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ANALYSIS OF MICROSTRUCTURE AND MICROTEXTURE IN GRAIN-ORIENTED ELECTRICAL STEEL (GOES) DURING MANUFACTURING PROCESS

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The final Goss texture in grain-oriented electrical steels (GOES) is affected by microstructure evolution and inheritance during the whole production process. This paper presents the results of detailed microtexture and microstructure investigations on GOES after the basic steps of the industrial AlN + Cu manufacturing process: hot rolling, first cold rolling + decarburization annealing, second cold rolling and final high temperature annealing. Microstructure studies showed that a copper addition to GOES affected solubility of sulphides. Copper rich sulphides dissolved during hot rolling and re-precipitated during decarburization annealing. An intensive precipitation of AlN and Si_3N_4 took place during decarburization annealing. No ε - Cu precipitation was detected. After high temperature annealing the misorientation of individual grains reached up to 8°.

Key words: GOES, microtexture, precipitation processes, Electron backscatter diffraction, Transmission electron microscopy

INTRODUCTION

Magnetic properties (easy magnetization, low hysteresis loss and low eddy current losses) of GOES depend heavily on the sharpness of the Goss texture ({110}<001>). It is agreed that the perfection of the Goss texture in final GOES sheets is closely affected by structure evolution and inheritance during the whole manufacturing process [1]. The X-ray diffraction results showed that Goss-oriented areas first occurred during the initial hot rolling as a friction induced shear texture close to the strip surfaces [2]. It is believed that these grains play, apart from inhibition minor phases, a significant role in the secondary recrystallization process [3, 4].

The conditions that are necessary for the growth of grains with the Goss orientation are provided by microstructural control. Particles of inhibition phases (MnS or AlN, depending on the technology) limit normal grain growth after the primary recrystallization. Coarsening and dissolution of these particles during the later stage of a high temperature annealing (HTA) create prerequisites for the growth of Goss grains. Furthermore, the texture components, which origin or are inherited during the individual manufacturing steps, eg. {111}<112>, have a great significance in terms of the sharpness of the final {110}<001> texture [3]. It was proposed that the strong Goss texture evolved from the Goss grains existing near the sheet surface. Despite long-term research activities there is still no generally accepted explanation of conditions determining formation of the Goss texture [4]. Understanding of the texture selection mechanism in the abnormal grain growth is important for the quality of final GOES sheets. Factors that are agreed to be important include the initial Goss grains size, the misorientation of these grains in relation to the neighbouring grains or significant texture components and inhibition effects of minor phase particles [3].

EXPERIMENTAL MATERIALS AND PROCEDURES

Chemical composition of a hot strip is shown in Table 1. The hot strip was industrially processed by an AlN + Cu production technology, where the first cold rolling was followed by the decarburization annealing (DCA), then a second cold rolling was applied and finally a high temperature annealing created the desired Goss texture [3].

Hot rolling was carried out at 1 250 °C to the thickness of 2,00 mm, After pickling, the first cold rolling to mid-thickness of 0,6 – 0,65 mm was applied and it was followed by decarburization annealing (DCA) at the temperature of 820u°C in the atmosphere containing N_2 + 20 %H_2. Carbon content of the steel after DCA was reduced to 0,003 wt. %. After the second cold rolling to the final thickness of 0,28 mm, a slow heating up to 1 200 °C was applied (HTA).

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Table 1 **Chemical composition of hot strip / wt. %**

C	Mn	Si	P	S
0,026	0,25	3,16	0,007	0,004
Cu	Al_{total}	N_2	Ti+Nb+V	
0,5	0,016	0,0088	0,009	

Cross sections parallel to the rolling direction (RD), comprising the whole thickness of strips/sheets, were studied. Metallographic samples were prepared by mechanical grinding and polishing. The final step of preparation was polishing on colloidal silica (grain size 0,04 µm). Microtexture analysis was carried out using Electron backscatter diffraction (EBSD) in a scanning electron microscope FEI QUANTA FEG 450. The software OIM (EDAX/TSL) was used for indexing of Kikuchi diffraction patterns and for evaluation of the orientation data. EBSD data were also employed for the grain size evaluation. Precipitation processes were studied on carbon extraction replicas in a transmission electron microscope JEM 2100. Electron diffraction and Energy-dispersive X-ray spectroscopy (EDX) techniques were applied for identification of minor phases.

MICROTEXTURE EVALUATION

Hot rolling of GOES slabs is carried out in two phase $\alpha + \gamma$ region. Trans-crystallization processes $\alpha \rightarrow \gamma \rightarrow \alpha$ affect the deformation texture. Redistribution of solutes between the coexisting phases supports heterogeneity of precipitation processes during the next steps of GOES processing. Figure 1a shows recrystallized and deformed fractions of grains in the hot strip. Grains near the sheet surfaces are recrystallized. Some elongated grains in the middle thickness remain deformed (misorientation inside grains is greater than 2° - Figure 1b). Thin bands of small ferritic grains parallel to the RD correspond to the areas which were austenitic during hot rolling. The average grain size in the middle thickness was larger than that under upper and bottom surfaces. Figure 2a highlights grains exhibiting a deviation from the Goss orientation less than 15°. A small number of such grains was scattered across the whole thickness of the hot strip. Grains with zero deviation from the ideal Goss texture were not found.

DCA after the first cold rolling resulted in a significant refinement of ferrite grains. Grains were equiaxed, with a small variation in size. Results of grain size evaluation through the thickness of samples investigated are summarized in Table 2. Figure 2 highlights grains with orientation close to the Goss in the sample after DCA. These grains are scattered through the whole thickness of the mid-thickness sample, some of them have a very small deviation from the exact Goss orientation.

The second cold rolling resulted in formation of heavily deformed grains elongated along the RD. Figure 3 shows microstructure through the thickness of the sheet after the second cold rolling. The microstructure

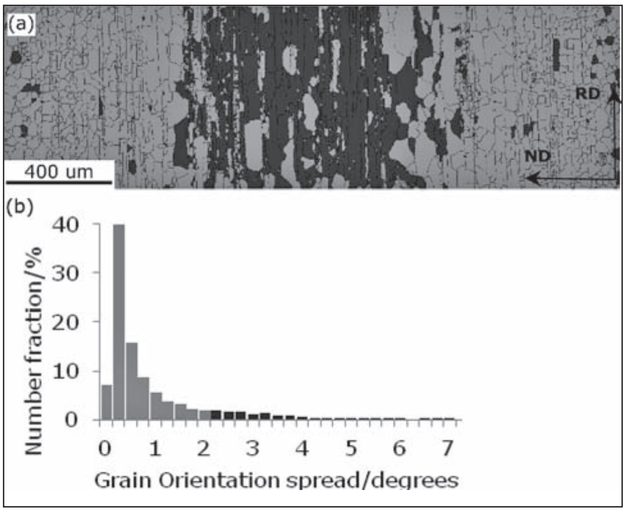

Figure 1 The cross section through the thickness of the hot strip parallel to the rolling direction: a) deformed and recrystallized fractions, b) grain orientation spread histogram

Table 2 **Results of EBSD grain size evaluation/ µm**

Sample	Grain size*
Hot strip	14,2 ±15,8
After DCA	8,3 ± 5,4

* Equivalent diameters ± standard deviations

consists of deformed ferrite grains (misorientation inside grains is greater than 2°) with a high density of shear bands. The fraction of low angle grain boundaries ($\theta < 15°$) was 20 %, most grains were separated by high angle grain boundaries ($\theta > 15°$).

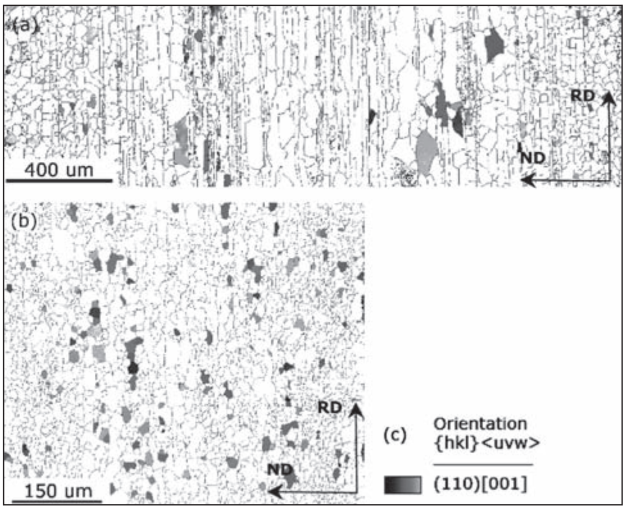

Figure 2 Discrimination of grains close to the Goss orientation + high angle grain boundaries: a) after hot rolling, b) after first cold rolling + DCA, c) the legend

The secondary recrystallization during the final HTA led to formation of grains up to about 15 mm in size. Figures 4a and 4b show the pole figures {100} and {110}. The intensity distribution in pole figures proves that the Goss texture is present. Large grains made it difficult to obtain good statistics of EBSD results on the sharpness of the Goss texture. The misorientation among individual grains reached up to 8°.

TEM INVESTIGATIONS

The role of a copper addition to GOES has not been fully understood yet. The following mechanisms have been proposed [3]:

- copper stabilizes austenite during hot rolling,
- precipitation of ε - Cu could positively affect distribution of AlN particles,
- precipitation of Cu_2S or complex sulphides of copper and manganese,
- segregation of copper atoms at grain boundaries,
- copper supports deformation by twinning and shear.

Precipitation processes play a crucial role in the development of the preferred orientation in GOES. Interaction of minor phase particles with defects of crystal lattice and grain boundaries affect recovery and recrystallization processes [5]. AlN is regarded as the most important inhibition phase in the AlN + Cu technology.

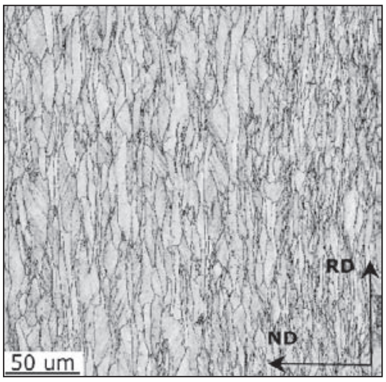

Figure 3 Image quality of the cross section through the thickness of the sheet parallel to RD, sample after the second cold rolling

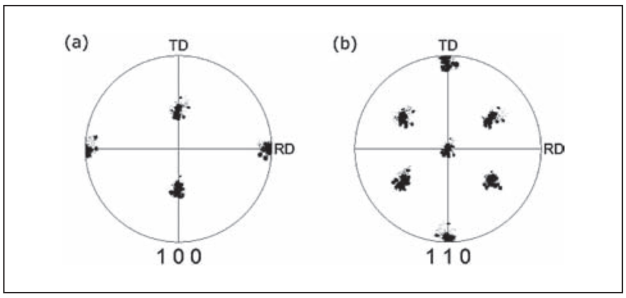

Figure 4 Characterisation of the Goss texture in the final sheet: a) pole figure {100}, b) pole figure {110}

In the hot rolled strip ferrite grain boundaries were decorated by cementite, which formed during coiling, Figure 5. Inside grains a very low number density of particles was observed. The following minor phases were identified: sulphides of manganese, complex sulphides of manganese and copper (up to 10 at. % Cu), AlN and TiN. The size of these precipitates reached up to several hundreds of nanometres. Copper rich sulphides dissolved during hot rolling (T_{sol} = ca 950 °C).

DCA at 820 °C was accompanied by very intensive precipitation processes. Re-precipitation of Cu_2S and complex sulphides of manganese and copper took place. The size of most these particles was less than 50 nm. In many cases nucleation of AlN on the surface of sulphides was observed. Two nitrogen-bearing minor phases were identified along ferritic grain boundaries: Si_3N_4 and AlN. Si_3N_4 phase dissolved some manganese (Si : Mn = 5 : 1), This metastable nitride should gradually transform to AlN phase [3]. EDX studies revealed particles with a variable ratio of Al and Si. These particles could represent a transient state. Electron diffraction experiments proved hexagonal unit cell and lattice parameters close to AlN in such particles. Typical AlN particles contained approx. (5 – 10) wt. % Si and some manganese. Intragranular precipitation of AlN was very intensive and heterogeneous, see Figure 6. Local differences in density of precipitation are probably a consequence of hot rolling in two phase region where some enrichment of austenite in carbon and nitrogen takes place. The typical size of nitrides reached several tens of nanometres. TiN particles were not affected by DCA. No ε - Cu particles were detected.

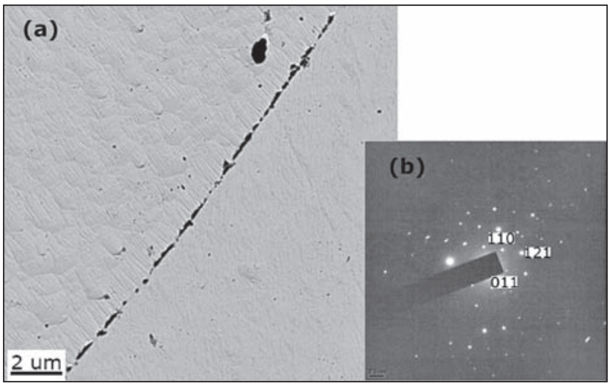

Figure 5 Precipitation in the hot strip: a) cementite particles at the grain boundary, b) SAED pattern of cementite, zone axis [-11-1]

After the second cold rolling the same minor phases were identified as in the sheet after DCA. High density of crystallographic defects in the matrix accelerated additional precipitation and the growth rate of precipitates during slow heating to the temperature of primary recrystallization at the beginning of HTA.

After HTA all nitrides and sulphides were dissolved, except for TiN. This phase is stable up to the temperature of 1300 °C.

CONCLUSIONS

Microstructure of the hot rolled strip consisted of slightly elongated ferrite grains. Grains close to the Goss orientation were scattered across the whole thickness of the strip. Hot rolling at 1 250 °C was accompanied by dissolution of copper rich sulphides.

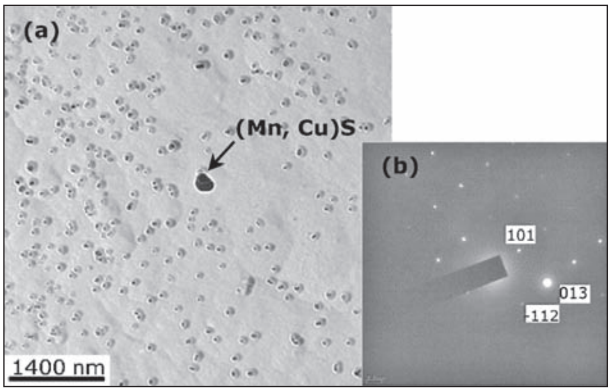

Figure 6 Intensive precipitation after DCA: a) AlN particles, b) SAED pattern of AlN, zone axis [13-1]

Cold rolling + DCA resulted in a pronounced refinement of the ferrite grain size across the sheet thickness and in an intensive precipitation of sulphides and nitrides. The typical size of nitrides reached several tens of nanometres. Copper rich sulphides re-precipitated from the matrix. No ε - Cu precipitation was detected. Grains close to the Goss texture were scattered across the whole thickness of the sheet.

The second cold rolling resulted in a high density of crystallographic defects in ferrite. These defects accelerated additional precipitation and the growth rate of minor phases at the beginning of HTA. HTA resulted in the formation of the Goss texture. Misorientation of individual grains in the final sheet reached up to 8°.

Acknowledgements

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Note: The translator for the English language is Boris Škandera