TEM - INVESTIGATIONS OF THE CELL BLOCK STRUCTURE AND DISCLINATION CONFIGURATIONS IN PLASTICALLY DEFORMED METALS

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ABSTRACT

The microstructures of f.c.c., b.c.c., and h.c.p. metals formed by cold – rolling at RT to more then 50% thickness reduction were investigated by TEM diffraction contrast imaging and by local disorientation measurements. In this connection typical configurations of partial disclination configurations can be observed by diffraction - contrast imaging in splittings of dense dislocation walls and in nodes of cell blocks or in torn-off dense dislocation walls.

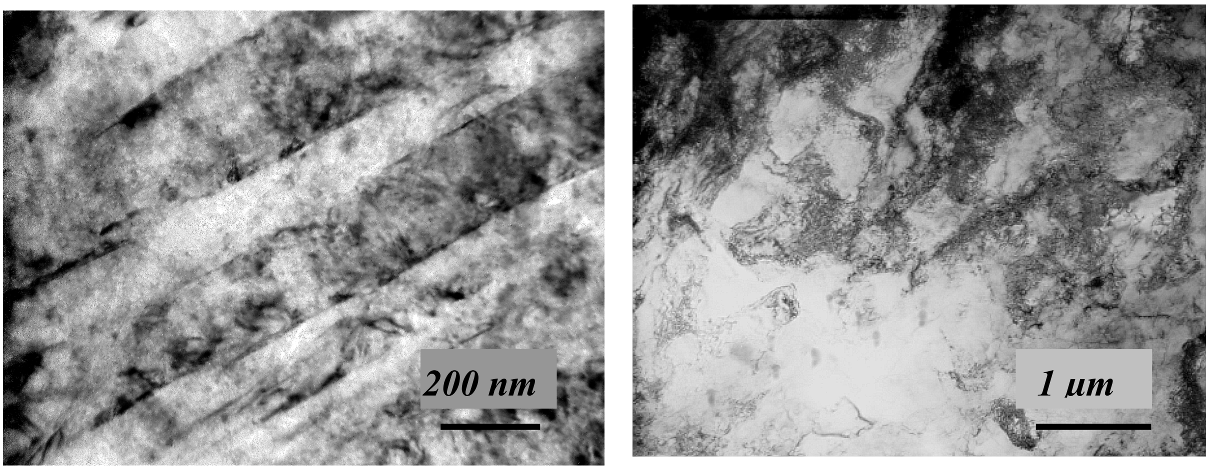
KEYWORDS

cell block structure, diffraction contrast, partial disclinations, plastic deformation, TEM

INTRODUCTION

During plastic deformation of metals inhomogeneous dislocation configurations evolve with dislocation rich boundaries separating regions with low dislocation density. These substructure development is characterised by the coexistence of two substructures on different size and disorientation scales: a cell structure and a cell block or fragment structure. The dislocation cells are only slightly disoriented. On the other hand the cell blocks are separated by dense dislocation walls with significant misorientations [1], [2].
Misorientation measurements by TEM - microdiffraction shows that there are two types of nodes within the cell block structure: (stress) compensated nodes and non-compensated ones [5], [6]. The latter are important sources of long-range distortion fields. These nodes may be described by means of disclinations [1], [3] introduced by Volterra into the theory of elasticity. The interactions among partial disclinations result in the formation of a cell block structure. While the cell structure saturates with respect to size and disorientation at higher strains, the mean cell block size continues to decrease and the mean disorientation between the blocks continues to increase. Consequently, the disclination concept may be used as a tool to describe collective phenomena in dislocation ensembles resulting in a rotational substructure on the mesoscopic scale [4].

In order to illustrate the deformation – induced microstrucure, TEM micrographs of titanium and α -iron with cell structures, cell blocks and microbands are presented in Fig. 1.



(a) (b)

Figure 1: TEM micrographs of the microstructure with microbands, cell blocks and a cell struc-ture in cold-rolled titanium (a) and iron (b)

Direct analysis of disclination arrangements is possible by TEM via diffraction - contrast imaging and the determination of local orientations or misorientations in the block structure by means of Kikuchi pattern by microdiffraction or CBED (convergent beam electron diffraction).

DESCRIPTION OF PARTIAL DISCLINATION CONFIGURATIONS

The characteristic parameters of a disclination are the line vector $\mathbf{l}_{\mathrm{Discl}}$ and the Frank vector $\mathbf{\Omega}_{\mathrm{Discl}}$. The Frank vector describes the rotation of a lattice vector left behind after a circuit around a disclination line.
In the case of non-compensated nodes of the cell-block structure the Frank vector is approximately obtained by summation of all misorientations across the cell block boundaries i meeting in the node:

$$\mathbf{\Omega}_{\mathrm{Discl}} = \sum \boldsymbol{\omega}_i \neq 0 \tag{1}$$

The type of the disclination is determined by the types of excess dislocations in dense dislocation walls and can be characterised by the angle between the Frank vector $\mathbf{\Omega}_{\mathrm{Discl}}$ and the line vector $\mathbf{l}_{\mathrm{Discl}}$ of the disclination. The line vector is tangential to the course of the line in 3D space. When the Frank vector $\mathbf{\Omega}_{\mathrm{Discl}}$ is parallel to the line vector $\mathbf{l}_{\mathrm{Discl}}$, for example, it is possible to translate a gradient in the density of excess edge dislocations on the microscopic scale into a wedge disclination on the mesoscopic scale. Moreover an inhomogeneity or a torn-off network of excess screw dislocations on the microscopic scale corresponds to a twist disclination on the mesoscopic scale with the Frank vector perpendicular to the line vector.
The orientation measurements require the generation of Kikuchi-patterns from spots beside the block boundaries in the immediate vicinity of a node [5]. The local misorientation between two regions A and B in the specimen can be obtained from the misorientation matrix $\mathbf{T}_{AB}$ describing the local specimen orientation of the crystal lattice S in the coordinate system of the microscope M:

$$\mathbf{T}_{AB} = \mathbf{T}_{MSA}^{-1} \bullet \mathbf{T}_{MSB} \tag{2}$$

with

$$\mathbf{n}_M = \mathbf{T}_{MS} \quad \mathbf{n}_S \qquad \mathbf{r}_M = \mathbf{T}_{MS} \quad \mathbf{r}_S$$
$$\mathbf{n}_M = (001) \qquad \mathbf{r}_M = [100]$$
$$\mathbf{T}_{MS} \quad \text{the orientation matrix}$$

Taking into account all local misorientations across all dense dislocation walls AB around a selected node, the resulting misorientation matrix of the node is:

$$\mathbf{T}_{\mathrm{Discl}} = \prod \mathbf{T}_{\mathrm{AB}} \neq \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \tag{3}$$

But for a self screening arrangement of several partial disclinations (index k) in a multipole, the relationship

$$\mathbf{T}_{\mathrm{Multipol}} = \prod \mathbf{T}_{\mathrm{Discl\text{-}k}} \approx \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \tag{4}$$

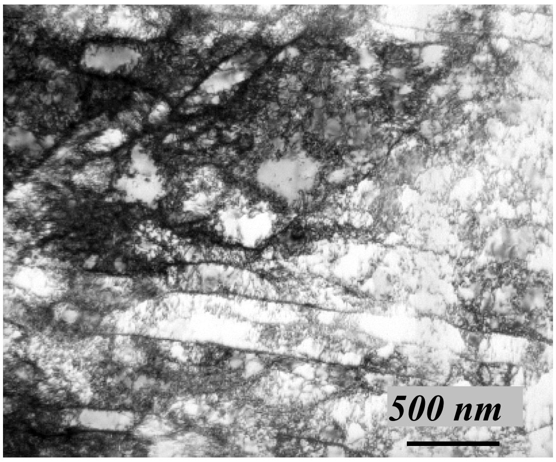
should be valid. Application of the equations (1) to (4) is illustrated in Fig. 3 and outlined in detail in [6].

DISCLINATIONS IN DEFORMED METALS

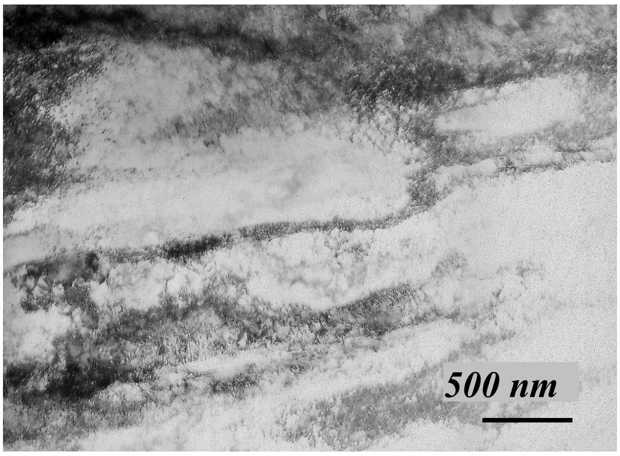
The sample materials of the present work were copper single and polycrystals, iron and iron-silicon single crystals, and titanium polycrystals deformed at RT by cold –rolling to more than 50% thickness reduction. Their microstructures were investigated with an analytical TEM Philips CM30 (acceleration voltage: 300 kV) equipped with a LaB_6 - cathode and a twin-objective lens. In the microdiffraction mode this equipment allows the registration of Kossel Moellenstedt patterns of sufficient intensity with a convergence angle smaller then 5 mrad and a spatial resolution much smaller than 20 nm ([5], [6]).

The TEM-foils represent only a two-dimensional cut of the non-uniform microstructure of a highly deformed material. To get a 3D impression of the defect arrangement it is useful to prepare the TEM-specimen in two perpendicular directions. As an example the Figures 2 (a) and (b) demonstrate this for the case of the preferred orientation of microbands of Cu in low indexed slip planes.

The long-range distortion fields of partial disclinations we can be observed directly in the splittings of dense dislocation walls within the microbands (Figure 3), at spiky nodes of the cell blocks (Fig. 5) and at torn-off dense dislocation walls (Fig. 6).



(a)



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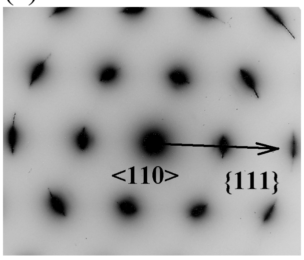
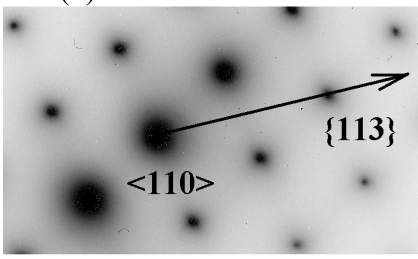
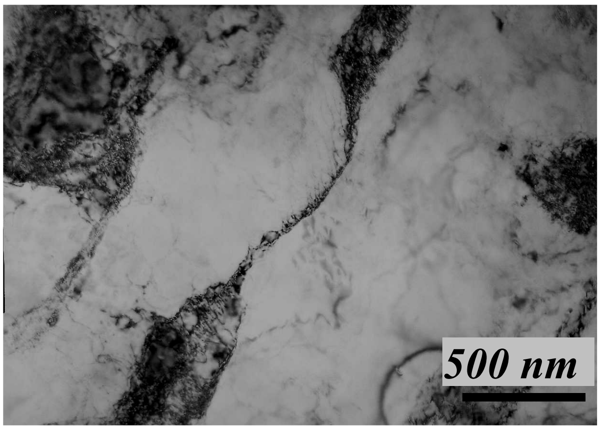


(c)



(d)

Figure 2: TEM micrographs (a) and (b) of the microstructure with microbands, cell blocks and cell structure in two perpendicular directions of a copper single crystal rolled down to 50 % thickness reduction at room temperature and the corresponding selected area diffraction pattern (c) and (d)



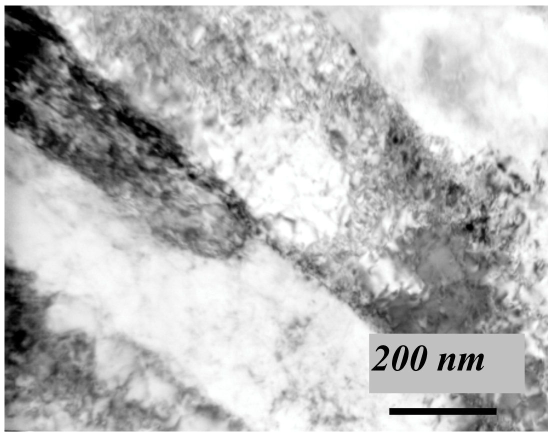
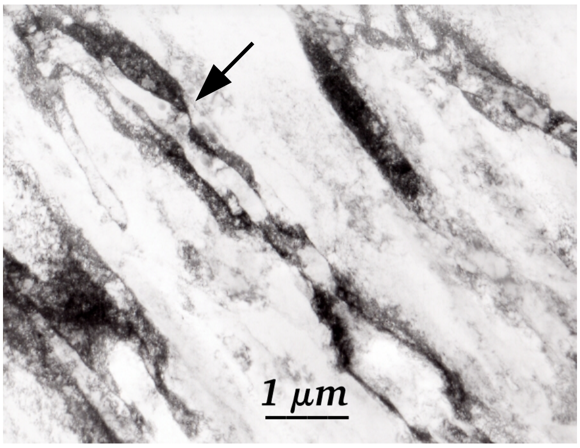
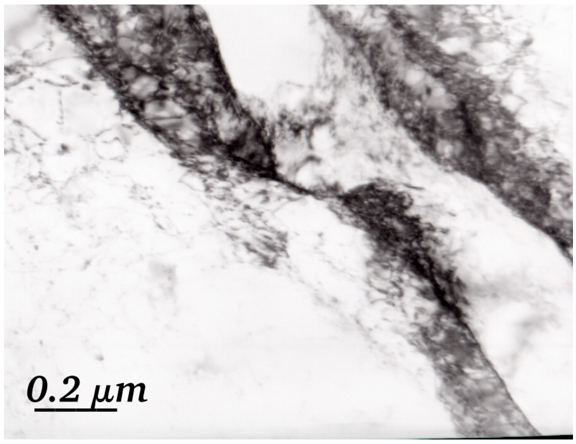
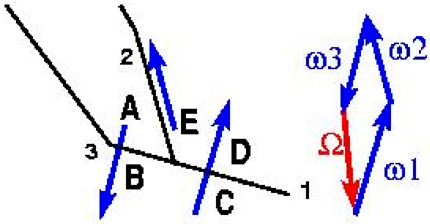


Figure 3: Splittings of dense dislocation walls with long range distortion fields as partial disclinations in microbands of iron rolled down to 89 % (a) and copper rolled down to 70 % thickness reduction at room temperature (b)



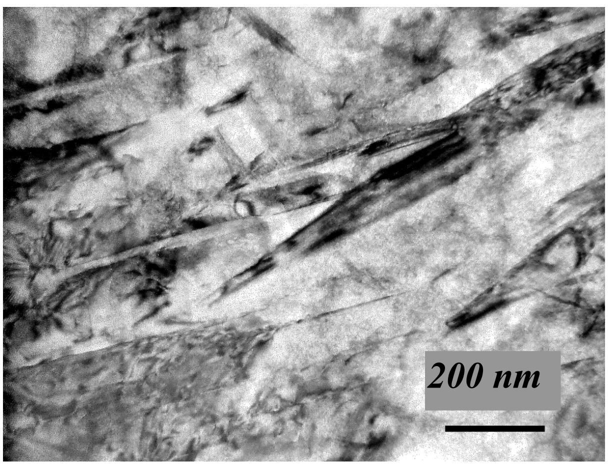


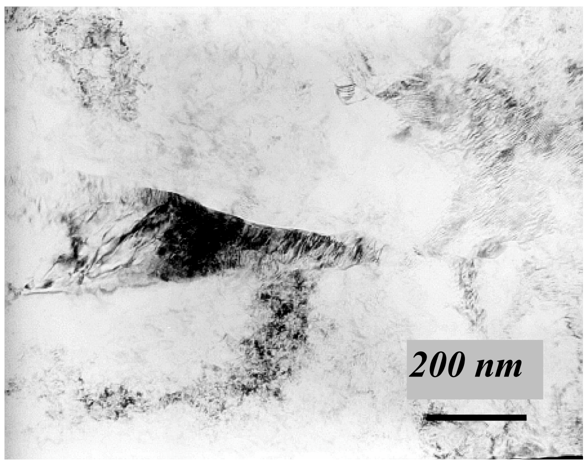
(a) (b)



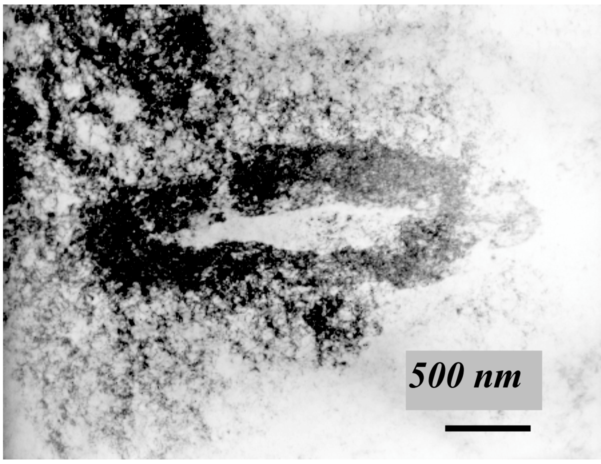
(c)

Figure 4: TEM micrographs (a) and (b) of a linear arrangement of partial disclination dipoles and quadrupoles in a microband of a copper single crystal rolled down to 70 % thickness reduction at room temperature and a schematic illustration (c) for the description of a non-compensated node as partial disclination with equation (1) with a Frank vector $\mathbf{\Omega}_{\mathrm{Discl1}}$ = (0.0024 0.0024 -0.0024) and an orientation misfit $(180°/\pi)\omega_{\mathrm{Discl1}} = 0.85°$





(a) (b)



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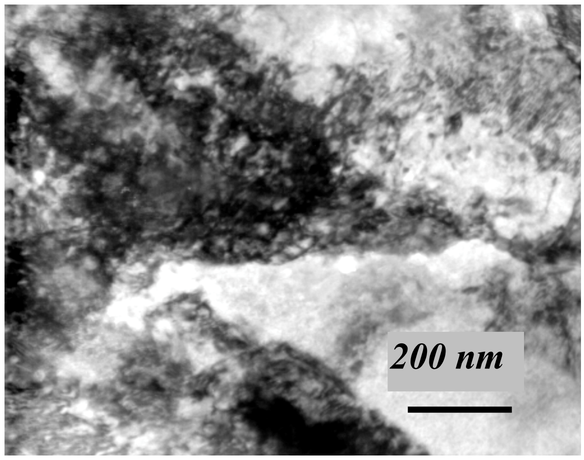
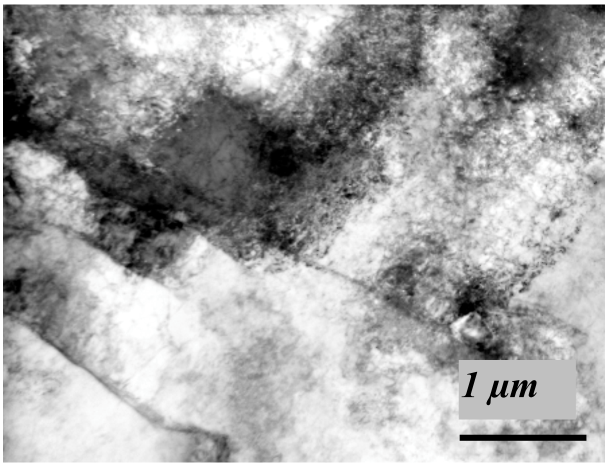
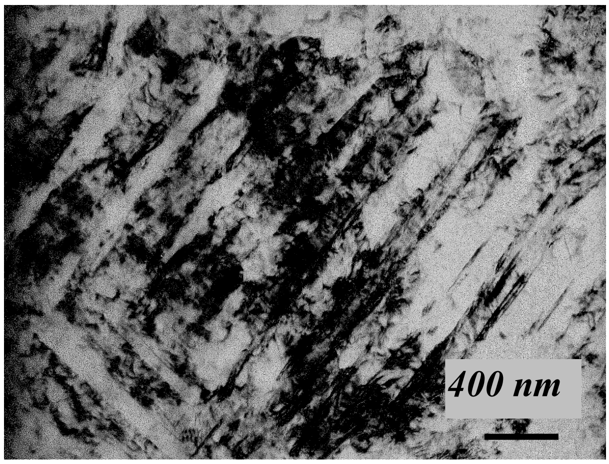


(d)

Figure 5: TEM micrograph of spiky nodes of cell blocks with long range distortion fields as partial disclinations in titanium (a), (b) iron-silicon (c) and copper single crystal rolled down to 70 % thickness reduction at room temperature



(a)



(b)

Figure 6: TEM micrograph of a linear arrangement of torn off dense dislocation walls as partial disclination multipoles in iron and titanium rolled down to more than 70 percent thickness reduction

The character of the disclination could not yet be determined in detail because the micrograph only gives the intersection point between the line vector $\mathbf{l}_{\mathrm{Discl}}$ and the thinned foil. But the solution of this problem should be possible, in principle, by simulating the diffraction contrast around the node and by comparing the result with (sensitive) changes of the image contrast in dependence to the local deviation vector **s** from the exact Bragg position. A prudent tilt of the specimen modifies the dislocation contrast and in the vicinity of a disclination the position of the bending fringes around the nodes are significantly changed (Fig. 7).

The TEM micrograph of Fig. 8 shows a disclination configuration with a torn-off dense dislocation wall in an extremely thin region of the TEM foil. Therefore a network of dislocations without superposition of the dislocation lines in the adjacent regions of the disclination becomes visible. Due to the strong bending of the foil in such a thin region it is difficult to measure local misorientations with sufficient accuracy. Nevertheless, the diffraction contrast indicates a misfit in the misorientation along the dense dislocation wall which indicates fluctuations of the dislocation density along the dense dislocation wall. A detailed characterisation of magnetic specimen requires the careful correction of the objective astigmatism and the alignment of the rotation centre of the objective lens after each change in the specimen position and in the specimen tilt. For the evidence of dislocation density fluctuations along a dislocations wall it is necessary to use misorientation measurements with a very small diameter of the electron probe. This could be done by a TEM with a field emission gun.

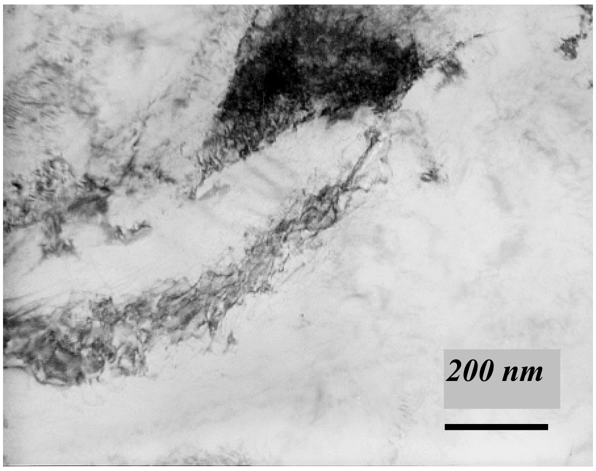


Figure 7: TEM micrograph of deformed titanium with non-compensated cell block nodes and bending fringes due to the long range distortion fields

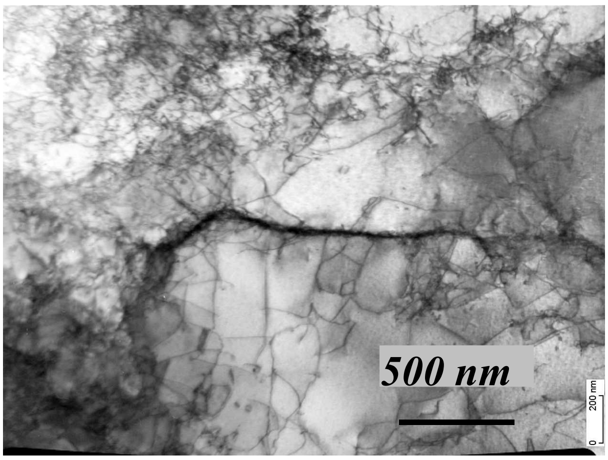


Figure 8: TEM micrograph of a dense dislocation wall in an iron single crystal rolled down to 92 percent thickness reduction at room temperature

In the examples given here, the misfit of the misorientation is in the range of a cup of degree. The measurement of smaller misorientation misfits in regions of a nearly regular cellblock structure is another still unsettled problem (see Fig. 1(b)). To solve it, on the one hand computer simulation of the diffraction contrast in the TEM micrographs is necessary, and on the other hand the distance of the electron probe from the selected node for the misorientation measurements has to be reduced. This can be done by a TEM with a field emission gun, too.

CONCLUSIONS AND OUTLOOK

The presented TEM diffraction contrast images illustrate that self - screening disclination arrangements with misorientation misfit due to long range distortion fields play obviously an important role in the microstructure evolution at larger strains. This confirms recent ideas for the interpretation of substructure evolution in severely deformed metals in terms of dislocation -disclination dynamics [4]. Moreover, new aspects of substructure characterisation as, for instan-ce, the determination of disclination densities and integrated measurement of mean lattice mis-orientations related to disclinations become important.

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