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KazNU after al-Farabi

THE RADIOGRAPHIC ANALYSIS OF THE COPPER NANOPOWDER IRRADIATED BY FAST ELECTRONS

The copper nanopowder irradiated by the high-energy electrons in the dose interval from 1 to 10 millirad by the X-ray structure analysis method. There have been found out a number of phase transitions/conversions.

The main physical characteristics of the metal nanoparticles drastically differ from the metal characteristics in their usual mass condition and in some cases re unique. The research of the mechanical, electric, optical and structural characteristics allow determining these differences [1,2].

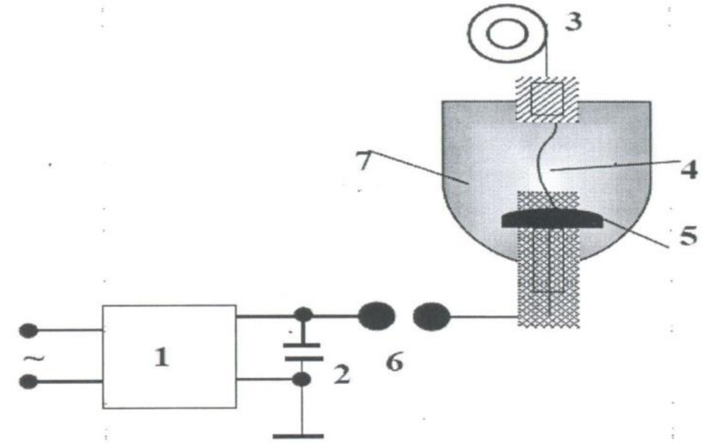
In this work we have described the results of the research of the copper nanoparticles structure.

The experimental part

The copper nanopowder was received by the method of the electric explosion of wire (EEW) [3]. The copper nanoparticles size was defined with the electron microscope Quanta 200i 3D. The achieved results have shown that the nanoparticles are of spherical form that corresponds to the data mentioned in the literature and have the size from 30 to 300 nm (picture 1). The distribution of the copper nanoparticles according to the size has been done by the method of the small-angle X-ray scattering on the diffractometer Hecus S3-Micro (picture 2).

In the picture one can clearly see that the after the explosion the nanopowder has uneven in size distribution of the nanoparticles. In the studied sample there are mainly the particles of the size ranging from 20 nm and 40 nm.

The copper nanopowder has been irradiated on the electron linear accelerator УЛ4.



Principle scheme of experimental setup: 1 – high-voltage power source, 2 – capacitor battery, 3 – wire supply unit, 4 – exploding wire, 5 – high-voltage electrode, 6 – commutator, 7 – explosion chamber filling with liquid

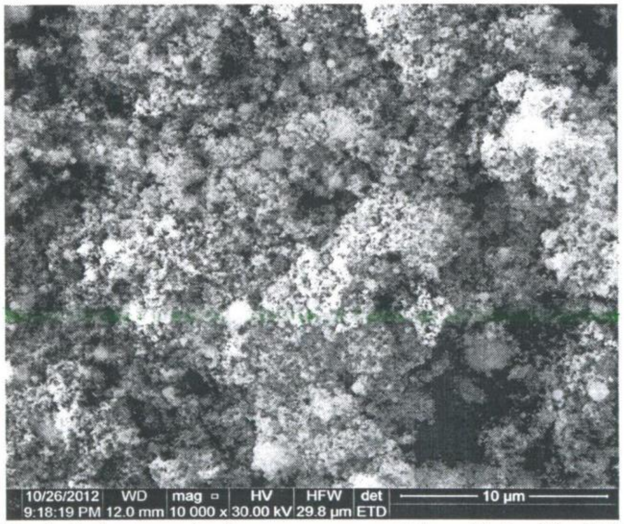


Fig 1. A photograph of copper nanoparticles, obtained by SEM

Using a scanning electron microscope Quanta 200i 3D, the sizes of copper particles, having a spherical shape of radii ranging from 30 nm to 300 nm (Fig), were determined.

The particle size distribution in the studied powders was determined by the method of small-angle X-ray scattering on the diffractometer Hecus S3-Micro. Fig. shows the average particle size distribution.

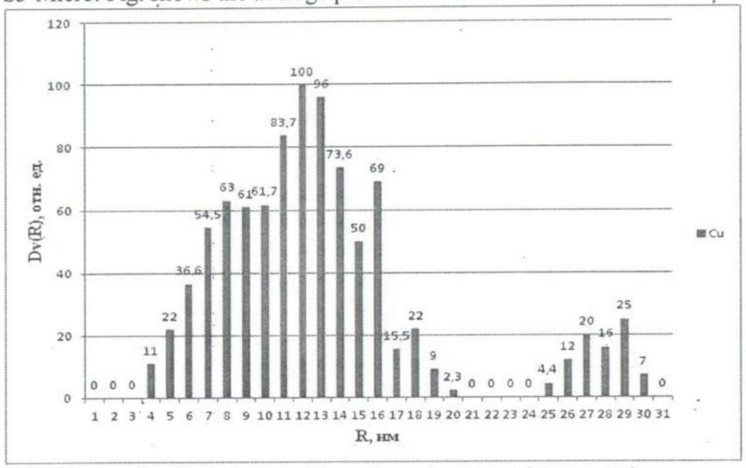


Fig 2. The dependence of the volume function of the particle size distribution

X-ray studies of copper nanopowders have been done by DRON-2M using monochromatic CuKα radiation. In the diffraction pattern of copper monolith (Fig. 3) are clearly visible peaks corresponding to reflections from the (111) (200) (220) and (311) fcc structure of copper with lattice size smaller than nano. Furthermore, this x-ray diffraction peak appears low at a small angle, which may correspond to defects in the crystal structure.

The received diffractograms are given in pictures 3-7: the diffractogram of the volumetric copper – picture 3, non-radiated nanopowder – picture 4, the copper nanopowder irradiated to an absorbed dose of 1 millirad – picture 5, the copper nanopowder irradiated to an absorbed dose of 5 millirad – picture 6, the copper nanopowder irradiated to an absorbed dose of 10 millirad – picture 7.

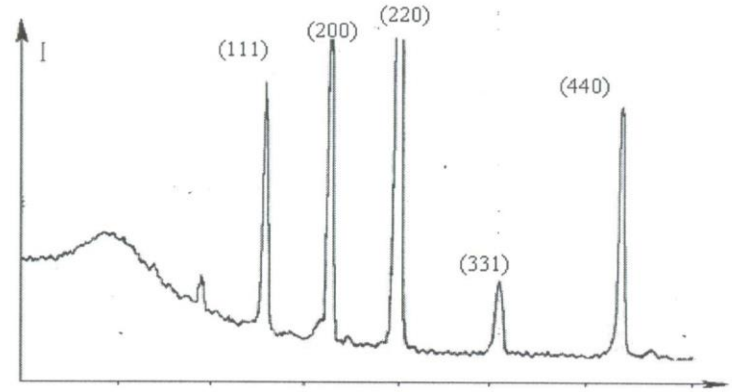


Fig 3. A diffraction pattern of the copper monolith

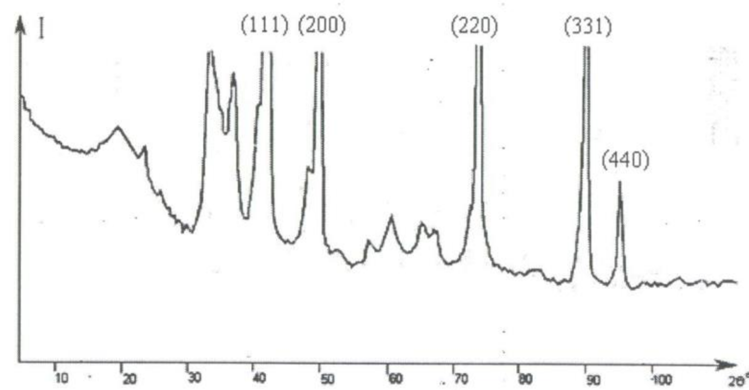


Fig 4. Diffraction pattern of unirradiated copper nanopowder

The splitting of the peaks can be attributed to the presence of clusters with different lattice parameters or the existence of several crystalline phases.

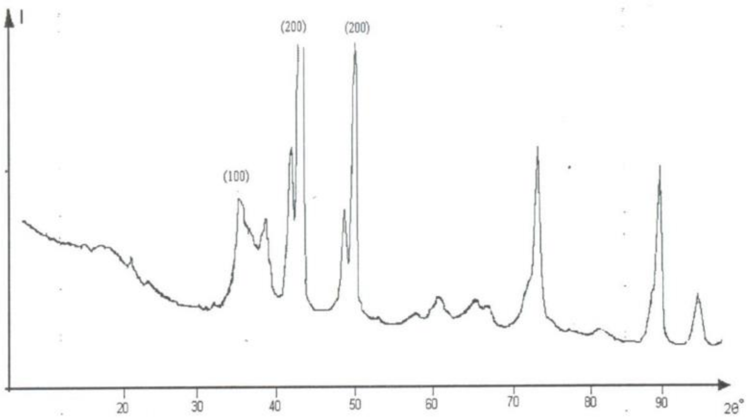


Fig 5. Diffraction pattern of copper nanopowder irradiated to an absorbed dose of 1Mrd

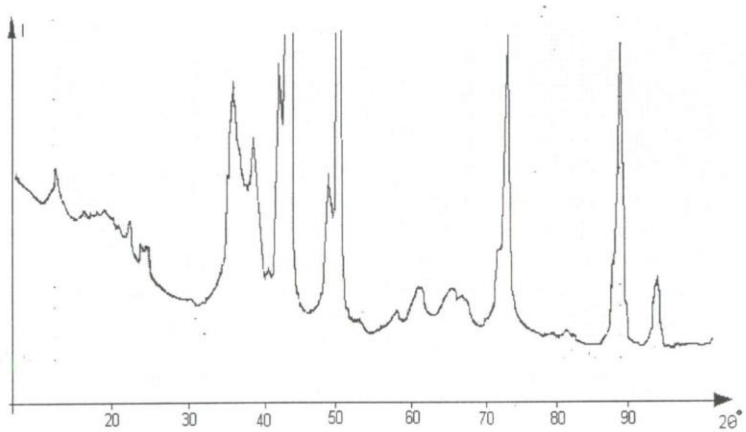


Fig 6. Diffraction pattern of copper nanopowder irradiated to absorbed doses of 5 Mrd

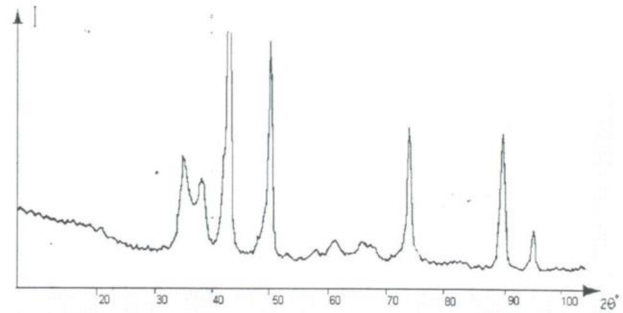


Fig 7. Diffraction pattern of copper nanopowder irradiated to an absorbed dose of 10 Mrad

Fig. 7 shows the diffraction pattern of copper powder, irradiated to a dose of 10 Mrad, which shows that the splitting of the peaks from the (111) and (200) vanishes, and the peak of (100) decreases. This intensive exposure to ionizing radiation leads to radiation fragmentation of nanoparticles, which results in the first place, the best nanoparticle size (magic number), secondly, to the agglomeration of nanoparticles.

Peaks at small angles, which are observed in all diffraction patterns and their calculations, nanopowders of copper may have a superlattice.

Such a superlattice can be formed by clusters which contain 55 atoms. The structure of the superlattice is also a face-centered lattice constant equal ace 13.722 Å.

Superlattice

$a_{superstructure}$=13.722 Å

$$R_{cu\ cluster} = \frac{a\sqrt{2}}{4} = \frac{13,722\sqrt{2}}{4} = 4.851Å$$

$$V_{cu\ superlattice} = \frac{4}{3}\pi r^3 = 646.36\ Å^3$$

$$V_{cluster} = \frac{V}{0,74} = 478.3\ Å^3$$

Volume of ion

R_{cu}=1.273 Å

$$V_i = \frac{4}{3}\pi r^3 = 8.641\ Å^3$$

Volume of ions in the elementary lattice

$$V=\frac{8{,}641}{0{,}74}=11{,}677Å^3$$

Number of atoms in the clusters (molecule)

$$N=\frac{646{,}36}{11{,}67}=55{,}36$$

Density of super atom

The experimental data analysis

The comparative analysis has shown that the parameters of the elementary copper cell of the volumetric material and nanopowders received by the EEW are partially different. So, the parameter of the elementary copper cell of the nanopowder is a little bigger than the volumetric material that corresponds to the literature data [1].

In the diffractograms of the copper nanopowder and the volumetric copper have been found the differences as follows between the characteristic peaks of the volumetric material in the nanopowder case there appear many small peaks. The peaks correspond to the copper oxides and dioxides the appearance of which can be explained by the fact that the nanosize particles have an increased reactionary capacity and are quickly oxidezed if they are not in the inert environment. Moreover, the diffraction peaks in the case of the copper nanopowder are deformed and there appear kinks. Such peaks deformation prove that in the nanopowders received by the EEW there appear two copper phases where both phases have a F-structure but they differ in the elementary lattice parameters.

At the irradiation of the copper nanopowders by the fast electrons with the energy more than 3 MeV the split into two phases take place more abrupto. At an absorbed dose of 1 millirad the peaks diffraction is harder that can be explained by the radial anneal mechanism of two phases because of the mechanic stress appearing after the nanoparticles condensation after the EEW. At an absorbed dose of 5 millirad both phases are nearing in content because one can observe the transition of one phase into another because of the electron structure change under the irradiation. At an absorbed dose of 10 millirad the phase with the bigger parameter is annealed completely and vanishes.

In the X-ray pictures one can see that the changes take place in the oxides and one can observe the character changes for the oxides peaks.

One should pay attention to the sphere of the diffusion scattering 2θ (5^0 + 30^0) that «breathes» that is changes depending on the electron irradiation dose. As a rule, the diffusion scattering sphere and the

background correspond to the small disperse material that is crystals with much broken structure or metal in the amorphous condition which is the link between the crystals in the volumetric material and nanopowder. It has been established that if irradiated by fast electrons of the dose 5 millirad the sphere of the diffusion scattering changes to the condition when its regulation takes place. It proves the process of its self-organization in the result of which the super crystal appears with the parameter of the elementary lattice that is three times bigger than the elementary lattice size of the volumetric material. The super crystal has a F-structure and the electron lattice parameter is a = 13,72Å according the diffractometer measuring. The lattice with such parameters should consist of the particles that are bigger than an atom but that possesses the atom properties. The calculation has shown that the radius of such a particle (superatom) equals 4,85Å. The atom number calculation that are in the nanoparticle structure has proved that the nuclear of such a superatom equals the «magic number» of 55 atoms Cu that correspond to the third coordination sphere of the copper structure. On the assumption of the nanocrystal radius the X-ray density and the elementary cell parameter of such a crystal have been calculated that are correspondingly ρ = 12,4 g/cm^3, a = 2, 74 Å, F a lattice. The nanocrystal with such parameters must leave a trace on the diffraction pattern. The design angles (F structure, a = 2, 74 Å) for the planes (111), (200), (220) equal correspondingly 2θ=58^0, 2θ=68^0, 2θ=114^0 and such angles have been found. At researching the nanopowders all diffraction patterns peaks have been identified that belong to the copper and copper oxides. Two angles 2θ =58^0 and 2θ=68^0 have not been identified because they belong neither to the copper nor the oxides. It proves that the superatom nuclear can have the crystal structure and correspond to the quantum dot.

Conclusions

It has been established:

1. The copper nanopowders received by the EEW have several phases and much depend on the crystal and electron structure.

2. At the irradiation of the fast electrons there happens the «self-organization» up to the superstructure.

3. The superstructure consists of the superatoms (quantum dots) that consist of 55 copper atoms in which the quantum dots have not metal characteristics.

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