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NUCLEATION DURING PRIMARY RECRYSTALLIZATION OF RGO ELECTRICAL STEEL SHEET OBSERVED BY THE EBSP-METHOD

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ABSTRACT

RGO electrical steel with low power losses and high permeability to be used in power transformers obtains its superior magnetic properties by a sharp Goss-texture developed by secondary recrystallization. RGO is produced by a two stage cold rolling process with intermediate annealing and subsequent primary recrystallization. By ODF analysis a high Goss intensity after primary recrystallization was empirically proved to be advantageous for the development of a sharp final Goss-texture in the following secondary recrystallization. This, in turn, requires a better understanding of how to achieve the beneficial primary Goss-texture formed by the basic processes of nucleation and growth. In the present study the early state of recrystallization was investigated. Only the EBSP-method (electron back scattering pattern), recently developed by Dingley, provides a sufficient submicron spatial resolution to measure the orientations of the early nuclei together with their cold worked and recovered environment.

1. INTRODUCTION

Except for "in situ"- and for "dynamical" recrystallization, the recrystallization of cold worked steels leads to textures being completely different from the preceeding cold worked texture /1-3/. Mainly two different models aim to describe the development of this recrystallization texture, called "oriented nucleation" and "selective growth".

The model of "oriented nucleation" is supported by results where, after the onset of recrystallization, nuclei with orientations being typical for the recrystallization texture were found very close to so called "transition bands" or "shear bands" /1, 2/. On the other hand experiments on deformed Fe3%Si single crystals being artificially nucleated at one end demonstrated the mechanism of growth selection between the variously oriented nuclei and for the fastest growing nucleus an orientation relationship to the deformed matrix characterized by a 27° <110> rotation.

In the following an experiment is presented which resulted in proving the selective growth mechanism for primary recrystallization in grain oriented silicon steel. Starting from cold worked sheet an early state of recrystallization was prepared by quenching after short time heating. Single grain orientations were measured in the SEM with the EBSP-method for the newly formed grains in a very early state. By giving a Gauss-type scattering to each measuring point, a continuous "nucleation texture " could be calculated, which was compared to the deformation texture and to that texture having developed at completion of recrystallization.

2. EXPERIMENTAL

2.1. Specimen preparation

The material used for the present investigation contained the following amounts of alloying elements in weight percent: Si: 3,16% / C: 0,029% / Mn:0,061% / S: 0,02% / P: 0,008% / N: 0,002% / + traces. The hot rolled strip of 2 mm thickness is "hot strip annealed" in a dry gas atmosphere at 1030°C for 150 s and then water-quenched and pickled. This is followed by a first cold rolling to the intermediate thickness of 0.65 mm, by a recrystallization annealing at 980°C for 180 s and by second cold rolling to the final thickness of 0.26 mm. There are three types of materials on which texture investigations were carried out:

a) Material "*coldroll*" is the starting material obtained by the above procedure.

b) Material "*fullrec*" is obtained from "*coldroll*" by heat treatment at 720°C in saltbath for 180 s which leads to complete recrystallization.

c) Material "*nuc*" is obtained from "*coldroll*" by heat treatment at 720°C but for the much shorter time of about 2 to 4 s in order to generate nuclei.

On the transverse sections (defined by sheet plane normal and rolling direction) of the annealed specimens a Nital etching was performed in order to evaluate the recrystallized portion in the SEM. For further investigations specimens having only few nuclei were selected, whereas those without any nuclei or with too many recrystallized grains already being in contact with each other were disposed of. Transverse sections were polished to a very fine grid of 0.5 µm and additionally sputtered with neutral argon atoms in order to avoid any structure on the surface. In this way accurately flat surfaces as required for EBSP-analysis were obtained. For materials "*coldroll*" and "*fullrec*", also flat specimens for X-ray texture measurements were cut out of the sheets.

2.2 Texture Analysis

For specimens "*coldroll*" and "*fullrec*" (200)-, (110)-, (211)- and (103)- pole figures were determined by an X-ray goniometer /5/. These were symmetrized and ODF's were calculated from this data by the series expansion method /6/ ($l_{max} = 22$).

At specimen "*nuc*" local single orientations were determined in the SEM, applying the EBSP-technique recently developed by Venables and Dingley /7, 8/. 203 orientation were measured for individual isolated nuclei distributed over the cold worked matrix. From the so obtained individual orientations a continuous ODF was calculated by superpositioning of the Gaussian scattering functions one for each measured orientation with a half width of 10° providing even and odd coefficients for true ODF - calculation. All ODF's are plotted in $\varphi_1 =$ constant sections through the Euler angle space $\{\varphi_1, \Phi, \varphi_2\}$.

2.3 The EBSP-method

Here in a modified SEM (JEOL 840) a stationary electron beam hits the highly tilted (70°) specimen sketching the backscattered electron image on a transparent phosphor screen. This backscattering electron image consists of sets of crossing lines due to the channeling contrast /7/. This image is detectable through a lead glass window by a low light TV-camera. After contrast improvement the backscattered image is displayed on a TV-monitor. The orientation of specific local areas can now be determined by positioning a cursor on each of two different zone axes of the diffraction pattern on the monitor in order to calculate the orientation by a microcomputer /8/.

Prior to orientation determination the investigation area was selected applying a special backscattering electron detector with a low aperture being highly sensitive for orientation contrast while the SEM is operating in the scanning mode. This orientation contrast image is rather weak due to a long working distance at a 70° tilted specimen stage. Therefore a 20 times integration and storing of the backscattering electron image at an image analyzer was used to significantly improve the contrast. After switching the SEM to spot mode the electron beam could be moved to different places inside the stored image, a stably operating SEM provided. Being controlled by the image analyzer computer the beam positioning was discrete. At a model material the spatial resolution could be proved to be better than 0,3 μm by crossing a grain boundary. Thus the local textures of very small nuclei and even of subgrain areas in a deformed matrix could be investigated /9/ but will be reported elsewhere /10/.

Because of this high spatial resolution sometimes it was not easy to distinguish between cold worked and recrystallized matrix from stationary pattern on the EBSP monitor. Therefore an infinitesimal beam movement was performed by shifting the cursor around the aimed position. A changing of the monitor image pattern indicates deformed matrix whereas stationary pattern ensures a recrystallized nucleus to be observed.

3. RESULTS

a) Cold work and recrystallization texture

These textures are shown in Figs. 1 and 2 as ODF's. The cold rolling texture (specimen "*coldroll*"), being typical for sheet steel /3/, mainly consists of a complete gamma-fibre ({111}<110>...{111}<112>, i.e. <111> ‖ ND) collecting the most important stable rolling orientations /10/. The recrystallization texture (specimen "*fullrec*"), being typical for those of RGO decarburized sheets, mainly consists of an eta-fibre ({001}<100>...{011}<100>, i.e. <100> ‖ RD) collecting all orientations from Cube to Goss and of some traces of the gamma-fibre /11/.

b) Nucleation texture

Fig. 3 presents the ODF calculated from the single orientation data of 203 isolated nuclei being distributed over the specimens cross section. This nucleation texture, although otherwise very weak, has a maximum very close to {111}<112>, i.e. to the maximum of the rolling texture located on the gamma-fibre (Fig. 1). The nucleation texture as a whole is completely different from the texture in Fig. 2 obtained after completion of primary recrystallization.

c) Environment of the nuclei

The early stage nuclei without exception were situated along the grain boundaries. At no other possible nucleation site such as transition bands /12/ or shear bands /2/ recrystallization nuclei were actually observed. (Also no "matching planes" reported to occur after the onset of secondary recrystallization /13/ could be found in the present investigation.) Between the nucleus' orientation and that of its direct environment always large angle orientation changes were found, but no pronounced orientation relationships were detectable. By scanning along the rolling and normal direction orientation changes of $\approx \pm 12°$ inside the deformed matrix grains could be observed.

4. DISCUSSION

The intensity maximum of the gamma-fibre of the nucleation texture (Fig.3) is very close to the major cold work texture (Fig.1) component and thus is assumed to be formed by "in situ" recrystallization. This is plausible also since the deformed matrix regions in gamma-fibre orientations (i.e. near $<111> \parallel$ ND) possess the highest amount of stored energy. Since all orientations contributing to the nucleation texture are measured on recrystallized grains isolated in the cold worked matrix and of less than 1 μm diameter, growth selection processes at that early state can be neglected. The $\approx <111> \parallel$ ND - oriented nuclei can thus be interpreted as being evolved by oriented nucleation in retaining their former orientation. The other nuclei are almost randomly distributed and some weak maxima are probably due to fluctuations resulting from limited statistics. In particular the weak maxima near cube and Goss must be decreased in their levels because of their high multiplicity due to their symmetry /14/. Thus the nucleation texture can be interpreted as a largely random texture resulting from heterogeneous nucleation at the grain boundaries superimposed by an oriented nucleation due to "in situ" recrystallisation of orientations near $\{111\}<112>$.

Since the final recrystallization texture (Fig. 2) is very different from the nucleation texture, it cannot be formed by oriented nucleation. Thus a selective growth mechanism must be responsible for the development of the recrystallization texture. During this selection process the $<111> \parallel$ ND components being the maxima of the nucleation texture decrease while those with $<100> \parallel$ RD increase. During this process apparently Goss oriented nuclei are preferred to grow into $\{111\}<112>$ regions which is in agreement with the well known growth relationship 27°-rotation around $<110>$ /4/. Therefore during recrystallization the nuclei of the Goss-component may grow stronger the higher the component $\{111\}<112>$ in the deformed state.

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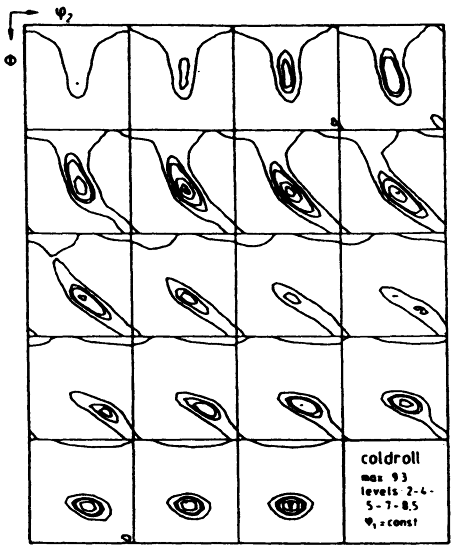

Fig. 1 Cold rolling texture

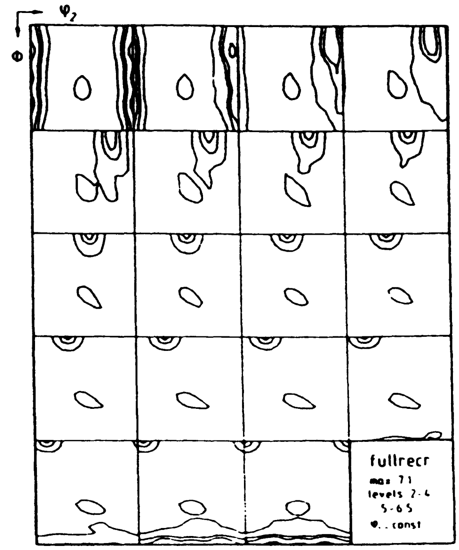

Fig. 2 Recrystallization texture

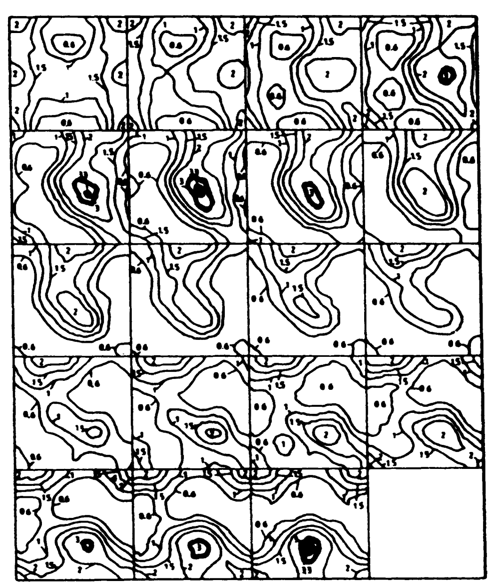

Fig. 3 Nucleation texture as superposition of Gaussian scattering functions of 203 measured single orientations