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SEPARATE CONSTRUCTION OF RECRYSTALLIZATION DIAGRAMS FOR GRAINS OF DIFFERENT TEXTURE COMPONENTS

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Abstract

Combination of the X-ray method for the grain size determination by intensity fluctuations with texture measurements allowed to reveal essential differences in growing of grains of different texture components by recrystallization. The procedure was used as applied to rods from tin bronze by elaboration of optimal annealing regimes for manufacture of Nb_3Sn superconductors. Rods of tin bronze were subjected to wire-drawing with deformation degree of 7 to 45% and annealed at temperatures 300 to 550°C. The axial texture was formed in rods, so that by X-ray measurement of disks, perpendicular to the rod axis, obtained pole figures represent an aggregate of concentric rings. For each of them an average value of intensity fluctuations was measured by corresponding diffractometric curves and the size of recrystallized grains was calculated with the minimal value near 2 μm.

Introduction

The production of modern Nb_3Sn-based superconductors consists in the multipass extrusion and drawing of the multifiber assembly of niobium and bronze rods followed by the diffusion annealing. Unlike the more ductile niobium, the bronze is hardened significantly during processing even at the little deformation degree, so it is necessary to subject the composite rod to the softening annealing to prevent its cracking. Herewith the annealing regimes should lead to the bronze softening, but prevent the niobium recrystallization. Although the bronze recrystallization diagrams is presented in the literature, the increasing of the tin content in the bronze up to 14 wt% and the addition of different alloying elements result in variation of temperatures of the onset and the finishing of recrystallization as well as the obtained grain size. Therefore, the recrystallization features of rods made of tin bronze (Cu-14wt% Sn), used for the low-temperature superconductors production, were studied in this work.

The X-ray methods are widely used for studying of the recrystallization processes of the materials. They give the opportunity to determine the temperatures of the onset and the finishing of recrystallization, the recrystallization degree, crystalline lattice parameters, the presence of defects in the lattice and other characteristics [1].

Furthermore, presently used metallographic studies for the grain size determination do not take into account the different grains orientations on the metallographic section; hence, they give results of some averaged estimation, including all texture components. At the same time the grains of different texture components are known [2] to differ in size by several times in some cases (for example, by presence of the multicomponent deformation texture, the appearance of annealing twins, under polymorphic transformations of the material and others). Moreover, different hardening degree of various texture components results in difference of their recrystallization process [3]. As opposed to metallographic studies, the method for the grain size determination by statistical fluctuations of X-ray intensity [4] due to its selectivity allows to separate characteristics of different texture components, belonging to the same phase, what allows a full and detailed investigation of the recrystallization process. In this paper, conventional procedures of X-ray characterization of the material as well as the method for the grain size determination reported previously in [5]. The method consist in the registration of X-ray intensity fluctuations at different positions of the sample during the measurement of texture pole figures (PF). When changing the position of the sample, the number of grains n within the irradiated area oscillates near some mean value $\bar{n}$, so that the measured intensity of X-ray reflection shows significant fluctuations. These fluctuations can be used for the grain size determination according to the expression $\left\langle (n-\bar{n})^2 \right\rangle = \bar{n}$, where brackets < > denote the mean value for all positions of the sample [4, 5].

Studied Samples and Experimental Procedure

The increasing of the tin content in the bronze up to 14 wt% [6] expands the temperature range of two-phase region of the phase diagram (Figure 1), what results in the formation of higher amount of intermetallic ε-phase or δ-phase, which can be fixed by rapid cooling. Besides, the phase composition within the high-temperature range (higher than 520°C) coincides with the border of two regions: (Cu) and (Cu)+γ-phase.

In the present study bronze rods (Cu-14wt% Sn), obtained by extrusion and drawing up to different deformation degrees, were subjected to the annealing within the temperature range 300 to 550ºC during 1 hour (Table 1).

The X-ray studies of rods were implemented on sections perpendicular to their axis in two stages:

– assessment of the structural state of samples was conducted by the full width at half-maximum (FWHM) of X-ray lines and lattice parameters of the main phase – a solid solution based on copper, having FCC crystalline lattice;

– for texture analysis by two incomplete PFs: {111}, and {200}, – were registered by means of standard procedure [7, 8]; grain sizes of different texture components were calculated using method described in [5].

Table I. Studied samples of bronze rods (Cu-14wt%Sn)

NN	Rod diameter, mm	Deformation degree, %	Annealing regime	
			T, °C	τ, h
1.	16.5	43.8	300 to 550	1
2.	9.41	25.1	350 to 550	1
3.	10.5	7.2	350 to 550	1

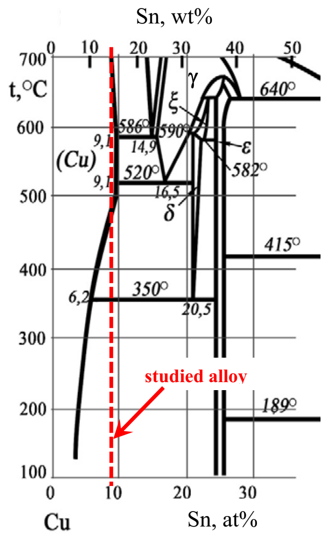

Figure 1. Phase diagram Cu–Sn [6]

Under annealing of metallic materials at temperatures below the recrystallization onset, they undergo a recovery and polygonization, accompanied by the decreasing of defects content and crystalline lattice distortions. This reduces the FWHM of X-ray lines and lattice parameters (Figure 2), but dropping of these parameters does not indicate the recrystallization onset because the nucleation of new grains, separated with high angle boundaries from the deformed matrix, are not correlated with the variation of the FWHM.

The recrystallization processes of FCC-metals can be accompanied with the fundamental texture variation, expressed in the arising of new and disappearance of initial texture maxima in the PF. This indicates that the grain nucleation occurs at the grain boundaries of the deformed matrix, so the textures of deformation and recrystallization differ. The temperatures of the onset and the finishing of recrystallization as well as recrystallization degree can be determined by the sharp variation of the ratios of texture components in this case, calculated by integral intensities of X-ray lines (Figure 2-b, c).

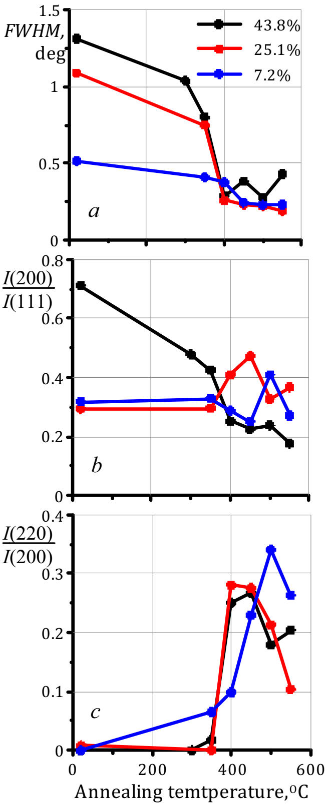

Figure 2. FWHM (*a*) and ratios of integral intensities of different X-ray lines (*b*, *c*) depending on the annealing temperature of bronze rods with different deformation degree: 7.2%, 25.1%, 43.8%

The texture of studied bronze rods has an axial character remaining after annealing (Figure 3). The deformation texture of bronze is formed by two typical for FCC-metals components, characterized by alignment of axes <111> and <100> in parallel with tensile direction (Figure 3-a). After annealing of rods their PFs retains the initial axial character, but in the course of annealing the circular texture maxima fragment and break into separate reflections, testifying about arising of new grains, i.e. nuclei of recrystallization. Resulting individual reflections are seen best of all in circular PF sections, corresponding to different tilt angles ψ (Figure 3-b). As the annealing temperature increases, these fluctuations grow-up, indicating the raising of the metal grain size.

When one of axes <111> of each grain is situated in the center of stereographic projection, other axes <111> due to crystallographic features of the cubic lattice must be homogeneously distributed by cones with apex angles 70.53 and 109.47°, and when in the center there is one of axes <100>, other axes of this type are situated at the cone with apex angle 90°. In the cubic lattice there are the following angles between main crystallographic axes: <100>–<110>=45º, <100>–<111>=54.74º, <110>–<111>=35.26º. Hence the PF section {111} with α = 55-60° characterizes grains of component <100>,

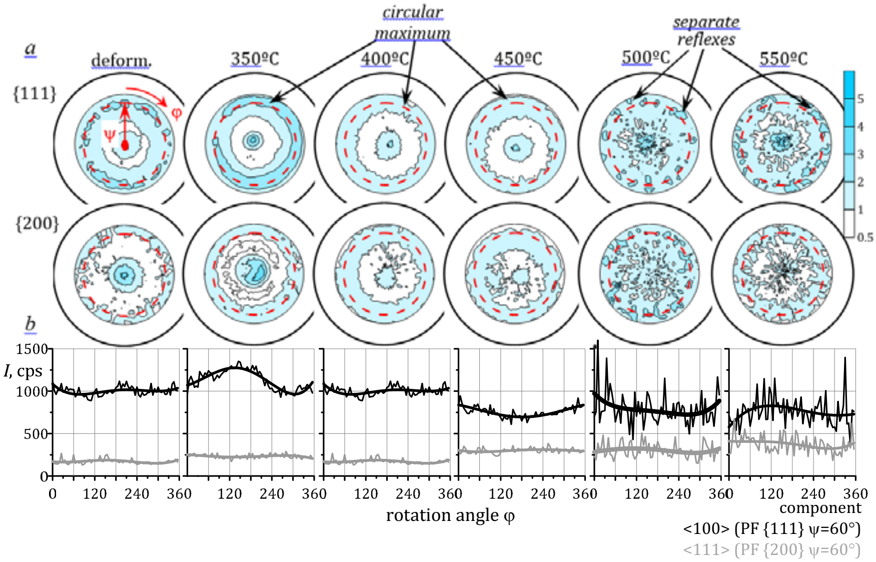

Figure 3. Fragmentation of circular maxima in texture of bronze rods (ε = 43.8%) due to annealing at different temperatures: *a* - PF{111} and {200}; *b* – circular PF sections at tilt angle ψ=60º

and PF section {200} with ψ = 55-60° – grains of component <111>. Due to scattering of the circular texture maxima in the radial direction of PF, the grain sizes were determined for three PF sections ψ = 55º and 60º for both components and then averaged.

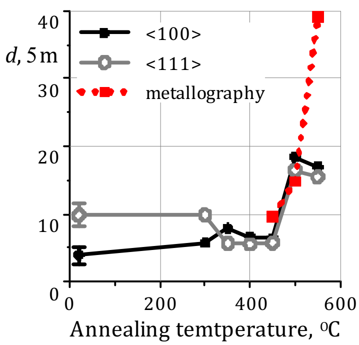

Figure 4. Dependencies of the grain sizes on the annealing temperature for texture components <100> and <111> in bronze rods with 43.8% deformation

The value of mean intensity of component <111> becomes closer to the mean intensity of component <100>, what testifies about the ratio variation of these components under recrystallization. Figure 2-b, c shows the different ratios of X-ray line intensities, which indicate the corresponding interrelations of texture components and temperatures of the recrystallization onset and finishing.

Variations of grain sizes for both texture components of rods with 43.8% deformation are presented in Figure 4. In the deformed state grains, corresponding to the component <100>, have a smaller size that indicates their greater deformation degree. Therefore, the recrystallization starts first in the grains of this texture component.

The decreasing of grain size for both texture components in the temperature interval 350-450ºC is seen in Figure 4. Such material behavior may be related

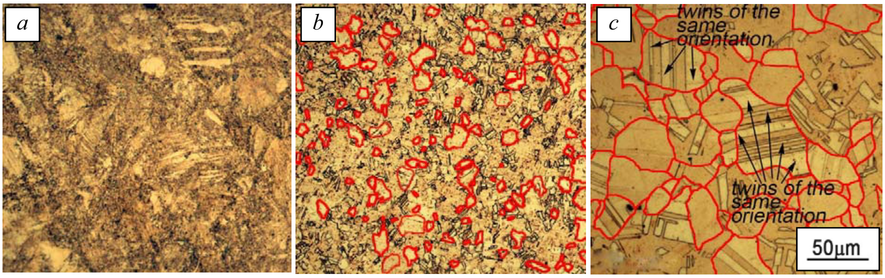

Figure 5. Microstructures of studied rods with 43.8% deformation degree annealed at 350ºC (*a*), 450ºC (*b*) and 500ºC (*c*)

to the phase transformations in the bronze at these temperatures. The texture within the mentioned temperature range is significantly scattered (Figure 3-1). Such a behavior of the material can be associated with features of the processes of grain refinement and simultaneous phase transformations under high temperature (Figure 1) with the formation of intermetallic δ-phase. Further increasing of the annealing temperature to the single phase region of the phase diagram Cu-Sn results in the raising of the grain size, which stops at 550°C. This temperature is associated with another two phase region (Cu)+γ-phase, possibly corresponding to the studied alloy composition.

Figure 5 shows microstructures of bronze for different annealing temperatures. It can be seen that grains of different sizes, which may differ in several times, are observed in rods especially at high temperatures. Moreover, the presence of annealing twins in some grains after annealing at 500ºC considerably obstructs the grain size determination by metallographic studies. The borders of grains, obtained by metallographic method, are drawn with red lines. Grains with different sizes as well as the arising twins are the regions of different orientations from the standpoint of X-ray methods, so due to the selectivity of X-rays the grain size of various components can be determined. It is seen (Figure 4) that after annealing at high temperatures grains of texture component <100> are larger than those of component <111>. And they are both much smaller than obtained by metallography.

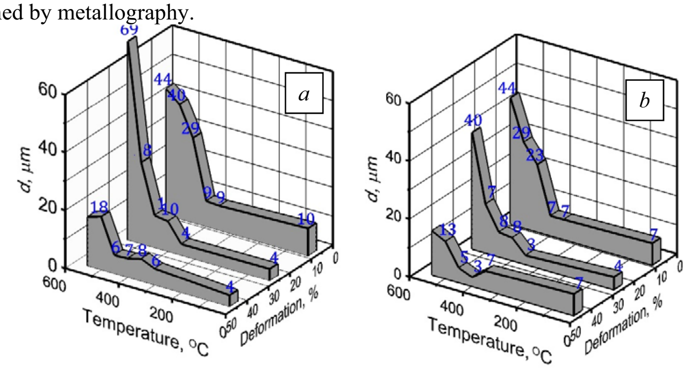

Figure 6. Fragments of the recrystallization diagrams for <100> (a) and <111> (b) texture components

Fragments of the recrystallization diagrams for both texture components are presented in Figure 6. In the course of texture measurement, the used method of grain size determination proves to be far less labour- and time-consuming, than any other. It allows to reveal with certainty details of the recrystallization process, such as the following. Temperature dependences of grain size are non-monotonical and change with increase of the pre-deformation degree. Because of the low density of recrystallization nuclei in rods with small deformation degree (7.2%) at 450°- 500ºC new grains of both components have time to grow up to larger size before they meet their neighbors, than it occurs in rods with higher deformation degrees. However, at 550ºC the situation changes. At temperatures of the two-phase region of the phase diagram, the grain size does not exceed 10 μm. When comparing temperature dependencies of grain size by different pre-deformation, one can see that at 500ºC primary recrystallization finishes in the rod with $\varepsilon = 43.8\%$, though continues still in other rods.

Conclusions

The decreasing of the temperature of the recrystallization onset of bronze Cu-14wt%Sn from 450ºC at 7.2% deformation to 350ºC 43.8% deformation at was established. The corresponding mean grain size after annealing at 500ºC is reduced from 44 μm (7.2%) to 15 μm.
The end stage of primary recrystallization occurs near 500ºC at 7.2% and near 450ºC at 43.8%.
The grains of texture component <100> are characterized with larger size than for component <111>.
The annealing at temperatures within two-phase region of diagram (350-450ºC) results in grain refinement in rods deformed up to 43.8% deformation, while at lower deformation degrees there is no decreasing of grain size because of smaller hardening degree.

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