Production of tantalum nanopowders by electric explosion of wire

A V Pustovalov¹, M N Vlasyuk¹, A V Korshunov¹
¹Institute of High Technology Physics, Tomsk Polytechnic University, Tomsk 634028, Russia

Abstract. The possibility of producing metallic tantalum nanopowders using electric explosion of Ta-wire was explored. The regimes of the electric explosion were determined which provided maximum level of energy brought into the wire. The basic properties of the obtained powders were examined using low-temperature nitrogen adsorption, atomic-emission spectrometry, as well as by means of transmission and scanning electron microscopes.

1. Introduction

Tantalum and its alloys are widely used in metallurgy, instrumentation, aerospace engineering, medicine, and nuclear power. But the main consumer of metallic tantalum is electronics, in particular in high-capacity tantalum capacitors. The tantalum powders of particle size 3-30 µm are used in the initial stage of capacitor production [1]. It is known that metal properties may change with decreasing powder particles size from micron to nanometer scale. The method of electric explosion of a wire (EEW) [2] is one of the promising and inexpensive methods for producing metal powders with a particle size of about 100 nm. The possibility of obtaining metal Al, Cu, Fe, Mo, Ni, W powders using this method is described in the literature in detail [3-5]. There was determined a number of distinctive properties of electroexplosive powders. However, the data on the possibility of obtaining tantalum powders by EEW and their properties are insufficient.

Thus, the main objective of the present work is to determine the possibility of obtaining metallic tantalum powders using electric explosion of wire and to explore the basic properties of the obtained powders.

2. Materials and methods

The experimental setup developed in Tomsk Polytechnic University was used to obtain the powders by the wire exploding method (Figure 1).

The setup works as follows. The distance between high-voltage and ground electrodes in reactor 3 must be fixed. The setup is evacuated and filled with argon to pressure of 2·10⁵ Pa. Then the high-voltage power supply charges the capacitor (C) of the impulse-current generator to the voltage level needed. After that the wire feeding system is activated. When the wire approaches the high-voltage electrode a switch goes off, the capacitor is discharged on the wire section between the high-voltage and grounded electrodes. As a result, the wire explodes. Then the process is repeated. The fan moves products of electric explosion first to the coarse particles filter and then to the fine particles filter. Coarse and fine powder particles settle in the containers of the respective filters, and the filtered argon is fed to the reactor again. After obtaining the necessary amount of powder the containers are removed and the powder is passivated in air-argon atmosphere.
The electric explosion of tantalum wire was studied using generalized variables of the theory of similarity of EEW [6]:

\[
e = \frac{CU_0^2 \cdot 10^{18}}{d^2 Z}, \quad \lambda = \frac{1 \cdot 10^{-6}}{d^2 Z}, \quad \nu = \frac{10^4 \cdot \sqrt{LC}}{d} \left[ \frac{s}{m} \right]
\]

(1)

where \( C \) is capacitance, \( U_0 \) is voltage of capacitor charging, \( Z \) is impedance of the circuit, \( d \) is diameter of exploding wire, \( L \) is inductance of the circuit.

To perform the study the following parameters of the circuit have been chosen: capacitance 0.78–2.2 \( \mu F \), charging voltage 16–32 kV, circuit inductance 0.7–0.85 \( \mu H \), diameter of Ta-wire 0.2 mm, wire length 40–120 mm. The energy input into the conductor was calculated using oscillograms of current run through the current shunt (\( R_s \), Figure 1) according to the equation:

\[
W_{EEW} = \frac{CU_0^2}{Z} = \frac{C(U_0 - E_0 \cdot \frac{1}{E_{EEW}} \int_{t} i(t) \, dt)^2}{Z} - \frac{L \cdot i(t_{EEW})^2}{Z} - R \int_{t}^{t_{EEW}} i(t)^2 \, dt
\]

(2)

where \( i \) is EEW current, \( R \) is circuit resistance, \( t_{EEW} \) is duration of EEW current impulse.

To simplify the analysis of the obtained data the energy input into the conductor was expressed in the form of dimensionless values:

\[
e/\varepsilon_s = \frac{W_{EEW}}{V_W \cdot e_s}
\]

(3)

where \( e \) is specific energy, \( V_W \) is volume of exploding wire, \( e_s \) is sublimation energy of tantalum (71.7 J/mm\(^3\)).

Additionally, the coefficient of energy transfer \( \eta \) from the energy power supply to the wire was determined according to equation (4):

\[
\eta = \frac{W_{EEW}}{W_0} \cdot 100\%
\]

(4)
Analysis of the EEW-regimes was performed using the following procedure: the modes of critical explosion [7] was studied first, and then the dependencies \( \lambda_b = f(\varepsilon) \) and \( \eta_b = f(\varepsilon) \) were determined. Secondly the EEW-regimes with conductor length less then critical were checked. After studying EEW of Ta-wire under different initial conditions the optimal mode of EEW was found. Under this mode a sample of tantalum powder was obtained. The following parameters were determined for all the obtained powders:

- specific surface area using low-temperature nitrogen adsorption (BET, "Sorbtometr-M");
- morphology and average particle size by means of transmission (TEM) and scanning (SEM) electron microscopes (Philips CM-12, SEM – 515);
- impurities by means of atomic-emission spectrometer "MAAS" and analyzers TSN 600, TC 844 LECO.

3. Results and discussion

Figure 2 shows typical oscillograms of current for electric explosion of Ta-wires.

![Figure 2. Oscillograms of current for electric explosion of Ta-wires: (a) and (b) the critical mode; (c) wire length above critical, (d) wire length less then critical](image)

The process of electric explosion of tantalum is similar to the process of electrical explosion of tungsten [8], and often is divided into three stages. First and second stages - EEW, the third – arc discharge. The arc discharge stage is present in all cases, but under some explosion regimes the moment occurs when EWW-current does not change (plateau current) (Fig. 1, a, b and c). The EEW-regimes with the lowest possible plateau current were chosen as a baseline – the critical modes of EEW.

Analysis of the waveform of the current allowed to determine the empirical relationships describing the critical modes of EEW:

\[
\lambda_b = 4.5 \varepsilon^{0.2}; \quad \eta_b = 27.9 \varepsilon^{-0.6}; \quad e_p/e_s = 0.55 \varepsilon^{0.2}
\]  

(5)

It should be noted that with increasing \( \varepsilon \) the energy, injected into the conductor in the critical regime of explosion, increased quite slowly. So for \( \varepsilon = 0.15 \) the input energy equals 0.4\( e_s \). The increase in \( \varepsilon \) up to 1.2 leads to an increase of energy up to 0.6\( e_s \). To increase the input energy when a wire exploded the regimes were studied when the wire length was less then critical under the other parameters were constant (\( C, U_0, d, L \)). This allowed us to determine the change in the energy properties of the explosion relative to the base – critical EEW.
Figure 3 shows the dependencies of the specific input energy and the coefficient of energy transfer on the wire length.

\[
\frac{e}{e_0}, \frac{\eta}{\eta_0}
\]

\[l/l_b\]

Figure 3. The dependence of the specific input energy and the coefficient of energy transfer from the power supply to the wire relatively the critical EEW

Decreasing the length of the exploding wire leads to decrease in \(\eta\) with simultaneous increase in the specific energy input into the wire. However, the value of \(e\) increases for all the studied wire lengths. When the length of wire was equal to 0.5\(l_b\) the maximum level of input energy was observed. Further decrease in the length of exploding wire leads to a decrease in specific energy which is due to the early development of the arc discharge stage.

To obtain tantalum powders the mode with the maximum level of input energy was chosen \((U_0 = 26\ \text{kV}, \ C = 2.2\ \mu\text{F}, \ l = 60\ \text{mm})\). This EEW-mode provided the level of input energy \(1.1e_c\). According to the data obtained using scanning and transmission microscopes the sample consists of two fractions.

The first fraction (Figure 4 a - c) is of micron spherical particles with an average size of about 2 \(\mu\text{m}\).

The average size of the particles of the second fraction is about 200 nm (Figure 4 d,e).

Figure 4. (a and b) SEM and (d) TEM photos of the obtained tantalum powder.
According to [2,3] formation of large micron particles caused by melting and splattering of a part of metal wire followed by crystallization of drops. Particles of nanometer size are formed by condensation of metal vapor. Specific surface area of the obtained powders was 2.3 m$^2$/g. Spectral analysis of the samples showed the presence of metallic impurities not exceeding 0.15 %. The amount of adsorbed gas was 0.94 % - O$_2$, 0.11 % - N$_2$ and 0.46 % - H$_2$. The presence of metallic impurities is determined by the initial purity of the tantalum wire. The increase of the adsorbed gas is caused by high reactivity of the obtained powder.

4. Summary
Based on the obtained data there has been determined the possibility of Ta nanopowders production using electric explosion of Ta-wire. The obtained powders have a bimodal distribution of particle size, a small amount of extraneous metallic impurities and adsorbed gases.

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