Formation and decomposition of Widmanstätten austenite in GOES belt-casted strips

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Abstract. This contribution deals with the formation and decomposition of Widmanstätten austenite during solidification of the thin belt-casted strips made of a grain oriented electrical steel (GOES). Solidification of liquid steel started with the formation of δ-ferrite. Cooling in the δ + γ phase field resulted in the formation of a small fraction of Widmanstätten austenite by displacive mechanism accompanied by carbon partition. Widmanstätten austenite laths formed flat low energy interface facets along ferrite grain boundaries. In order to minimize the interfacial energy, ferrite grain boundaries in the vicinity of flat austenite/ferrite facets migrated. It resulted in the formation of either straight or zig-zag ferrite boundaries. Intensive precipitation of sulphides along ferrite/austenite interfaces made it possible to study the early stages of austenite decomposition at the end of the δ + γ phase field. Complex sulphides containing chromium, manganese, iron and copper were identified as the Cr₂CuS₄ phase. Decomposition of austenite started with the formation of epitaxial ferrite. This was accompanied by further partitioning of carbon into remaining austenite. The growth of epitaxial ferrite into the flat ferrite/austenite interface facets along ferrite grain boundaries resulted in a wavy shape of these boundaries. Finally, remaining carbon rich austenite transformed either to pearlite or plate martensite.

1. Introduction

Strip casting is an efficient and economical technology for production of thin steel sheets [1]. It reduces the total hot rolling reduction during subsequent processing of as–cast strips. Recent progress in strip casting makes it possible to apply this technology for the production of a grain oriented electrical steel (GOES).

Recent results have demonstrated that the microstructure of belt-casted GOES strips can consist of ferrite, pearlite and a mixture of plate martensite and residual austenite [2-4]. GOES strips firstly solidify as δ-ferrite, and during subsequent cooling in the δ + γ field a small fraction of austenite and several minor phases precipitate in the parent phase. Austenite is a harder phase than ferrite and δ→γ transformation is accompanied by a volume shrinkage [3]. Austenite in GOES strips usually forms Widmanstätten-like patterns and its dominant morphology can be defined as laths [3]. At the end of the δ + γ field austenite decomposes. Products of austenite decomposition depend on the cooling rate,
chemical composition and size of Widmanstätten austenite laths. Minor phases play an important role in the production of the final Goss texture in GOES sheets [5]. Intensive precipitation of minor phases in the δ-ferrite makes it possible to study the beginning of austenite decomposition [4].

Formation mechanisms, morphology and crystallography of Widmanstätten ferrite in steels have been studied extensively but less is known about Widmanstätten austenite [6,7]. This paper deals with studies on the formation and decomposition of Widmanstätten austenite in the GOES belt-casted strip. The basic goal is a better understanding of the microstructure evolution in GOES strips produced by advanced strip casting technology.

2. Material and experimental procedures
The thin strip was manufactured at TU Clausthal using the horizontal belt casting process [1]. In this case, liquid steel flows from a ladle into a tundish system and is dispensed onto a water–cooled belt. The top surface of the solidifying strip is protected by argon. The cooling rate varies across the thickness of the strip. The calculated cooling rate on the top surface of the strip in the temperature range from 1573 K to 973 K was about 2 K s⁻¹. The width and thickness of the belt-casted strip was 0.30 m and 0.014 m, respectively. Chemical composition of the strip is shown in Table 1.

| Table 1. Chemical composition of the cast investigated, mass %. |
|-----------------|------|------|---|---|---|---|
| C   | Si  | Mn  | S  | Cr | Cu | Al |
| 0.034 | 2.81 | 0.06 | 0.024 | 0.20 | 0.15 | 0.002 |

Microstructure investigations were carried out on longitudinal sections in the middle width, across the whole thickness of the strip. Evolution of the microstructure during cooling of the solidified strip was studied using a combination of light microscopy (LM), X-ray diffraction (XRD), scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), X-ray microanalysis (EDX), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) techniques. Specimens for electron backscattered diffraction (EBSD) in SEM (Quanta 450 FEG) were prepared by mechanical grinding and polishing, the final polishing was done using colloidal silica. The OIM Analysis™ software was used for acquisition and indexing of Kikuchi diffraction patterns and for evaluation of the EBSD orientation data. Minor phases were studied in TEM (JEM 2100) on carbon extraction replicas using both selected area electron diffraction and EDX. DSC analyses were carried out on the Setaram MHTC 96 Line equipped with a 3D heat flux DSC-B type sensor. Measurements were performed using specimens with dimensions of φ5 x 8 mm in the temperature interval between 900 and 1650 K in an inert gas atmosphere (He).

3. Results and discussion
Microstructure across the thickness of the belt-casted strip consisted of coarse ferrite grains and decomposed Widmanstätten austenite laths [4]. Coarse ferrite grains were fragmented into a number of subgrains with misorientation less than 2°. The fraction of decomposed Widmanstätten austenite, as determined by image analysis, was about 2% [4]. Particles of Widmanstätten austenite preferentially nucleated at ferrite grain boundaries where they formed low energy flat austenite/ferrite interface facets. The formation of Widmanstätten austenite only on one side of a ferrite grain boundary resulted in its straightening, figure 1. Such straight grain boundaries are actually planar in three dimensions [8]. If Widmanstätten austenite laths nucleated on both sides of a ferrite grain boundary, two sets of flat austenite/ferrite interface facets arose [3]. The planar interface facets for both sets of Widmanstätten austenite laths were not parallel due to misorientation between the ferrite grains. In order to minimize the interfacial energy, segments of ferrite grain boundaries were forced to migrate. This was probably favoured by the high formation temperature of Widmanstätten austenite [3]. It resulted in zig-zag shape of ferrite grain boundaries composed of alternating segments of straight boundaries, figure 2. Furthermore, fine austenite particles also nucleated intragranularly.
Figure 1. The straight ferrite grain boundary decorated by decomposed Widmanstätten austenite “smithiomorphs” [6] growing into one ferrite grain, LM.

Figure 2. Widmanstätten austenite laths growing into two adjacent ferrite grains, migration of the ferrite grain boundary resulted in its zig-zag shape, LM.

Figures 1 and 2 also document heavy precipitation along grain/subgrain ferrite boundaries, ferrite/austenite interfaces and in ferrite grains. In the area of final solidification of the belt-casted strip, i.e. at about ¼ depth under the top surface of the strip, relatively coarse sulphides decorated ferrite grain boundaries, figure 3. EDX analyses revealed that these sulphides were rich in iron, table 2. Needle-like intragranular particles in the δ-ferrite were identified as cementite, figure 4. Most fine globular precipitates were complex sulphides. Chemical composition of fine sulphides was variable. Majority of these sulphides were rich in chromium, manganese, iron and copper. The average chemical composition of fine sulphides is shown in table 3. Electron diffraction studies proved that the crystal structure of fine sulphides was consistent with the Cr₂CuS₄ phase (cubic, Fd3m, a = 0.961 nm), figure 5.

Figure 3. Coarse iron sulphides along ferrite grain boundaries and fine intragranular complex sulphides, LM.

Figure 4. Needle-like particles of cementite, fine sulphides along subgrain boundaries and in ferrite grains, LM.
Table 2. Results of semiquantitative EDX analyses of coarse sulphides, mass %.

| No. | Si | S  | Ti | Cr | Mn | Fe | Cu |
|-----|----|----|----|----|----|----|----|
| 1   | 0.3| 33.6| 0.9| 8.9| 9.5| 46.0| 0.8|
| 2   | 0.5| 35.5| 1.9| 8.5| 2.0| 50.5| 1.0|
| 3   | 1.1| 25.8| 0.2| 2.0| 19.3| 51.0| 0.7|

Figure 5. Intragranular Cr$_2$CuS$_4$ sulphides, insert: zone axis [111]$_{Cr_2CuS_4}$, TEM.

Figure 6. Decomposition of Widmanstätten austenite to epitaxial ferrite and pearlite, arrows mark epitaxial ferrite, LM.

At the end of the δ + γ field Widmanstätten austenite decomposed either to pearlite or plate martensite. In some laths of decomposed Widmanstätten austenite both products coexisted. Discontinuous networks of sulphides at ferrite/austenite interfaces made it possible to study the early stages of austenite decomposition. The decomposition of austenite started with the formation of epitaxial ferrite with an identical orientation as the surrounding ferrite. Arrows in figure 6 mark the parts of Widmanstätten austenite laths which transformed to epitaxial ferrite. This transformation was preferentially observed in thin Widmanstätten austenite laths and especially close to the tips of these laths. It was accompanied by a further partition of carbon into remaining austenite. The remaining austenite in this area transformed to pearlite, figure 6.

Table 3. Results of semiquantitative EDX analyses of fine complex sulphides, mass %.

| No. | S   | Cr  | Mn  | Fe  | Cu  |
|-----|-----|-----|-----|-----|-----|
| 1   | 43.3| 33.3| 9.3 | 7.5 | 6.6 |
| 2   | 43.6| 32.4| 13.6| 4.5 | 5.9 |
| 3   | 42.1| 34.0| 13.5| 3.4 | 7.1 |
| AVE | 43.0| 33.2| 12.1| 5.1 | 6.5 |
| STD | 0.8 | 0.5 | 3.0 | 2.9 | 0.4 |

*AVE is the average value and STD is the standard deviation.

Arrows in figure 7 mark the original positions of austenite/ferrite interfaces decorated by fine
sulphides. Decomposition of austenite started with the formation of epitaxial ferrite and the remaining austenite in this case transformed to a mixture of pearlite and plate martensite. The formation of plate martensite suggested, that the Widmanstätten austenite lath was significantly enriched in carbon. Plate martensite usually forms in Fe – C alloys containing more than 0.6 wt.% C [9]. Plate martensite always coexists with some retained austenite, figure 8.

![Figure 7. Widmanstätten austenite lath decomposed to a mixture of pearlite and plate martensite. Arrows mark the original positions of the ferrite/austenite interface, decomposition of Widmanstätten austenite started with the formation of epitaxial ferrite, LM.](image1)

![Figure 8. Decomposition of Widmanstätten austenite laths in a mixture of plate martensite and residual austenite, LM.](image2)

Except for the straight and zig-zag ferrite grain boundaries, some ferrite grain boundaries exhibited in the vicinity of decomposed Widmanstätten austenite laths a wavy character. The IPF orientation map in figure 9 shows two parallel decomposed Widmanstätten austenite laths growing from the ferrite grain boundary into the grain A.

![Figure 9. IPF orientation map, partial decomposition of Widmanstätten austenite to epitaxial ferrite - arrows mark the growth of epitaxial ferrite from the grain B into the originally flat austenite/ferrite interface facet.](image3)

![Figure 10. Phase map of ferrite (red) and retained austenite (green) + high angle grain boundaries (α >15°). Retained austenite forms islands between martensite plates (area C) and fine intragranular particles.](image4)
Products of Widmanstätten austenite laths decomposition are plate martensite + retained austenite and pearlite. Arrows at the bottom lath show the growth of epitaxial ferrite with the orientation identical to the ferrite grain B across the originally flat interface facet into the Widmanstätten austenite lath. It resulted in a wavy character of the ferrite grain boundary. The phase map in figure 10 documents ferrite and retained austenite distribution in this area. Islands of retained austenite coexist with plate martensite in the decomposed Widmanstätten austenite lath – see area C in figure 10. Furthermore, fine intragranular particles of austenite were resistant to decomposition during cooling of the strip.

Figure 11 shows thermal effects recorded during the DSC experiment in the temperature range of 900 – 1650 K. Heating and subsequent cooling of the GOES specimen with the starting microstructure consisting of the δ-ferrite, pearlite and sulphides was carried out at the rates of 15 K·min⁻¹ and 40 K·min⁻¹, respectively. The heating curve shows the peak related to the change of magnetic properties of the δ-ferrite (Curie temperature, 1027 K) and two minor peaks. The thermal effects at 1093 – 1125 K can be related to the formation of austenite. The broad peak at 1442 – 1590 K probably corresponds to dissolution of sulphides and austenite.

![Figure 11. DSC curve showing peaks during heating and cooling of the GOES specimen between 900 and 1673 K.](image)

Nevertheless, the volume fraction of sulphides in the microstructure was too low to cause distinct thermal effects on the DSC curve during their dissolution. Thermal stability of complex sulphides in GOES steels increases with decreasing copper content [5]. On the cooling curve the peak at 1183 – 1093 K was observed. These thermal effects can be linked with the austenite decomposition to epitaxial ferrite and pearlite. The peak at 980 K corresponds to the change in magnetic properties of the parent phase.

4. Conclusions
Solidification of the belt-casted strip started with the formation of the δ-ferrite. During cooling of the strip a small fraction of Widmanstätten austenite precipitated in the δ-ferrite by displacive mechanism accompanied by partitioning of carbon. Widmanstätten austenite laths preferentially nucleated on ferrite grain boundaries and formed the flat low energy austenite/ferrite interface facets along the
ferrite grain boundaries, which were forced to migrate. If Widmanstätten austenite laths nucleated on both sides of the ferrite grain boundary, zig-zag boundaries arose. Heavy precipitation of complex sulphides developed in the δ-ferrite, grain/subgrain ferrite boundaries and ferrite/austenite interfaces. Majority of fine sulphides corresponded to the Cr₂CuS₄ phase. Decomposition of Widmanstätten austenite laths started with the formation of epitaxial ferrite. The growth of epitaxial ferrite into the flat ferrite/austenite interface facets along ferrite grain boundaries resulted in a wavy shape of these boundaries. The formation of epitaxial ferrite resulted in further enrichment of remaining austenite islands in carbon. The final step of Widmanstätten austenite decomposition resulted in the formation of pearlite or plate martensite. The decomposition mechanism was affected by the cooling rate, chemical composition and dimensions of Widmanstätten austenite. The fraction of retained austenite in the final microstructure of the belt-casted strip was very low (about 0.5 %).

Acknowledgment
The authors gratefully acknowledge the support by the project “Damage Prediction of Structural Materials”, No. CZ.02.1.01/0.0/0.0/17_048/0007373 within the Operational Programme Research, Development and Education financed by the European Union and by the state budget of the Czech Republic and the GAČR project No. 17-18668S.

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