ELECTRON BACKSCATTERED DIFFRACTION TECHNIQUE-PRINCIPLE AND APPLICATION

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Abstract
This paper deals with basics of Electron Backscattered Kikuchi Diffraction (EBSD) technique in scanning electron microscope, directed to explanation of main terms and specifying its field of application in materials engineering.

Examples from EBSD investigation of micro-orientation relationships in nirogen-added AISI 316LN austenitic stainless steel type are presented.

1. Introduction

Electron backscattered diffraction (or otherwise called ***BKD*** - Backscattered Kikuchi Diffraction or ***OIM*** - Orientation Imaging Microscopy or ***ACOM*** - Automated Crystal Orientation Mapping) is a recent addition to the group of electron- microscopic techniques, based on analysis of ***backscattered diffraction patterns*** (***EBSP***) from highly tilted bulk specimen, using EBSD equipment on a standard scanning electron microscope.

Although backscattered pattterns were in SEM first observed 50 years ago, a major new technology began in early 1980´s, when the real- time imaging of backscattered diffraction pattern using sensitive low light TV cameras was involved. Of particular importance in the emergence of EBSD as a experimental technique was the development of rapid automated pattern acquisition and analysis during the past decade. Due to consequent increase of spatial resolution of modern electron microscopes is this technique gaining more popularity in various materials analyses 1-3 .

Comparing to light microscopy for quantitative metallography EBSD enables improved spatial resolution, more accurate data and more complete microstructural characterisation. The main advantage over TEM is use of bulk samples rather than thin foils and therefore more available grains per specimen 2, 3 .

EBSD is now routinely applied to many polycrystalline materials - commercially produced metals and alloys, ceramics, semiconductors, superconductors and minerals. It is used in various fields of materials science- for investigations of plastic deformation, nucleation and growth during recrystallisation, in the determination of recrystalised fractions in partially recrystalised materials [4, 5]. Moreover, it can provide a lot of data concerning intergranular, brittle transgranular and fatigue cracking [6]. In multiphase materials, phases can be fast differentiated using this technique, based on significant crystal structure differences. The database of such information obtained can be further used for qualitative determination of phase contents as well as texture analysis for each phase [7, 8]. Also structural variations in minerals can be revealed in this manner [9].

Above all applications, the orientation and interfacial studies using EBSD are preferred- the modifications in grain boundary networks and grain size and morphology are located easily- on microscopic scale, still in a representative and statistically relevant sample area [10, 11].

In addition, relatively new for EBSD is the use of enviromental scanning microscopes for investigation of uncoated and non- conducting materials 12 .

2. Physical principle of experimental method

2.1 Basic requirement

EBSD is very surface sensitive technique because of very low backscattered signal depth. Therefore a very flat and even distortion free sample surface is needed in order to get EBSPs of good quality so that automatic computer evaluation is reliable.

There are many types of materials examined using EBSD. They can differ in basic properties and also in their processing history that results in different microstructure and therefore in a different preparation behaviour. The choice of the best suitable preparation will depend on this as well as on the available equipment and time 13 . Recommended practices for sample preparation are summarised in Fig. 1.

As is illustrated in Fig. 1, the majority of materials require some degree of mechanical polishing, usually followed by final polishing with fine- dispersed colloidal sillica.

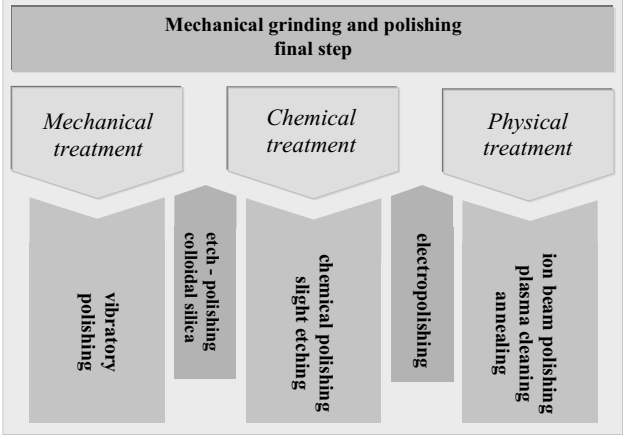

Fig.1 Preparation strategies for EBSD [13]

While mechanical polishing can produce good EBSD patterns, it is often necessary to use another step to improve pattern quality. Electropolishing or chemical etching are methods which may be used for metallic materials.

The relatively new for EBSD work is the use of ion etch of specimen surface, in some cases used also for removing undesirable, pattern quality decreasing oxide and surface layers. Similarly, to remove oxidative layers from the surface plasma cleaning can be helpful 13, 14 .

2.2 Other requirements

Except suitable sample surface preparation there are other essential steps needed for EBSD operation.

The first is that the angle between incident electron beam and the specimen surface should be small, in order to minimize the amount of signal, which is absorbed and to maximize the resulting backscattered intensities. Therefore, the sample is in microscope chamber highly tilted from horizontal, usually employing special designed sample holder. As a standard EBSD tilt angle 70 degrees was kept, Fig.2 2, 3 .

In addition, all samples investigated using this technique should be crystalline, have a grain size larger than 0,5 ěm and should be stable in electron beam.

2.3 Pattern formation

When a stationary electron beam in microscope strikes the sample, are the primary electrons scattered through large angles within the specimen and diverge from the point source in all directions. Part of incident primary electrons is subsequently colliding with specimen atoms and scattered out the sample. Some of these ***backscattered electrons*** are elastically scattered from the few tenth nanometers (~50 nm) bellow the material surface with nearly no energy loss (~1%) and are carrying informations used for local orientation measurements in submicron scale [2, 15].

By guiding the electron beam from point to point over the surface of highly tilted bulk sample, Fig. 2, forms this elastic scattering for each set of crystal planes two cones of

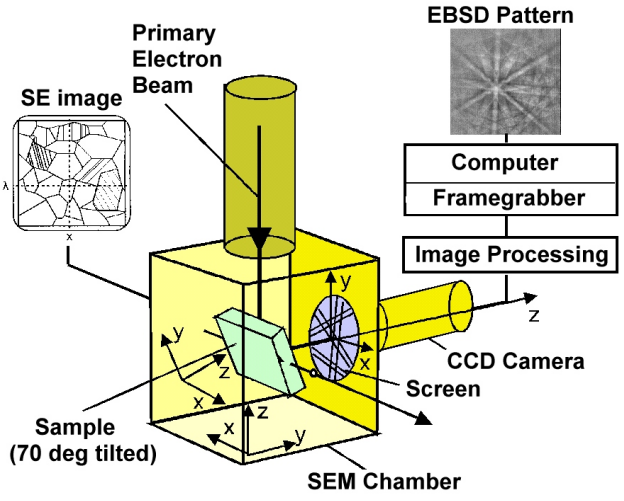

Fig. 2. SEM- EBSD composition with schematic illustration of information output [15]

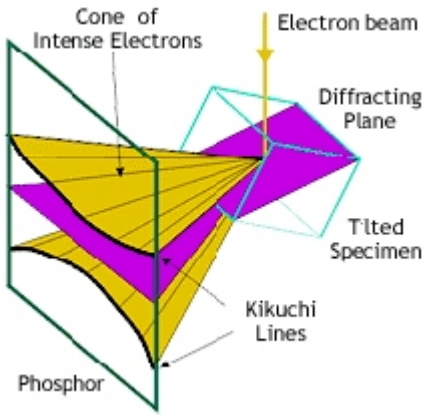

Fig. 3. Formation of one pair of Kikuchi lines from diffraction of the electron beam with one family of lattice planes [16]

relative intense radiation, whenever the Bragg condition is satisfied.

Placing the sensitive phosphor screen suitably near the sample, so as to intercept the cones, results in obtaining of conic sections, which appear as a pair of lines, called ***Kikuchi lines***, Fig. 3. Geometry of formed EBSP-an array of Kikuchi lines is determined completely by the crystal structure and orientation of the sample.

Obtained EBSPs are then captured using CCD camera connected to an image processor and finally fed into PC for display and evaluation. The computer at first has to determine the possition of the clearest Kikuchi bands and then to calculate interzone angles between zone axes located in the pattern. Mutual angles measured are next compared with a standard set of interzone axes of previously determined type of crystal structure present. When the best fit between experimental and exact angles in the database is found, the zone axes are automatically indexed. The orientation is then displayed, parallel with sitting the sampling probe on selected grains or points of microstructure image, as Miller's indices superimposition to the image of Kikuchi bands, Fig. 4 [2, 3, 17].

All parameters of the this process (e.i. phase allocation, coordinates of each point of investigation and others) are stored in a data file. The accuracy of measurement is better than 1°.

3. EBSD application possibilities

3.1 Grain orientation analysis

The largest interests in EBSD are concerned with *grain orientation* and *interface orientation studies*. Basic information on grain orientations and grain morphologies can be obtained in so-called ***orientation map*** or ***orientation image***. Example of such map from annealed austenitic stainless steel is given in Fig. 5a. Colour coding (in this case using a rainbow scale) allows to distinguish individual grain orientations and morphologies as well as their distribution over the sampled area. Computer routine also figures proportions of orientation components in the form of histogram, Fig. 5b.

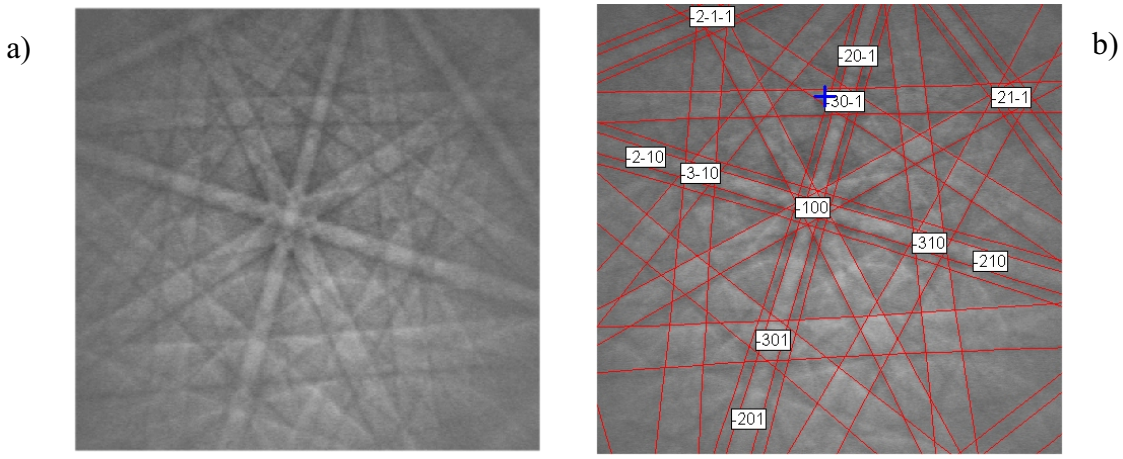

Fig. 4. Indexing consequence for one grain in basic state sample: a) example of band image captured by detector unit; b) the same image after overlay of Miller´s indices of zone axes

For statistical orientation analysis pole figures or inverse pole figures can be obtained. In the form of discrete data points or smoothed into contours present these plots important directions on a 2D stereographical projections for each individual measurement. Using this kind of data processing it is possible to reveal preffered orientation of present families of grains (texture)[3]. Examples of pole figures from stainless steel sampled area are given in Fig. 6.

The important consequence of orientation measurements is that misorientation between crystals can be accessed- in other words, the grain or phase boundaries can be investigated, if the orientations on either side of interface are known. Orientation changes revealed in orientation map depict all grain boundaries, so that grain boundary statistics can be extracted from it. This statistic usually quoted is the length of certain grain boundary types, such low angle, high angle, twin boundaries, which can be measured and outputted as the proportion of the total grain boundary length. As in the former case, it is attainable in the form of histogram 18, 19 .

Similarly, direct graphical visualization of grain boundaries in the map, choosing appropriate colours and line

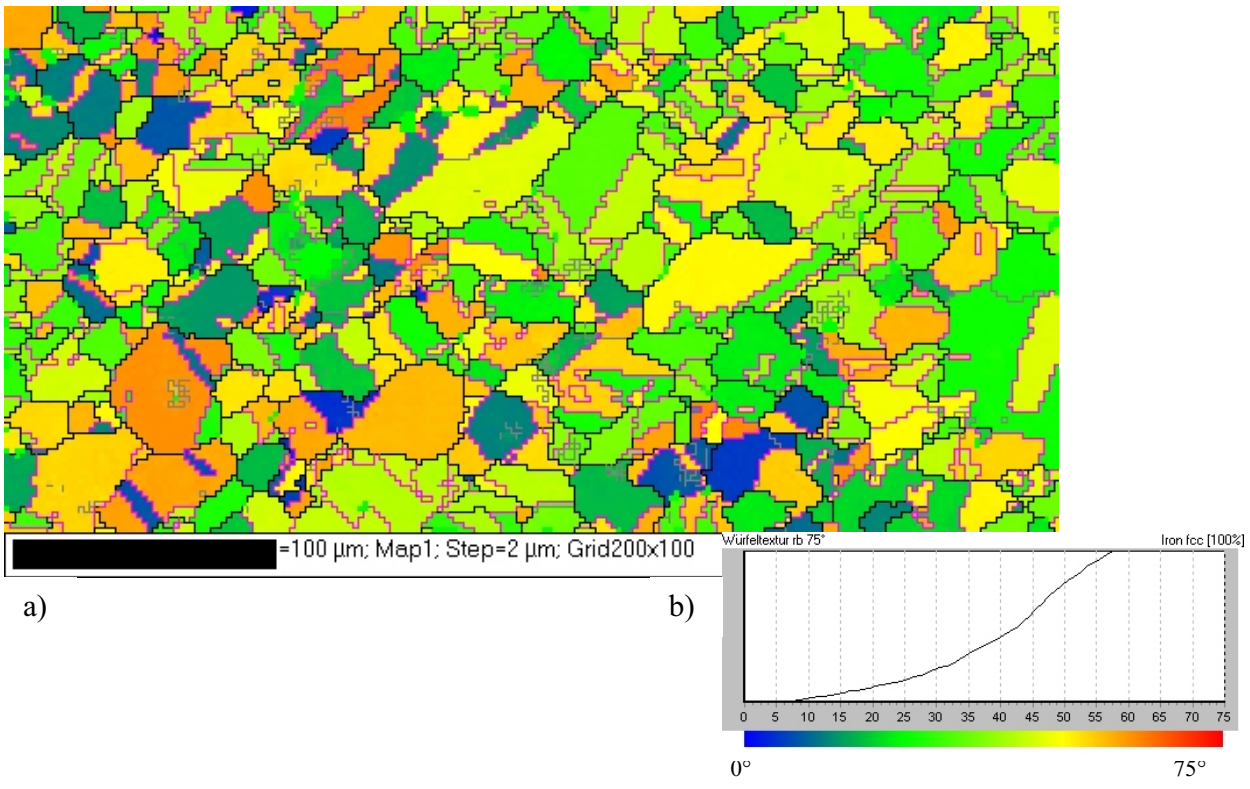

Fig. 5. Orientation map (a) from austenitic stainless steel sample annealed 30 min. at 700°C, with colour coding done with deviation angle from ideal cube texture using a rainbow scale. Low-, high-angle and twin boundaries colour classification are also shown. Distribution of individual grain orientations is illustrated in histogram (b).

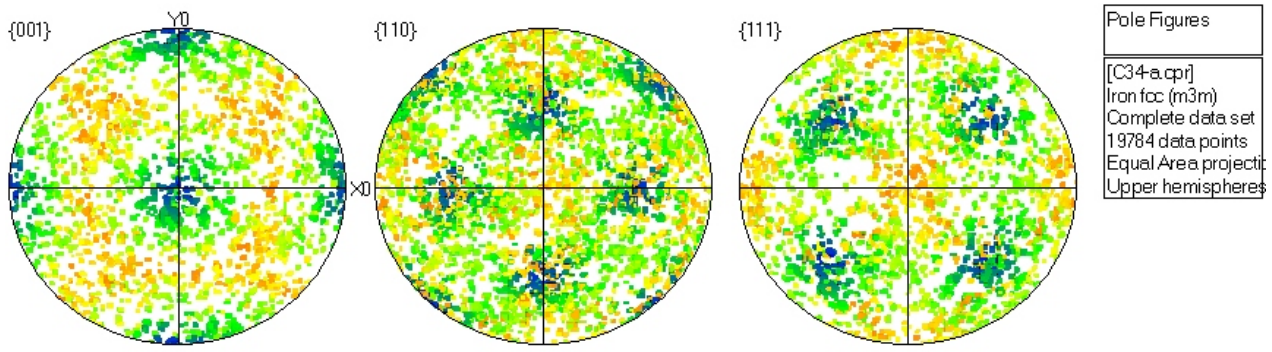

Fig. 6. Examples of discrete data point pole figures from sampled area in Fig. 5

width for individual grain boundary categories, is also possible (Fig.5).

Furthermore, EBSD can be used for grain boundary geometry studies. It is based on fact that neighbouring grains can be identified in the microstructure and selected for orientation measurement. The misorientation between neighbouring grains can then be calculated in terms of axis/angle pair. [18] The result of such misorientation measurement can be plotted into misorientation profile, represented as a function of angle vs. distance along the line between operator selected grain pair.

3.2 Special grain boundary statistics

If the aim of investigation is to analyze ´special´ or so-called CSL boundaries, additional graphical processing of obtained map is necessary. Relating to present CSL boundaries, similar data outputs as in the case of previous example are possible, Fig. 7.

3.3 True grain size

Traditional grain size measurements rely on observation of grain boundaries in an image of the microstructure, which is complicated in materials where the grain interfaces are difficult to observe, in the case of heavily twinned microstructures (fcc and hcp metals) or when are grain boundaries not revealed by conventional etching.

If the distance between sampled points on the surface has been properly chosen with respect to the microstructure, it is possible to make a direct measure of the true grain size and to determine distribution of individual grain sizes on the particular section through the microstructure using EBSD technique. Except mean, minimum or maximum grain size value represents computer routine grain size distribution in the form of histogram.[3]

4. Conclusions

Electron Backscattered Diffraction became very popular among materials sciencists in the last years as a far superior method for the rapid and accurate generation of crystallographic data in standard scanning electron microscope equipped with EBSD detector unit. This technique is valuable by recognition and misorientation analysis of grain boundaries, without any destruction of bulk sample and with minimal operator output using suitable software.

The theoretical basis and main application fields of EBSD in materials engineering were shown. Demonstration of examples from investigation of orientation relationships in nitrogen-added austenitic stainless steel was performed.

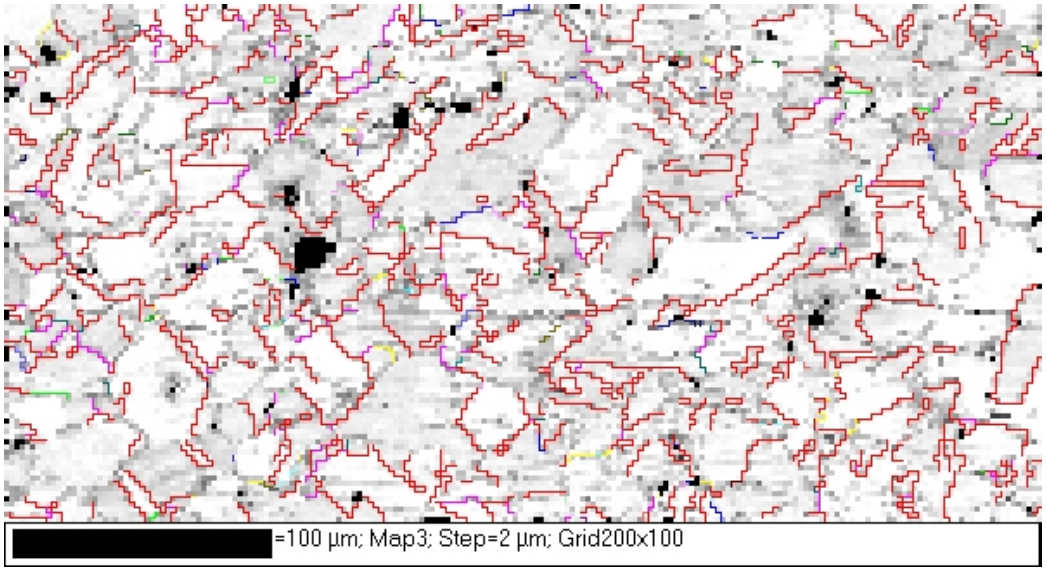

Fig. 7. CSL boundary map from the area in Fig.5, with delineation of chosen families of CSL boundaries.

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