Microstructure Characteristics of ECAP Processed 1050 Aluminum After Deformation and 5 Years Later

M. Wroński¹ · K. Wierzbowski¹ · R. Malik¹ · S. Wroński¹ · D. Wojtas¹ · A. Baczmański¹ · J. Tarasiuk¹

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Abstract

Modification of microstructure parameters of severely deformed 1050 aluminum after 5 years of recovery at ambient temperature was studied. The samples of 1050 aluminum were extruded using ECAP technique. The EBSD maps were recorded for the initial material and for the deformed samples. Next, the latter samples were left at ambient temperature during 5 years and a full EBSD study of the material was repeated. It was found that all microstructure parameters and textures have changed after this period. This is due to recovery and partial recrystallization, which took place in the samples during 5 years. The presented modifications of material microstructure are important, e.g., in the aspect of long time service of mechanical parts produced by severe plastic deformation techniques.

Keywords Severe plastic deformation · Equal channel angular pressing · Electron backscatter diffraction · Microstructure parameters · Texture · Recovery

1 Introduction

The ultrafine-grained (UFG) microstructure of materials and resulting important modifications of physical properties can be obtained applying severe plastic deformation (SPD) methods. This wide subject is reviewed in many works, e.g., in [1–3]. SPD process can be realized using different techniques: equal channel angular pressing (ECAP) [4–9], hydrostatic extrusion (HE) [10], accumulative roll bonding (ARB) [11], KOB method (extrusion with oscillations of the die) [12], high pressure torsion (HPT) [13], multiaxial forging (MF) [14] and some variants of the above techniques.

However, the microstructure of UFG materials (especially of soft metals) generally evolves with time, even at ambient temperature. The microstructure evolution versus time can modify some characteristics of the material, e.g., hardness [15] or creep behavior [16]. This effect is important in the aspect of life time of different mechanical parts processed by SPD techniques. A possible critical modification of these

parameters can lead to fracture and destruction of an applied device.

The evolution versus time of UFG microstructure can be influenced by temperature of deformation processing [15], by amplitude of applied deformation [16, 17] and also by residual stresses present in the material. The evolution of such microstructure is often examined in aspect of short-term thermal stability (e.g., [18]). The exhaustive review of long-term UFG microstructure stability of different materials (2 alloys and 15 pure metals) processed by the high-pressure torsion (HPT) is presented in [15]. The examined samples were annealed at room temperature during times up to 10 years. It was found that the effects of such annealing depend on a few factors, but predominantly on the melting temperature (T_m). The metals with high T_m remained stable, while in metals with moderate T_m (Ag, Au, Cu) a softening by self-annealing occurred. And finally, surprisingly, in metals with low T_m (Mg, Al–Zn, Mg–Li) an unusual hardening occurred. The authors of the cited paper explained these effects by behaviors of grain boundaries with respect to the dislocation activity or grain-boundary sliding.

Another works reported also other factors, which stabilize the UFG microstructure: the pores filled with gas [19], presence of interface-dominant multilayers [20], formation of twins [20] and possible appearance of precipitates.

✉ K. Wierzbowski
wierzbowski@fis.agh.edu.pl

¹ Faculty of Physics and Applied Computer Science, AGH University of Science and Technology, 30-059 Kraków, Poland

In the present work the equal channel angular pressing (ECAP) was used to produce SPD state of the 1050 aluminum alloy. The microstructure produced by this technique and its variation after 5 years recovery at the ambient temperature were examined.

The problem mostly concerns the metals with lower melting temperatures, like aluminum and copper, in which slow but continuous recovery occurs at the ambient temperature. As mentioned, there are not many works devoted to this subject. The present study was inspired by our previous work [21], in which the variation of properties of highly rolled polycrystalline copper after 8 years of recovery at the ambient temperature was examined. In contrast to copper, aluminum has lower melting temperature ($T_m = 660\text{ }^{\circ}\text{C}$ for aluminum and $T_m = 1085\text{ }^{\circ}\text{C}$ for copper) but also higher stacking fault energy ($E_{sf} = 166 \times 10^{-3}\text{ J/m}^2$ for aluminum and $E_{sf} = 45 \times 10^{-3}\text{ J/m}^2$ for copper). It is known that metals with higher stacking fault energy have lower density of defects. Different values of these two parameters can influence deformation and annealing characteristics. Therefore, in the present work the characteristics of frequently used aluminum alloy 1050, processed by ECAP technique, are studied.

2 Experimental

The examined material was 1050 aluminum, which was processed by ECAP technique using route C and 90° channel. Two types of processed samples were examined: after 4 ECAP passes and after 8 ECAP passes, with punch speed of 5 mm/min in each deformation step.

The EBSD orientation maps were recorded for the deformed samples directly after ECAP processing (cf. [8]) and, next, 5 years later. Moreover, selected samples were recrystallized at $250\text{ }^{\circ}\text{C}$ during 1 h. Their orientation maps were measured and compared with maps of the selected deformed samples. The EBSD measurements were performed using Cambridge S360 (W-GUN) microscope and analyzed with the TSL software. The EBSD orientation maps were recorded in the central parts of the samples from $100\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}$ regions and were scanned with step sizes of $0.2\text{ }\mu\text{m}$ and $0.3\text{ }\mu\text{m}$ in the y - z plane (Fig. 1). A very delicate cleaning procedure was applied, which consisted in elimination of clusters containing less than four points. Samples were polished using silicon carbide paper (down to grade 4000) and in next step electro-polished using the A3 solution from Struers.

The hardness measurements were performed using Vickers hardness tester for the initial material, for the deformed samples (0 and 5 years samples) and for the recrystallized final samples. The following parameters were used in

Fig. 1 Geometry of ECAP process and reference system connected with EBSD measurements

measurements: force 1000 G, loading time 30 s, pause 15 s, unloading time 30 s.

3 Results and Discussion

Various microstructure parameters and textures were determined from EBSD orientation maps. The results for the samples after 4 and 8 ECAP passes were compared for two situations: directly after ECAP processing and for the same samples, kept at the ambient temperature, 5 years later. Also, selected fully recrystallized samples were included in our analysis.

In the following sections the examined material characteristics are presented and discussed.

3.1 EBSD Orientation Maps

The determined orientation maps were measured in central regions of the y - z plane of the samples (cf. Fig. 1). Representative parts of the maps for the samples after 4 and 8 ECAP passes, directly after deformation ('0 years') and 5 years later ('5 years') are shown in Fig. 2. As can be seen, '5 years' maps are slightly modified as compared with '0 years' ones. Consequently, one can expect that microstructure parameters and textures are modified and these issues are discussed in the next sections.

3.2 Grain Sizes

One of basic microstructure characteristics is the average grain diameter. Its values obtained using the number average for the samples after 4 and 8 ECAP passes are shown in Fig. 3a. All results presented in this work concern grain boundary definition based on 15° misorientation ($\Delta\theta = 15^{\circ}$). A general conclusion is that '5 years' samples have smaller grains than the '0 years' samples.

Fig. 2 Parts of EBSD orientation maps for samples after 4 and 8 ECAP passes, directly after deformation ('0 years') and 5 years later ('5 years')

Fig. 3 **a** Average grain diameter (number average) for ECAP samples after 4 and 8 passes, directly after deformation ('0 years') and 5 years later ('5 years'), **b** shares of grains with diameters up to 10 μm (4 passes) and up to 7 μm (8 passes), directly after deformation ('0 years') and 5 years later ('5 years'). Results based on $\Delta\theta = 15^\circ$ grain boundary misorientation are shown

This reduction is more marked in the case of the samples after 4 ECAP passes than after 8 passes—Fig. 3a. It should be noted that the average grain diameter for the initial (as received) material was 17 μm . Therefore an important reduction of the average grain size was observed after ECAP processing as compared with the initial state.

A complementary calculation was also done illustrating the decrease of the average grain size after 5 years. The shares of grains with diameters up to 10 μm for the 4

ECAP samples and the shares of grains up to 7 μm for the 8 ECAP samples clearly increased—Fig. 3b.

The above effect is also visible, if grain size distributions are examined—Fig. 4 (area fraction). In the case of 4 ECAP passes the majority of larger grains with the sizes between 10 and 35 μm disappeared, and in the case of 8 ECAP passes this happened for grains with sizes between 7 and 15 μm , which is visible in Fig. 4. What is interesting, the grain size distributions calculated with 'number fraction' (Fig. 5) show

Fig. 4 Distributions of grain size (area fraction) for the samples after 4 and 8 ECAP passes, directly after deformation ('0 years')—**a** and **b**, and five years later ('5 years')—**c** and **d**. Results based on $\Delta\theta=15^\circ$ grain boundary misorientation are shown

only a slight modification after 5 years. This proves that a huge majority of grains after 5 years are very small ones but, there is still some number of larger grains, which are reflected in the area fraction (Fig. 4).

The numerical comparison of the average grain sizes at consecutive stages of the samples history are included in the Table 1 below.

The observed decrease of grain size after 5 years can be explained by a progressing recovery process, which occurs at ambient temperature for aluminum: recrystallization nuclei and new small grains appear. It is also seen that after full recrystallization (250 °C/1 h) the average grain sizes of both samples are distinctly higher than after deformation, but still smaller than for the initial material. Moreover, it can be noted that the average grain size of the recrystallized material is smaller in the case of the 8 ECAP passes sample.

3.3 Intra-granular Fragmentation, Density of Dislocations and Stored Energy

Besides decrease of the average grain size also the effects of grain heterogeneity, the overall average misorientation

and average misorientation inside grains have an important impact on material properties. Three parameters, which characterize the above effects, were determined from the measured EBSD orientation maps: grain orientation spread (GOS), kernel average misorientation (KAM) and grain average misorientation (GAM). These parameters were calculated for the initial (as received) material and for the '0 years' and '5 years' samples after 4 and 8 ECAP passes—Fig. 6.

GOS parameter expresses the mean orientation spread of measured points inside grains with respect to their average orientations. Therefore it characterizes the intra-granular heterogeneity of material. KAM parameter expresses the average misorientation between neighboring points for the whole sample. In contrast, GAM characterizes the average misorientation between neighboring points but only in interiors of grains (grain boundaries are excluded).

The first observation is that these three parameters have distinctly lower values for the '5 years' samples than for the '0 years' ones. This is the effect of recovery and partial recrystallization, which took place in the samples. It can be also noted that GAM and KAM parameters for the '5 years'

Fig. 5 Distributions of grain size (number fraction) for the samples after 4 and 8 ECAP passes, directly after deformation ('0 years')—**a** and **b**, and five years later ('5 years')—**c** and **d**. Results based on $\Delta\theta = 15^\circ$ grain boundary misorientation are shown

Table 1 Average grain sizes of the processed samples (μm)

	Initial material	0 years	5 years	Recrystallized
4 ECAP passes	17	1.51	1.21	11.12
8 ECAP passes		0.95	0.88	8.40

samples are approximately at the same levels as for the initial material; this means that '5 years' samples are already highly recovered.

It should be also noted that GOS values for the deformed and recovered samples ('0 years' and '5 years')

Fig. 6 Grain orientation spread (GOS), grain average misorientation (GAM) and kernel average misorientation (KAM) for the initial material and for the '0 years' and '5 years' samples after: **a** 4 ECAP passes and **b** 8 ECAP passes. KAM and GAM were calculated from misorientations up to 15°

are lower than for the initial material. This effect can be explained by smaller grain size in these samples as compared with the initial material (for the initial material the average grain area was around 17 μm but for the samples after 4 and 8 passes it was around 1 μm). Smaller grains are necessarily less heterogeneous than the coarse grains.

Another interesting data, which can be obtained from EBSD maps, are the dislocation density and the specific stored energy—cf. [22–25]. Using KAM value (calculated for misorientations up to 5°) one can estimate the average density of dislocations forming low angle boundaries in the material [23]:

$$\rho \approx \frac{\alpha\theta}{bd} \quad (1)$$

where KAM value (up to 5°) is substituted for θ , b is the value of Burgers vector (equal to 0.286 nm for aluminum), d is the step size in EBSD measurement and α is equal to 2 or 4 for boundaries of tilt or twist type character (it was put $\alpha=3$ for boundaries of mixed character in the present calculations). The dislocation density was calculated for the sample after 8 ECAP passes (with the following data: θ equal to 1.274° for ‘0 years’ sample, θ equal to 0.905° for ‘5 years’ sample, $d=0.2 \mu\text{m}$) and was compared with that for the initial material. The following values of the dislocation density were obtained: $1.17 \times 10^{15} \text{ 1/m}^2$ for the ‘0 years’ sample, $8.29 \times 10^{14} \text{ 1/m}^2$ for the ‘5 years’ sample, 1.39×10^{14} for the fully recrystallized sample and $3.81 \times 10^{14} \text{ 1/m}^2$ for the initial material.

Next, the stored energy per unit volume was calculated by the formula [22, 23]:

$$E = \frac{1}{2} \rho G b^2 \approx \frac{\alpha \theta G b}{2d} \quad (2)$$

where G is the shear modulus (equal to 26.1 GPa for aluminum).

The following values of the specific stored energy, E , were obtained: 4.07 J/mole for the initial material, 12.45 J/mole for ‘0 years’ sample, 8.85 J/mole for ‘5 years’ sample and 1.48 J/mole for the fully recrystallized material. These results are presented in Fig. 7 (the values of estimated dislocation density, ρ , are marked in parentheses). It can be noted that the dislocation density and stored energy (ρ and E) are three times higher in the deformed sample than in the initial material. Next, an important decrease of these quantities, of around 30%, is observed after 5 years. Therefore, the recovery process strongly reduced the dislocation density and, consequently, the stored energy. The above quantities are still lower for the fully recrystallized material—Fig. 7. The values of the stored energy for the ‘5 years’ sample are between these for the ‘0 years’ and for the recrystallized sample. It should be noted that also

Fig. 7 Stored elastic energy per mole for the initial material, for the deformed samples after 8 ECAP passes (‘0 years’, ‘5 years’) and for the sample after full recrystallization. The values of corresponding dislocation densities (expressed in $1/\text{m}^2$) are given in parentheses

the residual stress state (see e.g., [26, 27]) would give an additional insight into the examined problem and such the study is planned in the future.

The observed reduction of the dislocation density (and correspondingly of the stored energy) after 5 years is caused by rearrangement of dislocations, which leads to the formation of subgrains. Also, dislocation movements, facilitated by cross-slip (frequent in metals with high stacking fault energy, which is a case of aluminum), lead to the annihilation of dislocations of opposite Burger vector and also of point defects of opposite sign. On the other hand, selected material regions with lowered density of defects become the nuclei for new grains and the first order recrystallization starts. This mechanism also reduces the dislocation density.

3.4 Distributions of IQ Factor

The image quality (IQ) factor is another important parameter in the analysis of EBSD data.

It has high values in sample regions where crystal lattice is well defined (small amount of defects) and therefore diffraction lines are strong. The lowering of IQ factor is caused by pile-ups of dislocation and point defects, as well as by concentration of grain and subgrain boundaries. Consequently, IQ is low in deformed sample regions and high in annealed ones. It was shown that the distribution of IQ factor can be used to evaluate the recrystallized fraction of the material. The detailed scheme of this type of estimation is presented in [28].

The distributions of IQ factor for the initial material, for the deformed samples (‘0 years’), for the samples after 5 years recovery at ambient temperature (‘5 years’) as well as for the fully recrystallized ones are presented in Fig. 8. The distributions of IQ factor for recrystallized samples are strongly shifted to higher values as compared with the

Fig. 8 Distributions of normalized IQ factor for the samples after: **a** 4 ECAP passes and **b** 8 ECAP passes. Results for ‘0 years’, ‘5 years’ and for fully recrystallized (after ECAP processes) samples are presented. The distribution for the initial material is shown in **a**

Fig. 9 Determined minimal and maximal recrystallized volume fractions after 5 years for the samples after **a** 4 ECAP passes and **b** 8 ECAP passes

deformed samples (‘0 years’) both for the samples after 4 and 8 ECAP passes. In contrast, the IQ distributions for the ‘5 years’ samples are in intermediate positions. The departure of ‘5 years’ distribution from ‘0 years’ one is more marked for the sample after 4 ECAP passes than for the sample after 8 passes. It can be also noted that the maxima of IQ factor for the initial and fully recrystallized materials are approximately at the same position (Fig. 8). Basing on the algorithm presented in [28] one can evaluate the minimal and maximal recrystallized volume fractions for each sample. The results for the ‘5 years’ samples after 4 and 8 ECAP passes are presented in Fig. 9. The scheme of this evaluation is shortly presented in “Appendix”. The following values were obtained: minimal and maximal recrystallized volumes for the sample after 4 passes are 30.7% and 63.5%, and for the sample after 8 ECAP passes they are: 14.8% and 48.7%, correspondingly. Therefore, it is evident that the recovery and recrystallization processes at ambient temperature were more advanced in the sample after 4 ECAP passes.

3.5 Misorientations Between Grains and Boundary Types

Distributions of misorientations for defined grains were calculated for ‘0 years’ and ‘5 years’ samples after 4 and 8 ECAP passes—Fig. 10a, c. Misorientation distributions were also calculated for fully recrystallized samples after 4 and 8 ECAP passes as well as for the initial material—Fig. 10b, d.

When comparing misorientation distributions of ‘0 years’ and ‘5 years’ samples, a shift of misorientations is observed in the case of material after 4 ECAP passes—Fig. 10a. More misorientations between 20° and 45° appeared after 5 years at the expense of the misorientations between 50° and 63° . In contrast, the misorientation distributions for the analogous samples after 8 ECAP passes changed only very slightly—Fig. 10c. However, it should be underlined that misorientation distributions for the deformed samples are strongly different from that of the initial material—Fig. 10b. What concerns the distributions for fully recrystallized samples they are similar for the samples after 4 and 8 ECAP passes (cf. Fig. 10b, d) and are close to the random distribution (Mackenzie plot).

It can be therefore concluded that misorientation modification after 5 years is more visible in the sample after 4 ECAP passes. This confirms the result obtained on the base of IQ factor, that more intense recovery process occurred in this sample.

Another important characteristics are types of grain boundaries and their integrated shares. The boundaries were divided into two categories: low angle boundaries with misorientations between 2° and 15° and high angle boundaries with misorientations above 15° .

The first observation is that for the 8 passes sample (both for ‘0 years’ and ‘5 years’) the shares of low angle boundaries are smaller than for the 4 passes sample, and correspondingly the shares of high angle boundaries are higher—Fig. 11. Moreover, in both cases of deformation the shares of low angle boundaries decreased after 5 years and the shares of high angle ones increased. This shows that new grains

Fig. 10 Distributions of misorientations between defined grains (boundaries determined on the base of $\Delta\theta = 15^\circ$): **a** for ‘0 years’ and ‘5 years’ samples after 4 ECAP passes, **b** for fully recrystallized sample after 4 ECAP passes and for the initial material, **c** for ‘0 years’ and ‘5 years’ samples after 8 ECAP passes, **d** for fully recrystallized sample after 8 ECAP passes and for random orientation distribution (Mackenzie plot)

Fig. 11 Shares of low ($2^\circ \leq \theta < 15^\circ$) and high angle ($\theta > 15^\circ$) boundaries for ‘0 years’ and ‘5 years’ samples after: **a** 4 ECAP passes, **b** 8 ECAP passes

formed during recovery process have preferentially higher mutual misorientations than grains in the deformed samples.

3.6 Deformation and Recrystallization Textures

Textures of the deformed sampled (‘0 years’ and ‘5 years’) as well as those of fully recrystallized ones were determined using data from the EBSD orientation maps. The

Fig. 12 ODF sections at $\varphi_2 = 45^\circ$ for the initial material, for the samples after 4 ECAP passes ('0 years' and '5 years') and for the fully recrystallized sample after 4 passes. In the bottom part the ideal orientations observed for f.c.c. metals after ECAP deformation are shown (cf. Table 2)

Fig. 13 ODF sections at $\varphi_2 = 45^\circ$ for the initial material, for the samples after 8 ECAP passes ('0 years' and '5 years') and for the fully recrystallized sample after 8 passes

$\varphi_2 = 45^\circ$ sections of the orientation distribution functions (ODF) for the initial material and for the samples after 4 ECAP and 8 passes are shown in Figs. 12 and 13, respectively. The ideal orientations, typical for f.c.c. metals after ECAP processing, are presented in the bottom part of Fig. 12 and they are listed in Table 2.

Texture of '0 years' sample after 4 ECAP passes is composed of B , $\bar{B}$, A and $\bar{A}$ orientations—Fig. 12. In the '5 years' texture two new components appear: C and A_2^* , which are also present in the fully recrystallized texture—Fig. 12.

Generally, it is visible that the '5 years' texture of the sample after 4 ECAP passes converges towards that of fully recrystallized material.

Texture of '0 years' sample after 8 ECAP passes contains the same ideal orientations as the corresponding texture after 4 passes—compare Figs. 12 and 13. However, in contrast to the previous case, the '5 years' texture of the sample after 8 ECAP passes is only slightly modified and its convergence towards the texture of fully recrystallized sample is rather weak.

Table 2 Typical ideal orientations observed for f.c.c. metals after ECAP deformation

Component	φ_1	Φ	φ_2	Miller indices
A_1^*	80.26/260.26	45	0	$\langle 011 \rangle \{5 - 21 21\}$
	170.26/350.26	90	45	
A_2^*	9.74/189.74	45	0	$\langle 011 \rangle \{25 - 3 3\}$
	99.74/279.74	90	45	
A	45	35.26	45	$\langle 112 \rangle \{2 - 20 9\}$
$\bar{A}$	225	35.26	45	$\langle 112 \rangle \{-2 20 - 9\}$
B	45/165/285	54.74	45	$\langle 111 \rangle \{4 - 15 11\}$
$\bar{B}$	105/225/345	54.74	45	$\langle 111 \rangle \{-11 - 4 15\}$
C	45/225	90	45	$\langle 011 \rangle \{-17 - 12 12\}$
	135/315	45	0	

Euler angles and Milller indices are marked [8]

3.7 Hardness Measurements

The results of hardness measurements are presented in Table 3. The values are listed for the initial material, for the deformed samples (0 and 5 years samples) and for the recrystallized final samples.

It is visible that, within experimental uncertainty, the hardness of the Al 1050 samples did not change after 5 years of annealing at ambient temperature. The similar effect for high purity aluminum deformed by HPT (10 turns at 2 GPa) and stored during 2938 days was reported in [29] (even a small increase of the hardness was recorded: from 32 to 34).

An constant hardness, observed in aluminum after 0 and 5 years, can be explained by two opposite tendencies:

- the decrease of the defects density (and of the stored energy) favors the softening,
- the observed decrease of the average grain size favors the material hardening (accordingly to Petch–Hall law).

The two above trends act in opposite directions and as a result a material softening is not observed. This result is important in view of possible applications of SPD 1050 aluminum parts in mechanical devices: it means that their mechanical hardness is stable.

Table 3 Vickers hardness of samples at different stages of processing

	Initial material	0 years after ECAP	5 years after ECAP	5 years after ECAP and -recrystallized
4 passes	25.2 ± 0.3	45.2 ± 2.8	44.1 ± 0.5	22.7 ± 0.3
8 passes		44.3 ± 2.5	43.4 ± 1.1	21.8 ± 0.4

4 Conclusions

The microstructure parameters of 1050 aluminum determined directly after ECAP processing and 5 years later were analyzed and compared with fully recrystallized samples. The following conclusions can be done:

- A considerable decrease of the average grain size is observed for the ‘5 years’ samples as compared with ‘0 years’ ones for the materials deformed by 4 and 8 ECAP passes. This is also visible in the distributions of grain size, where the disappearance of large grains is observed. This effect is caused by the formation of new small recrystallized grains during slow recovery process occurring at the ambient temperature.
 - Intra-granular refinement and fragmentation is reduced after 5 years both in 4 and 8 ECAP passes samples. This is confirmed by the decrease of GOS and GAM parameters.
 - Dislocation density and stored energy connected with low angle boundaries (misorientations up to 5°) are markedly reduced after 5 years at the ambient temperature.
 - Distributions of IQ factor showed that higher volume fraction of recrystallized material was observed after 5 years in the sample after 4 ECAP passes as compared with this after 8 passes.
 - The shares of low and high angle grain boundaries change in a similar way for the 4 and 8 ECAP samples: the share of low angle ($2^\circ \leq \theta < 15^\circ$) boundaries decreased after 5 years and oppositely, the share of high angle ($\theta > 15^\circ$) boundaries increased.
- Distribution of misorientations between defined grains after 5 years recovery is markedly modified in the case 4 ECAP passes sample: a selected shift towards lower misorientations is observed. The misorientation distributions for the fully recrystallized samples are close to that of a material with random orientation distribution (MacKenzie plot).
- Characteristic strong texture components are observed for the samples after 4 and 8 ECAP passes. Texture of the sample after 4 ECAP, measured 5 years after deformation, tends towards that of the fully recrystallized samples. This effect is much weaker in the sample after 8 ECAP passes.
 - Hardness did not decrease after 4 years of annealing at ambient temperature.

Fig. 14 Characteristic areas in the IQ distributions for the samples after: **a** 4 ECAP passes and **b** 8 ECAP passes

A general conclusion is that more intense recovery and partial recrystallization was observed after 5 years in the sample after 4 ECAP passes than in that after 8 ECAP passes, in spite of the fact that 4 ECAP sample accumulated a smaller amount of deformation. This result can be explained by some amount of dynamic recrystallization, which occurred between the 5-th and the 8-th ECAP pass. What is interesting the hardness did not change (within experimental uncertainty) after 5 years, which was explained by two opposite microstructural mechanisms.

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Appendix

The calculation of recrystallized fractions was based on the algorithm proposed in [28].

The volume fractions of the recrystallized material were evaluated basing on the normalized distributions of IQ factors (IQ values were normalized to the range [0.0–1.0]). Also the area under each distribution was normalized to the value of 1.0.

The normalized distributions for ‘0 years’, ‘5 years’ and fully recrystallized (FR) samples are shown in Fig. 14. Two characteristic areas in these distributions are marked: S1 and S2. The surface S1 + S1 is the common area of distributions for ‘5 years’ and for FR samples, hence it represents the maximal value of the recrystallized volume fraction. On the other hand, S2 is the common area of distributions for ‘5 years’ and FR samples but with subtraction of that common for the FR and ‘0 years’ samples; therefore it represents the minimal value of the recrystallized volume fraction.

Accordingly, the recrystallized fractions were calculated as follows:

For the ‘5 years’ sample after 4 ECAP passes:

Minimal recrystallized volume fraction: $S2 = 30.7\%$

Maximal recrystallized volume fraction: $S2 + S1 = 63.5\%$.

For the ‘5 years’ sample after 8 ECAP passes:

Minimal recrystallized volume fraction: $S2 = 14.8\%$

Maximal recrystallized volume fraction: $S2 + S1 = 48.7\%$.

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