Influence of Deformation Temperature on Microstructure Evolution and Static Recrystallization of Polycrystalline Copper

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Static recrystallization was studied in warm and hot deformed polycrystalline copper (99.99%). Samples were compressed to a strain of 0.25 at 573 K and 723 K at a strain rate of $2 \times 10^{-1} \text{ s}^{-1}$, quenched in water, and subsequently isothermally annealed at 673 K. Static recrystallization kinetics were found to be faster in samples hot-deformed at 723 K in spite of the lower level of stored strain energy. In all cases nucleation at the early stages of annealing takes place by bulging of serrated grain boundaries. Deformation at 723 K introduces higher orientation gradients evolved along serrated grain boundaries than those at 573 K. It is shown that static recrystallization is controlled not only by the level of stored strain energy, but also by the specific features of sub-grain structures formed in local regions.

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1. Introduction

In the last few decades, the studies on recrystallization in both static and dynamic conditions have been carried out using a wide range of materials and deformation modes.¹⁻⁵ It has been shown that the basic steps of conventional static and dynamic recrystallization (SRX and DRX) are the formation of new grains with high angle boundaries followed by long range migration driven by stored strain energy in the deformed matrix.⁵ One of the accepted nucleation mechanisms of SRX and DRX is bulging of pre-existing grain boundaries and is usually observed in relatively low-strained materials.⁴⁻⁶ In addition inhomogeneous micro-regions, such as deformation bands and shear bands, can act as preferential site for nucleation of recrystallization. Recently, the use of orientation imaging microscopy (OIM) has proved a powerful technique for revealing structural inhomogeneities and microtextures evolved after cold working.⁷⁻¹⁰ It has been documented that evolution of local inhomogeneities in deformed microstructures is sensitive to deformation temperature, strain rate and the initial structure, *etc.*^{11,12} Although the evolution of these structures at ambient temperature is well documented there is a much less information of such structures formed under warm and hot deformation. The specific nucleation mechanisms operating in SRX and several features of SRX kinetics are particularly unclear in hot-deformed materials.

The aim of the present work is to investigate the evolution of microstructures in materials deformed at elevated temperatures, and to subsequently study the influence of deformed microstructures on SRX kinetics including nucleation. Particular emphasis is placed on the local inhomogeneous features developed under warm or hot deformation, and the relationship of such regions to grain nucleation. The use of OIM was a key experimental strategy for the study.

2. Experimental Methods

2.1 Material and deformation mode

The samples of a polycrystalline copper (99.99%), 10 mm in diameter and 12 mm in height, were annealed for 1.8 ks at 923 K, leading to an equiaxed recrystallized grain size of about 230 μm . Constant true strain rate compression tests were performed using a testing machine equipped with a water quenching apparatus⁶) to freeze the instantaneous deformation structure. Samples for static annealing test were deformed to a strain of 0.25 at 573 K ($= 0.42T_m$, where T_m is the melting point of copper) and 723 K ($0.53 T_m$) at a strain rate of $2 \times 10^{-1} \text{ s}^{-1}$. To reduce frictional effects during compression, graphite powder was used as a lubricant and this successfully eliminated barreling. The deformed samples were sectioned parallel to the compression axis into specimens approximately 1.0 mm thick. These were annealed in a salt bath held at 673 K for various periods of time from 1 to 1000 s.

2.2 Microstructural observation and orientation imaging microscopy

The progress of recrystallization was studied by a number of metallographic methods: optical microscopy (OM), scanning electron microscopy (SEM) incorporating an orientation imaging microscopy (OIM) system and transmission electron microscopy (TEM). Samples for optical microscopy were mechanically polished with a suspension of alumina powder and then electropolished in a 50% aqueous solution of phosphoric acid at 2.1 V for 60 s followed by electroetching at 0.5 V for 15 s. Microhardness measurements under a load of 2.9 N were performed at room temperature. The fraction recrystallized and the mean grain size in annealed specimens were determined by a point counting method and a linear intercept method with an aid of a computer software for image analysis, *Image-Pro Plus*TM. Special care was taken to exclude twin boundaries from grain size measurements. Grain misorientation distributions were obtained from electron backscattered diffraction pattern (EBSP) analysis by using a Hitachi

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S-3500H SEM with OIM™ software provided by TexSem Laboratories, Inc. The area scanned by the electron-beam, with a step of 5 μm , was always over $700 \times 900 \mu\text{m}^2$. This area included at least 20 grains for as-deformed specimens and over 50 grains for partially and fully recrystallized specimens. The misorientation distribution of sub-grains was calculated from misorientation profiles measured in 10–15 representative grains on over 330 scan points for each sample. TEM samples prepared by conventional twin-jet polishing in an 80% aqueous solution of phosphoric acid were examined in a JEOL 2000-FX electron microscope at an accelerating voltage of 200 kV. The dislocation density in the deformed specimen was determined in 5 to 6 different areas ranging between 0.4 and 1.5 μm^2 in grain interiors. The dislocation density was counted as number of dislocation-foil surface intersections per unit area. This method gives an “area density” of dislocations in units of dislocations/ m^2 .¹³⁾

3. Results and Discussion

3.1 Deformation behavior and microstructures

True stress-true strain (σ - ϵ) curves of 4 N Cu at 573 K and 723 K are presented in Fig. 1. The flow curves at 723 K exhibit the typical shape of a material undergoing dynamic recrystallization (DRX), *i.e.* a single peak followed by work softening. In contrast, almost no strain rate dependence is observed at 573 K and a steady state flow is achieved at high strains with no apparent softening. For convenience in this paper, deformation performed at above or below $0.5T_m$ will be defined as hot deformation (HD) or warm deformation (WD), respectively. It may be noticed that strain of 0.25 is far below the peak strain of about 0.6 at 723 K ($0.53T_m$) and $2 \times 10^{-1} \text{ s}^{-1}$ (*i.e.* under HD), which suggests that the deformation microstructures in the specimens provided for static annealing test do not have any DRX grains and so should be only dynamically recovered (DRV).^{4,14)}

From the σ - ϵ curves in Fig. 1 differences in average stored energies, as well as dislocation substructures, in the samples

deformed at 573 K and 723 K may be anticipated. Typical TEM micrographs resulting from deformation at 573 K and 723 K are shown in Fig. 2. Elongated, trans-substructural channels separated by dense dislocation walls (DDWs) have developed. The DDWs formed at 723 K are much more rearranged and the dislocation densities in the regions between the walls are lower than those at 573 K. The dislocation density in cells and the average cell or channel size were evaluated from these micrographs and the measurements are summarized in Table 1. Total dislocation densities (ρ_{tot}) and values of stored strain energies (E) were calculated using the equations.⁵⁾

$$\rho_{\text{tot}} = (\sigma/Gb)^2 \quad (1)$$

$$E = 0.5\rho_{\text{tot}}Gb^2, \quad (2)$$

where $G_{573\text{K}} = 3.75 \times 10^4 \text{ MPa}$, $G_{723\text{K}} = 3.5 \times 10^4 \text{ MPa}$ and $b = 2.56 \times 10^{-10} \text{ m}$ for copper.¹⁵⁾ It should be noted that at 723 K dynamic recovery is more advanced so that the stored strain energy accumulated is estimated to be 1.5 times smaller than that at 573 K.

OIM micrographs of as-deformed microstructures at 573 K and 723 K are shown in Fig. 3. Crystallographic orientations are related with colors defined in the inverse pole figure. Different color for each grain and in grain interiors correspond to different crystallographic orientations. The color code given for orientation of grains/scan points is consistent with compression axis. In Fig. 3 high-angle boundaries with misorientations more than 15° are delineated by thick-black lines, while low-angle boundaries in the range of 4° – 15° by thin-black lines and those in the range 1.8° – 4° by thin-gray lines. Histograms showing the frequency of angular misorientations between adjacent sub-grains developed in grain interiors at the two deformation temperatures are shown in Fig. 4. Although the distributions appear similar, deformation at 723 K results in a larger range of misorientations, up to 15° , and so the average value (2.10°) is larger than that at 573 K (1.67°). It can be also clearly seen in Fig. 3(b) that a various kinds of colors accompanied with a high density of thin-black and -gray lines are present in each grain interior of the HD sample. This indicates that yet more recovered microstructures are distributed more inhomogeneously in grain interiors in the HD sample comparing with these of the WD one.

In order to evaluate long range orientation gradients, a parameter of particular importance for the nucleation of recrystallization,¹⁶⁾ relative sub-grain orientation sequential measurements were made along a line passing through a grain center from boundary to boundary, as shown in Fig. 3. Cumulative disorientation is defined as the relative difference of crystal orientation between an arbitrary reference scan point (1) and a following point i ($i = 2, \dots, n$), measured for each pair separately. For convenience a scan point within a grain interior is taken as the reference point which has zero lattice rotation. Figure 5 shows a typical example of cumulative disorientations (*i.e.* lattice curvature) that were found, the data are typical ones of those obtained in each sample, although the specific profiles of lattice curvature may differ to some extent from grain to grain. It can be seen in Fig. 5 that high orientation gradients are developed near grain boundaries and the gradients are higher with increasing temperature. Another feature noted in the profiles is that changes in

Fig. 1 Effect of deformation temperature and strain rate on the true stress-true strain curves of 99.99% Cu.

Fig. 2 Typical TEM substructures evolved in copper compressed to a strain 0.25 at a strain rate of $2 \times 10^{-1} \text{ s}^{-1}$ at (a) 573 K and (b) 723 K. Note difference in dislocation density within the DDWs.

Table 1 Average cell size, dislocation density and stored energy for copper deformed to a strain of 0.25 at 573 K and 723 K.

Deformation temperature	True stress at $\varepsilon = 0.25$	Average size of transverse channel/cell	Dislocation density in cell interiors	Total dislocation densities (calculated)	Stored energy (calculated)
$T(\text{K})$	$\sigma \text{ (MPa)}$	$d \text{ (}\mu\text{m)}$	$\rho \times 10^{14} \text{ (m}^{-2}\text{)}$	$\rho_{\text{tot}} \times 10^{14} \text{ (m}^{-2}\text{)}$	$E \text{ (kJm}^{-3}\text{)}$
573	177	0.58	2.47	3.40	417.7
723	140	0.77	1.95	2.44	280.0

cumulative disorientations are more irregular in the HD grain interior. This means that there are higher disorientations between some sub-grain boundaries evolved at 723 K. It has been shown previously¹⁷⁾ that such high orientation gradients developed near grain boundaries are due to grain boundary sliding and/or shearing connected with grain boundary serration. Microstructural examination in OM of present samples⁶⁾ revealed that grain boundaries are more frequently serrated at 723 K and, in contrast, relatively smooth and/or straight at 573 K.

3.2 Changes in microstructure with annealing

Changes in deformed microstructures with annealing time are presented in Fig. 6 for a WD sample and in Fig. 7 for a HD sample, respectively. It can be seen in Figs. 6 and 7 that recrystallized grains evolve in irregular and discrete clusters mainly at grain boundaries and less frequently at twin bound-

aries. The density of new grains is higher and sizes smaller in the HD compared with those in the WD material. Such features are representative of the whole of a deformed sample, confirming earlier optical microscopy results.⁶⁾ It is clearly seen in Fig. 7(b) that progress of SRX within deformed grains is related with evolution of various crystallographic orientations (depicted by various colors) accompanied with the development of many low-angle boundaries.

The distribution of misorientations between sub-grain boundaries, based on 10000 to 17000 measurements for each condition, and changes with annealing time are shown in Fig. 8. Results are remarkably similar for both deformation temperatures. The distributions for as-deformed state (*i.e.* $t = 0 \text{ s}$) show a bimodal shape, with the two peaks corresponding to high angle boundaries (HABs, beyond 20°) and low angle boundaries (LABs, below 10°). The fraction of LABs developed in grain interiors is much higher than that for

Fig. 3 OIM micrographs of copper deformed to a strain of 0.25 at (a) 573 K and (b) 723 K. Crystallographic orientations are expressed in colors according to inverse pole figure and color code is consistent with compression axis. Thin-gray lines correspond to boundaries of misorientation $> 1.8^\circ$, thin-black lines- $> 4^\circ$ and thick-black lines- $> 15^\circ$, respectively.

Fig. 4 Subgrain misorientation distributions in as-deformed grain interiors. (a) 573 K, (b) 723 K.

Fig. 5 Typical examples of the distribution of cumulative disorientations developed in grain interior. The disorientations were measured from a grain boundary (G. B.) to the other grain boundary along the lines indicated in Fig. 3.

HABs. The fraction of LABs rapidly decreases with annealing time, leading to the formation of moderate to high-angle boundaries. It is also noted in Fig. 8 that the decrease in the fraction of LABs is slightly faster in the HD sample than that in the WD sample. The analysis of HABs type revealed that they are mainly twin boundaries of first order ($\Sigma 3^n$, $n = 1$). After 80% recrystallization in the specimens annealed for 300 s, the fraction of any given $\Sigma 3^n$ boundary as well as grain boundary distribution is essentially similar to those for a fully recrystallized fcc metals susceptible to annealing twinning.¹⁸⁾

3.3 Progress of static recrystallization

The fraction recrystallized-annealing time (X_V-t) curves at 673 K are shown in Fig. 9(a). The X_V-t curves for both of the samples are rather similar and, somewhat surprisingly, are not influenced by deformation temperature. The recrystallization rate in an early annealing stage of the HD is even slightly

Fig. 6 OIM of the sample deformed to $\varepsilon = 0.254$ at 573 K and annealed at 673 K. (a). $t = 30$ s, (b). $t = 70$ s. The colors and lines have the meanings as described in Fig. 3.

higher than that for the WD material. These results would be anticipated to be reversed since the average strain energy stored at 723 K is lower than that at 573 K, as shown by the results in Figs. 1, 2 and Table 1. Figure 9(b) shows the X_V - t relationships re-plotted in log-log scale utilizing the KJMA equation:⁵⁾

$$X_V = 1 - \exp(-\beta t^n), \quad (3)$$

where X_V is the volume fraction recrystallized, β and n are experimental constants. Two regions with different slopes (n) are readily distinguished. At early stages of SRX the n for warm and hot deformation is 3.1 and 2.0, respectively. The slope changes abruptly at an annealing time ~ 40 s ($X_V \cong 25\%$) to a value of $n \sim 0.8$ for both materials.

Figure 10 shows changes in mean grain size, including both

Fig. 7 OIM of the sample deformed to $\varepsilon = 0.25$ at 723 K and annealed at 673 K. (a) $t = 30$ s, (b) $t = 70$ s. The colors and have the meanings as described in Fig. 3.

as-deformed and new recrystallized grains, with annealing time. The mean grain size begins to decrease at around 5 s, rapidly drops to a minimum and then increases slowly due to grain growth as recrystallization is completed. It can be seen in Fig. 10 that the D- t relations for the both samples are roughly the same, however, change in the D- t curve is always faster in the HD than in the WD material.

The results for room temperature microhardness (H_V) vs. annealing time (t) are shown in Fig. 11(a). Each point shows the average value from 8 to 12 measurements randomly performed over the central part of the sample. H_V starts to decrease after annealing for 20 s at which X_V is about 10% (Fig. 9(a)). Then H_V rapidly drops and approaches to a value of fully annealed state after annealing for about 500 s in both of the samples. The scatter of H_V , ΔH_V , evaluated by the standard deviations is plotted against annealing time in Fig. 11(b). The ΔH_V - t curve for the HD sample shows a broad peak at around 30 s and the ΔH_V decreases above 100 s and

Fig. 8 Changes in misorientation distribution of (sub)grain boundaries with annealing at 673 K in the sample deformed at (a) 573 K and (b) 723 K. Arrows indicate the average misorientation.

Fig. 9 The recrystallization kinetics at 673 K of copper deformed at 573 K and 723 K. (a) Volume fraction recrystallized, (b) KJMA plot.

approaches to a constant value for a fully recrystallized state. In contrast, the ΔH_V - t relation for the WD sample scarcely changes until 10 s and then rapidly increases to a sharp peak at around 70 s, followed by a decrease to the constant value. If ΔH_V can result from the degree of inhomogeneities in microstructure,¹⁹⁾ the latter suggests that the degree of inhomogeneity is higher at early stages of annealing in the HD sam-

Fig. 10 Variation of mean grain size with annealing time at 673 K.

ple. This may be confirmed by the results in Figs. 3 to 7.

3.4 Effect of deformation temperature on recrystallization

The important features of the analytical work described above is that the formation and growth of new grains are influenced not only by the average strain energy stored but also by the local inhomogeneous (sub-grain) structures which act as recrystallization nuclei. The hot-deformed grains formed at 723 K contain more recovered dislocation substructures, exhibit more sub-boundaries with higher misorientations and greater orientation gradients adjacent to grain boundaries (Figs. 4, 5). The density of new grains is also higher at early stage of annealing for the sample deformed at 723 K (Fig. 7). To further illustrate this point the number of fine grains per unit area, N , less than 10 μm in diameter is plotted against X_V in Fig. 12, and is considered to be proportional to the nucleus density at early stages of annealing.²⁰⁾ N for the sample deformed at 723 K rapidly increases with increasing X_V and reaches a peak at $X_V \cong 20\%$, followed by a rapid drop to zero at $X_V > 40\%$. In contrast, N for the sample deformed at 573 K increases relatively slower, with a lower peak value at $X_V \cong 25\%$ before dropping to a stable value at $X_V \cong 50\%$ and approaches zero near the completion of recrystallization.

New grains form preferentially at existing boundaries and examples of such grains evolved in the sample deformed at 723 K are shown in Fig. 13. It can be seen in Fig. 13 that bulging takes place at serrated grain boundaries, followed by the nucleation of new grains. Grain boundaries become more frequently serrated with increasing temperature under hot deformation.⁶⁾ If grain boundary sliding and/or shearing takes place at such serrated boundaries, higher orientation gradients can be evolved along the boundary vicinities at higher temperatures. This is consistent with the results in Fig. 5.

It may be noted that nucleation under dynamic recrystallization frequently takes place by bulging of such serrated grain boundaries. The critical condition for the operation of

Fig. 11 Changes in (a) room temperature microhardness (H_V) and (b) the scatter of hardness (ΔH_V) with annealing time.

bulging can be expressed by the following equation:²⁰⁾

$$\Delta E = 4 K \gamma_{GB} / L, \quad (4)$$

where ΔE is the difference in strain energy across the migration boundary, γ_{GB} the grain boundary energy and L the length of portions of serrated grain boundary. The constant K is usually one for smooth grain boundaries originally proposed by Bailey and Hirsch.²¹⁾ In contrast, for serrated grain boundaries introduced by hot deformation the K value should be always smaller than one.²⁰⁾ Thus, the bulging mechanism for DRX nucleation can operate easier and more frequently at serrated grain boundaries in hot-deformed matrices. It is quite possible that, since grain boundary sliding takes place easier with increasing temperature, grain boundary serration as well as high orientation gradients can be developed at near grain boundaries at 723 K (Fig. 5). Under such conditions grain boundaries can be the most preferable nucleation sites for not only DRX, but also SRX. This is the reason why a higher density of new grains is formed (Fig. 12) and the recrystallization kinetics is accelerated at early annealing stages for the sample deformed at 723 K (Fig. 9(a)).

After annealing for over 40 s the X_V - t relationships are

Fig. 12 Effect of deformation temperature on the relationship between the number of fine grains (N), whose diameter is smaller than $10 \mu\text{m}$ per unit area, and fraction recrystallized (X_V).

Fig. 13 Typical examples of new recrystallized grains evolved by bulging of initial grain boundaries. The sample was deformed to $\epsilon = 0.25$ at a strain rate of $2 \times 10^{-1} \text{ s}^{-1}$ and at 723 K, followed by annealing for 10 s at 673 K.

essentially the same (Fig. 9). This may result from the lower strain energy stored and/or the more recovered microstructure present in the HD sample. On the other hand, the fine grain densities (N) in $X_V > 40\%$ are almost zero for the sample deformed at 723 K but, in contrast, a small and almost constant value for the one at 573 K (Fig. 12). It can be surmised that the later stages of SRX for the sample deformed at 723 K are

Fig. 14 Relationship between average twin density and volume fraction recrystallized.

controlled mainly by growth of new grains, while in SRX for the 573 K sample the structure develops by both new grain formation and grain growth. The more recovered substructures and lower density of dislocations developed in the HD sample may lead to the development of coarser recrystallized grains in the later annealing stage.

An analysis of the extent of twin boundary formation may support the above hypothesis that SRX in later stages can be mainly controlled by new grain growth. Twins are usually observed to evolve in lateral glide apart from the migrating boundaries of new grains as a result of partial dislocations reactions at the expense of grain boundary energy.²²⁾ The density of twin boundaries, defined as the number of twin intersections by a line of unit length, was measured during various stages of recrystallization. Results are presented as a function of the volume fraction recrystallized in Fig. 14 and are given as a ratio of current twin density to the twin density for the as-deformed WD sample. It can be seen that the twin density increases rapidly to a small plateau at early annealing stages and then rises quickly to a final saturation value at $X_V > 40\%$. The later increase may be related to boundary migration due to the extensive grain growth.

4. Conclusions

The influence of warm- and hot-deformation on static recrystallization (SRX) of copper was studied. The main findings are as follows:

- (1) At early stage of annealing static recrystallization takes place slightly faster in hot-deformed samples in spite of the lower level of stored strain energy.
- (2) Recrystallization kinetics are represented by two different lines in an Avrami plot. A higher slope at early stages of annealing is associated with a nucleation process controlled by the deformed microstructure, while the lower Avrami slope at later stage of annealing is controlled by grain boundary migration taking place in recovered microstructures.
- (3) A higher density of nucleation sites evolves in the vicinity of grain boundaries under hot deformation due to the development of grain boundary serrations and high ori-

entation gradients. Under such conditions, bulging of serrated boundaries frequently takes place during early annealing stages.

- (4) It is concluded that static recrystallization can be controlled not only by the amount of stored energy (due to the increase in average dislocation densities), but also by the density of nucleation sites and degree of dynamic recovery.

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