Assignment of Appropriate Conditions for Synthesizing Tungsten Nanopowder by Electric Explosion of Conductors

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Abstract. The paper provides the results of experimental research into properties of tungsten nanopowders synthesized by electric explosion of a conductor in argon at various energies, put into the conductor when exploding. The authors have studied how the conditions of synthesizing the tungsten nanopowder influence on the average size of particles.

1. Introduction
Powders of tungsten are used to synthesize high strength, dead-hard heat resistant alloys, high-speed tool steels and welding electrodes. At present special attention is paid to the possibilities of improving the technologies of synthesizing the tungsten powders and their physical and mechanical properties as well. One of the most promising procedures to produce the powders is electric explosion of a conductor (EEC) \cite{1}. This procedure as a method of synthesizing powders is distinguished by simplicity, low prices for the equipment, a relatively high efficiency, and low power inputs. Its principal advantage is a possibility to synthesize powders consisting of nanometer particles with a quite restricted range of their size distribution. These powders have a rather big specific surface area, demonstrating, therefore, their better reactivity. Nowadays tungsten powders produced this way are used for synthesizing tungsten disulphide when manufacturing solid-film lubrications on its base \cite{2}, as an active component in zeolite catalysts of non-oxidizing methane conversion into aromatic hydrocarbons \cite{3}, and as refractory modifying additives of a weld \cite{4}.

Physical properties of metallic tungsten, inter alia its low thermal conductivity, high melting and steaming temperatures complicate the assignment of appropriate conditions for EEC synthesizing the metal tungsten powder.

The purpose of this research is to study the influence of EEC initial conditions on the properties of the synthesized tungsten powder and determine appropriate EEC conditions for manufacturing the best quality powder.
2. Experimental
The research into electric explosion of the tungsten conductor and synthesizing the powders is carried out by the facilities, the general layout and operating principles of which are well-described in papers [5, 6]. Tungsten wire ВА-П-А with the diameter of 0.25 mm is used for EEC. The length of the exploded conductor is varied 40 to 100 mm. The capacitor bank capacity is varied 1.12 to 3.31 mkF, and its charge voltage is changed in the range from 20 to 30 kV. EEC is conducted in argon at the pressure of 2.5 atm.

The energy put into the conductor, energy, emitted at the arc stage of electrical discharge, current density and period of the EEC process are calculated according to the current oscillograms on the base of methodology in [7].

The properties of synthesized powders are studied via scanning electron microscopy (microscope JSM-7500F), X-ray diffraction analysis (diffractometer Shimadzu XRD-7000), the specific surface area is measured via thermal nitrogen desorption (analyzer “Sorbometr-M”).

3. Discussion of Results
Typical oscillograms of electric exploding the tungsten conductor are presented in Figure 1. Initial EEC conditions and some properties of the synthesized powders are given in Table 1.

![Oscillograms](image)

*Figure 1 – Current oscillograms of tungsten EEC. a) sample W-1; b) sample W-2; c) sample W-3*

In Figure 1 one can see that deceasing length of the exploded conductor for other conditions kept constant results in the increase of the maximal passing current from 12 to 23 kА, but the time of explosion is practically the same about 1.5 ms. It should be noted that the complete tungsten EEC process is at the arc stage of electrical discharge. It is possible due to physical properties of the metal to be exploded: low ionization energy (82 J/mm³) is the cause of bridging the early conductor electrical explosion by the arc stage of electrical discharge. Moreover, it blocks the further energy input into the conductor, so its higher values fail to be achieved as against EEC of other metals [1].

Having analyzed EEC modes with various initial conditions (Table 1) we revealed that shortening the conductor to be exploded at other conditions of the explosion kept constant causes a slight growth of the specific energy put into the conductor, (W-1 – W-2 or W-4 – W-5), but the energy emitted during the arc stage of electrical discharge increases far quickly. Furthermore, the rise of energy, put into the conductor when exploding, is possible in a certain range only. When exploding the conductor shorter 50-60 mm, decreasing $e/e_s$ and considerable increasing $e_{arc}/e_s$ are registered, being the consequence of the early stage of arc discharge.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$U$, kV</th>
<th>$C$, mkF</th>
<th>$L$, mkH</th>
<th>$l$, mm</th>
<th>$e/e_s$</th>
<th>$e_{arc}/e_s$</th>
<th>$(e + e_{arc})/e_s$</th>
<th>$S$, m²/g</th>
<th>$A_s$, nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>W-1</td>
<td>20</td>
<td>1.12</td>
<td>0.81</td>
<td>90</td>
<td>0.4</td>
<td>0.1</td>
<td>0.5</td>
<td>1.1</td>
<td>280</td>
</tr>
<tr>
<td>W-2</td>
<td>20</td>
<td>1.12</td>
<td>0.81</td>
<td>75</td>
<td>0.5</td>
<td>0.2</td>
<td>0.7</td>
<td>1.2</td>
<td>260</td>
</tr>
<tr>
<td>W-3</td>
<td>20</td>
<td>1.12</td>
<td>0.81</td>
<td>40</td>
<td>0.5</td>
<td>0.6</td>
<td>1.1</td>
<td>1.4</td>
<td>220</td>
</tr>
<tr>
<td>W-4</td>
<td>26</td>
<td>1.12</td>
<td>0.81</td>
<td>100</td>
<td>0.5</td>
<td>0.2</td>
<td>0.7</td>
<td>1.2</td>
<td>250</td>
</tr>
<tr>
<td>W-5</td>
<td>26</td>
<td>1.12</td>
<td>0.81</td>
<td>80</td>
<td>0.6</td>
<td>0.4</td>
<td>1</td>
<td>1.5</td>
<td>210</td>
</tr>
<tr>
<td>W-6</td>
<td>26</td>
<td>50</td>
<td>0.5</td>
<td>1.1</td>
<td>1.6</td>
<td>1.6</td>
<td>190</td>
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<td></td>
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<tr>
<td>W-7</td>
<td>30</td>
<td>100</td>
<td>0.5</td>
<td>1.5</td>
<td>2</td>
<td>1.5</td>
<td>210</td>
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<td></td>
</tr>
<tr>
<td>W-8</td>
<td>30</td>
<td>75</td>
<td>0.7</td>
<td>2.1</td>
<td>2.8</td>
<td>1.9</td>
<td>160</td>
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<td>W-9</td>
<td>30</td>
<td>60</td>
<td>0.5</td>
<td>2.9</td>
<td>3.4</td>
<td>2</td>
<td>150</td>
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<td>4.9</td>
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<td>3.8</td>
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<tr>
<td>W-12</td>
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<td>50</td>
<td>0.4</td>
<td>5.1</td>
<td>5.5</td>
<td>2.7</td>
<td>110</td>
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</tbody>
</table>

$U$ – voltage of the capacitor charge; $C$ – capacitor capacity; $L$ – circuit inductance; $l$ – length of the conductor to be exploded; $e$ – specific energy put into the conductor in the EEC process; $e_{\text{arc}}$ – specific energy emitted at the arc stage of electric discharge; $e_{S}$ – sublimation energy of tungsten (88.7 J/mm³); $S$ – specific surface area of powder; $A_{S}$ – average size of particles calculated according to specific surface area on the base of equation $A_{S} = 6/(S\rho)$, where $\rho$ – compact density of tungsten (g/cm³).

The study on synthesized powders has detected that specific surface area of powders is increased due to the growing energy put into the conductor when exploding and energy emitted at the arc stage of electrical discharge. The relation of $A_{S}$ to $e/e_{s}$ and $e_{\text{arc}}/e_{s}$ can be described by the function $A_{S} = f(e + e_{\text{arc}})/e_{s}$ (Figure 2).

![Figure 2](image)

**Figure 2** – The average size of particles vs. the sum of energy put into the conductor when exploding and energy, emitted at the arc stage of electrical discharge.

One can see in the plot, given in Figure 2, that the decrease in the average size of particles is typical for the whole interval under consideration. The significant decrease of $A_{S}$ from 300 to 150 nm is possible in conditions of growing total energy from 0.5 to 3.5 $e_{s}$, the increase in energy up to 5.5$e_{s}$ is the cause of the average size loss of particles to 110 nm.

Therefore, the appropriate conditions of EEC synthesizing tungsten powders are modes with the total energy, put into the conductor and energy, emitted at the arc stage of electrical discharge,
approximating to $3.5e^3$. The further increase of the energy has only a slight effect on the average size of particles.

The data obtained by the scanning electron microscope demonstrate that the sample synthesized in appropriate EEC conditions (sample W-9) consists of two fractions. The first fraction includes micron particles (Figure 3), the second one – nanometer particles (Figure 4). In accordance with the data presented in literature [8] two various fractions of particles are formed due to the specifics of the conductor destructing process under electrical explosion. Micron particles are formed from the bubbling drops when splashing the conductor. The nanometer particles are formed under vapor condensation.

Having processed the obtained data a conclusion is drawn that the size of micron particles ranges from 1 to 3 μm, and maximum of their distribution is 1.5 μm. The size on nanometer particles varies in the range 20 to 200 nm, the maximum is 40-50 nm.

![Figure 3](image1.png)

**Figure 3** – a) Photo of the particles, sample W-9; b) size distribution of micron particles.

![Figure 4](image2.png)

**Figure 4** – a) Photo of particles, sample W-9; b) size distribution of nanometer particles.

The powder under consideration consists of two phases $\alpha W - 68.8\%$ and $\beta W - 31.2\%$ (Figure 5) according to the data of X-ray diffraction analysis.
4. Conclusions
The research into the electric explosion of tungsten conductors in various initial conditions allows making the following conclusions:

1. The average size of particles calculated on the base of the specific surface area of powder is decreasing because of the growth of energy, put into the conductor when exploding, and energy, emitted at the arc stage of electrical discharge.

2. The appropriate EEC conditions are modes with the total energy, put into the conductor and energy, emitted at the arc stage of electrical discharge, approximating to 3.5\(e_s\). The further increasing of energy causes a slight decrease of the average size of particles and is not reasonable.

3. The tungsten powder synthesized in appropriate EEC conditions consists of two fractions: a micron one, having the maximum of distribution 1.5 \(\mu\)m and nanometer one with the distribution maximum of 40 - 50 nm. This tungsten powder is a blend of two phases of \(\alpha\)- and \(\beta\)- tungsten.

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References:
by conductor electric explosion method 685 pp 596-600.
