Local texture and microstructure in cube oriented Nickel single crystal deformed by equal channel angular extrusion

Journal:	<i>Philosophical Magazine & Philosophical Magazine Letters</i>
Manuscript ID:	TPHM-10-Apr-0145.R1
Journal Selection:	Philosophical Magazine
Date Submitted by the Author:	24-Aug-2010
Complete List of Authors:	Goran, Daniel; Bruker Nano GmbH Fundenberger, Jean-Jacques; Université Paul Verlaine-Metz, Laboratoire LETAM BOUZY, Emmanuel; University of Metz, LETAM Skrotzki, Werner; TU Dresden, Institut für Strukturphysik Suwas, Satyam; Indian Institute of Science, Department of Materials Engineering Grosdidier, Thierry; University of Metz, LETAM Toth, Laszlo; Metz University, Laboratoire de Physique et Mecanique des Materiaux
Keywords:	plastic deformation, shear bands, nickel
Keywords (user supplied):	Single crystal, ECAE, Microstructure

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**Local texture and microstructure in cube oriented Nickel single crystal
deformed by equal channel angular extrusion**

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Abstract

Local texture and microstructure have been investigated in order to study the deformation mechanisms during equal channel angular extrusion of a high purity nickel single crystal of cube initial orientation. A detailed texture and microstructure analysis by various diffraction techniques reveals the complexity of the deformation patterns in different locations of the billet. A modeling approach, taking into account the slip system activity, is used to interpret the development of this heterogeneous deformation.

Keywords: Single crystal, ECAE, Texture, Microstructure, Heterogeneity.

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1. Introduction

The processing of metals and alloys through equal channel angular extrusion (ECAE) [1, 2] has attracted much attention within the last decade primarily because this technique has shown the potential to achieve very large strains leading to ultrafine grain microstructures (≤ 300 nm) in bulk polycrystalline materials [3]. The process involves passing a billet through two channels of equal area of cross-section generally inclined at an angle of 90° . This imparts large deformation to the material in one pass itself and the process can be repeated to refine the grain size down to a value characteristic of the material.

The mechanisms of grain refinement through ECAE are, however, not uniquely agreed. The key phenomenon is the development of dislocation structures and their transformation into high angle boundaries. The principal difficulty is to find out the response of individual grains to such a deformation in terms of evolution of dislocation structure. The evolution mechanisms of dislocation structures during straining were examined, for example, by Hansen and co-workers [4-6]. It is, however, difficult to propose a unified description of the dislocation mechanisms governing both microstructure and texture evolution during large strain deformation. Correlations between dislocation structures and processing characteristics can be examined experimentally [7-9] or by statistical modeling [10-12]. For metals with medium to high stacking fault energy, the main feature of the dislocation structure development is the transformation of tangles into three-dimensional structures of dislocation cells or subgrains [4]. For higher strains, as applied in ECAE, the deformation microstructure is characterized by larger structural heterogeneities, e.g. deformation and shear bands. The formation of such deformation heterogeneities is a very important feature of ECAE and requires a systematic and rigorous analysis in order to improve the understanding of the

1
2 microstructural refinement process and provide a realistic input to deformation texture models
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4 that generally are limited to homogeneous grain deformation modes. Although, it is well
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6 documented that microstructure and texture after ECAE processing can be quite
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8 heterogeneous [13-18], the origin of such heterogeneities has not been sufficiently
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10 investigated from the viewpoint of microstructural development. When the deformation
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12 heterogeneities are to be addressed experimentally, single crystals are preferred instead of
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14 polycrystals as they can provide a simplified and deeper insight into the complex phenomenon
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16 of plastic deformation. Only a few studies have been carried out in this direction on copper
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18 [19-21], aluminum [22, 23] and nickel [24-26] as well as Nb [27] single crystals. On the other
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20 hand, different types of microstructures and textures were observed in these ECAE deformed
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22 single crystals, which were attributed to the formation of local deformation heterogeneities
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24 such as deformation and/or shear bands. It is therefore important to investigate the effect of
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26 the nature of such heterogeneities on the development of the texture and the microstructure
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28 during ECAE processing. With this goal, most of the studies on deformation structures in
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30 ECAE carried out so far have concentrated on the central area of the extruded billet which -
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32 being far from the walls where friction intervenes - was expected to be more representative of
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34 the response corresponding to the imposed deformation geometry. However, significant
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36 variations in texture and microstructure were also observed in ECAE deformed single crystals
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38 along the billet height [14, 24]. In addition, the recent analysis of a cube-oriented nickel single
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40 crystal extruded through a squared section die has also revealed strong differences in
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42 deformation pattern along the transverse direction of the extruded material [26]. These
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44 changes in deformation mechanisms resulted in significant macroscopic deformation
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46 heterogeneities on the overall cross-section of the sample even when a single crystal was used
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48 [26].

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The present work examines in detail the microstructure at different locations of an extruded Nickel single crystal in order to improve our understanding of the local deformation patterns. To this end, the crystallographic orientations are determined globally by neutron diffraction and locally by texture measurements carried out using X-ray and electron diffraction. These analyses are completed by a modeling of the slip system activities that helps to explain the development of the associated heterogeneity.

2. Material and experimental procedures

A bar of dimensions 40 mm × 10 mm × 10 mm was cut by spark erosion ~~technique~~ from a Ni single crystal (4N purity) grown by the Bridgman technique in the cube orientation. EBSD measurements were carried out to verify the crystal quality by precise orientation measurements at 18 locations on the polished {100} surfaces, revealing non-systematic deviations from the perfect cube orientation with maximum deviations of 4.5° and a mean value of 2°. Although only one sample was studied because of the complexity obtaining a Ni single crystal in cube orientation, the obtained results are expected to be representative for this orientation as the sample had relatively large dimensions and in general, the reproducibility of the testing by ECAE is very good.

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The ECAE experiment was carried out at room temperature using a Zwick 200 kN screw driven machine and a die set with rectangular intersection of the square extrusion channels, without any rounding of the corner region. A sketch of the ECAE setup with the coordinate systems is presented in Fig. 1a. Two specimens were cut from the middle of the billet length (Fig. 1c), one for optical microscopy (OM), EBSD analysis and local texture measurements by XRD and the other for global texture measurements by neutron diffraction. Two faces, the

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1 cross-section (ED-plane) and the right-side section (TD-plane), were analyzed with all the
2 techniques mentioned above. The analysis on the TD-plane was made at approximately 1.5
3 mm depth from the side face, in order to avoid the friction effects on the microstructure and
4 texture. OM was carried out using a Zeiss Axiophot optical microscope after mechanical
5 polishing and subsequent chemical etching with a solution of nitric acid (25.7 vol.%), glacial
6 acetic acid (67.5 vol.%) and distilled water (6.8 vol.%). Texture measurements were carried
7 out on a texture goniometer using Schultz reflection geometry with Co ($\lambda = 0.1789$ nm)
8 radiation. Local X-ray texture measurements were made at different locations on both sections
9 (see Fig. 2) using a pure (99.9%) annealed molybdenum mask, 100 μ m thick and containing
10 an aperture of 3 mm in diameter. Necessary corrections were applied to the raw experimental
11 data in order to reduce the effects induced by the mask. Four pole figures, namely (111),
12 (220), (200) and (311), were measured and from these data the orientation distribution
13 function (ODF) was calculated with the TexEval software developed by LETAM (University
14 of Metz) based on the series expansion method of Bunge [28]. No sample symmetry
15 restriction was imposed for the calculation of the ODF, that is, the ODFs were calculated
16 using triclinic sample symmetry.

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35 Global texture measurements were done by neutron diffraction at TEX-2 (GKSS Research
36 Centre, Geesthacht, Germany) on a cube of (10 mm $\times$ 10 mm $\times$ 10 mm) taken from the
37 middle section of the ECAE deformed bar (sample 2 in Fig. 1c). Complete (200), (220) and
38 (111) pole figures were measured. The orientation distribution function was calculated from
39 these pole figures in the same way as above.

47 Although the majority of the measurements were made on the ED-plane, the reference system
48 x , y , z for the Euler angles of the ODFs is kept as it is defined in Fig. 1b. Because all the major

components of the texture that develop in the cube oriented Ni single crystal could be observed in the section $\varphi_2=0^\circ$ of the ODFs, only this section has been displayed for each set of results. All the ODFs in this paper are in the full range (0-360°) of φ_1 due to the fact that apparently there is no monoclinic symmetry around the z-axis (TD), which represents the ideal case for simple shear [29].

EBSD analysis was carried out using a Jeol JSM 6500F microscope equipped with a field emission gun and an electron back-scattering diffraction (EBSD) detector. The specimen surfaces for EBSD investigation were mechanically polished with grit paper and diamond suspension solution up to 0.25 μm and subsequently electropolished in a solution of 70% glacial acetic acid and 30% perchloric acid 60%. The EBSD patterns were processed and analyzed with the CHANNEL 5 software (HKL Technology, Denmark). The colors in the EBSD maps relate to the color key expressed in the standard stereographic triangle while the characteristic fcc crystal direction is registered along the normal direction ND. When needed, a good resolution (0.1 μm step size) was used in the acquisition process. No noise reduction was applied, thus the maps represent the raw EBSD results (non-indexed points are shown in black).

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For TEM bright-field analysis, thin foils were prepared from the ED-plane using the polishing solution mentioned above. The TEM examination was done in a Philips CM 200 operated at 200 kV.

3. Results

3.1. Optical microscopy

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Figure 2 represents a 3D view of the specimen obtained by OM mapping on the ED- and TD-planes. The cross-section is characterized by a heterogeneous banded microstructure. As already depicted in [26], excluding the two layers experiencing the direct effect of the friction on the lower and upper die walls (they were 800 and 500 μm thick, respectively), the rest of the billet section can be divided into different zones characterized by different aspects of the deformation patterns.

The upper part of the section, zone I, depicts a very complex banded microstructure. A major feature in this area is the presence of very broad inclined bands. These bands are roughly inclined along two distinct directions. Comparatively, the microstructure in the lower part of the sample (zone III) is characterized by long parallel thin bands almost parallel to TD. These thin bands can also be seen on the TD-section extending over several millimeters along ED. The two lateral zones of the extruded billet are mirror like images (labeled II^{L} and II^{R} , respectively). They are essentially characterized by more or less featureless domains located next to the lateral wall from which very large parallel bands appear to originate and extend towards the centre of the sample. It is interesting to note that the central part of the sample (labeled II in Fig. 2) – which, being far from the die, is generally considered to be the most homogeneous and representative one - is in fact characterized by the most complicated deformation pattern. The microstructure here consists of an intermixed of different features such as thin bands parallel to TD and larger inclined ones. These sets of bands are also visible on the longitudinal section (TD-section) in the area labeled II^{RS} of Fig. 2. They clearly extend all through the billet length and are slightly inclined to ED.

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3.2. Global and local texture

Figure 3a shows the results obtained from the global texture measurement using neutron diffraction. ECAE textures for cubic materials were analyzed before and the location of ideal orientations was reported by Tóth et al. [29]; a representative key figure is given in Fig. 3g only for the $\varphi_2 = 0^\circ$ ODF sections. With respect to this figure the global texture in Fig. 3a is characterized by a partial fibre about the shear plane normal (SPN) having a strong texture component: the split C ideal orientation with components C_1 and C_2 . Another texture component identified is the TD-rotated cube orientation (here with a rotation of about 60° around the z-axis).

The results of the local texture measurements obtained by XRD at different locations (zones indicated by circles in Fig. 2) are presented in Figs. 3b-f. The local texture in the upper zone (Fig. 3b) of the cross-section (zone I) is characterized by the TD-rotated cube component and a second component (C_1) which is rotated from it towards the ideal C component. Zone II (Fig. 3c) shows C_1 and C_2 which have rotated symmetrically (in opposite directions) even further towards C. The local texture measured on the TD-plane in zone II^L (Fig. 3d) is given by the highly intense texture component C_1 very close to the C ideal orientation. Zone II^{RS} (Fig. 3e) shows a strong C_2 component mirror related to that of zone II^L. Moreover, a relatively weak fibre parallel to TD is visible in this part of the sample. The lower zone III (Fig. 3f) of the cross-section displays a texture that is totally different from the textures observed in the other zones. The texture in this part of the sample is principally characterized by a cube orientation that spreads around the extrusion direction. The strength of the textures is lowest in the upper and lower zone.

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2 The local X-ray texture measurements have shown that the components found by neutron
3 diffraction are not homogeneously distributed within the ECAP billet but are located in a
4 systematic manner to a certain extent within its cross-section. Similar results have been
5 obtained by more localized texture measurements with high energy synchrotron radiation in
6 the central section along ND of the same ECAP processed single crystal [30].
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14 **3.3. EBSD analysis**

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18 The main objectives of the EBSD analysis were to validate the OM and XRD texture results as

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19 well as finding the links between the microstructural features and the different texture

20 components observed in the local X-ray texture results. Trace analyses were carried out to

21 characterize the crystallographic alignment of the different types of bands.

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28 Figure 4 shows the EBSD map obtained from the upper part of the sample (zone I) and the

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29 corresponding $\varphi_2=0^\circ$ ODF section. The map depicts a quite complex banded microstructure

30 with different types of bands intersecting each other. These bands belong to the cube

31 component rotated by different angles around TD and extending somewhat towards the ideal

32 C and A_1 components (Fig. 4b).

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40 The very broad bands at the centre of the cross-section, as revealed by OM, were analyzed in

41 detail by EBSD. The EBSD map (Fig. 5a) shows the typical microstructure of the right side of

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42 zone II with a very broad band (colored in green) inside the matrix (colored in blue). The

43 corresponding $\varphi_2=0^\circ$ ODF section given in Fig. 5(b) shows that the orientation of the matrix

44 is C_2 , while that of the broad band except of a slight ϕ rotation is a TD-rotated cube

45 component. The border to the matrix does not have any crystallographic alignment. Also, fine

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bands are present in the matrix as well as in the broad bands. Trace analysis shows that there is a preferred alignment of these bands along the traces of two $\{111\}$ planes on the ED-plane. This indicates that they are related to the crystallographic shear planes and therefore may be called microbands or crystallographic shear bands (CSBs). The broad green band may be interpreted as deformation band (DB). The CSBs are deviated at the boundary between the matrix and deformation band giving rise to a kink-type configuration. The texture component given by the CSBs, as represented in reddish-pink color, is in a $-\Delta\phi_1$ TD-rotated position from the matrix and deformation band orientation. This is clearly seen in the ODF sections (Figs. 5c, d) belonging to selected areas within matrix and DB. Thus, the CSBs are just shear zones leading to heterogeneous overall straining or strain partitioning. A similar observation is made on the left side of zone II by analyzing a DB of a TD-rotated cube component (with a slight $-\Delta\phi_1$ rotation in the same manner as observed in the right side of zone II) in a matrix of C_1 (Fig. 6). Although the DBs described have mirror symmetry the CSBs have the same shear sense (compare ODF sections of Figs. 5c, d and 6c, d).

The results of EBSD measurements at the centre of zone II^{SR} of Fig. 2 are presented in Fig. 7a. The microstructure is characterized by thin parallel CSBs (colored in red) inside the matrix of component C_2 colored in blue. These CSBs are the same as those found in the matrix in the ED-sections of Figs. 5 and 6.

Similar microstructural features as depicted in Figs. 5 - 7 were reported by Basson and Driver [31] after plane strain compression of a cube oriented Ni single crystal up to a strain of 1.5. The authors described the features inside the large deformation bands as being micro-shear bands associated with TD (ϕ_1) and some RD (Φ) rotations which is exactly the case in the present work.

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The EBSD map shown in Fig. 8 is characteristic of the microstructure in the lower part of the sample (zone III in Fig.2). In this zone the microstructure is as complex as in the upper zone and possesses very high spreads in orientation. The characteristic features of this part of the sample are large horizontal bands either composed of a sequence of smaller bands colored in green and reddish-yellow or of a fine-grained structure, which according to a misorientation analysis contains a high frequency of high angle grain boundaries. The horizontal fine-grained bands are shear bands of non-crystallographic character. They produce the same shear as the ECAE process, however, leading to $+\Delta\phi_1$ rotation, i.e. higher shear. In terms of texture, the major component is the rotated cube component (red colored) that spreads about ED

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3.4 High resolution EBSD and TEM analysis

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To investigate the nature of the CSBs inside the matrix in more detail, high spatial resolution EBSD maps have been acquired with an ultra fine step size of 50 nm. The boundaries map corresponding to the scanned area is shown in Fig. 9a overlapped with the band contrast (BC) map. Boundaries with misorientations smaller than 15° degrees are shown in white while the high angle boundaries i.e. $>15^\circ$ are indicated by black lines. As can be clearly seen in Fig. 9a, compared with the matrix, the CSBs contain a higher density of both high and low angle grain/subgrain boundaries, thus, indicating a higher shear strain. This correlates with the microstructural features revealed by TEM analysis (Fig. 9b). The grain and/or cell size in the CSB shown in Fig.9b. is significantly smaller than in the matrix.

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4. Discussion

Deleted: To show the nature of the CSBs in the matrix in more detail, first of all the misorientation distribution has been mapped and superposed with the Kikuchi band contrast which is a qualitative measure of the degree of deformation (Fig. 9a). As can be clearly seen, compared to the matrix, the CSBs show a higher band contrast and contain a higher density of high angle grain boundaries, thus, indicating a higher shear strain. This correlates with the microstructure revealed by TEM (Fig. 9b). The grain and/or cell size in the CSB is significantly smaller than in the matrix.

The microstructure in ECAE deformation of a cube oriented Ni single crystal is quite heterogeneous at the macro-, meso- and microscale. The heterogeneity of deformation in ECAE is well documented for single and polycrystals [13-27, 29, 30], with some studies focusing on texture heterogeneities [13-16, 30] and others dealing with the deformed microstructures [19, 22, 23] without making a detailed study on both. To the best of our knowledge, all the literature published in this area mention the heterogeneity of ECAE deformation on polycrystals only along the height of the sample, i.e. along ND [13-16, 30], and therefore, the corresponding theoretical formulations are based on this. However, in the present work, heterogeneities in texture and microstructure were also observed along the transverse direction, indicating that phenomena taking place along all directions should be addressed in order to have a clearer view of the complex plastic deformation in the ECAE process. In the following, an attempt is made to interpret the formation of the macro-, meso- and microscale heterogeneities observed in the microstructure of the one pass ECAE deformed Ni single crystal and their effect on the texture development.

Heterogeneous deformation of materials may be of ~~various, different~~ origins. In the present case, the heterogeneities observed are related to the die geometry of the ECAE process and the single crystal orientation. In the following subsections, these aspects will be discussed in more detail. First of all, the slip activity in the cube component subjected to simple shear is analyzed. Then, based on the peculiarities found, the origin of the heterogeneities with respect to texture and microstructure in normal and transverse direction of the billet is discussed. Finally, a comparison is made with ECAE of cube textured polycrystalline aggregates.

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4.1 Slip system activity in the cube orientation during simple shear

Because of its highly symmetrical nature, the cube is one of the most studied orientations. It is generally accepted that the cube orientation is metastable during plastic deformation at room temperature and exhibits heterogeneous deformation as well as splitting into strongly misoriented microstructural features, e.g. shear bands and deformation bands [6, 31]. In order to get a clearer understanding of the stability of cube orientation, slip system activity calculations were made for the exact cube and rotated cube orientations subjected to simple shear deformation. To do this, the full constraints viscoplastic crystal plasticity model [29, 32] was employed on the initially cube oriented crystal using the $\{111\}\langle 110 \rangle$ slip systems (see Table 1). The ideal simple shear model was used with a shear rate of $\dot{\gamma} = -1.0s^{-1}$ applied in the 45° intersection plane of the ECAE die, i.e. in the $x'-y'-z$ reference system, see Fig. 1. The corresponding velocity gradient - when expressed in the $x-y-z$ frame - is given by [29]:

$$L = -\frac{\dot{\gamma}}{2} \begin{bmatrix} 1 & -1 & 0 \\ 1 & -1 & 0 \\ 0 & 0 & 0 \end{bmatrix}.$$

This strain mode was used in the simulations in small strain increments up to a total shear of $\gamma = -2.0$. The obtained slip rates are depicted in Fig. 10 for a strain rate sensitivity index of $m=0.05$. It is to be mentioned here that when smaller m values are used, the slip distribution remains nearly the same.

The lattice rotation rate for the initially cube orientation was found to be equal to the applied rigid body spin, meaning that only the ϕ_1 angle changes with a rate of $\dot{\phi}_1 = \dot{\gamma}/2$. Moreover, $\dot{\phi}_1$ remains constant for any rotated position of the cube. This result is in agreement with the analysis of Tóth and Jonas [33]. Consequently, the total rotation of the ideal cube component is 57.3° in the ϕ_1 angle while the other two Euler angles remain unchanged, see Fig. 10. The operating slip systems, however, change completely during this large strain rotation of the

cube component. Four slip systems are active in the exact cube component as well as in the $\phi_1 = 22.5^\circ$ position; however, in general, eight systems are active. Actually, a large number of active slip systems are necessary to produce zero plastic spin, which is the condition to have the lattice spin to be equal to the rigid body spin. It has been shown in Tóth et al. [34] that the minimum number of active slips systems for zero plastic spin actually is four.

It can be seen from Fig. 10 and Table 1 that the average slip vector remains always in the x-y plane. A consequence of this is that the stress state remains also plane stress state (like the strain state in the x-y plane) for any rotated position of the cube. This is an important finding for the discussion of the possible effects exerted by the walls of the channel on the crystal. One of the consequences is that lateral walls (with normal TD) do not have to exert forces on the crystal during its plastic deformation.

4.2 Influence of the ECAE process on the stability of the cube orientation

The stability of the cube orientation has already been discussed by Dillamore and Katoh [35] for rolling deformation and by Akef and Driver [36] for plane strain compression (strains in the range of 0.25–1.5). From their theoretical considerations, it follows that small deviations from the exact cube orientation lead to rotations in different directions, as has been observed in this investigation where the single crystal not only rotates about ϕ_1 (as seen above and as predicted by the Taylor theory for the exact cube orientation) but also about ϕ . This means that the small deviations ($2 - 3^\circ$) from the exact cube orientation existing in the undeformed sample may already lead to texture components spreading towards the C orientation.

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Another deviation from the exact cube orientation develops during shear due to deformation banding favored by the operation of a large number of slip systems. According to Humphreys and Hatherly [37], one of the origins of deformation banding is the ambiguity associated with the selection of the operative slip systems. In many cases the imposed strain can be accommodated by more than one set of slip systems and the different sets of slip systems lead to different lattice rotations. Another origin is given by inhomogeneous straining, i.e. different regions of a grain may experience different strains if the work done within the bands is less than that required for homogeneous deformation and if the bands can be arranged so that the net strain matches the overall deformation. These deformation bands are separated by geometrically necessary boundaries (GNBs). In addition, boundaries are formed by statistical trapping of dislocations forming so-called incidental dislocation boundaries (IDBs). In general, the misorientation of both types of boundaries increases with strain [38] the rate being larger for GNBs, i.e. GNBs accumulate dislocations at a higher rate than IDBs. Besides, the spacing of both types of boundaries decreases with increasing deformation indicating that grain subdivision continues to refine with increasing strain. With increasing misorientation this process leads to the formation of new high angle grain boundaries the percentage of which increases with strain. Grain fragmentation depends on grain orientation. Because of the symmetry of special orientations, one choice of slip systems leads to an opposite lattice rotation from the other choice. The orientation changes due to grain fragmentation lead to the destabilization of the cube component and support the formation of orientations close to C. The formation of the split C component has been successfully simulated with Tóth's flow line model by applying the viscoplastic self consistent polycrystal code on a cube texture represented by 1000 grains which were selected to represent a 6° Gaussian distribution around the cube position [30].

To explain the heterogeneous texture and microstructure observed in normal and transverse direction in the billet it is necessary to take into account the geometry of the ECAE process. In the present case a die has been used with an ingoing channel moving with the billet except the x-wall, thus reducing friction within this channel and reducing the pressing force. On the other hand, friction on the walls of the outgoing channel has been increased by narrowing this channel on each side by 0.1 mm and on the top by 0.2 mm in order to compensate for elastic rebound of the billet after extrusion [39]. Otherwise machining of the billet to the original size is necessary before performing another pass. As a consequence, friction at the side walls leads to shear processes producing rotations around the TD-rotated crystal x-axis, i.e. ϕ -rotations. Such rotations during shearing lead to orientation motion towards C. Indeed, the split C component in mirror symmetry is found on the left and right side of the billet in zones Π^L and Π^R . In contrast, high friction at the top and bottom wall leads to a TD-rotation, i.e. ϕ_I -rotation of the cube component. Friction on the top wall produces a shear sense coinciding with that of the ECAE process, while that on the bottom wall is opposite. Thus, cube rotations on the top are enlarged while on the bottom they are diminished.

As seen in Figs. 3, 4 and 8, as well as shown by [30], the rotation of the cube component progressively increases up to 90° from top to bottom. The theoretical simulated rotations show much less dependence on the position, see Fig. 10. Actually, they are limited to 57.3° which is the rotation value corresponding to the simple shear model. This theoretical rotation agrees with the rotation that can be obtained from only rigid body spin without any plastic spin in the lattice rotation. Such a behavior of the cube component for fcc crystals was already predicted by Tóth and Jonas [33] for simple shear.

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The 90° rotation in the bottom part, however, is more than the theoretical limit. This can be explained with the effect of friction which works against the plastic deformation process by reducing the plastic strain drastically. It is seen in finite element simulations [16] that in the bottom part there is practically no plastic strain. The material, however, is forced to flow, and an initial cube-shaped material element must rotate by 90° in rigid rotation. Thus, this rotation is not a rigid body rotation originating from plastic strain; it is just a kinematical rotation during which there is no shape change in the material element during flow. The relative weakness of the rotated cube in the experiment may be due to that process.

The shear bands passing through the entire sample change their character from the top to the bottom of the billet. In zones I and II they are more or less crystallographically oriented while in zone III they lie on the ND-plane disregarding deformation banding. The change in the character may be due to the high constraints given by the friction at the bottom wall. Crystallographic shear banding may be caused by the instability of the “homogeneous” deformation structure due to changes in the slip activity. As seen in Fig. 10 for the cube component the initially active slip systems (no. 3, 6, 9, 12) with Burgers vector in shear direction become less active after rotation from the ideal cube orientation. However, rotations in the $-\Delta\phi_l$ direction - as taking place in the CSBs - favor those slip systems which have shears without conflict with the side walls.

4.3 Comparison with ECAE of cube textured polycrystals

Various polycrystal simulations based on the simple shear model [2] or even the flow-line model [29] predict the formation of the C component for high to medium stacking fault energy materials. Suwas et al. [32] have reported that a strongly cube textured polycrystal of

copper - when subjected to ECAE - gives rise to a texture having a predominant C component while in weakly textured copper the texture evolves towards A_1 and C. In the aluminum alloy AA 5109, the pronounced cube component rotates about 65° and its intensity drastically decreases from about 13 to 3 mrd [15]. Simultaneously, all the typical shear components develop with low intensities (<4 mrd). Interestingly, the C component is split similar as here ($\Delta\phi_l \approx 5^\circ$, $\Delta\phi \approx 20^\circ$). However, in contrast, some other components also appear. This is due to a larger spread of the initial cube component compared to the present case, where the material is perfect cube oriented single crystal. The result of these differences is that grains rotate and fragment at the same time, leading to the formation of other shear components.

While the texture heterogeneity in the normal (ND) direction of ECAE deformed polycrystals is well proven, there are no studies known so far in the transverse (TD) direction. However, compared to single crystals, the influence of friction at the side walls, which strongly depends on the die geometry, should extend less into the interior of the billet in case of polycrystals.

5. Conclusions

In order to find the origin of deformation inhomogeneities in a cube oriented pure Ni single crystal deformed by ECAE at room temperature, a thorough analysis of microstructure and texture at macro-, meso- and microscale has been carried out using different complementary techniques. From the results obtained, the following conclusions can be drawn:

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- 1) A heterogeneous evolution of microstructure and texture was found to be the effect of both the initial cube orientation of the Ni single crystal and the geometry of the ECAE process.
- 2) The continuous change in the activity of the $\{111\}\langle 110 \rangle$ slip systems as well as the simultaneous activity of eight slip systems were found to be the principal reasons of the multistage heterogeneous deformation characterized by deformation and shear bands.
- 3) The deformation heterogeneity across the billet in the TD direction is due to friction of the sample with the side walls.
- 4) The microstructure and texture in the lower part of the billet evolved in a different way compared to the rest of the sample due to strong friction with the bottom wall.

Acknowledgements

The authors acknowledge Prof. A. Eberhardt (Metz, France) his help during the ECAE experiments. The neutron texture measurements were carried out by Prof. H.-G. Brokmeier (GKSS Geesthacht, Germany). D.G. acknowledges the doctoral scholarship accorded by the Conseil Régional de Lorraine, France.

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Figure captions

- Figure 1: (a) Schematic illustration of the ECAE die, (b) zoom on the intersection area of the two channels with the ECAE reference system and (c) sampling of the deformed bar. Sample 1 is used for XRD, OM and EBSD analysis on the ED- and TD-plane, sample 2 for global texture measurements by neutron diffraction. Deleted: OIM
- Figure 2: 3D view of the extruded billet showing the highly heterogeneous microstructure observed by optical microscopy on the TD and ED-plane. Different zones from top to bottom (I, II, III) and from left (II^L) to right (II^R) and right side (II^{RS}) are indicated. The circles match the approximate areas used for XRD local texture measurements while the white rectangles represent the approximate areas used for EBSD analysis. Deleted: OIM
- Figure 3: $\varphi_2=0^\circ$ ODF sections: (a) global textures measured by neutron diffraction, (b - f) local textures measured by XRD at different locations (zones indicated in Fig.2) on the ED- and TD-plane. (b) zone I, (c) zone II, (d) zone II^L , (e) zone II^{RS} and (f) zone III. The key figure with the ideal shear components and the initial cube orientation with a 6° spread are given in (g) and (h), respectively.
- Figure 4: (a) Orientations distribution map ($0.3\ \mu\text{m}$ step size) with (b) the corresponding $\varphi_2=0^\circ$ ODF section obtained on the top part of the cross-section (zone I); the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32). Deleted: OIM
- Figure 5: (a) Orientations distribution map ($0.25\ \mu\text{m}$ step size) of the right side of zone II (center of the cross section) with the corresponding $\varphi_2=0^\circ$ ODF sections: (b) whole image, (c) subsets 1 and 2; the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32). Dashed lines in (a) mark the approximate traces of the $\{111\}$ planes on the ED-plane for the corresponding C (blue) and rotated cube (green) components. Deleted: OIM
- Figure 6: (a) Orientations distribution map ($0.3\ \mu\text{m}$ step size) of the left side of zone II (center of the cross section) with the corresponding $\varphi_2=0^\circ$ ODF sections: (b) whole Deleted: OIM

image, (c) subsets 1 and 2; the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32). Dashed lines in (a) mark the approximate traces of the {111} planes on the ED-plane for the corresponding C (blue) and rotated cube (green/purple) components.

| Figure 7: (a) Orientations distribution map (0.2 μ m step size) of zone II^{RS} (middle part on TD-plane) with (b) the corresponding $\phi_2=0^\circ$ ODF section, the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32, 64). Dashed line in (a) marks the approximate trace of the {111} plane on the TD-plane for the C (blue) component.

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| Figure 8: (a) Orientations distribution map (0.15 μ m step size) of the lower part of the cross-section (zone III) with the corresponding $\phi_2=0^\circ$ ODF sections: (b) whole image, (c) subsets 1 and 2; the colors indicate the ND direction in the standard stereographic triangle (isovalues 1, 2, 4, 8, 16, 32).

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| Figure 9: (a) Boundaries map superimposed on a band contrast map (gray scale representation of the quality of Kikuchi patterns) given by EBSD (0.05 μ m step size) on the central area, (b) TEM micrograph of the same area showing the boundary between matrix and shear band.

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Figure 10: Slip activity in the rotated cube orientation calculated with the viscoplastic full constraints Taylor model for an applied macroscopic shear rate of -1/s in the 45° intersection plane of the ECAE die up to a total shear of 2.0. Numbers indicate the slip systems as defined in Table 1.

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Table 1: Slip system identification

Index	slip plane	slip direction
1	(1 1 -1)	[0 1 1]
2	(1 1 -1)	[1 0 1]
3	(1 1 -1)	[1 -1 0]
4	(1 -1 -1)	[0 1 -1]
5	(1 -1 -1)	[1 0 1]
6	(1 -1 -1)	[1 1 0]
7	(1 -1 1)	[0 1 1]
8	(1 -1 1)	[1 0 -1]
9	(1 -1 1)	[1 1 0]
10	(1 1 1)	[0 1 -1]
11	(1 1 1)	[1 0 -1]
12	(1 1 1)	[1 -1 0]

Figure 2: 3D view of the extruded billet showing the highly heterogeneous microstructure observed by optical microscopy on the TD and ED-plane. Different zones from top to bottom (I, II, III) and from left (II^L) to right (II^R) and right side (II^{RS}) are indicated. The circles match the approximate areas used for XRD local texture measurements while the white rectangles represent the approximate areas used for ~~OM~~ EBSD analysis.

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Figure 4: (a) **Orientations distribution** map (0.3 μm step size) with (b) the corresponding $\phi_2=0^\circ$ ODF section obtained on the top part of the cross-section (zone I); the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32).

Figure 5: (a) **Orientations distribution** map (0.25 μm step size) of the right side of zone II (center of the cross section) with the corresponding $\phi_2=0^\circ$ ODF sections: (b) whole image, (c) subsets 1 and 2; the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32). Dashed lines in (a) mark the approximate traces of the $\{111\}$ planes on the ED-plane for the corresponding C (blue) and rotated cube (green) components.

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Figure 6: (a) **Orientations distribution** map (0.3 μm step size) of the left side of zone II (center of the cross section) with the corresponding $\phi_2=0^\circ$ ODF sections: (b) whole image, (c) subsets 1 and 2; the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32). Dashed lines in (a) mark the approximate traces of the {111} planes on the ED-plane for the corresponding C (blue) and rotated cube (green/purple) components.

Figure 7: (a) **Orientations distribution** map (0.2 μm step size) of zone II^{RS} (middle part on TD-plane) with (b) the corresponding $\phi_2=0^\circ$ ODF section, the colors indicate the ND direction in the standard stereographic triangle (isolines in mrd: 1, 2, 4, 8, 16, 32, 64). Dashed line in (a) marks the approximate trace of the {111} plane on the TD-plane for the C (blue) component.

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Figure 8: (a) **Orientations distribution** map (0.15 μm step size) of the lower part of the cross-section (zone III) with the corresponding $\phi_2=0^\circ$ ODF sections: (b) whole image, (c) subsets 1 and 2); the colors indicate the ND direction in the standard stereographic triangle (isovalues 1, 2, 4, 8, 16, 32).

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