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Recrystallization and Grain Growth in Ultrafine-Grained Materials Produced by High Pressure Torsion**

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Ultrafine-grained (UFG) materials processed by severe plastic deformation are known to exhibit good mechanical properties. Much about the annealing behavior of such materials is still unknown, and this work aims to provide a better understanding of the thermal properties of UFG materials. For this purpose a Cu–0.17 wt% Zr alloy was subjected to high pressure torsion (HPT) with a maximal pressure of 4.8 GPa at room temperature. The microstructures of the specimens were characterized using electron back scatter (EBSD) measurements, transmission electron microscopy (TEM), and hardness measurements. During annealing of the samples, dispersoids were formed which improved the thermal stability of the alloy. At higher strain levels the fraction of high angle grain boundaries (HAGBs) increased above 70% of the total grain boundaries.

Promising mechanical properties of ultrafine-grained materials processed by severely plastic deformation (SPD) methods make it necessary to investigate the thermal stability during annealing.

Up to now several studies have been performed on recrystallization behavior of heavily deformed pure Al and Al-alloys as well as on pure Cu.[1–5]

It has been known for some time that heavily deformed metals with a high fraction of high angle grain boundaries (HAGBs) can continuously recrystallize during annealing.[1] Humphreys has also shown that a cellular structure with ≈ 70% of HAGBs becomes nearly resistant to discontinuous recrystallization, as on average the cell boundaries approach the condition where they all have similar energies on mobility.[6]

It is difficult to give the conventional recrystallization terminology to severely deformed materials that contain high amounts of HAGBs.[7] We should also take into consideration that the recrystallization behavior is strongly dependent on the deformed microstructure, the existence of second phase particles in the microstructure.

The aim of this work is to study the recrystallization behavior in a CuZr alloy, subjected to high pressure torsion to an equivalent strain of up to 27, using high resolution electron back scatter diffraction (EBSD) measurements. During annealing of the samples, dispersoids were formed which improved the thermal stability of the alloy. At higher strain levels the fraction of HAGBs increased above 70% of the total grain boundaries. At this level, most of the grain boundaries exhibit equal mobility, which means that the microstructure should be stable against discontinuities. Nevertheless, there are discontinuities observed during annealing of these microstructures. The exact recrystallization behavior was examined by investigating the same area of microstructure (grain boundary character, local orientation, and texture evolution) during different stages of annealing to determine whether the discontinuities in the microstructure are due to discontinuous grain growth or recrystallization.

Experimental

The material used in this study is a Cu–0.17 wt%Zr alloy homogenized at 950 °C for 10 h. Disks of 10 mm diameter and 0.8 mm thickness were cut by spark eroding. The disks were then subjected to high pressure torsion to different twisting angles (up to 720°). The isothermal annealing of the samples were performed successively at a temperature of 550 °C in a salt bath.

A cylindrical sample with the dimensions diameter 10 mm and thickness 0.8 mm is fixed between two stamps with a

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pressure of 4.8 GPa and undergoes torsion through a definite angle of rotation. We analyzed samples after 120, 240, 360, and 720° rotations, corresponding to one-third rotation, two-third rotation, full rotation, and two full rotations, respectively.

The chosen angles of rotations lead to equivalent strains at a radius of 3 mm of: $\varepsilon = 4.5$ for 120° rotation, $\varepsilon = 9.0$ for the 240° rotation, $\varepsilon = 13.5$ for the 360° rotation, and finally $\varepsilon = 27.0$ for a rotation of 720°.

The equivalent strain is defined by the following equation:

$$\varepsilon = \frac{\gamma}{\sqrt{3}} \text{ with } \gamma = \frac{2\pi N r}{h} \quad (1)$$

where ε is the equivalent strain, γ the applied shear strain which is defined by N number of rotation, r the radius (distance from center of sample), and h is the thickness of the sample.[5,7,8]

After the deformation samples were cut from the disk, which is shown schematically in Figure 1 (right side). The dimensions of the samples are 5 mm in length and 3 mm in width. Therefore, the distance of the measurement plane from the center of the disk is 3 mm.

Results and Discussion

The Deformed State

Figure 2 shows the microstructure of the material after an equivalent strain of 4.5 and 13.5. As the strain increases the original HAGBs and the new deformation induced grain boundaries rotate and a fibrous microstructure develops. This fibrous microstructure becomes finer by further increasing of strain as can be observed from Figure 2(b).

The amount of HAGBs increases to a fraction of about 80% with increasing strain and then reaches a steady state (Figure 3). After this stage the amount of HAGBs does not show significant changes with increasing strain.

The same behavior could be recognized from the micro-hardness measurements and grain size evolution at different levels of strain (Figure 3).

During high pressure torsion the texture components of simple shear develop [Figure 4(a)]. These texture components are represented by two fibers: the *A*-fiber ({111} parallel to shear plane) and the *B*-fiber (<110> parallel to shear direction). At a strain of 4.5 the characteristic orientation is an orientation between A_1^* and B with further increasing of the strain the characteristic orientation becomes the $\overline{B}$ component, see also the list of texture components given in Figure 4(g).

Annealed State

The microstructure evolution during isothermal annealing of Cu–0.17 wt%Zr deformed to a strain of 13.5 at 550 °C in a salt bath is shown in Figure 5.

From the inverse pole figures one can observe that after 600 s there are some discontinuities developing in the

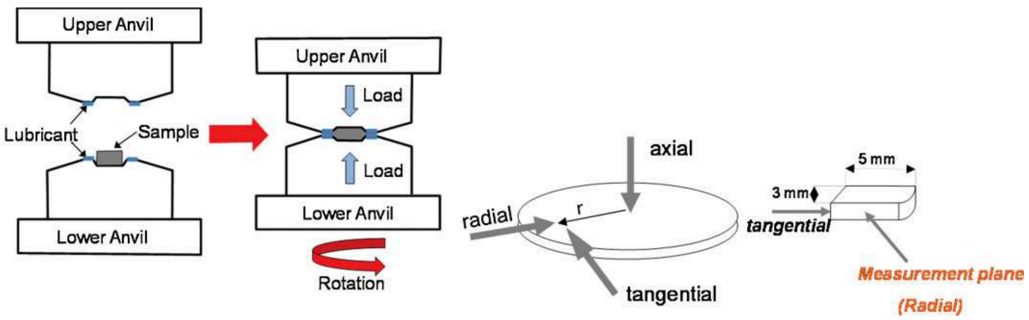


Fig. 1. Left side: schematic illustration of the HPT method.[9] Right side: sample geometry and dimensions of the analyzed HPT samples.

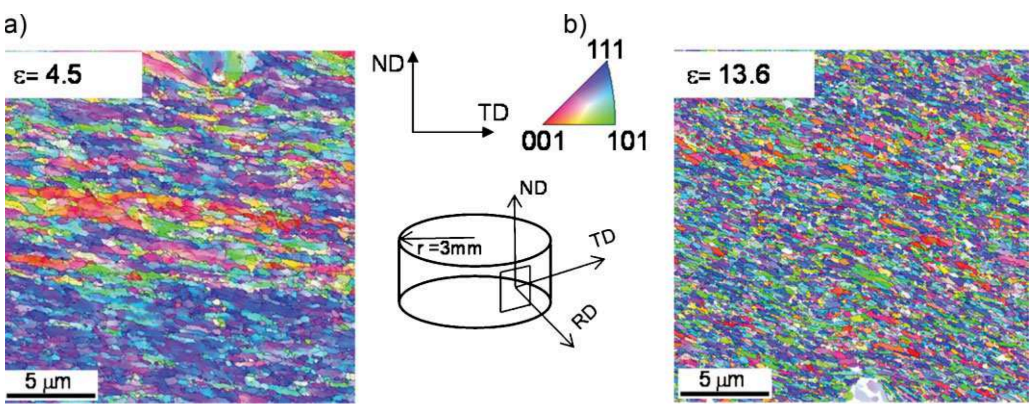


Fig. 2. Inverse pole figure map of a Cu–0.17 wt%Zr alloy subjected to high pressure torsion at equivalent strains of (a) 4.5 and (b) 13.5. The color coding indicates the crystal directions parallel to the radial direction.

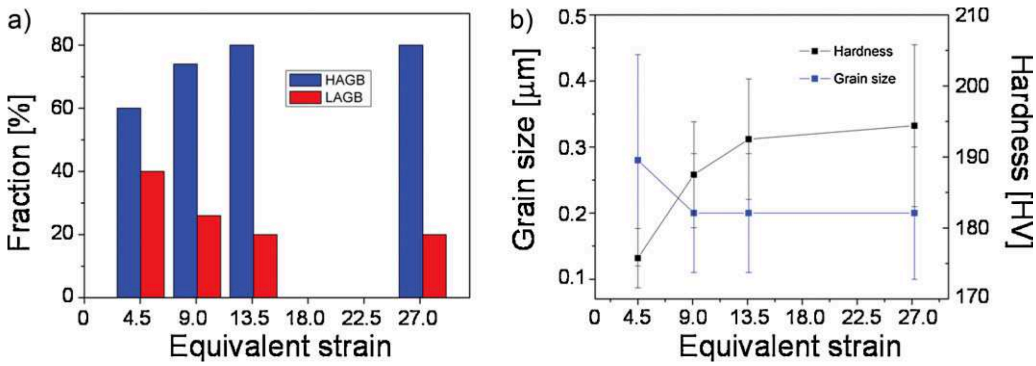
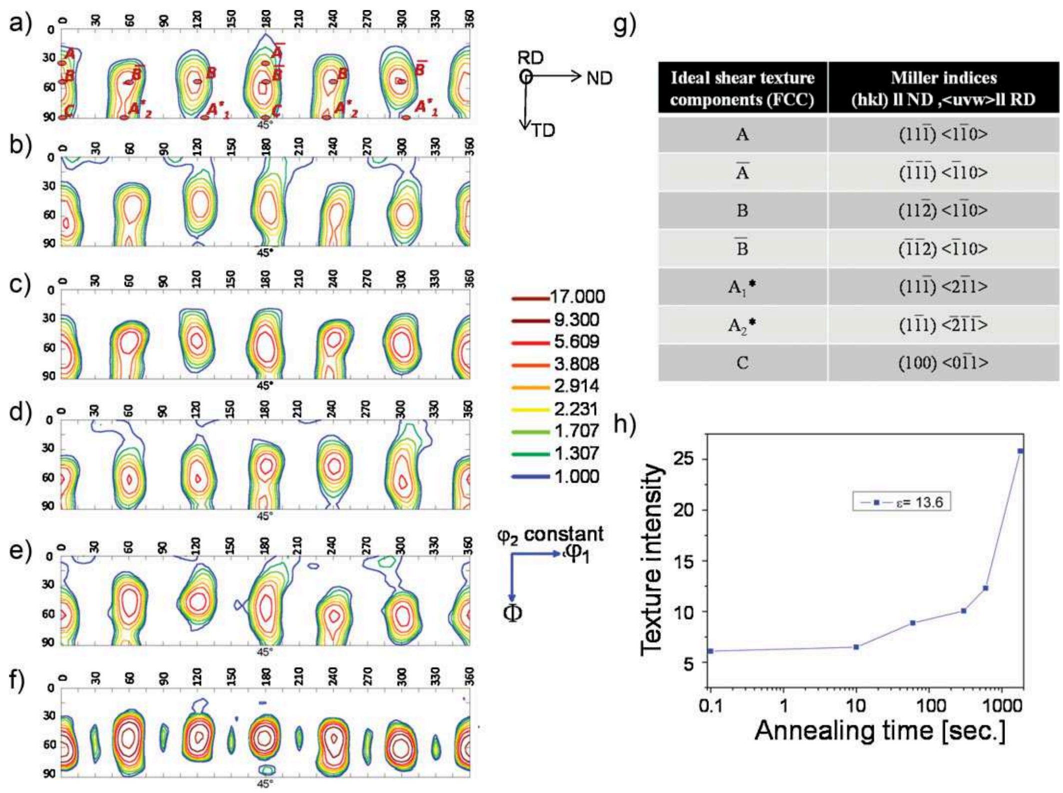


Fig. 3. Evolution of (a) HAGB and low angle grain boundaries (b) microhardness and grain size at different levels of strain subjected by high pressure torsion.

microstructure. According to the grain size and grain count evolution during annealing [Figure 6(a)] we can recognize that at the early stages of annealing the grain count decreases and the grain size increases up to annealing times of 60 s. Afterwards there is only a slight change in the grain size with increasing annealing time from 60 to 600 s and from there on the grain size increases again significantly.

The aspect ratio during the first stages of annealing increases from 0.44 to 0.51 and with further increasing of the annealing time the aspect ratio stays constant. There are only small changes in the fraction of HAGB area with annealing time [Figure 6(b)].

The grains in the deformed microstructure are in the range of submicrons and almost 80% of the grain boundaries existing in the microstructure are HAGBs. The increase of grain size at the very early stages of annealing (up to 60 s) is because of grain coarsening. It means that the existing grains with HAGBs, developed during deformation, start to grow slightly and develop a microstructure with more equiaxed grains. The grain size does not show significant changes from 60 to 600 s. It can be provoked by pinning effects of the dispersions on the grain boundaries. This should be investigated in detail using transmission electron microscopy (TEM).



Ideal shear texture components (FCC)	Miller indices (hkl) ll ND ,<uvw>ll RD
A	$(11\bar{1})$ $<1\bar{1}0>$
$\bar{A}$	$(\bar{1}\bar{1}1)$ $<\bar{1}10>$
B	$(11\bar{2})$ $<1\bar{1}0>$
$\bar{B}$	$(\bar{1}\bar{1}2)$ $<\bar{1}10>$
A_1^*	$(11\bar{1})$ $<2\bar{1}1>$
A_2^*	$(1\bar{1}1)$ $<\bar{2}\bar{1}\bar{1}>$
C	(100) $<0\bar{1}1>$

Fig. 4. Orientation distribution function of the sample during annealing at 550 °C (a) deformed after, (b) 10 s, (c) 60 s, (d) 300 s, (e) 600 s, (f) 1800 s, (g) ideal shear texture components for face center cubic materials. (h) Evolution of the maximum texture intensity during annealing (at 550 °C) of the samples subjected to a strain of 13.5.

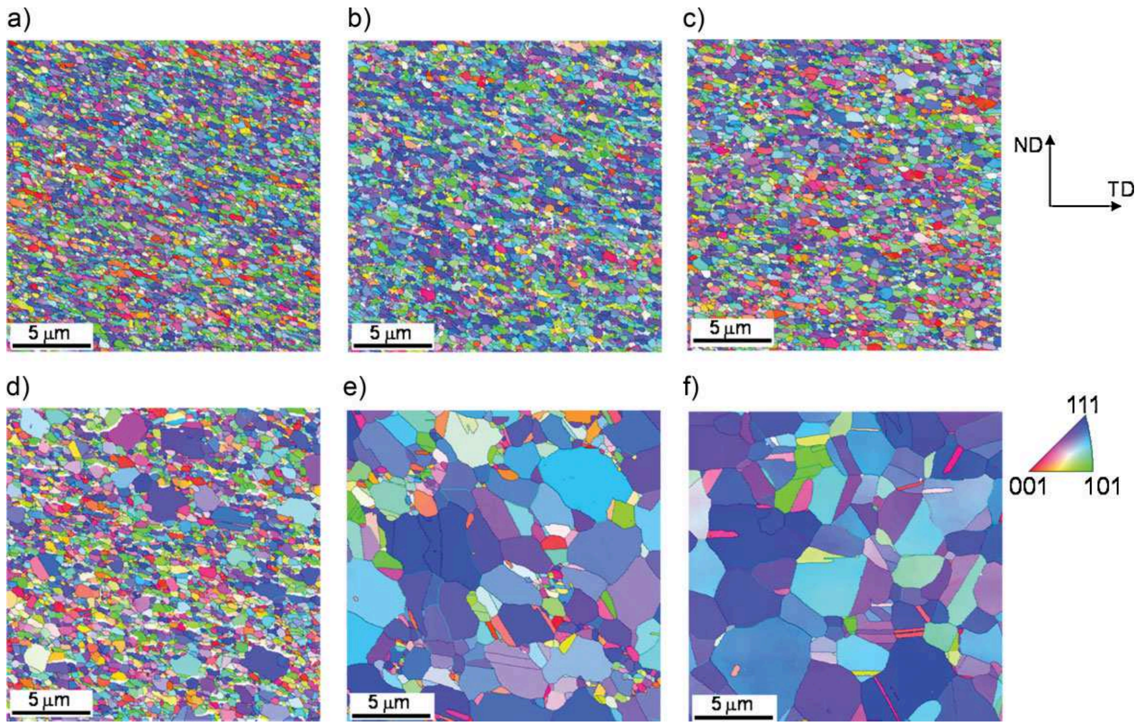


Fig. 5. Inverse pole figure maps of an isothermal annealing sequence of Cu–0.17 wt%Zr subjected to a strain of 13.5 at 550 °C for (a) 10, (b) 60, (c) 300, (d) 600, (e) 1800, (f) 3600 s.

The EBSD data also provides the Kernel average misorientation (KAM) which is a good quantity to evaluate the strain or the stored energy for a given volume element. The KAM is defined for a given volume element as the average misorientation of that point with respect to all of its neighbors. The higher the KAM, the larger the density of geometrically necessary dislocations (GNDs) in that point.

The KAM maps presented in Figure 7 show that with increasing annealing time, the stored energy from GND decreases. It shows that the recovery process is running parallel to grain coarsening at early stages of annealing. In Figure 7(e) we can see that there are some areas, which reduce their stored energy faster than other areas. These grains grow in the still deformed areas with a higher density of dislocation and discontinuities develop at this stage.

The texture evolution during annealing of the samples subjected to a strain of 13.5 shows that the texture components developed during the high pressure deformation exist after annealing at 550 °C and the position of texture components does not change during annealing up to 300 s [Figure 4(a–f)]. The maximum texture intensity increases during annealing [Figure 4(h)].

We also investigated the fraction of different texture components and how their stored energy decreases during annealing to see if there are priorities for them to grow or to reduce their energy during annealing process. Figure 8(b) shows that there are slight changes in the fraction of shear texture components by annealing at 550 °C up to annealing times of 600 s. During this time the strongest texture components are the B and $\overline{B}$ and after 600 s the fraction of both components increases significantly. The evolution of GND density in each texture component during annealing shows the same behavior [Figure 8(c)]. The higher rate of growth in some texture components is probably caused by the character of grain boundaries.

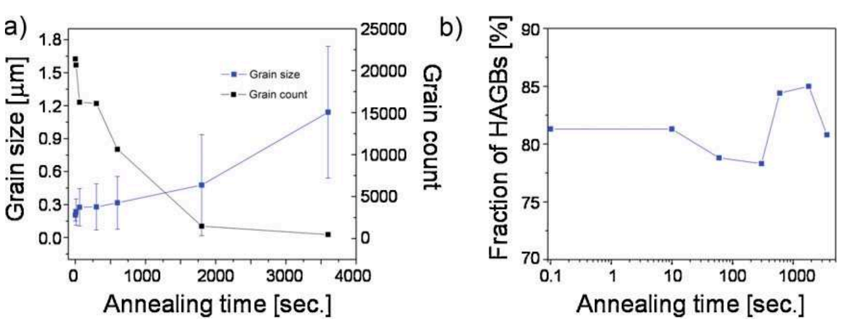


Fig. 6. (a) Average grain size, grain count and (b) fraction of HAGB area, calculated from EBSD data, of Cu–0.17 wt%Zr subjected to a strain of 13.5 at 550 °C.

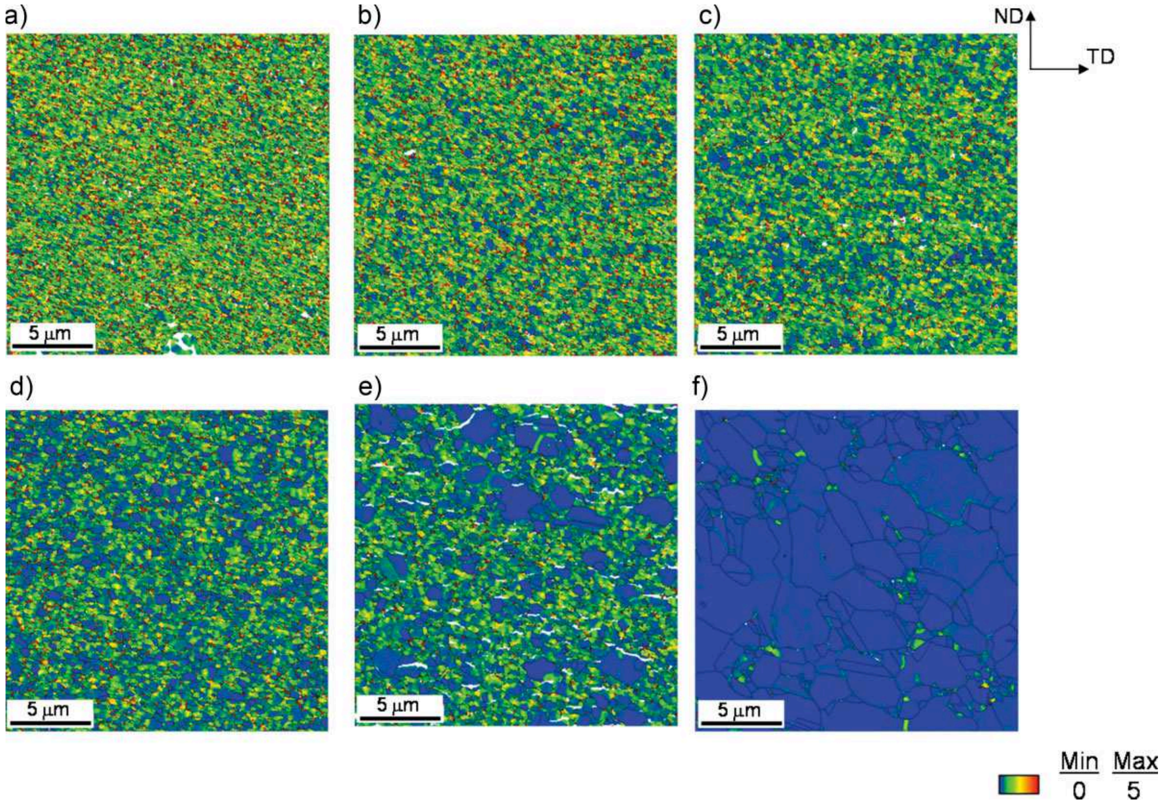


Fig. 7. KAM maps (3rd neighbor, step size of 50 nm) during annealing at 550 °C (a) deformed, (b) 10 s, (c) 60 s, (d) 300 s, (e) 600 s, (f) 1 800 s.

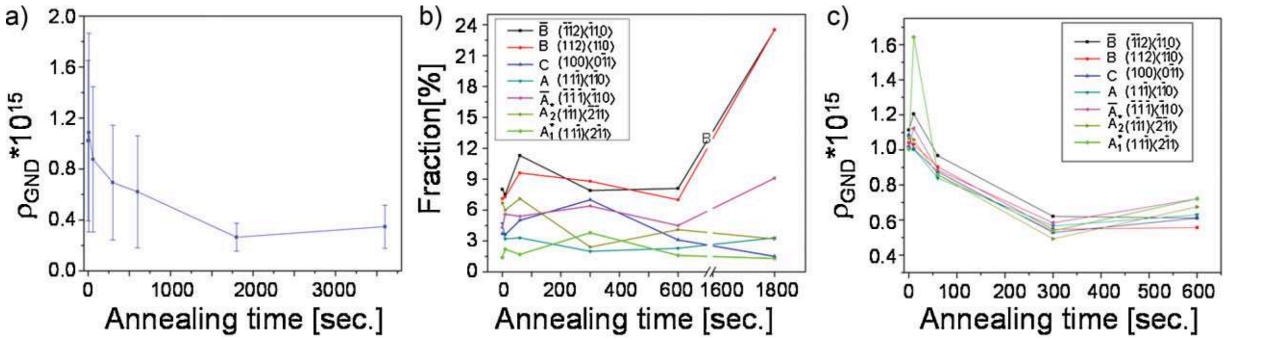


Fig. 8. Evolution of (a) overall density of GNDs, (b) area fraction of shear texture components, (c) mean density of GNDs in each shear texture component, in the samples subjected to an equivalent strain of 13.5 during annealing at 550 °C.

Conclusions

Investigation of the microstructure evolution of ultrafine-grained Cu–0.17 wt%Zr alloy subjected to strain of 13.5 by high pressure torsion method showed that at early stages of annealing extended recovery is taking place and then some grains can reduce their stored energy faster than others, due to the fact that they develop discontinuities in the microstructure and then after the microstructure is completely recrystallized normal grain growth takes place. The texture investigations showed that during annealing the strongest texture components are B and $\overline{B}$. The reduction of stored energy in different texture components showed the same behavior and other reasons should be responsible for the observed discontinuities for example different character of grain boundaries.

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