ON THE LOW-TEMPERATURE RECRYSTALLIZATION OF COPPER ROLLED AT 77 AND 20°K

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It is known that lowering the deformation temperature leads to an increase in the stored energy of deformation, which characterizes the degree of distortion of the crystal lattice ([1]). In this connection one may expect that, upon heating metals deformed under conditions of deep cooling, recovery processes in the lattice will occur at comparatively low annealing temperatures. In previous works ([2],[3]) a substantial decrease in the recrystallization temperature after deformation at $77 \div 4.2$°K was shown. In these cases the activating deformation was not uniform, and therefore the formation of new grains was observed only in places of the strongest lattice distortion. The present communication gives new data on the recrystallization of polycrystalline copper, occurring at room temperature throughout the entire volume of the specimen, and on the activation energy of this process after large homogeneous deformation by rolling at 20 and 77°K.

The investigation was carried out on specimens of oxygen-free technical copper of purity 99.98% ($R_{4.2^\circ\mathrm{K}}/R_{293^\circ\mathrm{K}} = 1.1 \cdot 10^{-2}$), with a cross section of 0.8×1.2 mm^2 and an initial grain size of 100 μ. Deformation by rolling was performed at 293, 77, and 20°K reversibly at a rate of 10 mm/min ([4]). The degree of reduction in all cases was 90%; the reduction per pass was 10%. Rolling at 77 and 20°K was carried out under a layer of cooling liquid. The rolled specimens were annealed in the temperature interval 293—468°K. Taking into account the known influence of intermediate anneals on recrystallization processes, the specimens were heated rapidly from the deformation temperature to the specified annealing temperature, without pauses or intermediate rests. The onset of recrystallization was determined from the appearance of the first point reflections (“spots”) on the diffraction rings of X-ray patterns of the specimens studied, taken by the back-reflection method.

The investigations showed that recrystallization can proceed at temperatures considerably lower than the recrystallization-onset temperatures usually cited in the literature. In copper rolled by 90% at 77 and 20°K, recrystallization occurs even at room temperature, and the more rapidly the lower the deformation

Figure 1

Figure 1: Figure 1

temperature. If after rolling at 20°K recrystallization begins after 19 h, then after rolling at 77°K it begins after 2.5 months.

Figure 1 gives X-ray patterns taken from one and the same specimen immediately after rolling at 20°K and after holding at room temperature for 19 and 100 h. Deformation at low temperatures produces considerable microdistortions in the lattice and a finely dispersed structure, as evidenced by the strong blurring of the lines in the X-ray patterns (Fig. 1a). However, already after a 19-hour hold at room temperature the doublet begins to be resolved, and individual point reflections appear on the (331) and (420) lines (Fig. 1b); their number steadily increases with increasing holding time (Fig. 1c).

Thus, despite the low purity of the copper studied, low-temperature deformation leads to the occurrence of recrystallization

Fig. 1. X-ray diffraction patterns taken from a polycrystalline copper specimen: **a** –immediately after rolling at 20° K by 90%, **b**, **c** –after holding at room temperature for 19 h (**b**) and 100 h (**c**). The appearance and growth in the number of "spots" indicate the occurrence of the recrystallization process.

throughout the entire volume of the specimen even at room temperature over a comparatively short period of time.

A more important kinetic parameter of recrystallization is the activation energy, which does not depend on the annealing time. In Fig. 2, in semilogarithmic coordinates, the dependences are given of the time to the onset of recrystallization τ on the reciprocal annealing temperature. From the relation $\tau = \tau_0 e^{Q/RT}$, the activation energy of the onset of recrystallization was determined from the slope of the straight lines $\ln \tau = f(1/T)$. Since recrystallization is characterized by complex kinetics governed by the formation and growth of nuclei, the energy determined in this way represents an averaged effective activation energy Q_{eff} (5).

The following values of the activation energy of the onset of recrystallization were obtained for copper deformed at different temperatures:

Rolling temperature, °K	293	77	20
Q_{eff}, kcal/g-atom	33	25	18

It is evident that lowering the deformation temperature reduces the activation energy. The activation energy of the onset of recrystallization after deformation at 20° K is almost 2 times smaller than after deformation at room temperature. This circumstance, as well as the decrease in the recrystallization temperature,

Fig. 2. Dependence of the time to the onset of recrystallization on the reciprocal temperature for copper specimens deformed by 90% rolling at 293, 77, and 20°K

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is due to the growth of the stored deformation energy and the increased degree of metastability of the crystalline body.

The decrease in the temperature and activation energy of recrystallization after low-temperature deformation can be explained from the standpoint of modern ideas about structural changes in a strain-hardened material and about the processes of formation and growth of recrystallization nuclei. At present, the dislocation concept is the most widely accepted and experimentally confirmed; according to it, recrystallization nuclei are formed as a result of redistribution (and partial annihilation) of dislocations in places where their density is highest, with the subsequent creation of large-angle, easily migrating boundaries.

Low-temperature deformation creates very favorable conditions for the occurrence of recrystallization processes, since:

a) with a decrease in the deformation temperature, the size of the cells decreases and at the same time the angle of their misorientation increases (6), i.e., large-angle boundaries with high mobility are formed;

Fig. 2. Dependence of the time to the onset of recrystallization on the reciprocal temperature for copper specimens deformed by 90% rolling at 293, 77, and 20° K

b) a decrease in the cell size leads to an acceleration of nucleus formation owing to a decrease in the dislocation path length;

c) during low-temperature deformation, a large number of highly mobile point defects (vacancies) are formed, which enrich the subgrain boundaries during annealing and thereby increase their mobility (7, 8).

These processes apparently affect not only the initial stage of recrystallization, but also the formation of a stable microstructure.

In connection with the phenomenon of low-temperature recrystallization, the possibility opens up of studying recrystallization diagrams of metals while taking into account the temperature regime that activates work hardening, and of creating structures with special physical properties.

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Note: Figure translations are in progress. See original paper for figures.