ANALYSIS OF PRECIPITATION AND DISSOLUTION PROCESSES IN THE CONTEXT OF GRAIN ORIENTED HIGH SILICON STEEL PRODUCTION

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Abstract

High-silicon grain oriented steels require strict requirements for chemical composition. The steelmaking process must therefore be extremely tightly controlled due to the need to achieve elemental compactness within strictly defined ranges. Particularly important in this regard is the Al/N ratio. The hot-rolling process is also subject to special requirements, being carried out at high temperatures due to the favorable effect of temperature on the solubility of AlN which is an inhibitor of grain growth in the secondary recrystallization process occurring in the subsequent stages of the electrižcal steelmaking process. The processes of precipitation and solubility of non-metallic inclusions such as AlN play a key role in the formation of the desired texture. To determine the occurrence of precipitation processes of non-metallic inclusions, studies on precipitation processes were performed: thermocalk under equilibrium conditions, dilatometric studies on heating and cooling and cooling according to the technological process. It was found that despite the potential for the occurrence of austenitic region, in practice, there is no entry into the austenitic range. On the other hand, the effects of AlN precipitation can be observed.

Keywords: Grain oriented high silicon steel, precipitation, dilatometric analysis, production processes

1. INTRODUCTION

Grain-oriented high silicon steels are widely used in electrotechnical industry. Magnetic properties of grain oriented electrical steel (GOES) depend strongly on sharpness of the Goss texture (110) [001]. Despite of much research, various advancement in performance and production route, the exact mechanism of the evolution of Goss texture in high silicon grain-oriented steel is still debatable [1]. Factors which are crucial for the formation of the desirable texture during high temperature annealing includes the size the initial grains with the Goss orientation and role of second phase particles keep the ferrite grains small during early stages of the production process, maintain the driving force for secondary recrystallization and play the critical role in abnormal growth of Goss grains in this process. It is well known that aluminium nitride (AlN) has been extensively used as grain growth ''inhibitor'' in electrical steel [2-4]. Compared to MnS, AlN is a more stable inhibitor in the high temperature range due to its higher melting point leading to a slower decomposition rate [5]. The solubility product of AlN in the γ phase (fcc) is 1-2 order higher than in the α phase (bcc), that is why the addition of a small amount of carbon allows the α → γ transformation to occur at high temperatures [6]. There are several basic processes to produce the electrical steel. The procedures include steel making, hot rolling, normalizing annealing (hot band annealing), cold rolling, first recrystallization annealing, secondary recrystallization annealing and heat flattening coating [7]. This paper focuses on the analyse of precipitation and dissolution processes in the first stages of the production route. Despite the widely described textural development during cold rolling and annealing process, very little research work has been performed in relation to the precipitation and dissolution process during hot rolling part. The part of manufacturing technology for grain-oriented silicon steel is the slab high temperature reheating method. The heating temperature needs to reach as high as 1400 °C [8]. After heating the slabs in the furnace hot rolling process is carried out at significantly higher temperatures compared to the rolling process of classic low carbon steel, with reduced cooling at each stage of the process. At this stage, the selection of the right temperatures is particularly important since the size of the precipitates formed has a significant impact on the recrystallisation process.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

All investigated materials were of similar chemical composition for the same grade of steel (**Table 1**).

Table 1 Chemical composition of the investigated material.

Element	Range (wt%)
C	0.028 – 0.040
Si	3.00 – 3.25
Al	0.010-0.018
Cu	0.048- 0.55
Fe	Bal.

Dilatometric analysis was performed on a steel sample obtained after initial stage of hot rolling process. The test specimen was prepared by electrical discharge machining to obtain a cylinder with a diameter of 3 mm and a height of 10 mm. The dilatometer test was conducted on a RITA dilatometer at the local cathedral.

3. RESULTS AND DISCUSSION

3.1. Thermo-calc simulation

The phase volume fraction on equilibrium phases in the temperature interval from 0 to 160 °C was simulated using Thermo-calc software AB. The TCFE 10: TCS Steels/Fe-Alloys Database was used. The calculated phase diagrams are shown in **Figure 1.** The results obtained show that in the temperature range of hot rolling about 30 % α → γ transformation occur (**Figure 2a)**. It can be also concluded that high temperature reheating slabs allows for complete dissolution of AlN, according to the simulation total AlN should be dissolved in temperature about 1220 °C (**Figure 2b)**.

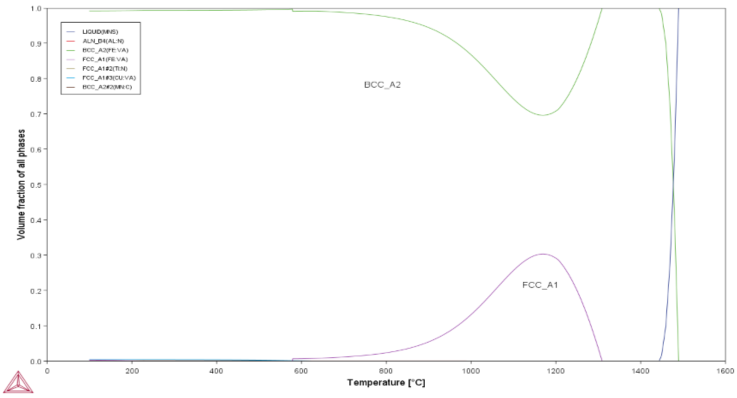

Figure 1 The predicted volume fraction of all phases

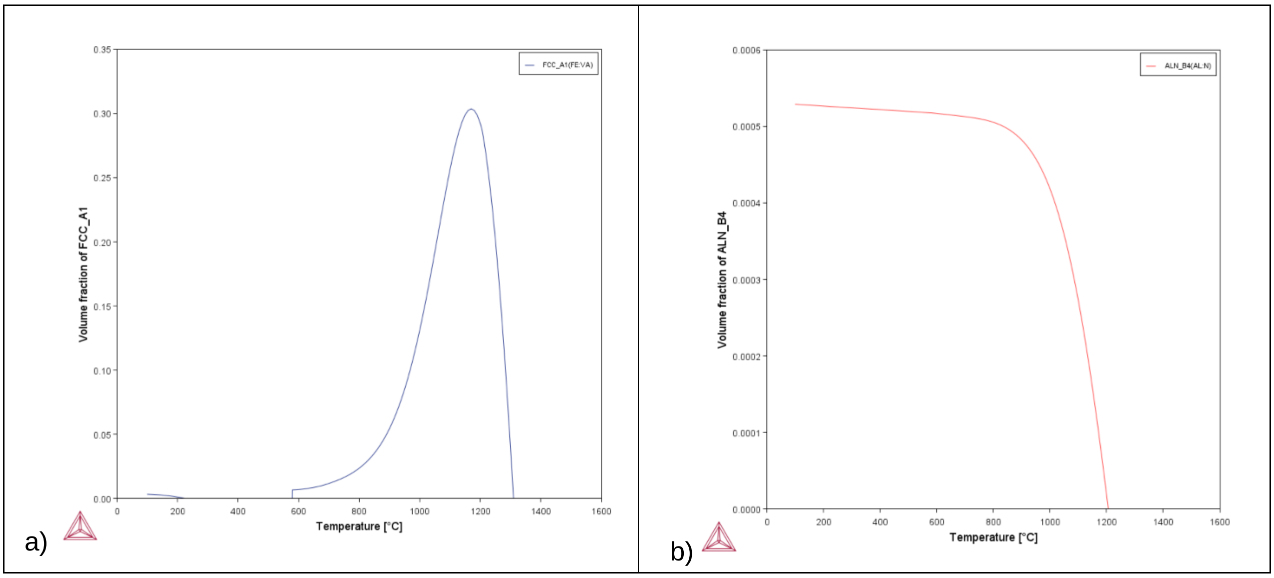

Figure 2 The predicted volume fraction of: a) fcc phase and b) AlN

3.2. Dilatometric analyses

Dilatometry is used to monitor phase transformations which involve changes in lattice parameters. Lattice vibrations (phonons) of the crystal lattice play a major part in the thermal expansion of the material, two phases in an alloy system with the same crystal structure may present different thermal expansion coefficients which is noticeable during dilatometry experiment [9]. For this study dilatometer tests were carried out using two different cooling and heating rates. The first experiment was carried out with constant heating rate and cooling 0.1 °C/s. Interesting effects were observed which may be related to phase separations (**Figure 3).** Intense effect from the temperature range 740-780 °C can be associated with magnetic transformation as evidenced by the change in power during heating processes. Distinct dilation effects recorded around intense power change indicating the effect of magnetic transformation are marked with arrows on the **Figure 4.**

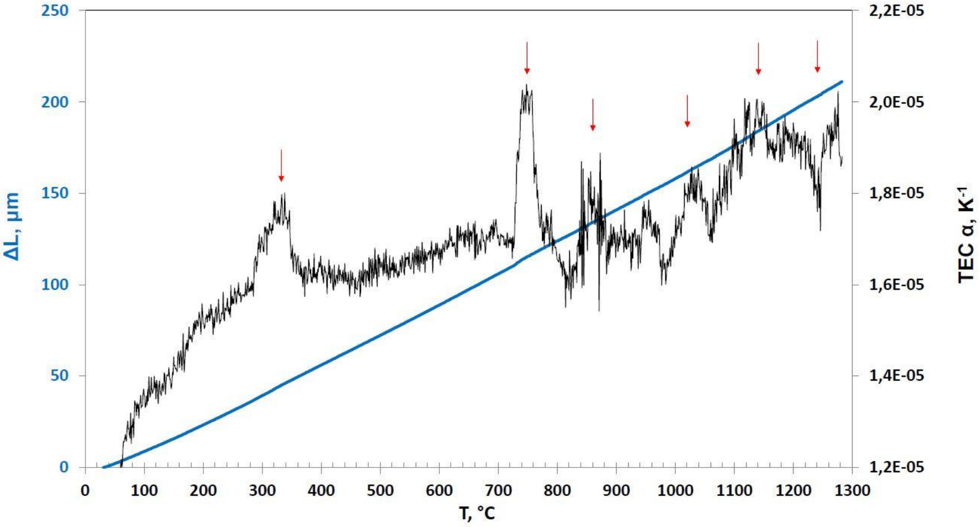

Figure 3 Dilatometric curve with differential curve for the process of heating steel at a rate of 0.1 °C/s

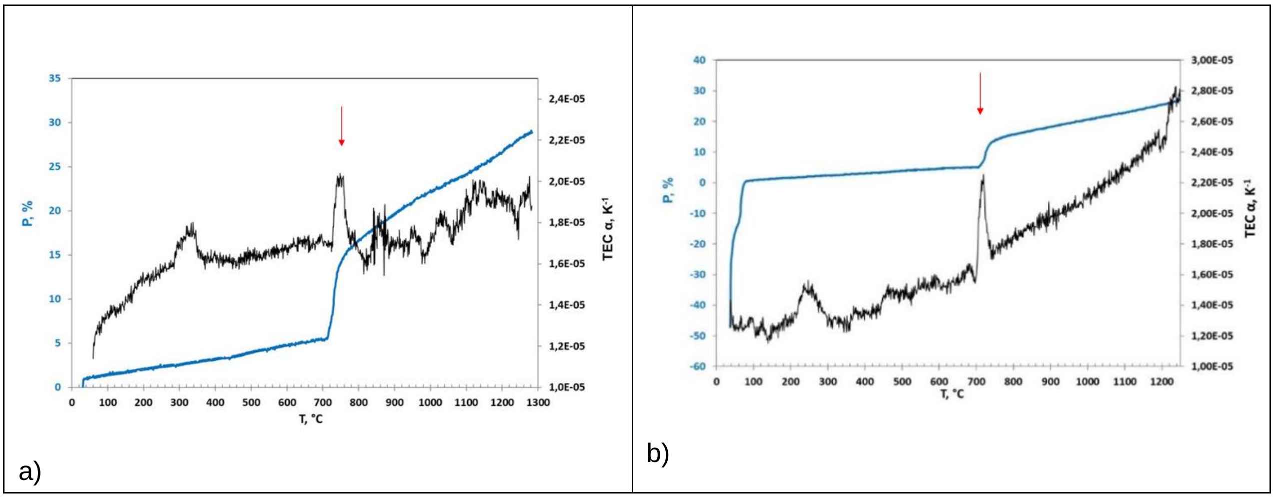

Figure 4 Power curve with thermal expansion coefficient changes for rate 0.1 °C/s a) heating, b) cooling

Similar magnetic transformation effects occur during cooling **Figure 5.** There is a recurring X effect that correlates with the technology used to not allow slab cooling below these temperatures.

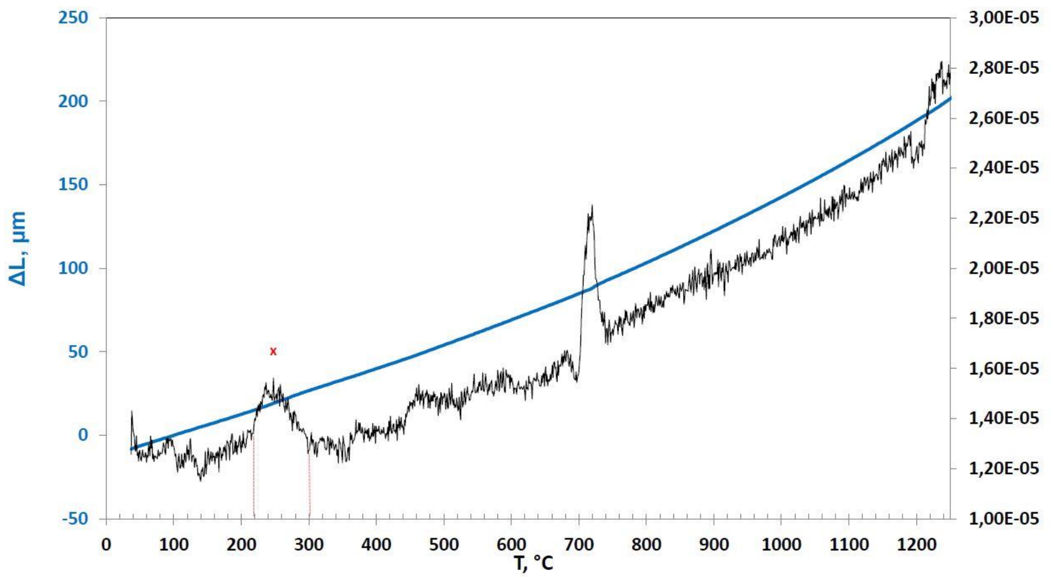

Figure 5 Dilatometric curve with differential curve for the process of cooling steel at a rate of 0.1 °C/s

To relate the remaining effects more accurately during heating to the Thermo-calc analysis, heating of the sample at a lower heating rate was performed **Figure 6**. At temperatures around 300 °C, an effect marked as X is observed that could be related to the fcc phase appearing in small amounts in Thermo-calc analysis. Numerical calculations performed for austenite and ferrite using Carb Nit software have also been used to analyse of AlN precipitation and have been reported in the literature [10].

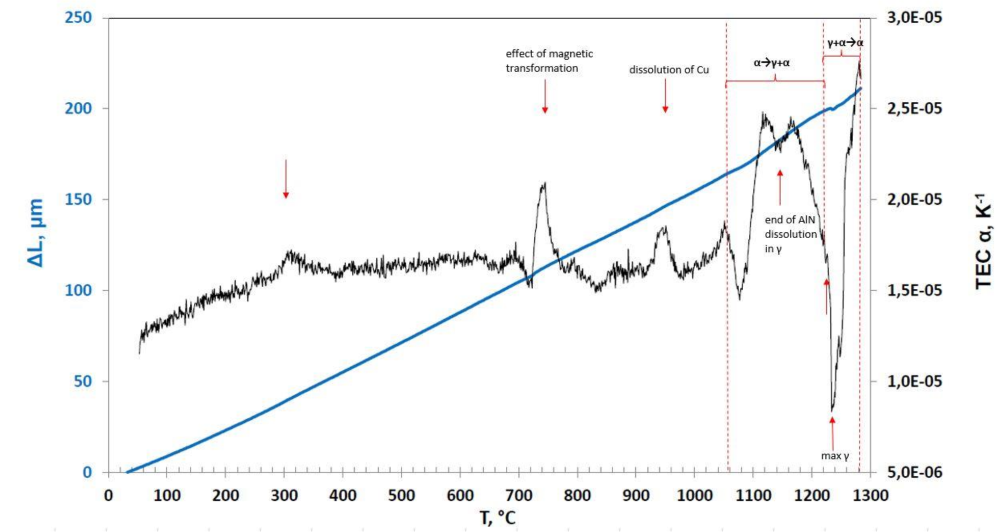

Figure 6 Dilatometric curve with differential curve for the process of heating steel at a rate of 0.08 °C/s

Since in the actual process there are changes in the cooling rate at different stages, a dilatometric curve with a representation of the cooling rate in the process was also developed **Figure 7**. The effect marked as X is also noticeable around 300 °C similarly to heating analyses.

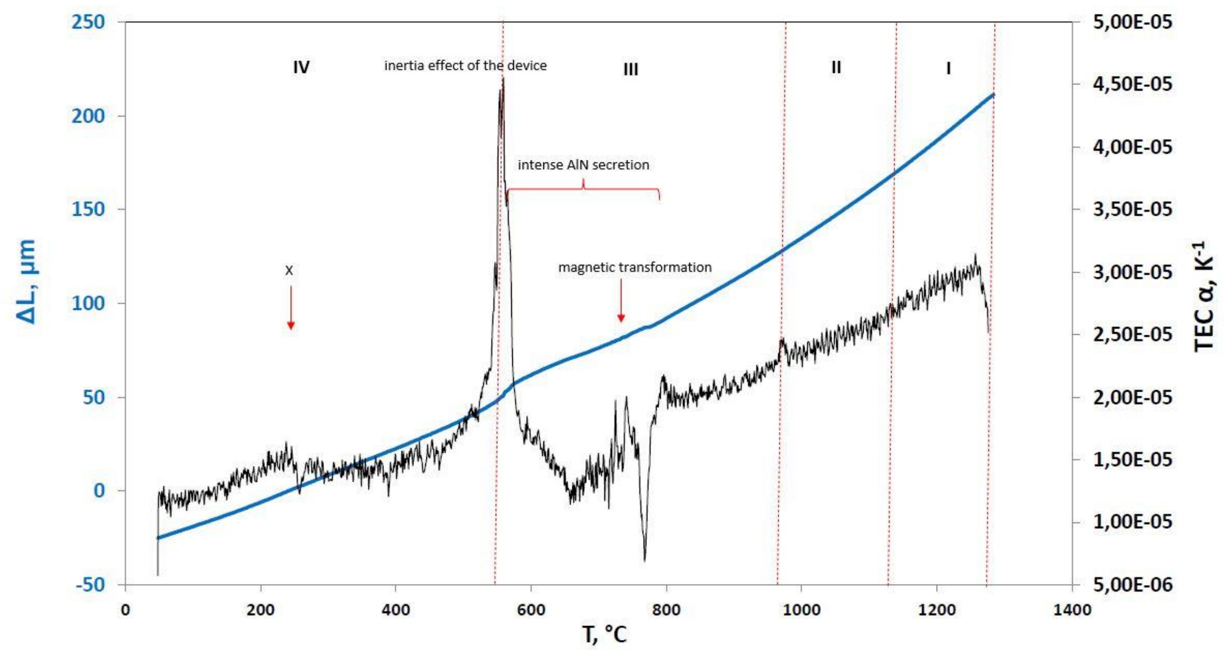

Figure 7 Dilatometric curve with differential curve for the process of cooling steel at a rate of: I -1,15 ˚C/s, II-3 ˚C/s, III - 26 ˚C/s.

4. CONCLUSION

Simulated Thermo-calc analysis for the equilibrium state indicates the possibility of the following phases in the investigated steel: bcc comes from ferrite, fcc probably comes from austenite, AlN, it can be also noticeable little FCC probably comes from Cu. Dilatometric studies have indicated the following effects during heating: transformation $\gamma+\alpha\rightarrow\alpha$ at 1250-1300 °C, transformation $\alpha\rightarrow\gamma+\alpha$ at 1175-1250 °C, dissolution of AlN between 1100 – 1250 °C. Dilatometric studies corresponding to the technological process (cooling) indicated the effect of intense AlN secretion. During heating and cooling, a dilatation effect was found in the range 740-780 °C, which may be associated with magnetic transformation. Near the temperature 300 °C dilatation effect appeared, which may be related to fcc phase based on Thermo-calc analysis and correlating with requirements to maintain slab temperature to prevent cracking of the material. This effect is weakened when using variable cooling rates applied to the process.

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