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Orientation Microscopy in the Transmission Electron Microscope - Investigations of Small Orientations Changes by Means of Orientation Mapping in TEM

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1. Introduction

Orientation Microscopy (OM) is a technique for determining the crystallites orientations in the automatic way using systems for acquisition and indexing diffraction patterns in grid of points refers to sample coordinate system. In this way, plenty of additional information about examined material can be provided. As a conventional method of orientation visualization, color code map is used; however, data behind each pixel is applicable for further computing. Quantifying parameters and characteristics based on the sets of measured orientations provide essential information about grains, grain boundaries and about local crystallographic lattice deformation.

OM was introduced in the Scanning Electron Microscope (SEM) in the 1990s [Adams, 1993]. Nowadays OM or Orientation Mapping technique is a very well known and common technique in the SEM. There exist several commercial systems for creating and processing data obtained in the SEM. In order to improve spatial and angular resolution introducing the OM in the Transmission Electron Microscope (TEM) was considered.

2. Crystallographic orientation and measurements methods

The study of the microstructure can be carried out using many techniques eg. light and electron microscopy. Most of the classical methods allow only the qualitative analysis. The use of crystallographic information enables more advanced qualitative and quantitative description of microstructure. Of particular importance are information obtained from systematic measurements of crystallographic orientation of the sample in defined areas of the investigated material. Knowledge of the orientation distribution can be linked with the properties characterizing the polycrystalline material.

Crystallographic orientation can be defined as function of coordinates (x,y,z) correlated with points in the sample of investigated material. Since measurements are usually carried out in the plane of the sample, the function depends on coordinates x, y. Then crystallographic orientation at each point with coordinates x, y can be defined as the rotation transforming

the coordinate system centered at the point (x,y) associated with the sample of the material in a coordinate system associated with a single crystal [Bunge, 1982].

To describe the orientation can be used a number of parameterization. The basic form of presentation of crystallographic orientation is a matrix of rotation. In addition, the orientations can be expressed through the axis and angle of rotation, Euler angles, Rodrigues parameters, quaternions et al. [Bunge, 1982; Morawiec, 2004, Frank, 1988]. The most commonly used are the Euler angles parameterization, where the orientation is expressed by three angles $\varphi 1$, Φ, $\varphi 2$, respectively: $0 < \varphi 1 < 2\pi$, $0 < \Phi < \pi$, $0 < \varphi 2 < 2\pi$ (the representation introduced by Euler in 1775) [Wenk, 2004; Morawiec, 2004]. The term crystallographic orientation is also related to the concept of crystallographic texture. Texture is called crystallographic orientation distribution in polycrystalline material [Kocks, 1998]. As defined by the texture of the material can be determined unambiguously from the measured single crystallographic orientation. Collection of individual crystallographic orientation can be used to determine the microstructural characteristics such as orientation distribution functions (ODF), misorientation distribution functions (MDF) of grain boundaries having the same or different phases. Based on the data obtained it is possible to analyze the stereological parameters of microstructures and phase distribution in the material. Proper selection of the data obtained using different measurement methods can provide the full characterization of the microstructure and determine its impact on the important, from a technological point of view, material properties.

To measure the crystallographic orientation can be used various techniques, from optical methods to diffraction methods [Wenk, 2004]. The most commonly used methods are diffraction: X-ray diffraction, neutrons and electrons.

The phenomenon of diffraction on the crystal can be described by the Braggs' equation.

X-ray diffraction methods are widely used to global texture measurements. To advantages of this method belong:

-    Simple methods of sample preparation,
-    Large area of investigation, global texture can be obtained,
-    Nondestructive method.

Spatial resolution of diffraction methods by means of the diffractometer depends on the used method and ranging from 50 μm to 0.1 mm [Schwarzer, 1998].

In order to increase spatial resolution instead of the traditional lamp as a source of the x ray radiation can be applied synchrotron. In that way can be obtained spatial resolution about 5 μm. Using the synchrotron radiation it is possible to obtain 3D microstructural characteristic 3DXRD (*Three-dimensional X-ray Diffraction*). This method was developed in Risø National Laboratory (Denmark) [Poulsen, 2004].

For texture characterization of the large volume of bulk materials the most proper is using neutron diffraction. But the access to the neutron diffractometer is limited.

New intensively developed methods are methods of local oreintation measurements by means of electron microscopes.

Due to the progress in equipment and fast data collecting computers methods of orientation measurements in scanning and transmission electron microscopy have been developed

rapidly in the recent years. Especially in the case of SEM orientation measurements become almost as widely used as X-ray methods. The basic idea of the local orientation measurements is of recording diffraction images, and then finding a crystallographic orientation corresponding to the image.

2.1 Orientation microscopy in SEM

Scanning electron microscope has become in the recent years versatile tool to observe microstructure. It can be applied for visualization of the topography of the surface of the sample for analyzing chemical composition or for crystallites orientation determination in specific area of the sample.

Exist several types of difraction patterns in SEM eg.:

- MKXD (*Micro Kossel X-ray Diffraction*),
- ECP (*Electron Channeling Pattern*), SAECD (*Selected Area Channeling Diffraction*),
- EBSD (*Electron Back-Scatter Diffraction*).

Currently, most developed and most commonly used diffraction patterns for the measurement of local crystallographic orientation in the SEM is EBSD technique. Diffraction pattern is formed under Braggs' law. Kikuchi lines or pairs of lines originating from different crystallographic planes on the pattern.

Automating the processes of acquisition and indexing diffraction images has allowed the creation maps by systems to measure the orientation in the SEM. The development of such systems was associated with the development of modern research equipment including cameras CCD (Charge Coupled Device) for recording microscopic images, and numerical methods allow for automatic indexing diffraction images based on supplied data. It initiated the development of a new field of electron microscopy, called the orientation microscopy [Adams, 1993]. OM or OIM™ acronym was introduced in the 90s of last century and concerned the distribution of crystallographic orientation in the material in terms of the coordinates of the sample by means of SEM. The best known commercially available systems for measuring the crystallographic orientation is OIM™ TSL - EDAX using the method of indexing diffraction images created by Wright and Adams [Wright, 1992] and HKL Channel 5 - Oxford Instruments based on indexing algorithm developed by Schmidt et al. [Schmidt, 1989]. Own system, named ACOM (Automated Crystal Orientation Mapping) was developed by Schwarzer [Schwarzer, 1997]. The principle of operation of all systems is based on the automatic recording of diffraction images obtained from the particular sample area, indexing and mapping of orientation. The orientation map is a graphical representation of the distribution of orientation on the sample surface. Mapping the orientation is the acquisition of diffraction images in a specific grid of points with a given step [Randle, 2000]. Received orientations are represented on the map by colors matched according to previously defined key colors. In the EBSD system grid step is done by controlling the electron beam position or the stage position. The introduction of a new FIB technique (Focused Ion Beam) in scanning electron microscopes equipped with gallium gun for cutting thin film of a desired size and thickness allowed to extend to three dimensions of orientation maps. With combining FIB and EBSD measurements, it is possible to cut successive samples (with an area of approximately 50x50 microns and a thickness of about 150 nm) of bulk material, and then make orientation measurements [Zeafferer, 2005;

Konrad, 2006]. In this way, based on measured sequence of two-dimensional maps one can obtain three-dimensional image of the microstructure. Modern commercial systems used in SEM, are also equipped with the possibility of a combined analysis of EDS (Energy Dispersive X-ray Spectrometry) and EBSD allowing obtaining information about chemical composition, crystallographic phase and orientation in a certain area of the sample. An important parameter for mapping is the spatial resolution of the method. The spatial resolution can be defined as possibility of distinguishing between two points in which orientation is different. In scanning electron microscopy measurement spatial resolution depends on investigated material and the type of electron source and is limited by the volume at which the beam interacts with the sample. In case of field emission gun (FEG) spatial resolution is the best and is about 30 nm. However, in the case of standard measurements to obtain the resolution of less than 100 nm is very difficult. While the angular resolution of EBSD images is above 0.5 ° [Schwarzer, 1998]. The advantages of EBSD systems are speed, full automation possible and very good quality maps for single-phase materials. Difficulties occur in the analysis of multiphase materials. Due to the slope of sample (>60 °) during the EBSD measurements, the important role plays the good preparation of the sample surface [Wenk, 2004]. Multiphase material, with different susceptibility to polishing and chemical reagents for the various phases may cause difficulties in obtaining equal sample surface, which affects the quality of the diffraction images, and thus the ability to automatically detection lines in the image. In case of materials after severe plastic deformation with nano and submicrocrystalline grains obtain the good quality of orientation map in SEM is challenging. These methods are also not effective for the analysis of local, small changes in orientation (<1 °) on the border of the angular-resolution diffraction EBSD.

2.2 Orientation microscopy in TEM

There is a strong need for development the method of orientation investigation of nanomaterials with angular and spatial resolution better that can be offered by EBSD/SEM.

This can be attained by means of transmission electron microscopy (TEM) Where the spatial resolution of the measurement is more limited by thickness of thin foils then the parameters of beam (diameter of electron beam can be lower then 2 nm) [Wu ,2009 Zaefferer, 2011]. This allows for analysis materials with ultra crystalline grains.

Automation of the systems in transmission electron microscope (TEM) is difficult. There exist several systems but most of them are not commercial available.

In TEM there are several different types of diffraction patterns [Carter & Williams, 1996]. The most common and wide used for phase discrimination are spot patterns (fig 1.).

Taking into account only geometry of spot patterns, orientation can be determined with accuracy ~5°. Higher accuracy (up to ~0.5°) can be assured by taking into consideration intensity on the diffraction patterns.

Investigation used local orientation measurements in TEM were performed in 1983 by Sztwiertnia and Haessner [Haessner, 1983]. Based on manual collected spot patterns were calculated local partial orientation and misorientation distribution function in cold rolled copper.
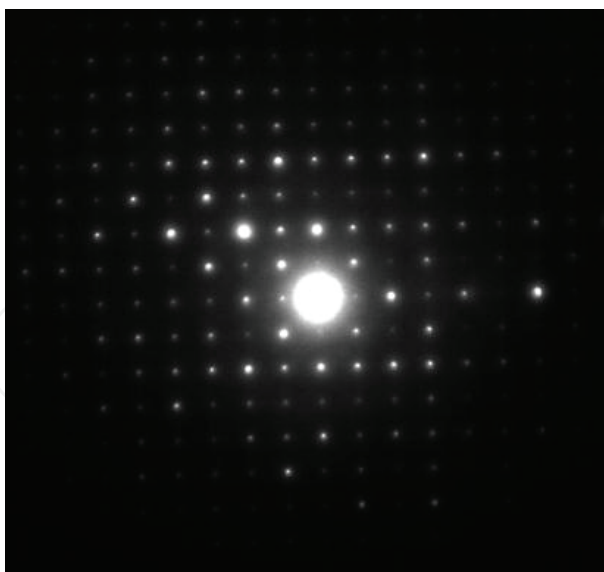
Fig. 1. Spot pattern in TEM.

Exemplary applications of spot patterns for investigation of the materials after severe plastic deformation are presented in [Zeafferrer, 2000]. In this work both geometry and intensity of spot diffraction patterns are used for determination crystallographic orientation. Acquisition of images is performed manually by microscope operator.

Another system based on spot pattern was presented by Rauch [Rauch, 2006]. Further development of this system resulted in commercially available system called Astar offered by NanoMEGAS company [Rauch, 2008].

In this case, the main goal of indexing is to match acquired spot pattern to the previously calculated database of patterns.
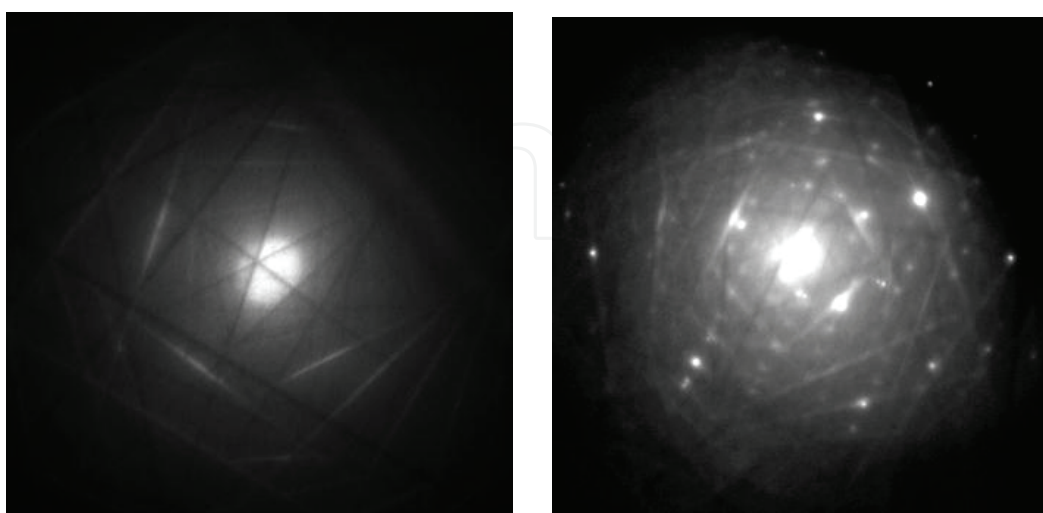
(a)   (b)

Fig. 2. Kikuchi type diffraction patterns: CBED a) and TKP b).

Better angular resolution has Kikuchi types diffraction patterns (Fig. 2). They allow to specify orientation with accuracy 0.1°.

To creating orientation map can be applied Kikuchi types patterns obtained in transmission beam (TKP – Transmission Kikuchi Diffraction Patterns) and convergent beam (CBED – Convergent Beam Electron Diffraction Patterns).

Method for automatic measurement of local crystallographic orientations in the TEM in defined areas of thin film using diffraction CBED images developed as one of the first Haessner and Sztwiertnia [Sztwiertnia, 1991; Pospiech, 1991; Haessner, 1992]. This method has been used to study recrystallization textures in the alloys with cubic symmetry. The first semi-automatic system for mapping the orientation of the TEM was created by Schwarzer and Zeafferer [Zeafferer, 1994] and then developed by Zeafferer [Zeafferer, 2000]. System operation is based on direct analysis of the collected diffraction images: spot patterns and Kikuchi type patterns. Besides the automatic indexing of images of diffraction system also contains procedures for computer processing of images and to determine the crystallographic dependencies such as: disorientation between grains, Burgers vector, slip systems and twinning systems.

Since systems for measuring the orientation maps in transmission microscopy are much harder to automate, most of these systems are created without the automation of image acquisition diffraction [Zeafferer, 1994; Schwarzer, 1997a]. System relies on recording diffraction in automatic way was built at the University of Metz [Funderberger, 2003; Fundenberger, 2003a]. These systems are not commercially available and must be individually adapted to a specific laboratory.

In addition to the different diffraction image analysis technique in TEM orientation map are created using Dark Field Image technique [Dingley, 2006]. This technique was introduced as a commercial system in 2000 by TSL-EDAX [Dingley, 2000]. For the measurements are used large series of dark field images recorded at different diffraction conditions. Intensity analysis is made on each dark field image. This allows the reconstruction of diffraction images and calculation the orientation at that point. The resolution of such measurements is dependent on the magnification used and the resolution of the microscope in dark field images [Zeafferer, 2002]. The advantage of this system is a fast measurement time of about 15 - 20 minutes. However, there are problems associated with drift of the sample and the need to use a large range of intensity (required for high-quality CCD camera).

All these methods and the system built in the IMIM PAS, whose description will be presented in the following subchapter, are used in various aspects of research in materials science. Together with X-ray difractometry and EBSD measurements techniques are complementary methods that allow for a detailed description of the microstructure of materials.

3. Elements of the system

In IMIM PAS the system for orientation mapping on TEM was built [Bieda, 2008]. The main idea of this system is automatic recording of diffraction images and save them on computer

hard disk in the form of graphic files using a digital CCD camera from a few to tens thousand of diffraction images and their subsequent indexing and analysis of the crystallographic orientation set.

The system (Fig. 3 and 4) includes the following elements:

- Philips CM20 microscope - transmission electron microscope with a maximum accelerating voltage 200 kV and spatial resolution (the possibility of distinguishing between two points on the microscopic image) value of ~ 0.2 nm. The microscope is connected to a PC via the serial port (RS232-C).
- CCD camera Gatan Dual Vision Company - 12 bit (4095 gray scale) digital CCD camera with a resolution of 1300 x 1030 pixels and exposure time from 0.01 to 10 s, mounted on the side of the microscope column. The camera is connected to the computer using a cable card and DMA enabling fast data transfer.
- Software Gatan Digital Micrograph - the software used for communication between a computer and a camera and a microscope. Allows to save images on a computer, has a built-in tools for image analysis, and allows to create own programs in developing capabilities of Digital Micrograph.
- Software EP for indexing Kikuchi diffraction patterns developed by A. Morawiec [Morawiec, 1999], as the result of cooperation between the IMIM PAS and the University of Metz (France).
- Software for analysis of data containing information about the distribution of orientation in a sample of material such as Tango (part of Channel 5 system for the analysis data from EBSD in SEM), programs to analyze the components of the texture, to calculate the ODF, etc.
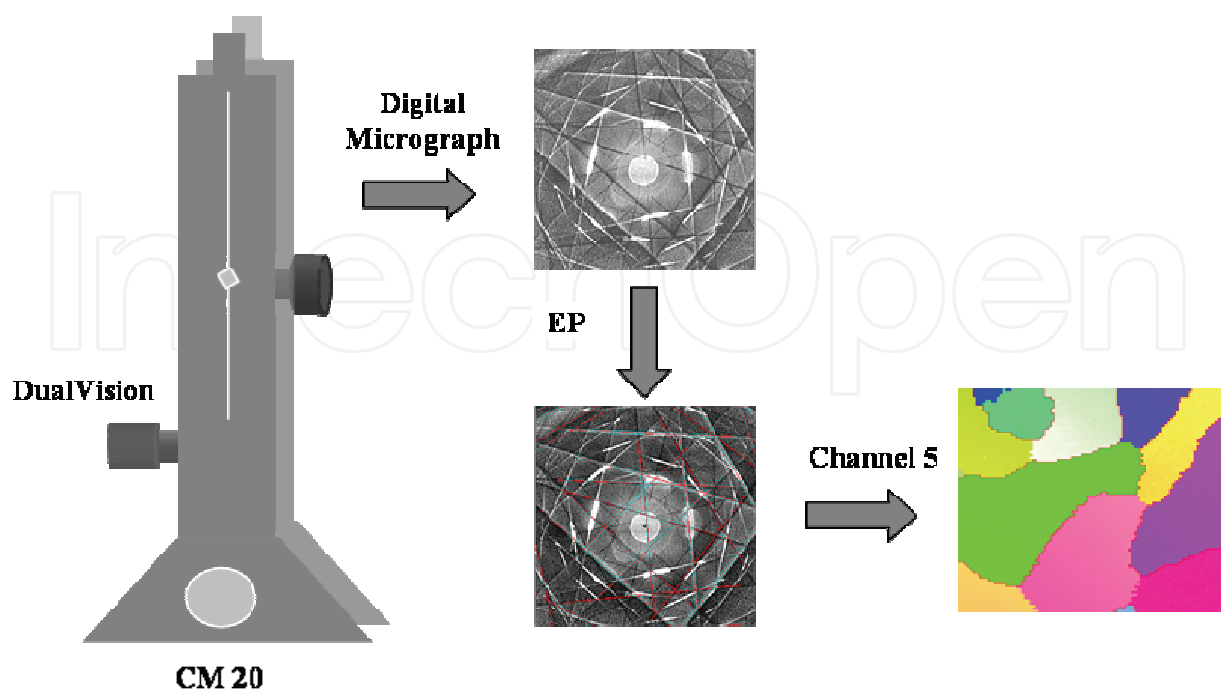

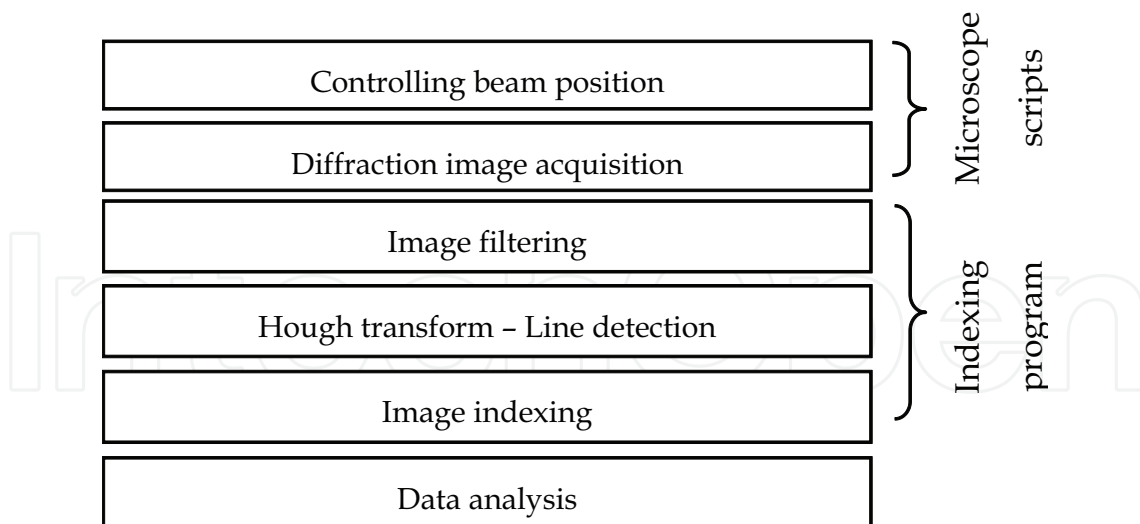Fig. 3. Elements of orientation mapping system.

Controlling beam position	} Microscope scripts
Diffraction image acquisition	
Image filtering	} Indexing program
Hough transform – Line detection	
Image indexing	
Data analysis	

Fig. 4. Schema of orientation mapping system.

4. Application of the system

One of the possible applications of the OM measurements in the TEM are presented. Deformed pure (4N) aluminum was chosen as an exemplary material. The new quality and quantity of supplied data made possible better microstructural characterization and observation of very small orientation changes produced characteristic subgrains structure (fig. 5.).
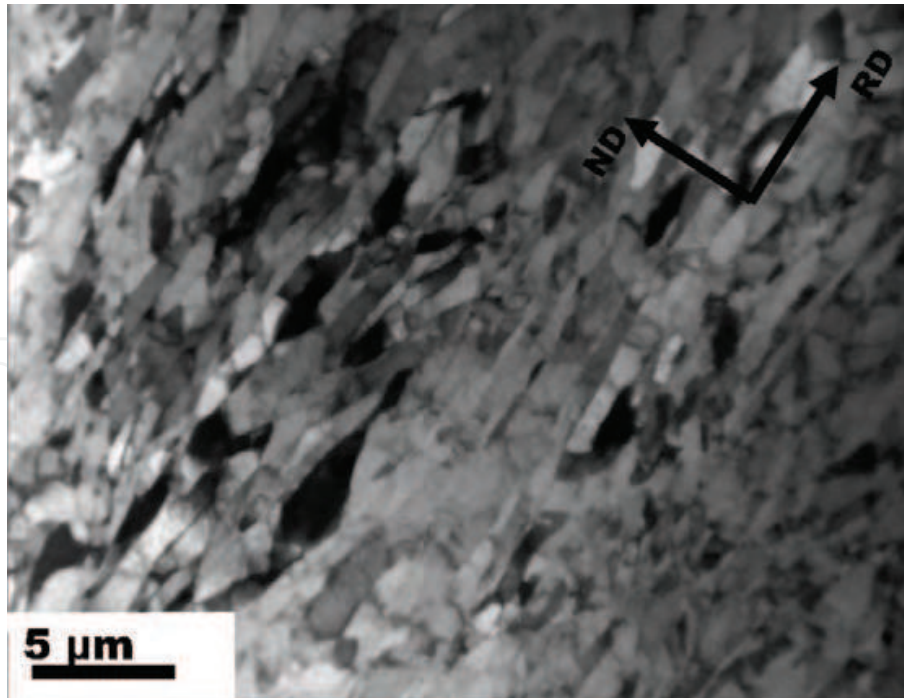
Fig. 5. TEM bright field image of cold-rollling up to 90% pure alluminium

Measurements of the local crystallographic orientation were carried out in aluminum with a purity of 99.99%. Thin foils were prepared from cross-rolled up to 90% sheet of metal. That

represented characteristic layered microstructure for the deformed material consisting of grains and subgrain bands lying almost parallel to the rolling plane, Figure 5. In previously published works such as [Pospiech, 1998; Sztwiertnia, 2006a] was presented the study of the microstructure of aluminum rolled on the basis of that single crystallographic orientation manually recorded along the direction normal to the plane of rolling (ND). The results obtained (about 1000 crystallographic orientation) were analyzed due to the contribution of individual components of texture in the plane perpendicular to the direction of the transverse plate (TD). An example of the orientation distribution function calculated from the measured orientation is presented in Figure 6. Pure aluminum after cold rolling to 90% deformation texture is characterized by copper with fiber type α and β. In the main β fiber are S and Copper components, and in the fiber α can be distinguished Brass and Goss components.
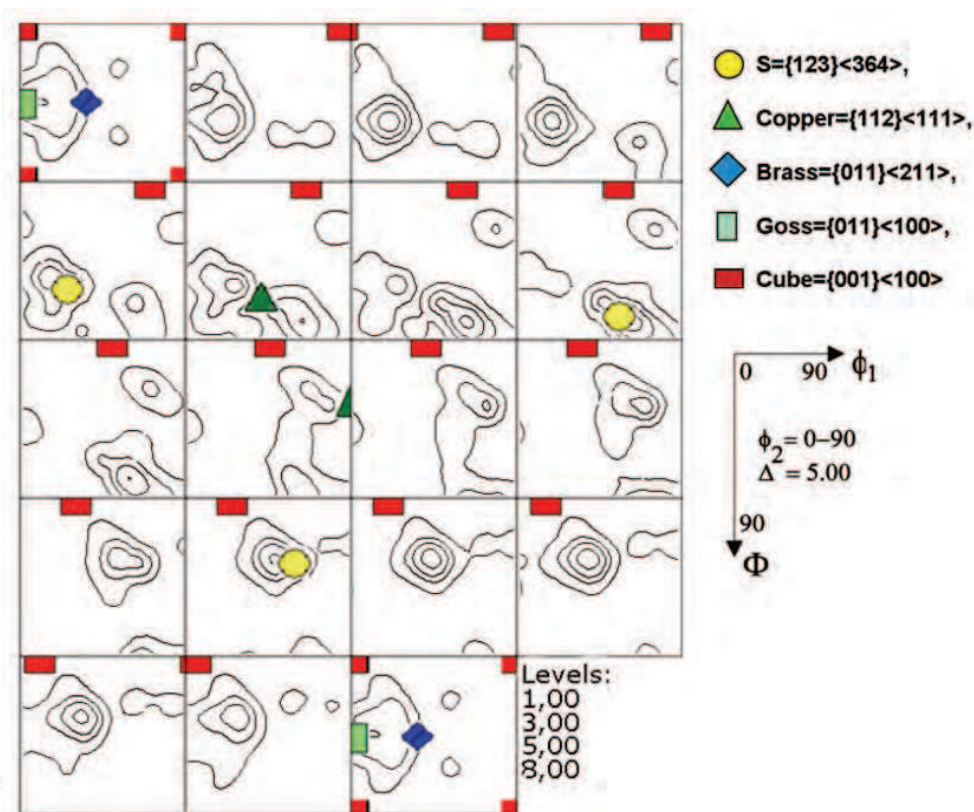
Fig. 6. Exemplary local orientation distribution function (ODF) for cold rolled up to 90% pure aluminium counted on the base of 1106 of orientation with marked texture components.

5. Results and discussion

Local crystal orientation measurements using the system to create orientation maps, allowed the registration of several thousand orientation at one measurement with a fixed step controlling electron beam displacement. This allowed obtaining the orientation maps in areas with a width from a few to several microns. With the ability to measure diffraction at the measuring points distant from each other by several nanometers, could be observed subtle changes in the crystallographic orientation in very small distances.

Orientation maps were measured for the longitudinal section (perpendicular to transverse direction TD) in such a way as to include as much information about the boundaries lying parallel to the plane of the aluminum sheet, Figure 7, 8 and 9. The analyzed areas of the samples were above 10 microns of thickness (along normal direction ND). In such areas piles of elongated subgrains in rolling direction RD were observed, which boundaries in the ND were characterized by disorientation angles generally less than 5 °.
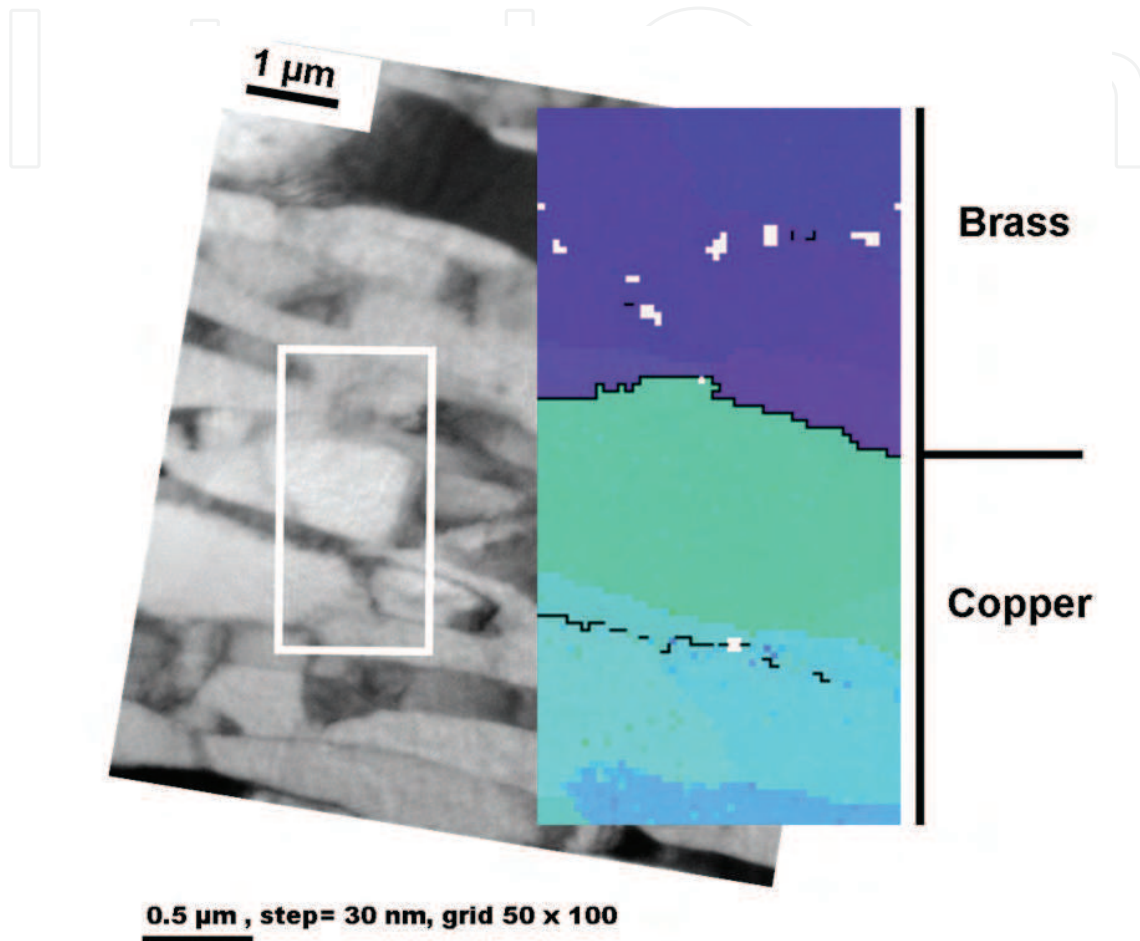
Fig. 7. Bright field image (TEM) of aluminum cold rolled up to 90% and orientation map with colors from inverse pole figure, the black lines are misorientations angles > 5° and corresponding texture components. Unresolved diffraction patterns are presented as white points.

Cumulative disorientation angles, less than 5 °, occurred between the orientations of subgrains, corresponding to one component of the texture. In Fig.7 on orientation map is marked boundary misorientation angle of > 12 ° occurs between the areas representing the type of texture components, respectively Copper and Brass. In the area of Copper component, we can distinguish the boundary disorientation angle of approximately 5 °. Since the border between the two components of the texture observed a gradual shift in orientation towards the ideal position of component-type Copper. Larger changes in the angle of disorientation identify the extent of occurrence of individual components of the texture (Fig. 7 and 10), while only minor changes were observed within the same component.
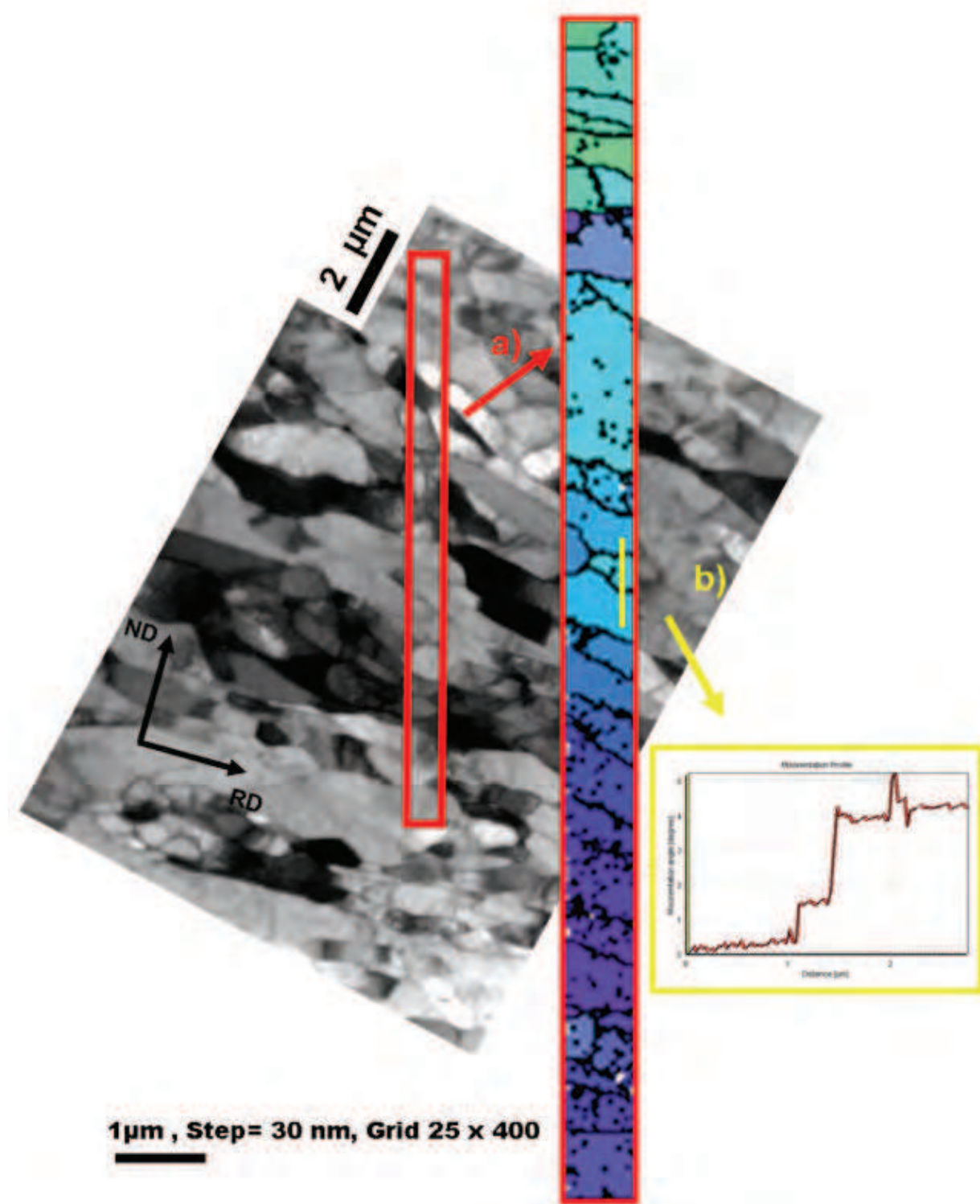
Fig. 8. Bright field image (TEM) of aluminum cold rolled up to 90% and a) orientation map with colors from inverse pole figure, the black lines are misorientations angles > 1°. Unresolved diffraction patterns are presented as white points and b) misorientation profile across three subgrains.

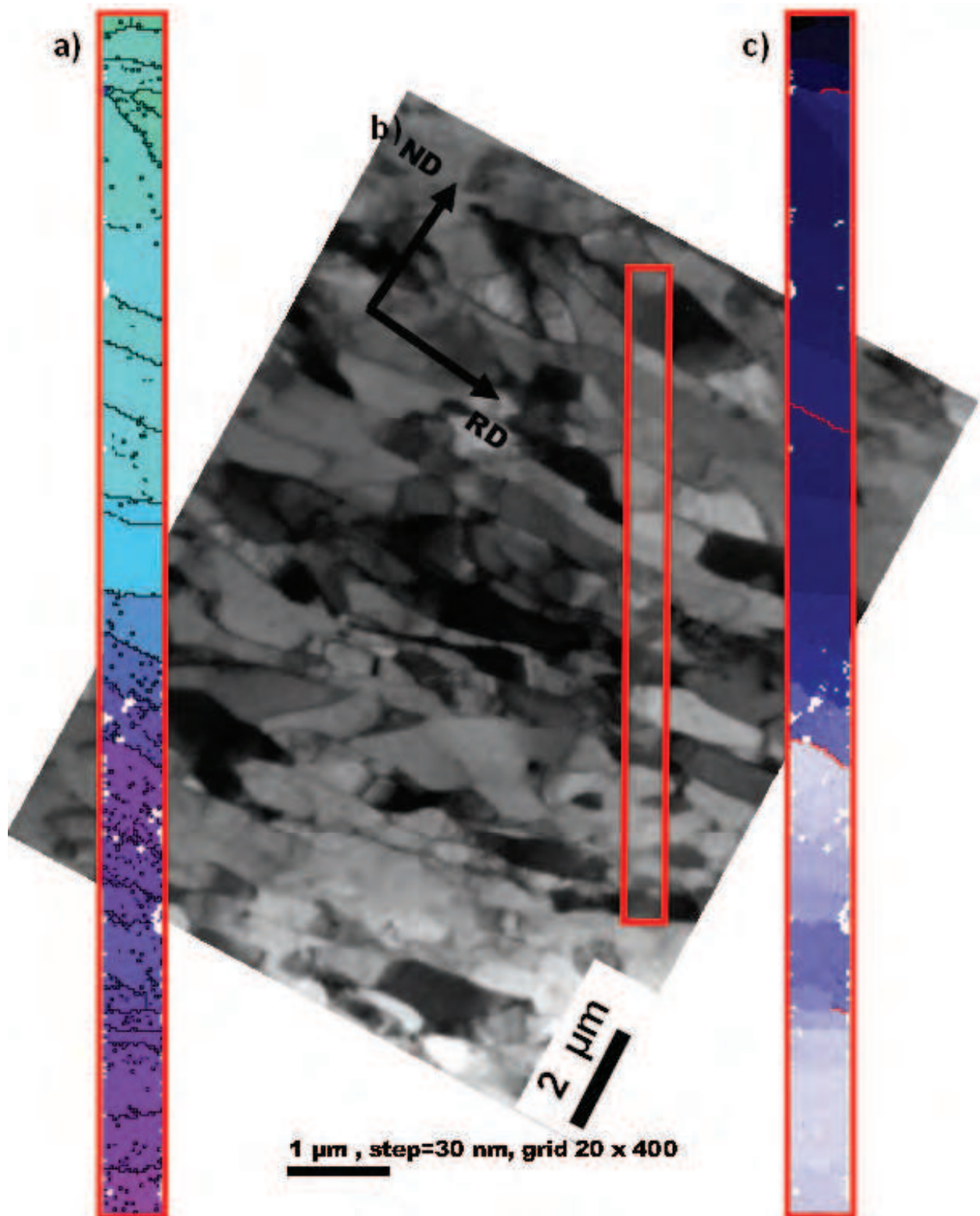Fig. 9. Bright field image (TEM) of aluminum cold rolled up to 90% b) and orientation map with colors from inverse pole figure a), the black lines are misorientations angles > 1°. Unresolved diffraction patterns are presented as white points and c) color map where gradient of color means deviation from the first orientation up to 20°, the red lines are misorientations angles > 5°.

Bands shown in the orientation maps (Fig. 8 and 9) are composed of bundles of parallel, highly elongated in the RD subgrains, between which there are relatively small (between 1 ° and 10 °), cumulative disorientation angles. In addition, in Figure 9c, is showed that the color gradation is related to a gradual change from the initial orientation. On this map red are marked boundaries, where the orientation changes were> 5 °. Typical course of changes in the angle of disorientation between adjacent subgrains along a line drawn parallel to the length of the orientation map is shown in Fig. 10a. Again, it can be seen that the differences in orientation are often very small on the boundaries of two subgrains, and may be cumulative, with the angle of disorientation between the first and last subgrains of the band may exceed 20 °. For example, total disorientation angle across the transition band width of about 12 microns, Fig. 10c, is ~ 20 °. Identifying the orientation of the corresponding components of the texture, it appears a transition from a stable orientation of S in the direction of another stable orientation Brass (Fig. 10).
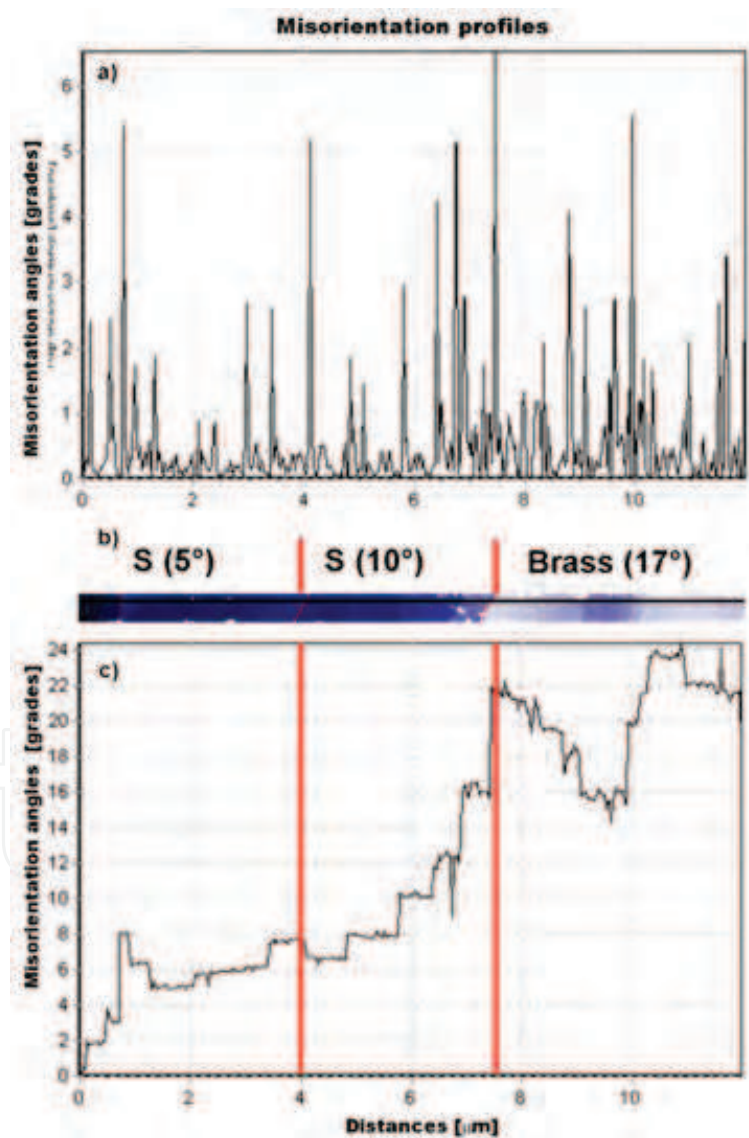

Fig. 10. Misorientation profiles across the line marked on the orientation map b) between particular grains a) and relative to the first orientation c) with marked texture components, the numbers in bracket means deviation from ideal orientation.

Accumulation of disorientation angles between subgrains across the spectrum is characteristic for the transition band [Hjelen, 1991], which distinguishes it from other elements of the microstructure of similar morphology. Transition band separates the fragments of the original grains, in which, during deformation, act various combinations of slip systems as a result of what they turned toward different, stable end orientations [Hu, 1963a]. With increasing strain - as the fragments of deformed grains are close to the stable end orientation - total disorientation angle increases, e.g. [Hjelen, 1991]. It should be emphasized that the transition band is often observed in deformed single crystals. In polycrystalline materials, their identification has been difficult because there were no methods to study small changes in orientation at short distances. In earlier work, in which orientation measurement was performed [Pospiech, 1998; Sztwiertnia, 2006a], the transition bands in deformed polycrystalline microstructure could not be noticed, because the diffraction at certain points of measurement were recorded only when followed their apparent change in that way it was easy to "lose" their subtle changes within the band.
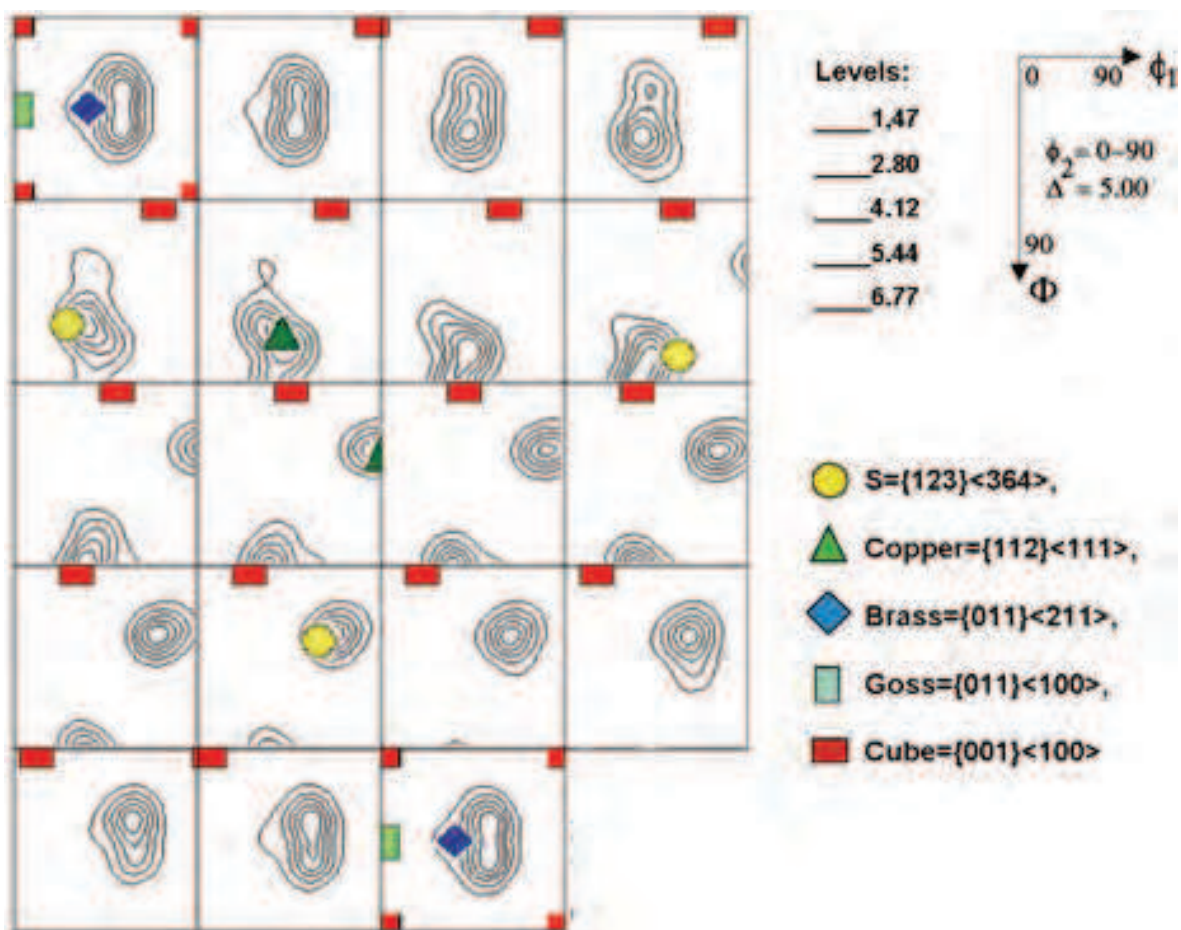

Fig. 11. Local orientation distribution function (ODF) for cold rolled up to 90% pure aluminium counted on the base of 56 000 of orientation measured in TEM with marked texture components.

On the basis of 56 000 measured orientation orientation distribution function (ODF) was calculated (Fig.11) and texture components as S, Brass and Copper were identified. Their respective percentage share was: 35.3%, 29.7% and 24%. Transitional areas between the bands in which there are no large misorientation are called deformation bands [Dillamore, 1972]. An example of such spectrum is shown in Fig. 12. Here we do not observe a smooth change of orientation angle combined with the accumulation of misorientaton angles, but only the fluctuations around a certain position. Thickness of the deformation bands having the same orientation was a few micrometers, and the angles of misorientation between adjacent bands were not large. Distribution of orientation along the ND was not accidental. Cells or subgrains were often concentrated in clusters containing up to 15 crystallites having the same type of orientation.
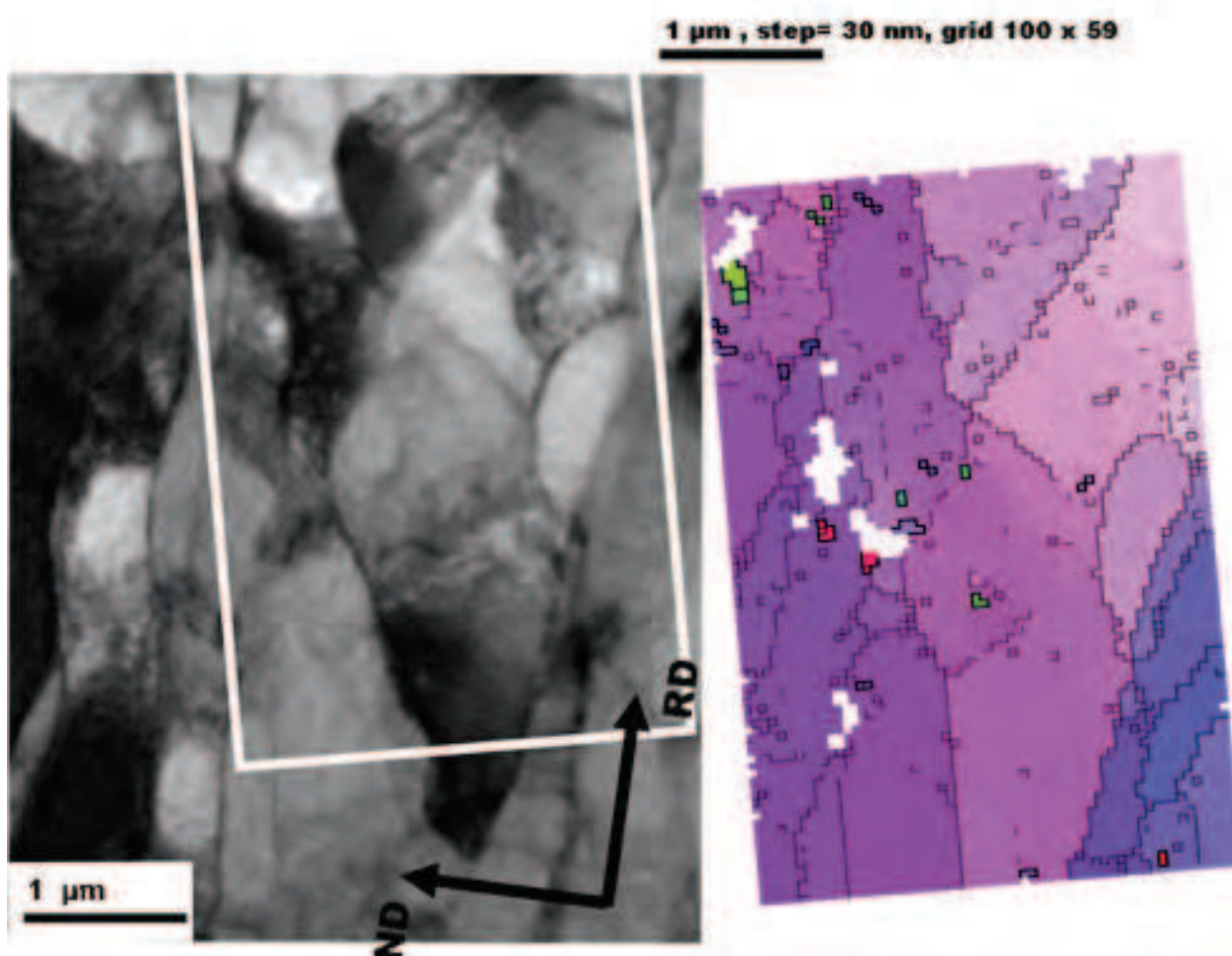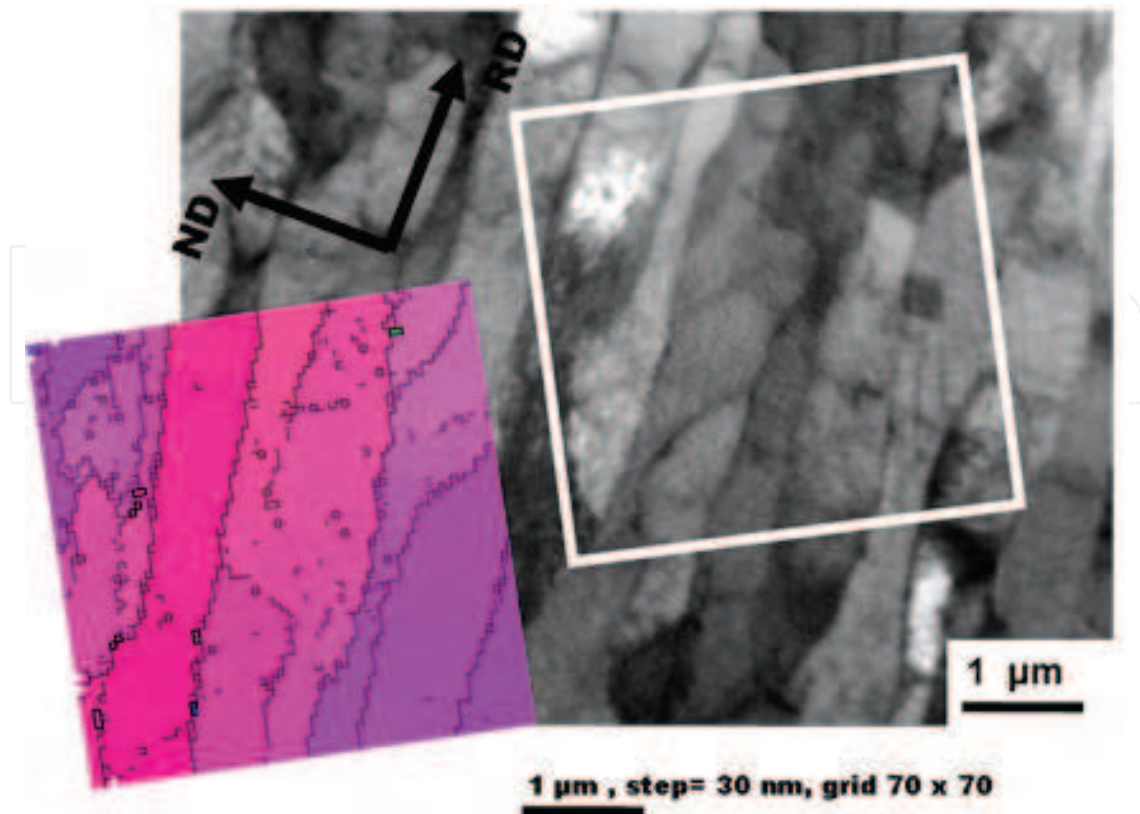

Fig. 12. Bright field image (TEM) of aluminum cold rolled up to 90% and orientation map with colors from inverse pole figure, the black lines are misorientations angles > 1°. Unresolved diffraction patterns are presented as white points.

Detailed maps obtained at higher magnification allowed to show the microstructure of subgrains in bands around 100 nm in widith (Fig. 13) with an angle of disorientation ~ 1 °.

Fig. 13. Bright field image (TEM) of aluminum cold rolled up to 90% and orientation map with colors from inverse pole figure, the black lines are misorientations angles > 1°. Unresolved diffraction patterns are presented as white points.

6. Conclusions

Principles of diffraction patterns used for orientation measurements in the TEM and short survey of existed systems for orientation mapping in the TEM was introduced. Pros and cons of TEM systems and comparison with SEM systems was presented in this chapter including spatial and angular resolution.

Difficulties in automation and variety of diffraction patterns used in the TEM made the existing systems, with some exceptional, hard to commercialize. Generally, methods in the TEM and the SEM can be used as complementary methods for investigating microstructure in nano and micro scale.

In spite of better spatial resolution in the TEM than in the SEM, the more important is better angular resolution in the TEM (about 0.1°), which can be assured of using Kikuchi type diffraction patterns and make possible investigation of small orientation changes in material in micro and nano-scale.

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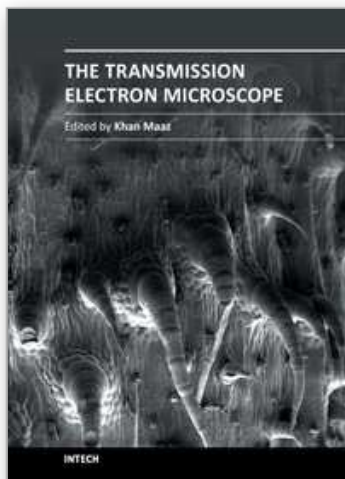
The Transmission Electron Microscope

Edited by Dr. Khan Maaz

The book "The Transmission Electron Microscope" contains a collection of research articles submitted by engineers and scientists to present an overview of different aspects of TEM from the basic mechanisms and diagnosis to the latest advancements in the field. The book presents descriptions of electron microscopy, models for improved sample sizing and handling, new methods of image projection, and experimental methodologies for nanomaterials studies. The selection of chapters focuses on transmission electron microscopy used in material characterization, with special emphasis on both the theoretical and experimental aspect of modern electron microscopy techniques. I believe that a broad range of readers, such as students, scientists and engineers will benefit from this book.

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