MICROSTRUCTURE EVOLUTION IN PLASTIC DEFORMATION OF Fe - Si SINGLE CRYSTALS BY COMPRESSION AT ROOM TEMPERATURE

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ABSTRACT

In order to find out interrelations between the mechanical behaviour and the microstructure evolution of metallic materials at larger strains, measurements of substructure parameters as the mean total dislocation density, the density of excess dislocations of one sign, mean lattice misorientations etc. are necessary. This can be done, for example, by analysis of the broadening of both the radial intensity distribution (ϑ -2ϑ scans) and the rocking curves (ω - ϑ scans) of X-ray diffraction peaks and, in the case of the misorientations, also by evaluation of electron – backscattering patterns (EBSP). In the present paper the procedure is illustrated for [001] – oriented Fe–Si single crystals, which were compressed at room temperature. The results of the integrated substructure analysis are discussed in connection with TEM observation of the disclocation structure of the crystals.

KEYWORDS

Fe-Si, diffraction–peak broadening, dislocation density, EBSD, misorientation, single crystal, substructure, X – ray analysis

INTRODUCTION

Plastic deformation of metals to large strains is characterised by dislocation patterning, i.e. the development of complex mesoscale substructures (e.g. cell block structures containing dense dislocation walls and microbands, deformation bands, disclinations), which cannot be described in a simple manner in terms of individual dislocations. A common feature of such substructures is the formation of significant local lattice rotations. In the case of cold working of f.c.c. this phenomenon can be explained by means of a model of substructure formation based on dislocation – disclination dynamics [1,2]. The predictions of this model are in satisfactorily agreement with experimental investigations. In particular, the existence of numerous disclinations can doubtless be illustrated by TEM for all important metallic structures [3-5].

In order to find out interrelations between the microstructure evolution and the mechanical behaviour of the materials, measurements of substructure parameters as the mean total dislocation density, the density of excess dislocations of one sign, mean lattice misorientations etc. are necessary. At lower strains this may be done directly by means of TEM, but in the case of complex defect structures the analysis of X–ray diffraction peak broadening (e.g. [6-10]) seems more promising, and especially for the determination of misorientations the EBSD technique can be used, too. This paper presents experimental data concerning the evolution of the dislocation-induced substructure of Fe-Si single crystals with

[001] orientation during plastic deformation by compression at RT, which were mainly obtained by means of high – resolution X – ray diffractometry and EBSD.

EXPERIMENTAL INVESTIGATIONS

Sample material and techniques of investigation

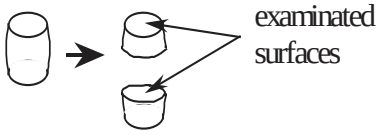

Fig.1: Sample preparation

The sample material of the present work were [001] – oriented cylindrical Fe-Si single crystals containing 3,05 at.%., which were plastically deformed at room temperature in compression up to strains ε = 0.09, 0.22, 0.45, and 0.50 at a strain rate $\dot{\varepsilon} = 10^{-3}$. After the deformation the crystals were cut electroerosively in the central part perpendicular to the compression axis (Fig. 1) and the surfaces were ground with a 4000 - grit paper. The remaining deformation zone was removed electrolytically. Because the deformation becomes locally inhomogeneously already at low strains, for each specimen the microstructure was investigated at the outer surface as well as at the surface related to the crystal centre (Fig. 1).

Integrated analysis of the defect structure of the deformed crystals was based on high-resolution XRD, measuring the peak – broadening of the reflection 002 with CuKα_1 – radiation by means of a double crystal spectrometer (DCS) equipped with a flat perfect Ge (111) crystal as monochromator. In every case the radial intensity distribution I(2ϑ) and azimuthal intensity distributions (rocking curves) $I_\varphi(\omega)$ related to different directions within the plane perpendicular to the normal direction of $\mathbf{n}_h$ of the reflecting lattice planes (002) were recorded.

In order to complete and to control the X-ray analysis, the deformation-induced microstructure of the single crystals was also studied directly by TEM (PHILIPS CM30; U = 300 kV) and by local orientation measurements via EPSD (SEM LEO 15 with software package CHANNEL+ [11]).

Evaluation of the XRD data

Broadening of an X-ray diffraction peak **h** = {hkl} is influenced by both the dislocation-induced lattice strains related to the mean total density and its spatial fluctuations (i.e. the arrangement of the defects) and to the lattice rotations caused by excess dislocations of one

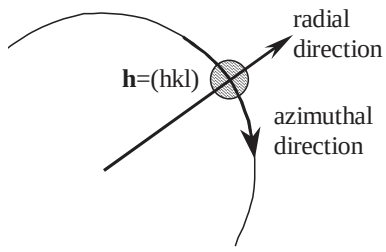

Fig. 2: Interrelation between the observable intensity distributions of an X - ray reflection and Ewald´s sphere

sign stored in dislocation walls ([12-15], for instance). This results in an anisotropy of the scattering domains around the corresponding nodes **h** = {hkl} of the reciprocal lattice, which in the case of single - crystal diffraction can be studied by measuring of radial and azimuthal intensity distributions (Fig. 2). The radial intensity distribution (RID) I(2θ) as obtained in θ-2θ scanning is an intersection of the scattering domain along the direction of **h** and reflects the effect of the lattice strains ε. Azimuthal intensity distributions (AID; rocking curves) I(ω) as

measured in ω-θ scanning and representing intersections of the scattering domain along directions perpendicular to **h** indicate essentially the effect of lattice rotations (misorientations).
The mean total dislocation density ρ_d can be determined from the inclination of a linear branch occurring in the Krivoglaz – Wilkens plot [10]

$$\Psi(ln\,L) = -\frac{ln\,A(L)}{L^2} = \frac{\pi}{2} b^2 h^2 \langle \chi_h \rangle \rho_d \; ln \frac{L_0}{L} \tag{1}$$

of the Fourier coefficients A(L) of the physical line – shape of the RID at large L. The symbols of this formula have the following meaning:

L – measuring length defined perpendicular to the reflecting lattice planes (hkl),

$\langle \chi_h \rangle$ – orientation factor depending on the orientation of the operating diffraction vector **h** with respect to the slip system of the dislocations and on the elastic constants of the scattering crystal,

$L_0 = \eta R_c$ – distance proportional to a correlation radius R_c of the strain fields of the dislocation arrangement

The correlation radius R_c is obtained from the intersection of $\Psi(ln\,L)$ with the abscissa.
Dislocation - induced line broadening of rocking curves I(ω) was treated theoretically by KRIVOGLAZ et al. [8, 12] and more recently, for instance, by BARABASH & KLIMANEK [13, 14]. From the papers follows that a well defined bell – shaped rocking curve is obtained, if the scattering volume consists of a large statistical ensemble of dislocation walls with sufficiently small misorientations of adjacent lattice cells. Otherwise the shape of the intensity distribution I(ω) is irregular and represents the sum of the rocking curves of individual cell blocks or subgrains, respectively, .
Assuming an arrangement of randomly distributed dislocation walls with the mean distance D_c, which is formed by excess dislocations of one sign with equal module of the Burgers vector b and the density $\Delta\rho_{exc}$, equal fractions of cell boundaries with opposite sign of the misorientation Θ, and statistical independence of the individual misorientations, one finds that the rocking curve $I_\varphi(\omega)$ related to a direction φ within the plane perpendicular to **h** is a Gaussian distribution with the integrated width

$$\beta_\varphi = \Delta\omega_\varphi = \sqrt{\frac{\pi}{2} \cdot \frac{T}{D_c}} \langle \psi_h \rangle h \left| \Theta_\varphi \right| = \sqrt{\frac{\pi}{2} \cdot \frac{T}{D_c}} \langle \psi_h \rangle h b \Delta\rho_{exc} \tag{2}$$

Analogously to the quantity $<\chi_h>$ in equation (1), $<\psi_h>$ is an orientation factor, and T denotes the irradiated area on the sample surface.

Evaluation of the EBSD data
The EBSD technique ([16] gives the orientations of a grid of equidistant measuring points at the sample surface in terms of triples g = (φ_1,Φ,φ_2) of Eulerian angles defining spatial vectors $\vec{g}_k$. The orientation differences Δg between adjacent surface elements with respect to a selected direction φ within the sample surface can be calculated from them by projection of the spatial vectors $\vec{g}_k$ and $\vec{g}_{k+1}$ onto this direction (Haberjahn et al. [17]). In order to compare the results of EBSD and XRD, the direction of the projection was chosen equal to that used in the measurement of the rocking curves.

Applying a step width Δx = 0.5 μm, the orientations g of 300×50 measuring points were determined and used for the calculation of the mean misorientation, the frequency distribution $f_{\Delta x} = f_{\Delta x}(|\Delta g|)$, the mean misorientation $<|\Theta|>$, and the mean chord length ℓ of uniformly oriented lattice regions. It can be assumed that such areas are related to the dislocation cells or subgrains. However, in this connection an uncertainty $|\Delta g_{exp}| \leq 0{,}2°$ due to the limitations of the orientation determination must be taken into account. Accordingly, all orintation differences < 0,2° were eliminated.

EXPERIMENTAL RESULTS AND DISCUSSION

An overview of the dislocation–induced substructure of a Fe-Si single crystal after compression to ε = 0.45 is given in Fig. 3. It can be interpreted in terms of an arrangement of incidental dislocation-cell boundaries with small random misorientations and a cell - block structure consisting of geometrical necessary boundaries as carriers of significant misorientations. Such geometrically necessary boundaries appear as dense dislocation walls and microbands.

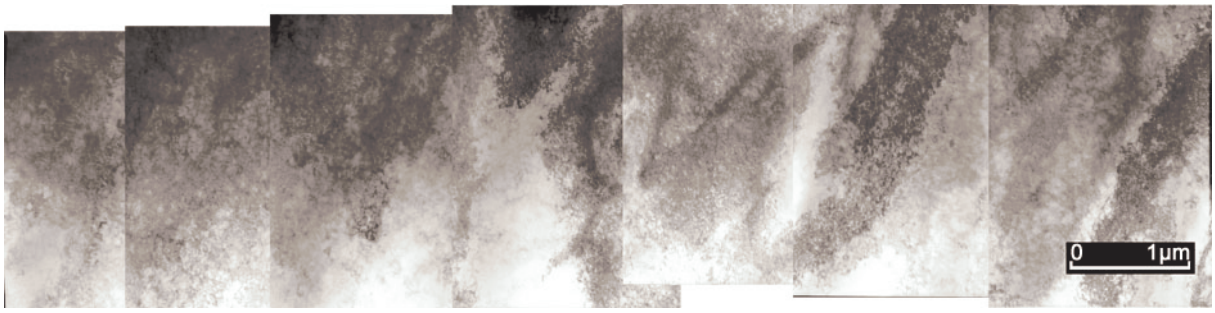

Fig. 3. The microstructure of a Fe-Si single crystal after compression to ε = 0.45 (centre)

The results of the X – ray analysis of the peak broadening are summarised in Fig.4.

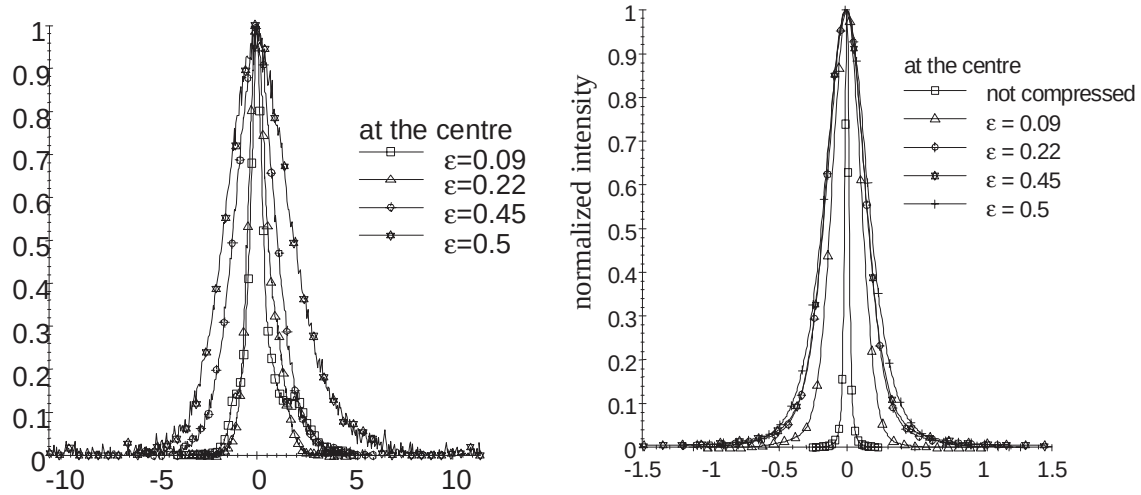

Fig. 4: Rocking curves I(ω) (left) and radial intensity distributions I(2Θ) (right) of the reflection 002 of the Fe – Si single crystals at different strains (sample centre)

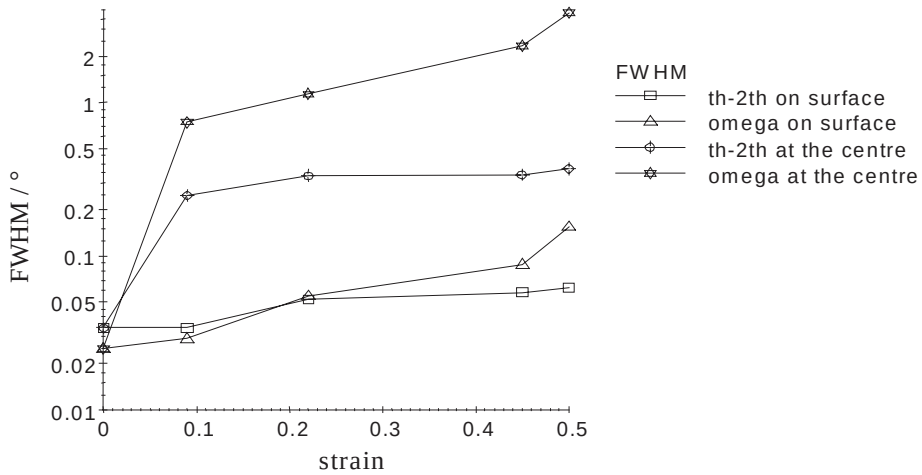

Fig. 5. Line widths (FWHM) of radial intensity distributions I(2Θ) and the rocking curves I(ω) of the reflections 002 measured at the outer surface and at the centre of the compressed Fe-Si single crystals

Fig. 5 shows in logarithmic scale the line widths (FWHM) of the θ - 2θ and the ω - scans, which were measured at the outer surface (Fig 1) and in the centre of crystals. Corresponding to the development of the deformation process in compression tests, the course of the curves indicates that the evolution of the misorientations and, consequently, of the dislocation arrangement must be quite different in both regions of the samples.

The radial intensity distributions I(2θ) could well be fitted by PEARSON VII functions. The mean total dislocation densities as calculated from their Fourier coefficients are presented in Fig. 6. In the calculations it was assumed that crystals contain 80% screw dislocations and 20% edge dislocations.

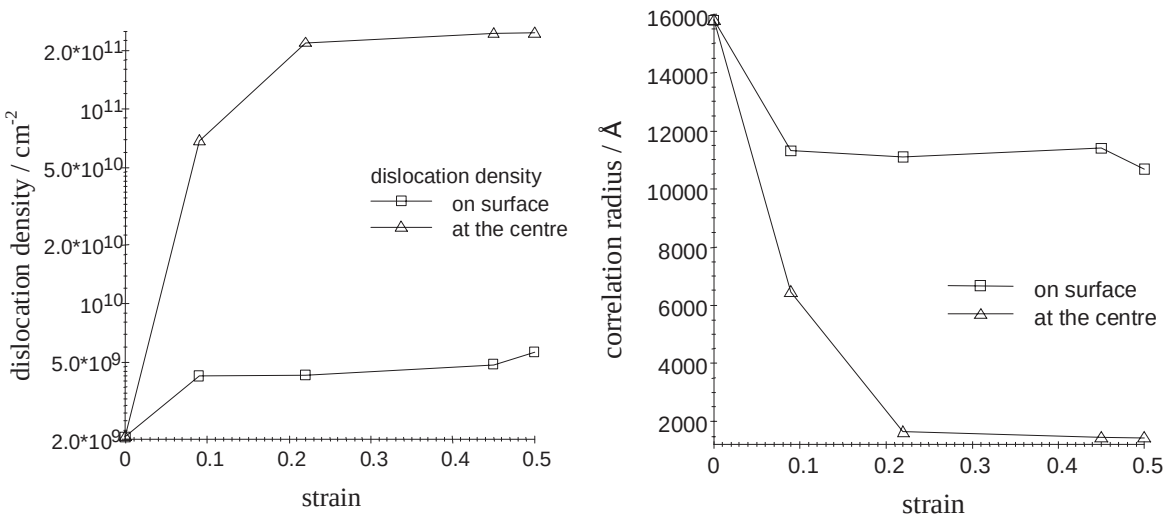

Fig. 6. Mean total dislocation densities (left, in logarithmic scale) and correlation radius (right) measured at the centre and at the outer surface of the Fe-Si single crystals.

In the central part of the compressed sample the defect content is by about a factor 50 higher than at the outer surface and already at the strain ε = 0.22 a dislocation density is estimated, which was estimated in cold-rolled Cu single crystals after 90% thickness reduction [15] Corresponding to the changes of the dislocation density and to the small changes of the line widths of the rocking curves, the decrease of the correlation radius of the dislocation

distribution is only small at the outer crystal surface. In contrast, in the central part it decreases by nearly one order of magnitude, indicating significant changes of the type of the dislocation arrangement.

The rocking curves I(ω) were measured with a cross section $A_0 = 0{,}09 \times 1{,}0 = 0{,}09\ mm^2$ of the primary beam, which obviously fulfills the presumptions for the application of equation (2). However, the rocking curves are asymmetric. As directly confirmed by the results of the EBSD measurements presented in Fig. 7, this indicates the existence of several peaks in the misorientation distribution $f_{\Delta x}(|\Delta g|)$ due to large subgrains within the growth structure of the single crystals. The phenomenon is visible, in particular, in the frequency distributions $f_{\Delta x}(|\Delta g|)$ measured at the outer surface of the crystals compressed up to strains ε = 0.09 and ε = 0.22. In the central part of the crystals the influence of the initial substructure on the misorientation distribution is much smaller because of the rapid formation of relatively large misorientations during the deformation process.

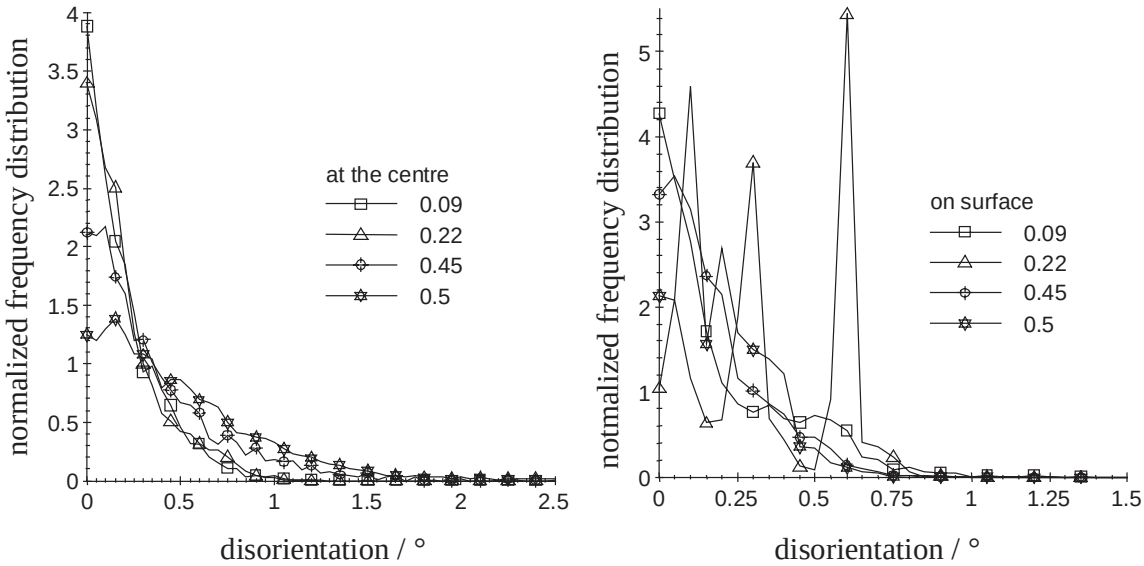

Fig. 7 Normalized frequency distribution calculated at the centre (left) and on surface (right) of crystals.

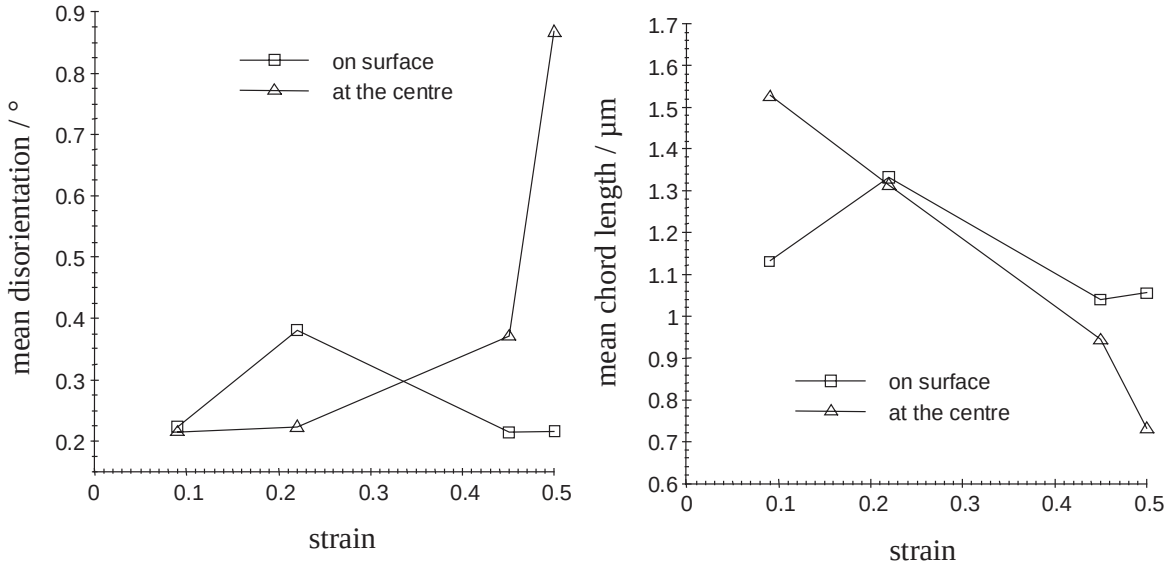

Fig. 8. Mean misorientations (a) and mean chord lengths (b) measured at the centre and at the outer surface of the deformed crystals.

The mean disorientation (fig. 8, left), which was calculated from EBSP-data, is practically constant on surface, in contrast to mean disorientation at the centre, where it alter very much. It correspond to alteration FWHM of rocking curves. The disorientation between neighbouring points was determined in volume. For comparison with rocking curves must be calculated the projection on the direction, which was measured with rocking curve. The similar situation is for mean chord length.

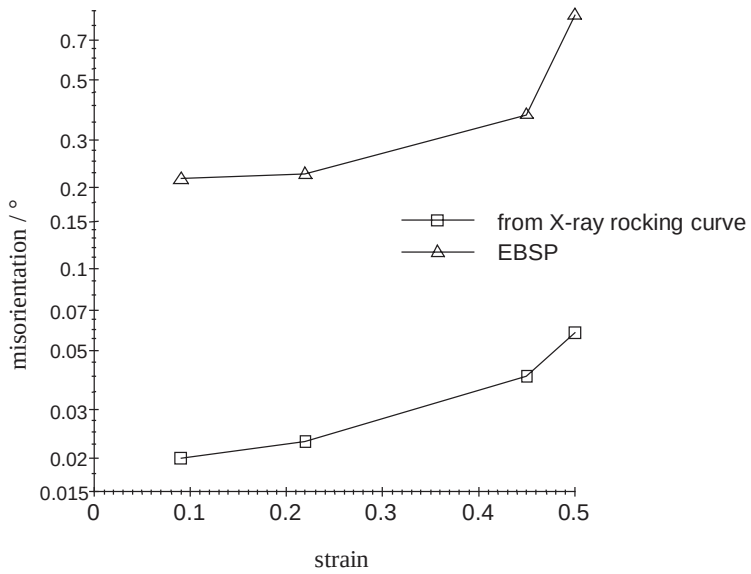

Fig. 9. Mean misorientation (in logarithmic scale) calculated from broadening of X-ray rocking curve and from EBSD - data.

In agreement with the behaviour of the line widths of the rocking curves, the mean misorientations (Fig.8) calculated from the EBSD data are practically constant at the surface and in-crease significantly at strains $\varepsilon > 0.2$. However, as already observed in investigations of cold-rolled Cu single crystals [10,15], their numerical values are higher by a factor 10 than the misorientations obtained from the rocking curves. One of the reasons may be that the meaning of the averages $<|\Theta|>_{EBSD}$ and $<|\Theta|>_{XRD}$ is different because of the different way of measuring the lattice rotations. Of course, the nearly parallel course of the curves $<|\Theta|>$ *vs.* ε is also an indication that the substructure model underlying the equation (2) is too simple. Based on a model for the evolution of misorientations during hot-working, in [10] it was shown that the correspondence between $<|\Theta|>_{EBSD}$ and $<|\Theta|>_{XRD}$ can be significantly improved by the assumption of weak correlation between the misorientations of the dislocation cell. Unfortunately, hitherto no information concerning such correlation in the substructure of cold – worked materials seems to be available.
As it should be mentioned still, an unexpected correspondence is observed in both the changes and in the order of magnitude of the correlation radius R_c estimated from the broadening of the radial intensity distribution and the mean chord lengths ℓ derived from the EPSD data. In order to interpret it correctly, considerations concerning both the theoretical background and the methodology of peak-broadening analysis are necessary.

SUMMARY AND CONCLUSIONS

The present work illustrates once more that combination of high-resolution X-ray diffractometry and local orientation analysis by EBSD can well be used for a systematic investigation of deformation-induced substructures of single crystals and (studying individual

grains) also polycrystalline materials especially at large strains. In this connection the examination of both the lattice strains and the evolution of the local lattice rotations seems indispensably. Corresponding to former work investigations of Cu [17] the results of the present work show, that the growth of the misorientations is accelerated at strains where the mean total dislocation density remains nearly constant (Fig. 6, 9) and an increasing number of disclinations can be detected in TEM images [3-5]. The experimental observations are also in correspondence with predictions of the disclination – based model of cold – working presented in [1].

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