Article

Twin Roll Casting and Secondary Cooling of 6.0 wt.% Silicon Steel

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Abstract: Iron–silicon alloys with up to 6.5 wt.% Si offer an improvement of soft magnetic properties in electrical steels compared to conventional electrical steel grades. However, steels with high Si contents are very brittle and cannot be produced by cold rolling. In addition to solid solution hardening, it is assumed that the B2- and DO3-superlattice structures are responsible for the poor cold workability. In this work, two cast strips with 6.0 wt.% Si were successfully produced by the twin roll strip casting process and cooled differently by secondary cooling. The aim of the different cooling strategies was to suppress the formation of the embrittling superlattice structures and thus enable further processing by cold rolling. A comprehensive material characterization allows for the understanding of the influence of casting parameters and cooling strategies on segregation, microstructure and superlattice structure. The results show that both cooling strategies are not sufficient to prevent the formation of B2- and DO3-structures. Although the dark field images show a condition which is far from equilibrium, the achieved condition is not sufficient to ensure cold processing of the material.

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Keywords: twin roll casting; secondary cooling; electrical steel; silicon steel; B2 and DO3 ordering; strip casting; rolling; microstructure; segregation

1. Introduction

Non-grain-oriented electrical sheets are widely used in the field of electrical engineering, for example in electric motors, transformers and power generators. The use of electrical steel with 6.5 wt.% Si exhibits excellent soft magnetic properties. Si increases electrical resistivity and therefore lowers eddy current losses [1]. Furthermore, the magnetostriction reaches a minimum with this alloy [2]. However, increasing Si contents are accompanied by decreasing ductility and thus workability. This is due to the increase in solid solution strengthening and the formation of embrittling B2 (Pm-3m) and DO3 (Fm-3m) superlattice structures [3]. Since the aforementioned eddy current losses are also increasing with the sheet thickness, alloys for application in high frequency electrical drives are usually produced in thicknesses of 0.2–0.35 mm. Due to embrittlement at high Si contents, the preferred electrical steel thicknesses for industrial use in the relevant application areas cannot be achieved with conventional methods at such high Si contents. In commercially available grades, a Si content of less than 3.5 wt.% is realized as a compromise between good properties and economical production [4].

The workability decrease is associated with an increase in Si content. In Fe–Si alloys with more than 4 wt.% Si dislocations lose their ability to cross-slip and predominantly move via planar slip mode [3]. Additionally, the formation of long-range order B2 and DO3 occurs with Si contents more than 5.4 wt.%, which are responsible for further plasticity deterioration [5]. B2 is a state in which all Si atoms try to surround themselves with iron atoms in the nearest-neighborhood. The DO3-lattice, on the other hand, is characterized by

the fact that the Si atoms tend to be mixed only with iron atoms both in nearest- and next-nearest-neighborhood [3,5,6]. B2 is related to form dissociated superlattice dislocations and intragranular stresses. The combination of high intrinsic Peierls–Nabarro friction force and the restricted possibility for dislocations to glide on the {110}-planes lead to cleavage fracture [7]. Especially in the two-phase and DO3 regions, the material is hardly deformable [3]. In Fe–Si alloys with 6.5 wt.% Si, the disordered A2 phase is only present above 800 °C [8]. The possibility of suppressing the embrittling B2 and DO3 in high Si alloys by rapid cooling has already been the subject of various research efforts [3,5,7–10]. While in Fe–6.5Si alloys in most cases the DO3-structures were avoided due to fast cooling strategies, some authors stress the limitations since the B2 structure could not be suppressed [7–9].

In this work, the producibility of an Fe–6.0Si alloy with 1.7 mm thickness by means of the twin roll strip casting process was investigated. In the second step, the possibility of undercooling embrittling phases out of the strip casting process by two different secondary cooling strategies was examined. On the one hand, air cooling was carried out without coiling, and on the other hand, water-cooling was carried out with subsequent coiling. The influence of different secondary cooling strategies in terms of suppressing the superlattice structures was investigated by TEM. Furthermore, the segregation formation and the microtexture were determined in detail by means of EPMA and EBSD. The resulting formability of the produced strip was explored in cold and warm rolling tests.

2. Materials and Methods

2.1. Material Processing

Two casting trials with different secondary cooling strategies were proceeded on the laboratory twin roll caster of the metal forming institute (IBF) at RWTH Aachen university, Germany. A more detailed explanation of the caster, the cooling device and the process regulation can be found elsewhere [11,12].

In each case 150 kg of a Fe–6.0Si steel alloy were smelted in an induction furnace and the melt was poured via the melt feeding system between the counter-rotating casting rolls. The melt feeding system consists of a refractory channel, a tundish and a submerged entry nozzle (SEN). The melt superheat refers to the temperature difference between the investigated Fe–6.0Si alloy and the tundish temperature and was adjusted to 50–80 °C. The internally water-cooled casting rolls are made of copper. In order to control the heat flux, the casting rolls are coated with 0.5 mm nickel and the surface roughness was set by shot blasting. Furthermore, the melt pool was covered by nitrogen, which has also an inertization purpose. The strip thickness was set to 1.7 mm and kept constant during the trials. The casting process is stabilized at a constant roll separating force (RSF) by the automation of the test plant. The RSF was adjusted to 10 kN for both tests, which corresponds to a width-specific RSF of 66.7 N/mm for a casting roll width of 150 mm. Two secondary cooling strategies with high cooling rates were pursued. In the trial AC1378 (internal reference number) the coiling operation was stopped, so that the cast strip could cool down uncoiled from roll nip exit temperature to room temperature. On the other hand in the trial AC1379 a water-cooling device was utilized, which operated with a water/air mixture. However, in order to utilize the cooling line the coiling mode is necessary, which means that the cooling process is slowed down in the range from coiling to room temperature.

In order to demonstrate the principle rollability of the material, the cast material was descaled by means of sandblasting, edged and both cold and warm rolled at 500 °C.

2.2. Materials Characterization

The Si content of the as cast strips was confirmed by means of inductively coupled plasma optical emission spectroscopy (ICP-OES) with the SPECTRO ARCOS device (SPECTRO Analytical Instruments GmbH, Kleve, Germany). With the aim to measure other accompanying chemical components, a spark emission spectrometer (S-OES) OBLF QSG 750 (OBLF Gesellschaft für Elektronik und Feinwerktechnik mbH, Witten, Germany) was used. Additionally, carbon content was quantified utilizing ELTRA CS 2000 (ELTRA GmbH,

Haan, Germany) combustion analysis device, whereas nitrogen contents were measured by carrier gas hot extraction method with the help of ELTRA Elementrac ONH-p (ELTRA GmbH, Haan, Germany).

The segregation of Si over the sheet thickness of the as cast strip was investigated by electron probe micro analysis (EPMA). Area and line scans were performed on the field emission electron probe microanalyzer JEOL JXA-8530F (JEOL Ltd., Tokyo, Japan) applying 15 kV beam energy and 100 nA beam current. Element-specific X-rays were analyzed by a wavelength dispersive detector, which is equipped with a PETH crystal amongst others. Electron backscattered diffraction (EBSD) analysis was also performed in the interest of microstructure and microtexture. Therefore, samples were water cut from each casting trial and EBSD analyses were conducted at the longitudinal section of casting direction (CD) and normal direction (ND). For this purpose, the EBSD data were generated at the GeminiSEM 300 by Carl Zeiss Microscopy (Carl Zeiss AG, Oberkochen, Germany), which is equipped with the EBSD-camera Symmetry by Oxford Instruments (Oxford Instruments plc, Abingdon, England). The measurements were performed with an accelerating voltage of 20 kV and a step width of 2 μm . Orientation distribution functions (ODF) were calculated from EBSD data using Matlab[®]-based MTEX-Toolbox. For further microstructure investigation samples of the cast samples were prepared and etched with 4% nital for light optical microscopy (LOM).

The existence of ordered B2 and DO₃-phases was examined by transmission electron microscopy (TEM). Therefore, from each casting trial a focused ion beam (FIB)-lamella was cut out. With the intention of neglecting segregation effects, the FIB-lamellae were removed with a distance of 500 μm from the surface, as will be reasoned later. TEM diffraction patterns and dark field images were received by a FEI Tecnai F20 (FEI Company, Hillsboro, OR, USA) operating with 200 kV beam energy.

3. Results and Discussion

3.1. The Casting Process

The results of the chemical analysis are given in Table 1. Due to the insufficient melt inertization of the casting system some Si loss appeared. Therefore, the cast alloys have approximately 0.5 wt.% less Si than originally presumed. The carbon content is limited to a maximum of 56 ppm, which is beneficial to the magnetic properties of electrical steels. Although the melt pools were inertized with nitrogen, an increase in nitrogen content in the cast strips was not notable.

Table 1. Chemical composition of the cast trials AC1378 and AC1379.

Trial Number	C	Si	Mn	Al	P	S	N
unit	ppm	wt. %	wt. %	ppm	ppm	ppm	ppm
method	CA	ICP-OES	S-OES	S-OES	S-OES	S-OES	TCA
AC1378	56	6.04	0.06	40	46	48	30
AC1379	43	6.03	0.06	20	39	42	31

The casting parameters of the cast trials can be found in Table 2. Operating the casting process with a RSF of 10 kN and thickness of 1.7 mm led to a very smooth casting process. No force oscillations leading to an inhomogeneous temperature distribution, e.g., as reported in [13,14] could be observed. According to [13] high RSF and low yield strength increase the tendency for such process oscillations. The small width-specific RSF of 66.7 N/mm and the increasing effect of Si on the yield strength have a beneficial impact on process behavior. Thus, a very homogeneous temperature profile over the band length and width could be received. The cooling water sensor data revealed heat flux densities in the range of 6.4–7.9 MW/m² resulting in contact times in a range between 0.39–0.45 s.

Table 2. Casting parameters of the cast trials AC1378 and AC1379.

Trial Number	RSF (kN)	Strip Thickness (Mm)	Melt Superheat (°C)	Heat Flux Density (MW/m ²)	Contact Time (s)	Roll Nip Exit Temperature (°C)	Secondary Cooling	Coiling Temperature (°C)
AC1378	10	1.7	50	6.4	0.45	1230	Air	No coiling
AC1379	10	1.7	80	7.9	0.39	1200	Water/air	700

3.2. Segregation

Figure 1a–c show the elemental distribution of Si in the air-cooled sample AC1378, in a map view from edge and sheet center, received from EPMA. Grain boundaries were removed from the corresponding SEM-picture and are plotted in the EPMA-maps. The EPMA resolution of 0.5 μm revealed a homogeneous distribution of Si in the edge area. No dendritic structures were distinguishably measured with a step size of 22.5 nm. Thus, the solidification structure of the present strip casting differs in principle from the dendritic solidification structures already observed in strip casting for conventional electrical steel [15] and high manganese steel [16]. Besides, a Si enriched phase (with the color red in Figure 1a) could be measured, which was identified as fayalite. In contrast, the center of the sheet revealed an inhomogeneity with a spotty, non-uniform appearance. The Si diminished domains, here shown in color blue, exhibit a size of approximately 50–100 μm . However, it is evident, that the observed Si segregation is smaller than the magnitude of the grain size. Therefore, the differences in Si content are based on microsegregations inside the grain and do not correspond to a macrosegregation. Figure 1d represents the EPMA line scan of Si related to the grain structure. The Si content remains static in the range of 0–550 μm distance from surface with a level of approximately 6.38 wt.%. While the highest and lowest measured Si content can be quantified to 6.85 and 5.56 wt.%, respectively, the average content was determined to be 6.34 wt.%. Thus, the average content received from EPMA shows a value that is approximately 0.3% higher than received from ICP-OES. However, it should be noted that the ICP-OES-method is more meaningful because a more representative amount of the sample material is examined. Due to the microsegregation the curve in the range between 550–850 μm distance from surface is strongly dependent on the location of the measuring line. Assuming a diffusion coefficient of Si in α -iron of $6 \times 10^{-13} \text{ m}^2/\text{s}$ at 1100 °C as reported in [17], a homogenization annealing of 10 h at 1100 °C would be required to reduce the segregation to 10% of the initial segregation. However, such a homogenization annealing would contribute to undesirable grain growth. It is generally known that a coarse grain reduces the ductility of a material. With the aim of ensuring processability of the alloy, such a homogenization annealing was therefore not performed.

Figure 1. Results of EPMA scans for Si measured at sample AC1378. (a) Map scan with overlaid grain structure at the edge of the sheet with step size $0.5\ \mu\text{m}$ and (b) the magnified area detected with a step size of $22.5\ \text{nm}$; (c) map scan overlaid grain structure in the middle of the sheet; (d) line scan over the half thickness of the sheet with the corresponding SEM-picture in the background. (Image source: GfE RWTH Aachen).

3.3. Microstructure and Microtexture

The microstructures of the cast samples AC1378 and AC1379 received from EBSD analysis are shown in Figure 2. The microstructure of the air-cooled sample AC1378 exhibits columnar grains in the edge area and equiaxed grains in the middle of the sheet. In contrast the water-cooled sample AC1379 is composed of more equiaxed grains. However, much finer grains are received in the edge area, where the highest cooling rate by the water-cooling has been achieved. The constitution of the grain population is demonstrated in the grain diameter histogram (Figure 2c). It can be observed that the grain size distributions follow a right skewed-behavior, whereas AC1379 shows a bigger distribution asymmetry than AC1378. The median grain sizes are $259\ \mu\text{m}$ for AC1378 and $135\ \mu\text{m}$ for AC1379. ODFs were calculated from EBSD data and are displayed as inverse pole figures with respect to the normal direction of the sheet for AC1378 and AC1379 in Figures 2d and 2e, respectively. Both trials show a preferential formation of $\langle 001 \rangle // \text{ND}$ oriented grains. The influence of melt superheating on grain formation in the strip casting process has already been investigated by other authors [18–20]. As studied in [19] using a ferritic chromium steel, the transition from an equiaxed to a columnar microstructure occurs from a superheated temperature of $40\ ^\circ\text{C}$ upwards. Columnar grains tend to adopt a $\langle 001 \rangle // \text{ND}$ orientation with a deviation of $0\text{--}15^\circ$ [19]. This can be verified using the air-cooled condition in AC1378 at $50\ ^\circ\text{C}$ superheating temperature.

Figure 2. Results of the EBSD measurements represented as IPF-map. The color-coding refers to directions parallel to the normal direction (ND) of the sheet: (a) cast microstructure of AC1378 and (b) AC1379. The positions of the FIB-lamellae for TEM-measurements are marked with crosses. (c) illustrates the corresponding grain size (diameter) classes as histogram received from EBSD data. Calculated ODFs are presented as inverse pole figures referring to the normal direction of the sheet for (d) AC1378 and (e) AC1379. (Image source for (a,b): GfE RWTH Aachen).

Figure 3a–c indicate the kernel average misorientations (KAM). When the angle of the KAM increases from 0 – 3° , the color changes from blue to red. While AC1378 (Figure 3a) exhibits a relatively flat and distortion-free surface, water-cooling shows a negative impact on the flatness of sample AC1379 (Figure 3b,c). The high level of misorientations reveals the distortions in the surface area of AC1379. These distortions could also be shown with the help of light optical microscopy (Figure 3d,e). The larger view of the section in Figure 3e exhibits near-surface grains with a bandlike structure. The inclination angle of approximately 45° to the surface suggests a plastic deformation by shear banding. As already observed from the EBSD data, finer grains in the edge zone could also be made visible by etching with nital. There are three possible explanations for the finer grains in the water-cooled sample AC1379. Firstly, the rapid passage through the high-temperature range prevents coarse grain growth. This means that the fine grains that have formed in the casting gap are better preserved, since they cannot be consumed by growing, neighboring grains. Secondly, the different superheating causes different grain formation. However, this effect can be excluded, since higher superheating would favor a coarser structure. A finer microstructure is observed with AC1379 despite 30°C higher superheat. Third, recrystallization is induced. Entering the cooling line, the cast strip had a temperature of approximately 1000°C and was cooled down to 700°C in 2 s, which corresponds to a cooling rate of 150°C/s . However, due to the very low thermal conductivity of 25 W/mK at 1000°C (data received from JMatPro[®] Version 8.0) of the Fe–6.0 Si alloy and the surface cooling, the edge fibers contracted in a larger extent than the relatively warmer core fiber. This phenomenon might induce a stress, which is relieved by plastic deformation of near-surface grains. Both KAM- and LOM-pictures confirm the presence of plastic deformations in the edge area. The high plastic strain in the surface area of the sheet and the high coiling temperature of 700°C would provide the opportunity for recrystallization.

Figure 3. KAM-maps of the surface areas from (a) AC1378 and (b) AC1379. (c) shows a magnified section of (b). Microstructure of water-cooled AC1379 cast sample obtained by LOM is represented in (d) and with enlarged detail in (e). (image source for (a–c): GfE RWTH Aachen).

3.4. Superlattice Structure

The superlattice formation is strongly Si dependent. Therefore, the influence of Si segregation had to be excluded. As already shown in Section 3.2, the Si content is constant at approximately 6.38 wt.% from the edge up to $500\text{ }\mu\text{m}$ into the sample. Furthermore, edge and grain boundary effects should be eliminated. For this purpose, the FIB-lamellae for TEM investigations were extracted at a distance of $500\text{ }\mu\text{m}$ from the surface. Figure 4

be detected (blue arrows in Figure 5a), they are absent in the water-cooled sample AC1379. Additionally, the water-cooled sample shows a more homogeneous distribution of the B2 and DO3-domains. According to previous TEM-investigations [1,6] four cases for Fe–Si alloys must be distinguished. These are A2, B2, DO3 and B2 + DO3. Due to the fact that all reflections of the B2-phase are also included in the DO3-pattern, a distinction between the cases B2, DO3 and B2 + DO3 is nontrivial. Therefore, a magnified dark field image of AC1379 was captured, once with a B2/DO3- (Figure 5c) and once with a DO3-reflection (Figure 5d). It can be seen that some reflections are additional to the isolated DO3-reflection in Figure 5c. Therefore, it can be assumed that both B2 and DO3 are attendant in the sample. It should be noted that despite the presence of the superlattice structures, the specimens are not in the near-equilibrium state reported in [6]. The biggest B2/DO3-domain was detected in AC1378 with an extent of about 50 nm (Figure 5a). The average domain size can be compared to a water-quenched state for the same alloy as reported in [5].

Figure 5. Dark field images received from TEM investigation: (a) AC1378 with a B2 and DO3 reflection; (b) AC1379 with a B2 and DO3 reflection; (c) magnified area of b with B2 and DO3 reflection and (d) same picture section as c with only DO3 reflection (Image source: GfE RWTH Aachen).

Superlattice structures, such as B2 or DO3, are assumed to decrease the ductility in Si steels drastically and therefore prohibit the cold workability of these grades. Consequently, rapid cooling aims to avoid the formation of these superlattices. By strip casting, high cooling rates of around 1000 K/s can be achieved in the mold region. However, it should be noted that the high cooling rate in the mold has no influence on the suppression of

the critical superlattice structures. This is because the strip leaves the nip point at a temperature of around 1200 °C, but with the present Si contents the formation of the superlattice structure starts at a temperature of 800 °C [1,6]. Therefore, secondary cooling strategies in the range of 800 °C until room temperature are required. The two cooling strategies investigated did not show effective suppression of the undesirable superlattice structure. The cooling in air is not fast enough for the given thickness. The advantage of the faster cooling rate of water-cooling could not be fully exploited in the given experiment. This is based on several aspects. First, continuous water-cooling requires the cast strip to be coiled in the given setup. This entails a slower cooling rate in the range between the coiling temperature and room temperature. The coiled strip cooled down very slowly from 700 °C coiling to room temperature, which promotes DO3-formation. Secondly, due to the continuous strip guidance, the passage speed through the cooling section cannot be decoupled from the casting speed. Therefore, only a dwell time of the strip of approx. 2 s in the cooling section could be realized. Thirdly, the cooling capacity of the existing cooling section is limited due to limited water supply and limited installation space.

In principle, a more powerful secondary cooling would be appropriate in order to reduce the formation of B2 and DO3 to a smaller extent. However, as can be obtained from the microstructure observations, the material is strongly distorted by water quenching and causes morphological defects in the final strip. A too severe cooling would probably lead to damage, e.g., by cracks as already reported by [7,8]. Therefore, a smoother cooling, in which a more effective suppression of the DO3 structure can be effected at the same time, would be desirable.

3.5. Rollability

The results of the rolling tests are illustrated in Figure 6. The samples shown are from cast trial AC1379, but are also representative of AC1378. As expected, the material already broke in the first cold rolling pass. The cracks originate from the center of the sheet and run transversely. A significant improvement in rollability could be achieved by heating the material to 500 °C and subsequent rolling. Nevertheless, it should be noted that these rolling tests were accompanied by edge cracks, some of which were critical, despite edging of the specimens prior to rolling. The lowest achievable thickness by warm rolling was 0.5 mm.

Figure 6. Macroscopic images of the cast and rolled samples of AC1379: (a) as cast state; (b) cold rolled sample; (c) warm rolled sample at 500 °C.

The maximum Si concentration measured in EPMA was 6.85 wt.% Si. It can therefore be assumed that a state of higher long-range order has been established in the areas, which is less favorable for plastic deformation than the state observed in the TEM investigations. In this case, the damage to the material would presumably first occur at the point of maximum segregation and then propagate as a crack.

4. Conclusions

The results from the current research can be summarized as follows:

- It was shown that a Fe–6.0Si alloy can in principle be processed by means of twin roll casting. In both trials, strips with a stable process point free of process oscillations could be produced.
- With the aid of the EPMA measurements, a largely homogeneous Si distribution was measured over 70% of the cross-section of the strip. Furthermore, no dendritic solidification could be observed. Nevertheless, microsegregations occur, especially in the center of the strip with maximum measured Si contents of 6.85 wt.%.
- The microstructure is refined by water-cooling, especially in the area close to the surface. However, too severe water-cooling induces thermal stresses in the sheet that lead to plastic deformations.
- Both cooling strategies led to a predominantly $\langle 001 \rangle$ / ND texture, which is desirable for electrical steels.
- The coexistence of B2 + DO3 structures was proven both in the air-cooled state and in the water-cooled and coiled state. However, a state far from equilibrium with finely distributed domains was set.
- Rollability at room temperature is poor and can be significantly improved by raising the rolling temperature to 500 °C. The maximum achievable sheet thickness before catastrophic failure was 0.5 mm by warm rolling.

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