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Structural characteristics of copper nanoparticles produced by the electric explosion of wires with different structures of metal grains

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ABSTRACT

This work studies the impact of the size of grains/crystallites on the structural characteristics of the nanoparticles formed as a result of the electric explosion of wires. The temporal dependencies of the current and voltage in the conditions of EEW experiments were analyzed. It was concluded that an increase in the grain/crystallite size does not result in a significant change in the electric resistance of the wires that are in solid state. It was demonstrated that an increase in the grain/crystallite size does not result in an increase in the average nanoparticle size. The structure of the nanoparticles with the size between 80 and 110 nm is close to a monocrystalline one. This data allows for making an assertion that certain models suggested in research papers to explain non-equilibrium phase transitions cannot be applied when describing the electric explosion of wires.

1. Introduction

The mechanisms of non-equilibrium phase transitions of metals/alloys under pulse heating by high-energy fluxes (laser radiation, plasma, electric current, etc.) are a trending object of fundamental and applied research. Depending on the heating parameters, the phase of a substance may change widely, between complete atomization and a two-phase system (metal gas plus condensed phase clusters) [1–4]. Here, short heating times are an implication for the experimental research of the evolution of metals/alloys during non-equilibrium phase transitions. The electric explosion of wires (EEW) is one of the methods that enable us to research the phase state of metals and alloys. EEW is the explosive destruction of metal caused by exposure to a current pulse with the density of 10^6–10^9 A/cm^2 [4].

EEW is used to produce metal nanopowders [5–8], alloys [9–12] and chemical compounds [13–15], generate shock waves [16] and X-ray radiation [17–20] and to study phases of matter under extreme conditions [21–25]. The use of EEW in the production of nanopowders provides such advantages as a narrow distribution of nanoparticles by size (as compared to other approaches), a high efficiency factor of energy transfer into the wire, the absence of harmful by-products of synthesis and the production of nanoparticles based on metastable phases of metals [26,27].

As of current, no generally accepted model of EEW exists [4]. This fact complicates the uncovering of the mechanism of nanoparticle formation in the conditions of EEW. Fast-explosion mode EEW is peculiar for its fast input of energy (E) into the wire, the input energy exceeding the energy of wire sublimation (E_s). When $E \geq E_s$, the expanding products of EEW have complex aggregate states where condensed-phase nanosize particles (clusters) coexist with weakly ionized plasma [28,29]. Several mechanisms of non-equilibrium phase transitions in fast-explosion mode have been suggested to explain the presence of clusters in the expanding products of EEW.

According to [30], in the course of current pulse transition through a wire, energy dissipates in multiple ways. Energy dissipation causes chemical bonds to destruct while creating a new surface (i.e. the metal decomposes into clusters). Energy dissipation also causes ionization. The authors assume that less energy transfers into heat than postulated by the Joule-Lenz law. The research has shown that average nanoparticle size (a_s) asymptotically approaches the average size of crystallites (i.e. the size of coherently

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scattering regions (d_{csr})). The authors of the present work assume that the correlation of dependencies $a_s(E/E_s)$ and $d_{csr}(E/E_s)$ implies that the wires are destructed into particles less than 10 nm in size.

The authors of work [31] conclude that the presence of clusters in the expanding products of EEW may be due to the clustered structure of liquid metals. The authors of this research assume that this hypothesis is confirmed by the correlation between nanoparticle sizes d_{csr} and the cluster sizes in liquid alloys (with values up to 10 nm).

The nanoparticles sized between 100 and 200 nm produced as a result of EEW have a polycrystalline structure, with the size of crystallites between 3 and 12 nm [30,31]. The authors assume that the polycrystalline structure of nanoparticles forms as a result of the coagulation of clusters that emerge directly from the liquid phase. However, the polycrystalline model of nanoparticle structure does not comply with the data presented in work [5], for example. The analysis of cobalt nanoparticles has shown that the values of a_s and d_{csr} are close to each other (55 and 48 nm, respectively), which is characteristic of a monocrystalline structure.

As per [32], the presence of clusters in the expanding products of EEW is the result of non-uniform heating of the wire with a current pulse. Non-uniform heating is caused by the existence of such structural defects as crystallite borders in the metal. During wire heating by a current pulse, local steam-gas regions are formed; those are characterized by increased electric resistance and energy deposition. Non-uniform wire heating results in the formation of a heterogeneous structure consisting of clusters and gas/plasma. The probability of metal phase transition taking place according to the above described model was suggested by the direct dependency between wire size d_{csr} and the size of nanoparticles formed in the course of EEW [32]. However, a research into brass and zinc wire heating by a pulse of current with the density of 3.3×10^7 A/cm^2 has shown that the borders of crystallites do not contribute to the formation of a heterogeneous structure in the wire's metal [33].

The above works suggest that EEW products are a dual-phase system comprising condensed-phase clusters and a metal gas. In the meanwhile, the above mentioned concepts are subject to discussion: one regarding the influence of the size of wire metal's grains/crystallites on the structural characteristics of the nanoparticles, and another one, regarding the mechanisms of cluster formation in the expanding products of EEW. The dependencies between the cluster sizes in the expanding products of EEW and nanoparticle sizes d_{csr} (as determined in Refs. [30–32]) do not have sufficient substantiation and contradict data from other works. Also, in science literature, there is no data about the impact of grain/crystallite size on the temporal dependencies current and voltage in the conditions of EEW. This data is required in order to study the evolution of matter under extreme conditions.

In this regard, it is important to determine how the size of grains/crystallites of wire metal influences the average size and size d_{csr} of nanoparticles. Not only is this correlation a trending object of fundamental research, it is vital for applied research that studies the use of EEW for producing metal and alloy nanoparticles with pre-determined structure and phase state.

2. Materials and methods

Copper wire (diameter: 0.3 mm) was used in the experiments. The microstructure of metal in unmodified (non-annealed) wires was altered through thermal annealing at 600 °C over an hour. The research of the structure of the unmodified and the annealed wires was performed using the NEOPHOT-21 optical microscope with the magnification factor of 200, 400 and 1000. The average size of the grains in the unmodified wire in skewed and right-angle cross sections was 1.9 ± 0.7 μm and 3.7 ± 1.6 μm, respectively. The average size of the annealed wire grains in skewed and right-angle cross sections was 63.7 ± 24.4 μm and 109.5 ± 31.2 μm. The average size d_{csr} of wires and nanoparticles was determined via the Debye-Sherrer method [34]. The graph of nanoparticle distribution by size was plotted from the microphotograph data obtained via TEM on the JEOL JEM-100CX II microscope. Number-average nanoparticle size (a_n) was determined from the data obtained using the nanoparticle size distribution function. Values a_s were determined from the specific surface of nanoparticles [14].

An *RLC* circuit was used for electric wire explosion. The parameters of the *RLC* circuit and the description of the system for detecting temporal dependencies of the current and the voltage are provided in work [33]. The temporal dependencies of the current and the voltage in Fig. 1 were plotted from the readings that were taken 9 times. Current density in the experiments varied from 4.6×10^7 A/cm^2 to 5.6×10^7 A/cm^2.

Copper nanoparticles were produced in argon atmosphere at 2×10^5 Pa. Energy $E(t)$ was calculated using expression (1).

$$E(t) = \int_0^{t_{\exp}} U(t) \times I(t) dt, \tag{1}$$

where t_{exp} is the time elapsed from the start of current flow through the circuit and up to the moment of wire explosion that corresponds to the maximum voltage (U_{max}).

3. Results and discussion

Fig. 1 shows the results of analyzing the microstructure of the unmodified and the annealed wires in long section. The research was performed on the Quanta 200 3D microscope using electron backscatter diffraction data (EBSD). In the unmodified wire's structure, the grains are stretched longwise. As a result of cold-hardening, grain deformation takes place (Fig. 1a and c). Such a structure is formed as a result of wire-drawing during its production. Thermal annealing of wires results in the increased grain size (re-crystallization). It also results in a decrease in the relative content of low-angle boundaries and the emergence of annealing twins (a peak of 60°) (Fig. 1b and d).

Nanoparticle samples were obtained from unmodified (non-annealed) wire at three different values of E/E_s. Nanoparticle samples were obtained from the annealed wire with the same *RLC* circuit parameters and at the same values of E/E_s, as those for the non-annealed wire. The parameters of the electric explosion of wires are given in Table 1.

Fig. 2 shows the oscillographs of the current and voltage characteristic of the electric explosion of the unmodified and annealed wires. By analyzing the data presented in Fig. 2 we distinguish 3 phases of wire heating by current pulse. During the first phase ($0 < t < t_1$), the wires are heated while in solid state. During the second phase ($t_1 < t < t_2$), the wires are melting. Wire resistance in this time range approximately doubles. During the third phase ($t > t_2$), the wires are heated while in liquid state. In the first stage, at different E/E_s values, no pronounced difference in the temporal dependencies between current and voltage is observed. This peculiarity of the temporal dependencies suggests that there is no significant impact of the size of metal grains/crystallites on the electric resistance of wires in the conditions of the experiments we conducted.

The data provided in Fig. 2 clearly shows that the electric explosion of unmodified and annealed wires takes place at different t_{exp} and $U_{max.}$ Maximum voltage pulse for the unmodified wire is reached later as compared with the annealed wire (Table 1). In our