity criteria, identical for all metals, have been found for the energy introduced into the wire and the maximum value of current through the wire:

$$\frac{w}{w_0} = \left(\frac{h_e}{h_0}\right)^{0.5}, \quad h_e \le h_0;$$

$$h_0 = j_0^2 (LC)^{0.5}/2 = CV_0^2/(2S^2 z),\ z = (L/C)^{0.5}, \qquad (3)$$

$$\frac{j_m}{j_0} = \frac{i_m}{i_0} = \frac{i_m z}{V_0} = \left(\frac{h_e}{\pi h_0}\right)^{0.25}, \quad \frac{h_e}{h_0} \le \pi. \qquad (4)$$

In Fig. 2, the graph of regression (3) and experimental points from open literature are plotted.

The data above suggest that the current density is a significant factor in explosion. The current density determines the rate of Joule heating, $dw/dt \sim j^2$ in accordance with (1). The energy $w_0$ stored in the capacitance $C$ with the initial charging voltage $V_0$, $w_0 = CV_0^2/2Sl$, and "store" of the specific action, $h_0$, are also essential factors. Experimental points are agreed with regression (3).

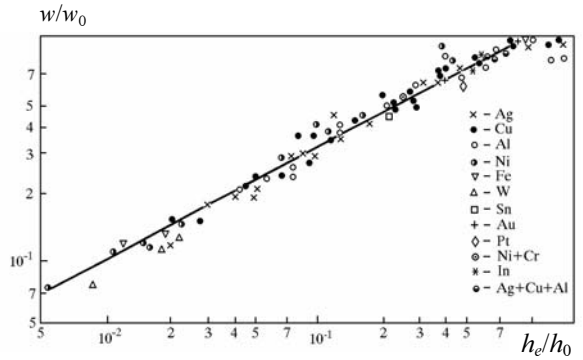


Fig. 2. The energy ratio introduced into the wire of various metals depending on the action ratio

Important consequence from (3) and Fig. 2 is the so-called "matched" explosion mode. In the matched mode, the energy $W_0 = CV_0^2/2$ completely dissipates in the wire metal during the first pulse of current. This takes place with $h_e/h_0 = 1$, and the energy-transfer coefficient, $w/w_0$, is equal to unity.

Using dependence (3), not only the energy consumption factor and the matched mode but also the overheating achievable under given conditions, i.e., the ratio of the energy introduced into the metal to the metal sublimation energy $w_s$, can be determined. A reverse problem (estimation of generator parameters needed to explode a given wire with required overheating $w/w_s$) can be solved as well.

The specific action integral is easily estimated from the energy (temperature) dependencies of the metal specific resistance or by use of several current oscillograms, in accordance with (2). The values of $h_e$ for some of the metals investigated with the current density ~ $10^7$ A/cm$^2$ are presented in Table 1.

Table 1. Properties of some metals

| Metal | Ag | Cu | Ni | Ti | In |
|---|---|---|---|---|---|
| $w_s$, kJ/cm$^3$ | 27.8 | 47.5 | 65 | 10 | 20 |
| $h_e$, $10^9$ A$^2$ · s/cm$^4$ | 1.0 | 2.0 | 0.7 | 0.3 | 0.2 |

Investigations show that it is possible to decrease the size of particles using conditions of fast electrical explosion and reduced pressure of surroundings. Under the conditions of fast electrical explosion the heating occurs uniformly over the wire length and radius. In this study, conditions for uniform heating [2] were fulfilled.

### 3. Principal regulations of the particle formation

The energy density introduced in the EW is one of the most important parameters of method. Recall that there exists a critical value, $w_{cr}$, such that with $w < w_{cr}$ the explosion fails to occur, and that for $w \ge w_s$ the velocity of expansion of products is given by $v = (W/m)^{0.5}$, $m$ is the mass of wire metal. It is clear that the mean particle size and the particle size distribution function will vary with increasing input

energy density. This has been demonstrated by a number of researchers. The average numerical size of the particles can be easily established [10] according to the dependence:

$$d = 0.3 \cdot 10^{-6} (w / w_s)^{-3}, \text{ m}. \quad (5)$$

Dependence (5) is obtained for the boundary conditions: $0.7w_s < w < w_i$, $w/w_{sp} < 1$, $w_{sp}$ is the energy of the secondary spark, and the time of explosion is more then skinning time and less then time of magnetohydrodinamic (MHD) instabilities development, providing conditions of uniform heating [2].

Because the surrounding medium hinders scattering of the material, the probability of attachment of a new atom to a condensation nucleus increases and an increase in the density of the surrounding medium γ should increase the particle sizes. From this viewpoint, the work of Glazunov *et al*. [9] is illustrative. These workers investigated the formation of powders in an inert gas at pressures of 1–600 atm. It has been shown that as a gas pressure increases, the medium size of the particles is increased and size distribution becomes wider.

The object of the present work is to investigate possibilities of ultra-fine powders production at lower pressures. Some results for Al powders are given in Fig. 3, here *d* is a surface median diameter of particles determined using *S* BET.

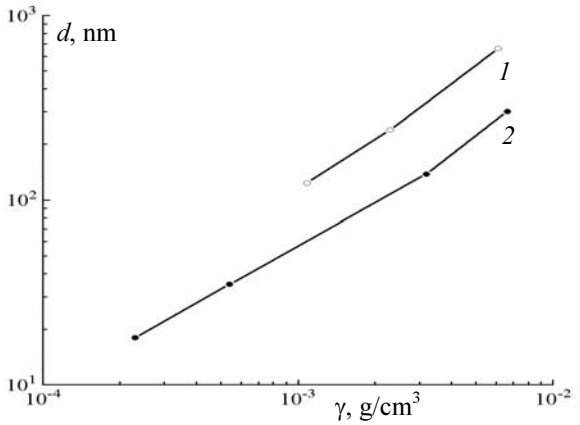


Fig. 3. Experimental dependencies between particle size of the Al powder and the density of surrounding gas under $w/w_s$ = 0.7–0.75 (*1*) and 1.0–1.1 (*2*)

From the data of Fig. 3 an influence of the introduced energy density (the particle size is more for the lesser energy) and an influence of density of the surrounding medium (the particle size decreases with decreasing of the gas density) are seen.

The experiments were implemented in the absence of the shunting discharge and the control was performed by the current oscillograms (Fig. 1, *b*).

To exclude an influence of heat effects of chemical reactions, inert gases and nitrogen were used (as shown in [11], molecular nitrogen is inert at the lower pressure).

The current density *j* defines a uniformity of Joule heating and is essential factor for the powder production. If $j < j_{\text{MHD}}$, the sausage-type MHD instabilities develop [2], and the wire is divided into drops along its length. From the other hand, the current density determines the heating rate: in accordance with (1), $dw/dt = j^2 \overline{\rho}$.

In Table 2, the data for the indium and aluminum powders with various specific energy introduced, $w/w_s$, and current density, $j/j_{mhd}$, are given. Here, the powder fineness is characterized by the magnitude of the specific surface area, *S*.

Table 2. Action of current density on fineness of powders

| No. | Metal | $w/w_s$ | $j/j_{mhd}$ | $S$, m$^2$/g |
|---|---|---|---|---|
| 1 | Indium | 0.6 | 1.33 | 3.6 |
| 2 | – „ – | 0.8 | 1.33 | 4.5 |
| 3 | – „ – | 0.8 | 0.47 | 3.0 |
| 4 | – „ – | 0.9 | 2.45 | 5.8 |
| 5 | Aluminum | 0.8 | 1.4 | 26 |
| 6 | – „ – | 0.8 | 1.0 | 14 |

From Table 2, it can be seen that the current density is also significant factor. For instance, with equal introduced energies ($w/w_s$ = 0.8), the specific surface area is more for the powder produced at the higher current density (see Nos. 2 and 3 for In and Nos. 5, 6 for Al powders). The uniform Joule heating allows producing powder with a higher fineness with lower energy expenditure (Nos. 1 and 3).

It is known that the metal structure is none uniform. Boundaries of grains, defects, impurities, and dislocations determine the resistivity and may cause the heterogeneity of ohmic heating. This point requires examination, and one of the principal objectives of this study is to investigate the influence of initial microstructure of metal on the particle size.

Figure 4 represents the dependence of the median-surface particle sizes $d_s$ for aluminum and other metals as function of the size of the regions of coherent scattering (RCS).

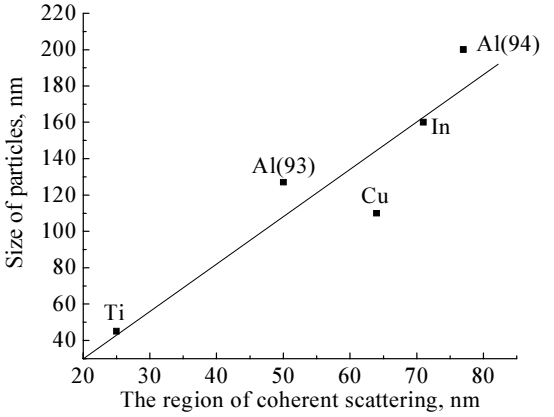


Fig. 4. The particle size versus the initial crystallite size

The most representative are the samples of Al (93) and Al (94) obtained at the electric explosion of an aluminum wire. An aluminum wire was exploded under the same conditions (wire parameters, electric circuit parameters, and environment). The introduced energy was equal to the sublimation one; the current density through the wire was $2.9 \cdot 10^7$ A/cm$^2$. But before the running of the sample Al (94) the wire was preliminary annealed. The average size of the crystalline particles increased and the CSR dimension enlarged approximately 1.5 times.

As a result, the average size of the particles obtained at the electric explosion increased and the specific surface area decreased from 17.5 to 11.0 m$^2$/g. The changes of the particle sizes are also confirmed by the electron-microscopic measurements.

## 4. Synthesis of carbon nanomaterials

A wide spectrum of carbon nanomaterials (CNM) was realized using electrical explosion of graphite and such metals as Fe, Co, and Cu in different surrounding organic medium (toluene, hexane, ethanol, kerosene and other organic liquids). Some results are presented in Figs. 5 and 6.

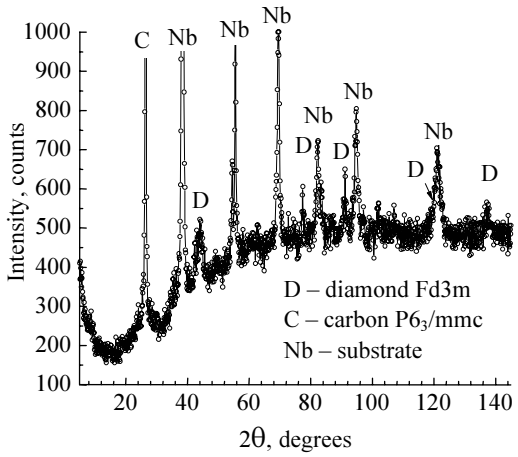


Fig. 5. X-ray diffraction patterns of the EW products in hexane, Cu $K_\alpha$ radiation

It's obvious that the phase composition of CNM produced consist from the ordinary graphite (space group P63/mmc) and cubical diamond (Fd3m) (if hexane was used as a surrounding medium, Fig. 1). In other case (when surrounding medium was changed to toluene), phase composition of CNM is consisting from the carbon nanotubes (Fig. 6).

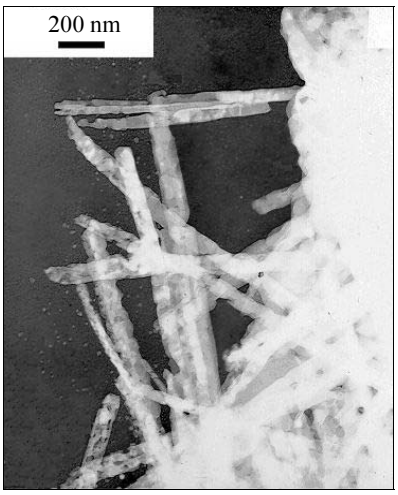


Fig. 6. Bunch of nanotubes, TEM photo of graphite EW products in the toluene)

In conclusion, it may be said that exploding wire method is a very promising method of ultra-fine powders production.

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