

Impact of nanosecond proton beam processing on nanoblocks of copper

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Abstract. X-ray studies in conjunction with the method of recoil nuclei and electron microscopy of irradiated plates polycrystalline Cu by nanosecond high power density proton beams ($E = 120$ keV; $I = 80$ A/cm², $t = 50$ ns) showed nano block nature of the formation of structure in the surface layer target and condensed-formed film.

1. Introduction

Analysis of the X-ray diffraction and electron microscopy pictures of the crystals' electron density distribution in the surface layer includes establishing the causes of the contrasting line [1, 2], nano blockiness [3], modulating the structure [4] and other phenomena caused by various influences [5, 6]. A number of technical difficulties obstruct the direct use of formulas to estimate the scattering intensity of X-ray and electron beams at a certain point of space [7]. We calculate only the integral intensities of the diffraction maxima caused by the small size of the crystallites, the ultimate high wide, non-periodical scattering centers, a large beam divergence and etc. [7-9]. When we calculate the theoretical intensity of the polycrystalline substances that consist of nano blocks (≤ 1000), we use crystal mosaic model. However, a good matching is observed for weak reflexes that were calculated by the kinematic theory. Differences are associated with mismatched shapes and sizes of coherent scattering and nano blocks. The integral intensities calculation of the diffracted crystal layers doped with protons, which the authors conducted [10], showed a decrease in these differences. As in the model of crystal mosaic, nano blocks in protonated layers are slightly disoriented, similar in size of coherent scattering regions and have few defects. It seems necessary to study the features of X-ray diffraction by nano block structure of the surface layers doped with protons and condensed films.

2. Experimental methods

Irradiation of polycrystalline Cu plates by nanosecond high power density proton beams ($E = 120$ keV; $I = 80$ A / cm², $t = 50$ ns) led to increasing proton concentration to a depth of 10 microns, depending on the resulting number of pulses. The nuclear-recoil method found that in the layer depth ≤ 1.2 m at 5 irradiating pulses the concentration of hydrogen nuclei ¹H reaches $8 \cdot 10^{19}$ cm⁻³. X-ray measurements were performed on a DRON-3 diffractometer in monochromatic Cu-K α radiation by sliding-beam method.



3. Results and Discussion

Except reflexes CuO, Cu₂O and face-centered cubic-Cu strong oscillations of the background and the redistribution of the intensity of reflection (111) Cu are observed on the radiogram of X-ray irradiated plates (Figure 1). It is taken into account that the integrated intensity of the diffraction reflections is proportional to the thickness of the film and the penetration depth of X-rays at a target. Depending on the number of irradiated pulses (N) and the observed values of X-ray modulation periods (v), calculated values of ribs of nano blocks Cu (α) which are based on the Fibonacci sequence of steps (F_n) are arranged in series (Table 1).

Table 1. The sizes of nano blocks after radiation of plates of copper.

| N, pcs | 1 | 2 | 5 | 11 | 31 | | | | | |
|----------------|-----|-----|-----|-----|-----|------|-----|------|------|-----|
| v, Å | 118 | 400 | 501 | 133 | 800 | 71.4 | 400 | 48.7 | 43.8 | 200 |
| F _n | 7 | 9 | 10 | 7 | 11 | 6 | 9 | 5 | 5 | 8 |
| a, Å | 110 | 289 | 468 | 110 | 757 | 68.3 | 289 | 42.1 | 42.1 | 179 |

In the series (Table 1) fluctuations in the calculated values of the size of nano blocks based on the step of Fibonacci sequence from periods of the observed X-ray modulation ranges from 2 to 15%, which may be due to the presence of nano blocks CuO, Cu₂O and inaccurate selection of modulation oscillations. Despite the differences in the experimental and calculated values of nano blocks' sizes, we can take a structural element (SE) with the volume *V* as the minimum volume of scattering X-ray diffraction

$$V = V_{e.c.} \sqrt{r} \quad (1)$$

where *V_{e.c.}* – elementary cell volume;
r = 1,618 - the golden ratio.

Use of scattering volume of the SE in the expression of the kinematic theory leads to the coincidence of the calculated and experimental values and intensities. That is indicated by the authors in [10] for polycrystalline Al. Applying experimentally determined value of the interplanar distance of the most densely packed plane with a maximum intensity line (d_{max}) we can determine the size of the SE from

$$A = d_{max} N^{1/2} \tau^{1/6} K^{-1/3} \quad (2)$$

where *N*=*H*²+*K*²+*L*² – is the sum of square indexes of high packed plane;
K - is a shape coefficient of nanoparticles (*K_{cub}* = 1; *K_{oct}* = 0.4714; *K_{tetr}* = 0.1222179).

X-ray diffraction analysis of condensed films generated by spraying Cu on K-8 glass substrates were carried out by the method of the moving beam. We analyzed the intensity distribution of the diffracted beam in the reflection area of (111) Cu.

Depending on the number of pulses irradiating a target, changes in the modulation period values are observed on the X-ray films. The calculated value of the edge lengths with *a* = 5.64 Å, that are included in the cubic-shaped nano blocks of the SE varies for the series (Table 2).

Table 2. The sizes of nano blocks after radiation of the condensed copper films on substrates from K-8 glass.

| N , pcs | 1 | | 2 | | 5 | | 11 | |
|-----------|------|-----|-----|------|-----|------|-----|-----|
| v , Å | 71.4 | 133 | 200 | 71.4 | 200 | 48.8 | 160 | 501 |
| F_n | 6 | 7 | 8 | 6 | 8 | 5 | 8 | 10 |
| a , Å | 68.3 | 110 | 179 | 68.3 | 179 | 42.1 | 179 | 468 |

The series (Table 2) shows that some percentage of the nano blocks from the SE maintained their sizes when transferring substance to a substrate. The amount of substance transferred for 31 pulses forms typical film structure of polycrystalline copper. The first layer (single-pulse irradiation of the target) is divided into small parts when it is separated from the substrate. This points at a poor adhesion of deposited and periodically connected nano blocks of the SE with the substrate. Maximum continuity of condensable films is achieved when a target is sprayed with two pulses. Modulation with a period of 71.4 Å observed in the single-layer and double-layer films can be attributed to the presence of the densest hexagonal packings of atomic layers in the transition between the nano blocks and the substrate areas. Hexagonal structure of these transitional layers' material with the substrate is observed at the electron- microscopic picture. Long-wave modulation of the film lattice increases in the multi-pulse mode and voids and cracks appear. When target is sprayed with five pulses, growth of crystal Cu, with size of the SE nano blocks is 199.8 Å begins in the film. A further increase in the number of pulses provides formation of polycrystalline copper with a modulation period 499.5 Å in the film.

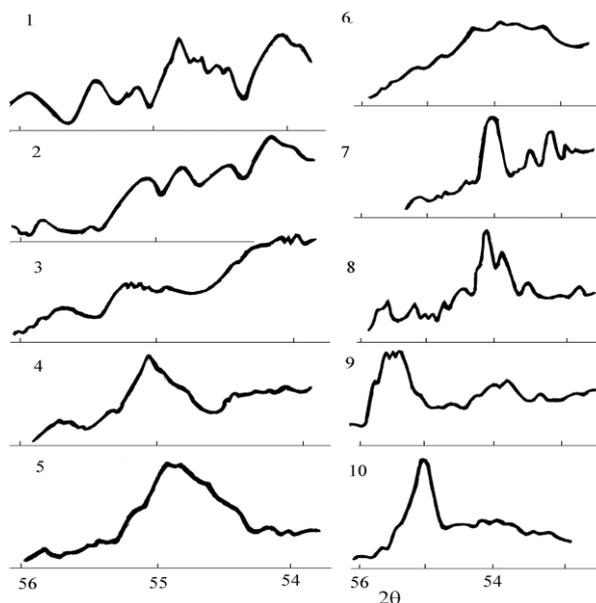


Figure 1. A portion of the Cu X-ray picture near reflex (111) of the subsurface layer of the target (5.1) and condensed film (6-10) during irradiation with nanosecond pulses of protons (pieces) 1 (1,6) 2 (2,7) 5 (3,8), 11(4,9), 31(5,10).

4. Summary

Thus, the conducted radiographic study in conjunction with the method of recoil nuclei and electron microscopy showed the nano block nature of the structure formation in the surface layer of the target and in the formed condensed film.

Due to the high concentration of protons, it can be noted with sufficient approximation that micro blocks mostly are formed from the structural elements.

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