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PRESENTATION METHODS OF TEXTURES MEASUREMENTS

NAČINI PREDSTAVITVE TEKSTURNIH MERITEV

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The microstructure of materials is usually described by size coordinates - xyz, reflecting the size, shape and the arrangement of the microstructural elements and by angular coordinates - $\varphi_1\Phi\varphi_2$, which show the texture. Texture is defined as the orientation distribution of crystallites in a polycrystalline material and results from many kinds of anisotropic solid-state process which can affect the material's anisotropic properties. The texture can either be measured grain-by-grain or by diffraction from the crystalline material, with the majority of all texture studies being performed by X-ray diffraction. The function *f(g)*, which describes the volume fraction of grains with a particular orientation *g* is discussed. In metallic materials the main features of texture are described by using adequate solutions of the three-dimensional orientation distribution function (ODF), which is usually calculated from two-dimensional X-ray pole figures. In the ODF the whole volume fraction of the crystals having a particular orientation is quantitatively described. The interpretation of the full ODF is difficult for a non-specialist and may not always be the most suitable way of presenting the texture; but it does allow the identification of texture fibres, their quantitative analysis, as well as the selective projection and plot of characteristic orientation fibres. Different methods for the presentation oftextures are compared and discussed using the development of *Goss* and *cube* texture in electrical steel alloyed with Sn and Sb as an example.

Key words: texture, orientation distribution function, pole figure, texture fibre, electrical steel

Mikrostrukturo materiala opišemo s koordinatami - *xyz* (velikost, oblika in razporeditev mikrostrukturnih elementov) in s kotnimi koordinatami - $\varphi_1\Phi\varphi_2$ (ki opredeljujejo kristalografsko orientacijo v prostoru, teksturo). Tekstura je orientacijska porazdelitev kristalnih zrn v polikristalnem vzorcu. Pri vseh anizotropnih procesih v trdnem nastane tekstura, ki ima za posledico anizotropijo lastnosti materiala. Teksturo opredelimo z določanjem orientacije posameznih zrn ali z uklonom rentgenske svetlobe na polikristalnem vzorcu. Večina vseh teksturnih meritev je narejena z rentgensko uklonsko metodo (XRD - X-ray diffraction). Opisana je funkcija *f(g)*, ki podaja volumski delež zrn z orientacijo *g*. S presekom tridimenzionalne orientacijske porazdelitvene funkcije (ODF), ki se ponavadi izračuna iz dvodimenzionalnih polovih figur, se lahko v primeru kovinskih materialov jasno in natančno opišejo glavne značilnosti določenega tipa teksture. Celotno ODF-funkcijo je zelo nerodno prikazovati, poleg tega pa je njena interpretacija zelo zahtevna. Omogoča identifikacijo teksture vlaken, kvantitativno analizo teksture, selektivno projekcijo in slike karakterističnih orientacijskih vlaken. Prikazani so različni načini predstavitve tekstur pri razvoju *Gossove* in *kubične* teksture v elektropločevini, legirani s Sn in Sb.

Ključne besede: tekstura, orientacijska porazdelitvena funkcija, polova figura, tekstura vlaken, elektropločevina

1 INTRODUCTION

Over the last decade tremendous progress has been made in texture investigations due to improved measuring techniques and newly developed methods for texture-data evaluation, both of which depend on computer processing[1-6]. For a complete description of a polycrystalline material it is necessary to consider the crystal structure, composition, the size and shape of the grains and their orientation: texture investigation is just an extension of metallography. Accordingly, the definition of the texture is the orientation distribution of crystallites in a polycrystalline aggregate. Like the grain sizes, the orientations are also usually considered in the form of a statistical distribution, i.e. the volume (V) fractions of material containing grains of a certain diameter or orientation (g) (equation 1)[2].

$$\frac{dV/V}{dg} = f^{vol}(g) = f^{vol}\left((hkl)[uvw]\right) = f^{vol}\left(\{\varphi_1\Phi\varphi_2\}\right) \quad (1)$$

The grain orientation (*g*) is specified by the Miller indexes (hkl)[uvw] or by the Euler angles $\{\varphi_1\Phi\varphi_2\}$.

Textures are presented in different forms such as the pole figure[7], the inverse pole figure, or an orientation distribution function in Euler[1,8] or Rodrigues-Frank space[9,10]. With the orientation distribution function (ODF) in equation 1 the equi-level surfaces (or lines) in the chosen orientation space are represented. When a large area of the sample is analysed the macrotexture is obtained. In recent years the rapidly growing fields of texture investigation are in microtexture and mesotexture[11]. **Figure 1** presents schematically the relationship between macrotexture, microtexture and mesotexture.

Texture is determined either with grain-by-grain measurements or by polycrystalline diffraction. The most straightforward method is to measure all the grains in the sample individually. This can be achieved using the etch-pit method[12], TEM Kikuchi patterns, or SEM Kikuchi patterns (electron back-scattered diffraction method - EBSD)[11,13]. The vast majority of texture studies

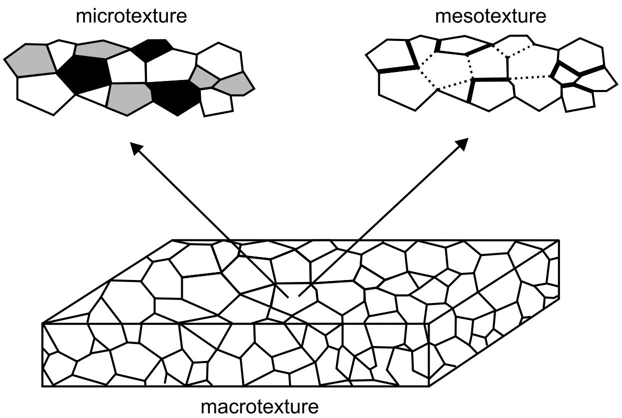

Figure 1: Schematic diagram illustrating the relationship between macrotexture, microtexture and mesotexture. The macrotexture represents the grain orientation of the sample, the microtexture represents the grain orientation of a smaller area, in this case fifteen grains with three different orientation categories and the mesotexture where, for example, three boundary types are indicated

Slika 1: Shematski diagram, ki prikazuje povezavo med makroteksturo, mikroteksturo in mezoteksturo. Makrotekstura pomeni določitev orientacije kristalnih zrn analiziranega vzorca, mikrotekstura pa analizo orientacije manjšega področja, v tem primeru petnajstih zrn, ki predstavljajo tri različne orientacijske kategorije, in mezotekstura, kjer lahko npr. določimo tri različne tipe mej med zrni.

are being made using polycrystalline X-ray diffraction, with neutron diffraction being used for thicker samples. Modern instrumentation allows both the the gathering of data and the preparation of the final pole figure using a computer.

The most frequently used and the most complete way of representing the texture is the orientation density in a three-dimensional orientation space (e.g. Euler angle space) called the orientation distribution function - ODF, which is obtained after suitable mathematical transformation from several pole figures. Using individual sections through the orientation space a good impression of the structure of the orientation distribution is obtained, however, this is usually too abstract and confusing for less-experienced users. The tendency in metallurgy is to describe the texture in a simpler and more comprehensive manner which enables a quantitative evaluation of the texture data. In the literature two such concepts have been proposed and are already in use[5]. The first is to compare the texture with an ideal texture, the areas of the highest orientation density are identified and their texture defined. The second method is to describe the texture by means of texture fibres or orientation tubes, particularly in the case of an axially-symmetric (fibre) type of texture. A three-dimensional orientation distribution function allows a quantitative presentation of the texture fibre.

In case of electrical steels alloyed with Sn and Sb the different choices of texture-measurement presentation are shown and explained with the emphasis on the Goss {110}<001> and cube {100}<001> texture formed in electrical steel during decarburization and recrystallization processes.

2 EXPERIMENTAL

Two different electrical steels were produced in the laboratory with the same concentrations of alloying elements, but with different contents of Sn and Sb in order to study the influence of these two elements on texture development. The textures were measured on samples of size 2.5 x 2.5 x 0.5 mm. A detailed description of the experimental procedure is published elsewhere[14].

The texture was measured using computer-controlled texture diffractometer and MoKα radiation at the Max-Planck Institute for Iron Research in Düsseldorf, Germany. The samples were 0.5 thick and the measured thickness was five times smaller. The samples were electrolytically polished and mounted in a texture goniometer which can rotate in three directions to bring a lattice plane (hkl) into the reflection position for any crystal orientation. The complete ODF functions were calculated from three pole figures (200), (110) and (211). The intensity of the diffracted beam was measured by normal counting methods and normalised to that obtained from a randomly oriented standard specimen.

3 RESULTS AND DISCUSSION

3.1 Macrotexture

3.1.1 Pole figure

A pole figure is a stereographic projection which shows the distribution of a particular crystallographic direction in the assembly of grains that constitutes the specimen (**Figure 2**). The pole figure contains some reference directions that relate to the material e.g. drawing direction (D) in wires or rolling direction (RD) and transverse directions (TD) in rolled sheets. The pole figure can be shown as a number of poles (**Figure 3**) or as lines of the same pole density (**Figure 4**). By using a

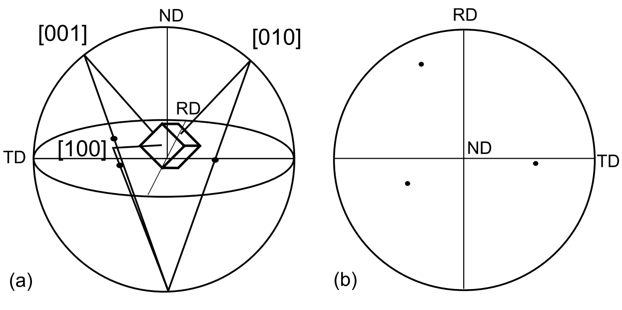

Figure 2: (a) Family of {001} poles in a cubic crystal projected onto the reference sphere of a stereographic projection (b) Pole figure of one crystal

Slika 2: (a) Družina {001} polov v kubičnem kristalu, ki so projecirani na referenčno sfero stereografske projekcije (b) polova figura enega kristala

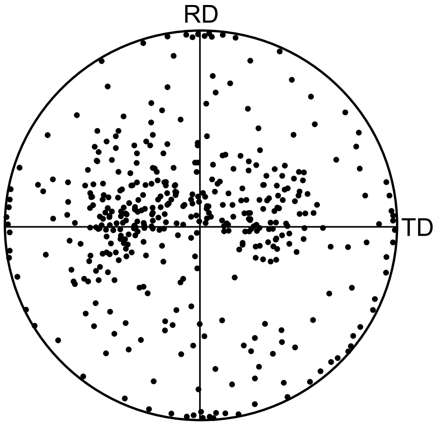

Figure 3: {001} pole figure of electrical steel alloyed with 0.05 %Sb
Slika 3: {001} polova figura elektropločevine, legirane z 0,05 %Sb

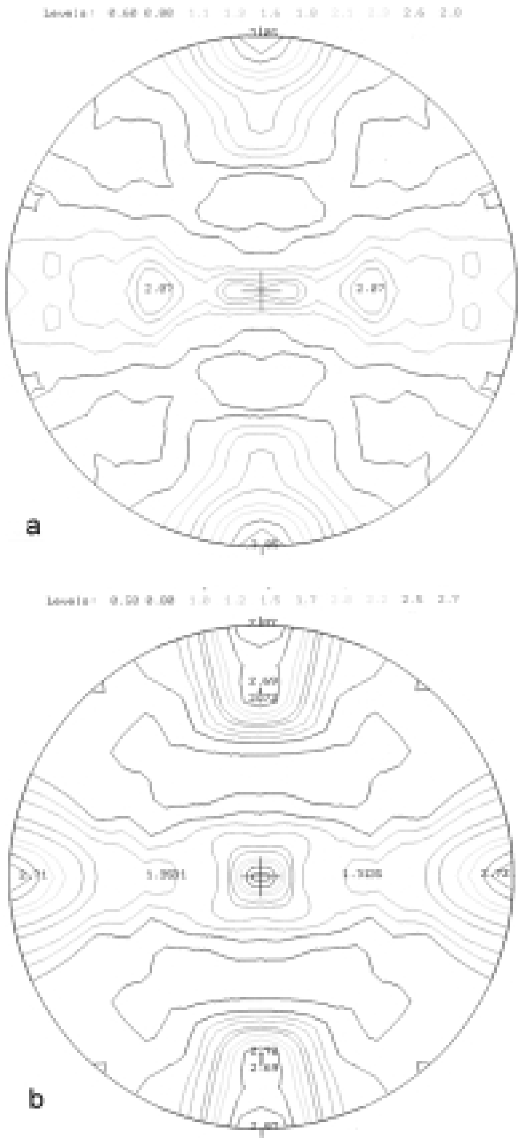

Figure 4: {200} computed pole figures measured on the surface of electrical steel (a) without Sn and (b) with 0.05 %Sn
Slika 4: Polovi figuri {200} po računalniški obdelavi, merjeni na površini vzorca elektropločevine (a) brez kositra in (b) z 0,05 %Sn

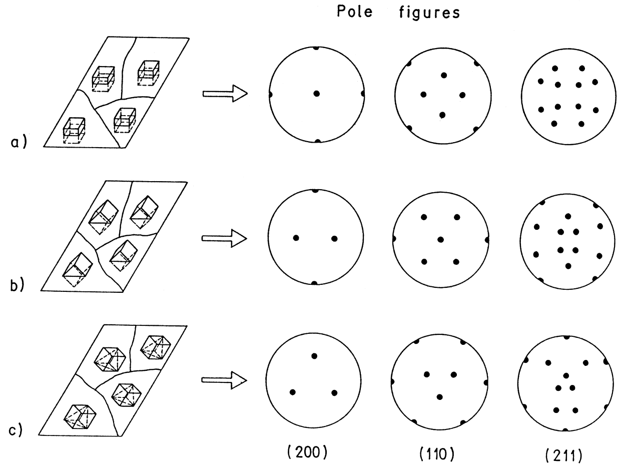

Figure 5: Schematic {200}, {110} and {211} pole figures for steel sheet with idealised textures (a) {100}<001>, (b) {110}<001> in (c) {111}<112>
Slika 5: Shematičen prikaz {200}, {110} in {211} polovih figur za pločevino z idealnimi teksturami (a) {100}<001>, (b) {110}<001> in (c) {111}<112>

model for idealised pole-figure textures {100}<001>, {110}<001> and {111}<112> shown in **Figure 5** some texture information from a real pole figure for electrical steel in **Figure** 4 can now be obtained. During the recrystallization process the cube texsture increased in the steel alloyed by Sn, partially at the expence of the Goss texture.

3.1.2 Inverse pole figure

The inverse pole figure is a single unit triangle of stereographic projection which shows one type of lattice plane normal. The reference axes of the inverse pole figure become the 100, 010 and 001 crystal axes (plane normal). Because the symmetry of the crystal is repeated within an inverse pole figure it is possible to express all the directions (e.g. <100>) with only one unit triangle

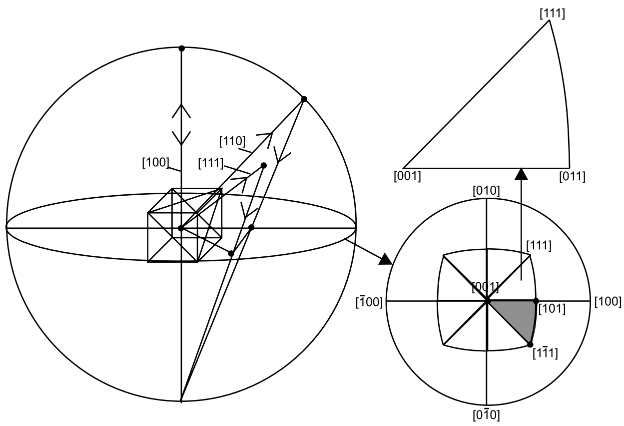

Figure 6: The inverse pole-figure plots direction in the sample related to fixed crystal directions in a single unit triangle of the stereographic projection
Slika 6: Smeri inverzne polove figure v odvisnosti od nekaterih smeri v kristalu. Inverzna polova figura je osnovni trikotnik v stereografski projekciji.

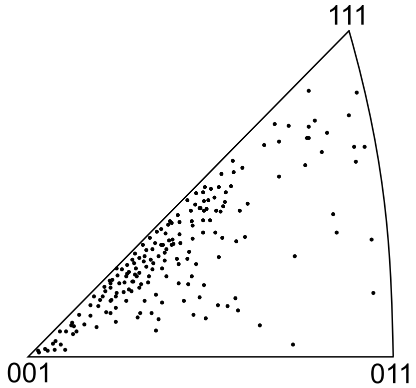

Figure 7: Inverse pole figure of the electrical steel alloyed with 0.05 %Sb
Slika 7: Inverzna polova figura elektropločevine, legirane z 0,05 %Sb

(**Figure 6**). The orientation of one crystal is presented with only one dot. An inverse pole figure is used when the orientation relative to aspects of the specimen's geometry is of particular significance. In **Figure 7** an inverse pole figure of the electrical steel alloyed with Sb is shown. **Figure 8** shows a single unit stereographic triangle divided into 51 fields marked with the closest Miller indexes[12]. The comparison of these two images shows that there are nearly no grains which have planes (111) and (110) parallel to the steel sheet surface. On the other hand, there are a lot of grains whose planes (100), (118), (115) and (227) lie parallel to the steel sheet surface.

3.1.3 Orientation distribution function and Euler space

The description of texture by pole figures is incomplete. The information provided refers only to the statistical distribution of a single direction. For example,

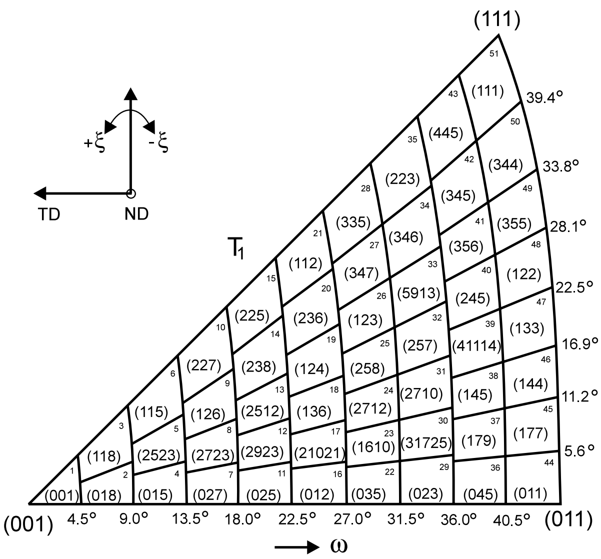

Figure 8: The areas of determined orientations in the unit stereographic triangle
Slika 8: Področja posameznih orientacij v osnovnem stereografskem trikotniku

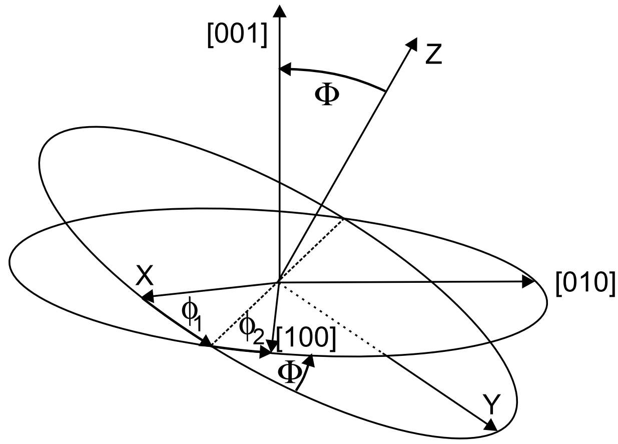

Figure 9: Definition of the Euler angles
Slika 9: Definicija Eulerjevih kotov

the (100) pole figure only gives us the statistical distribution of the volume share of grains with (100) lattice plane parallel to the sample surface. By introducing the Euler space (**Figure 9**) with its three angles, $\varphi_1\Phi\varphi_2$, the orientation distribution function can be calculated from several pole figures. Due to the very inconvenient presentation of the three-dimensional ODF function, usually only the sections through the Euler space at one fixed angle are used. The method is comparable with computer tomography, only it works in orientation space. **Figure 10** shows the sections through the Euler space at the fixed angle φ_1 with typical orientations, and **Figure 11** shows the texture of the electrical steel alloyed with 0.05 %Sn.

The complete ODF function should be presented in three-dimensional space, which demands a very abstract way of thinking. It does, however, the identification of texture fibres and a quantitative description along these fibres. **Figure 12** shows the Euler space with marked α,

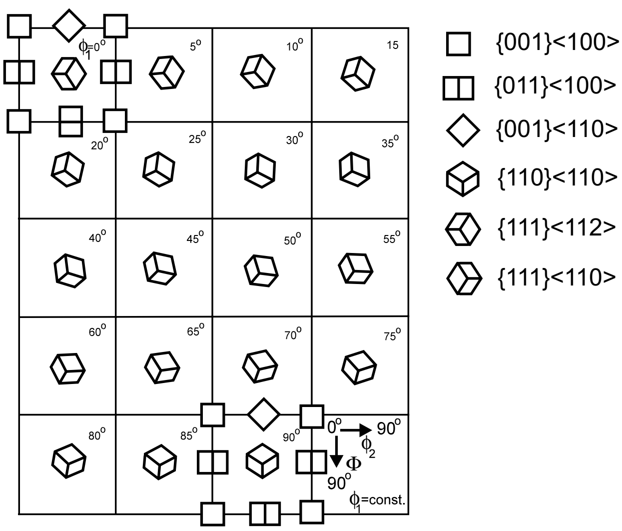

Figure 10: ODF sections showing the location of same orientations
Slika 10: ODF-prerezi s prikazi nekaterih orientacij

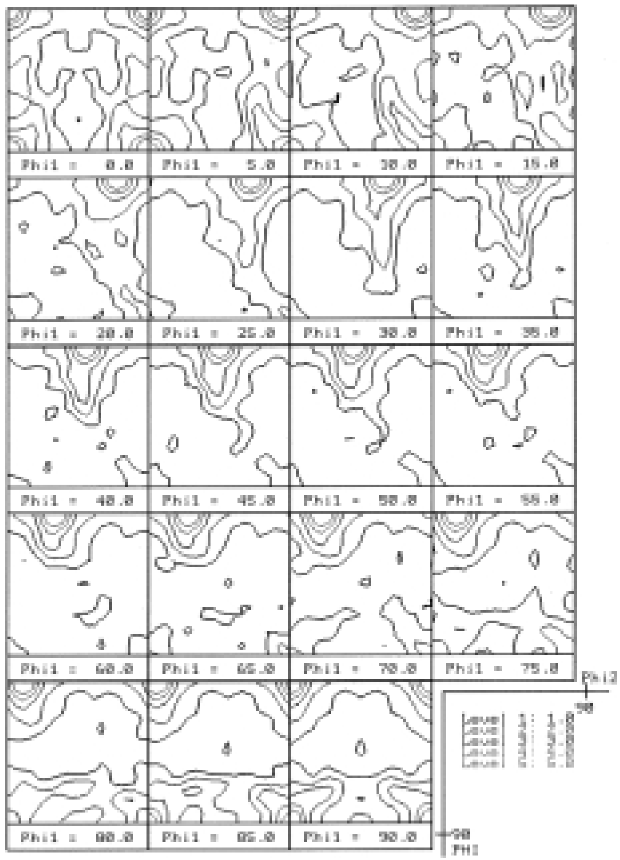

Figure 11: ODF sections at constant angle φ_1 determined on the surface of electrical steel alloyed with 0.05 %Sn

Slika 11: Tekstura elektropločevine, legirane z 0,05 %Sn, prikazana s prerezi skozi ODF pri konstantnem kotu φ_1

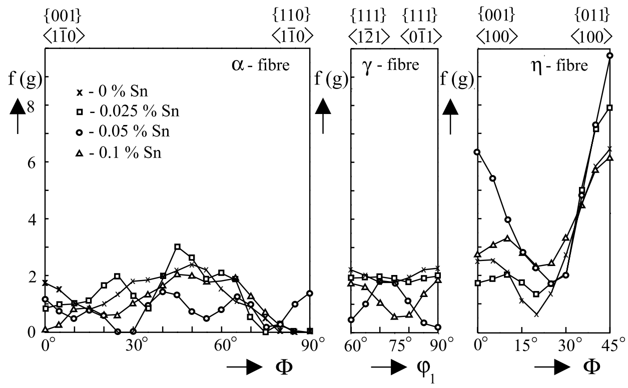

Figure 13: Quantitative description of α, γ and η fibre intensity which allows the direct comparison of a certain orientation density, in this case four electrical steels alloyed with different Sn content

Slika 13: Kvantitativen prikaz intenzitete vlaken α, γ in η. Ta način podajanja teksture omogoča neposredno primerjavo gostote določenih orientacij oziroma primerjavo volumskega deleža določenih orientacij, v tem primeru štirih elektropločevin, legiranih z različnimi vsebnostmi Sn.

γ and η fibres which give some information about Goss, cube and {111}<uvw> textures. In **Figure 13** the fibre texture of electrical steel alloyed with different amounts of Sn is shown. The y axis indicates the orientation density or orientation probability f(g). In the case of a completely isotropic sample, with randomly oriented grains of the same size, f(g) is equal to one. When f(g) is greater than one there are some preferred orientations. An analysis of **Figure 13** suggests that Sn promotes the development of Goss and cube texture and decreases the number of grains with {111}<uvw> texture. The texture analyses was performed in the middle plane of the steel

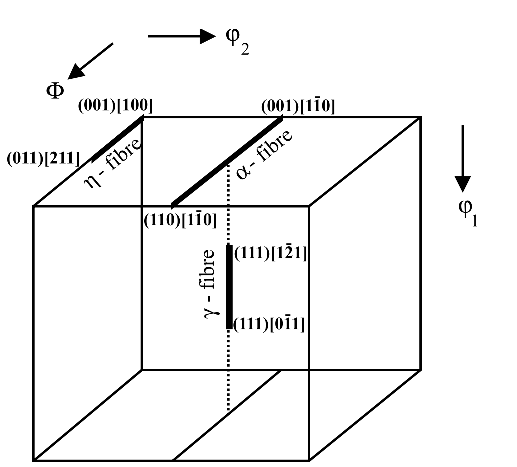

Figure 12: Euler space with marked α, γ and η fibres

Slika 12: Eulerjev prostor z označenimi vlakni α, γ in η

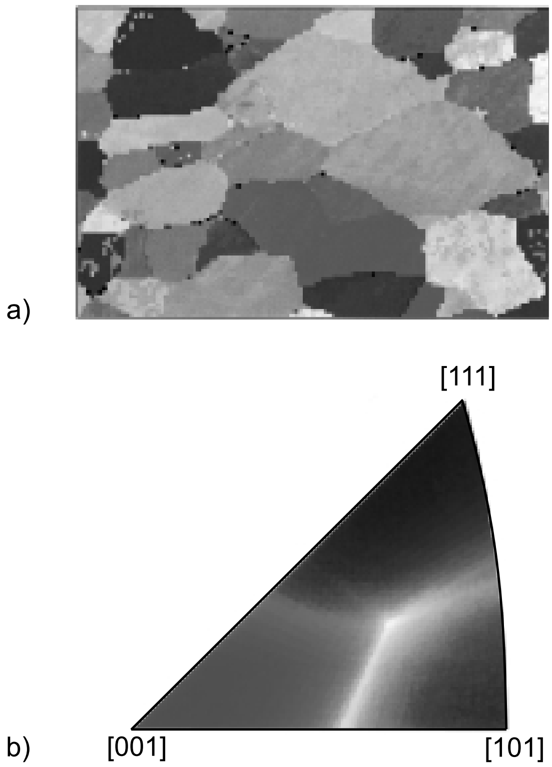

Figure 14: (a) Computerised electron back-scattered image (b) Colour key where certain colour represents the orientation[18]

Slika 14: (a) Računalniško obdelana slika odbitih elektronov, (b) barvni ključ, kjer posamezna barva podaja orientacijo[18]

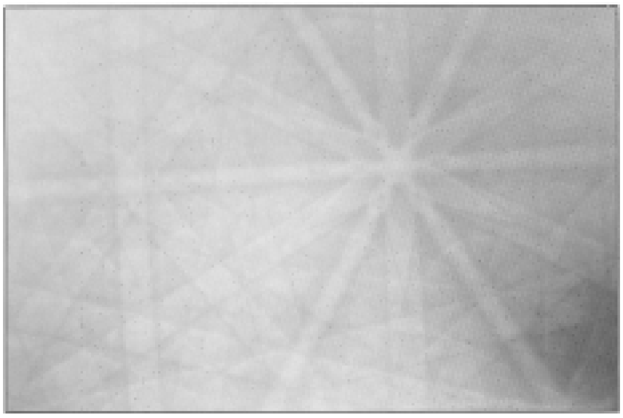

Figure 15: Electron back-scattered pattern (Kikuchi pattern) obtained by interaction of the primary electron-microscope beam with austenite specimen[17]
Slika 15: Slika odbitih elektronov (Kikuchi-slika), ki je nastala pri interakciji primarnega elektronskega pramena v elektronskem mikroskopu z vzorcem avstenita[17]

sheet. The results also show that the Sn grain-boundary segregation somehow influences the texture development during the recrystallization.

3.2 Microtexture

3.2.1 Crystal Orientation Mapping

The microtexture can be determined by different methods, providing a measurement of a certain grain orientation. When a large number of grain orientations is measured using a sophisticated fully automated specimen stage it is possible to construct the ODF function[15]. To determine the surface distribution of a smaller area a SEM Kikuchi method (EBSD) is most frequently used. In the EBSD method the back-scattered electron from the tilted specimen forms the diffraction Kikuchi patterns (**Figure 15**), which are transferred to a phosphor-coated glass screen, digitised and analysed by comparing with low-index Kikuchi patterns. In this way, the grain orientation distribution can be clearly demonstrated (**Figure 14** (**a**)). The crystal grains are coloured depending on the position of the inverse pole figures (**Figure 14** (**b**)).

3.3. Mesotexture

3.3.1. Coincidence grain boundary

The grain orientation is usually expressed relative to reference axes which are related to the specimen geometry. In the case of mesotexture the axes of one crystal are compared with the neighbouring grain. This is done only when the orientation of one grain with respect to the other is important and the type of grain boundary is of interest. The coincidence site lattice (CSL) is a model which has been widely adopted for the classification of grain-boundary geometry, especially in cubic material. Two neighbouring grains which are differently oriented in one direction have, at a determined angle, an identical lattice site and a coincidence grain boundary[16]. These boundaries are marked with an Σ value. The smaller Σ value means better site-lattice coincidence. The results of such texture measurements are shown as a grain-boundary skeleton with differently coloured grain boundaries or as a distribution diagram of Σ boundaries.

4 CONCLUSIONS

Texture is one of the fundamental structural parameters in all polycrystalline materials including metals, ceramics and polymers. Textures are formed and transformed by different solid-state processes, such as crystallization, recrystallization, plastic deformation, grain growth, phase transformation etc. For the complete description of a material, in addition to metallographic parameters, such as the size, shape and the distributions of grains, their orientation must also be considered. A great deal of progress has been made in texture measurements especially in the field of quantitative texture-data evaluation. The future development of new materials with more sophisticated crystal structures will mean that texture measurements will become increasingly important.

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