Effect of hot rolling conditions on the nitride precipitation process in low carbon steel strips

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Abstract. The nitride precipitation process plays an important role in the final mechanical and aging behaviour of low carbon steels in hot rolled state as well as in cold rolled and annealed state. The nitride precipitation process is investigated in an indirect manner: instead of measuring the amount of nitrogen compounds, the free nitrogen content of the samples was measured using a special thermomechanical treatment and a thermoelectric power tester. It was found, that the free nitrogen content in a steel strip strongly depends on three main factors: on the position in strip transverse direction, on the coiling temperature and on the cooling method at the run-out table of the hot rolling mill.

1. Introduction

Low carbon steels are frequently used for cold forming applications in hot rolled as well as in cold rolled and annealed state [1]. Low carbon steel grades usually consist of elements in w.t. %: 0.02…0.05 C, 0.15…0.3 Mn, 0.01 Si, 0.03-0.06 Al, 0.004…0.006 N, 0.015…0.05 Cr, 0.01…0.06 Cu, and low amount of Ti, Mo, Ni, Nb. The basic production technology is shown in Fig. 1.
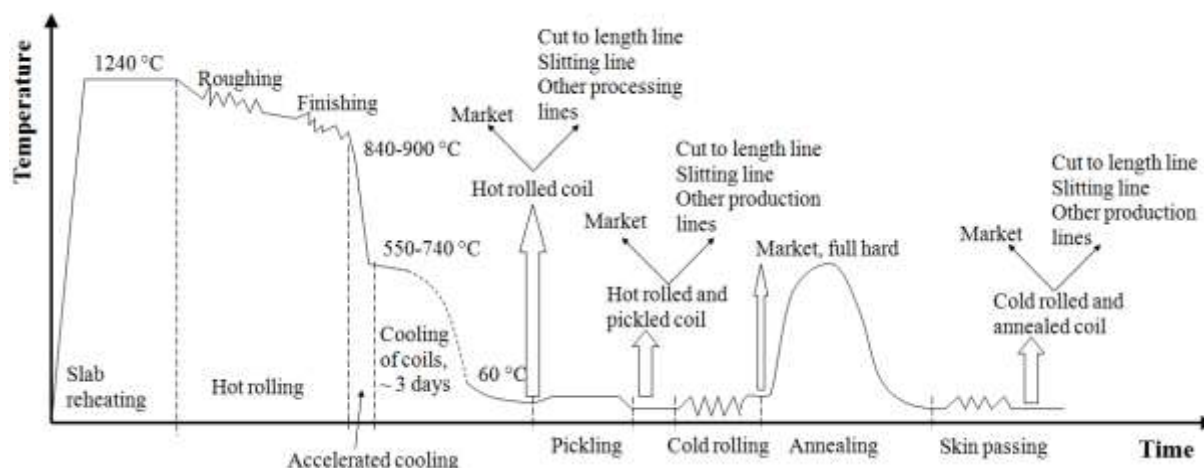

Figure 1. Basic production technology of low carbon steel strips

The dissolution of nitrogen containing precipitates (usually aluminium-nitride and aluminium-chromium nitride) occurs at the slab reheating temperature (~1240 °C) [1-3]. After reheating the slabs

to the suitable temperature, the roughing mill rolls the material to an intermediate thickness, ranging usually between 20 and 30 mm. The precipitation of nitrides in this austenitic region is very slow compared to the duration of roughing [2]. The final strip thickness is reached on the finishing mill, where the most important parameters are the finish rolling temperature (FT), the cooling method after rolling and the so-called coiling temperature (CT, the temperature of the strip before reaching the coiling equipment). The hot rolled coils are stored for 3-4 days to reach the suitable temperature (~60 °C) for further processing. During the hot rolling process the 10-20 % of the nitrogen content could precipitate depending on the temperature history and alloying elements in the steel [4-6]. But during the slow cooling after coiling a large amount of nitrogen containing precipitates could form, since the temperature and the time available is large enough to precipitate nucleation and growth [1, 4, 6]. The amount of nitrides formed depends strongly on the coiling temperature [1] and on the cooling rate after coiling [4]. Since the sides of the coil (strip borders) cools down more rapidly than the center of the coil, it is expected to be a large difference in the amount of precipitated nitrides (and in the free nitrogen content) along the transverse direction. The aim of this study to estimate the free nitrogen content along the transverse direction and investigate the role of cooling method and coiling temperature.

2. Experimental

The material investigated was a low carbon steel containing 0.056 C, 0.3 Mn, 0.042 Al, 0.046 Cu, 0.072 Cr, 0.034 Ni, and 0.0050 N (expressed in wt%). In order to study the effect of hot rolling thermal history, three different hot rolling conditions (FT=900 °C and CT=570°C, FT=900 °C and CT=730 °C with rapid and delayed cooling, FT=811 °C and CT=667 °C without strip cooling) were applied. The final strip cross section dimensions were 3 x 1000 mm. The free nitrogen content was determined at 50 m from the head end and along the width of the strip.

The determination of the amount of precipitated nitrides is based on the change in the thermoelectric power (S) caused by the precipitation of interstitials from solid solution. The thermoelectric power of the specimens was measured according to the concept shown in Fig. 2 [4].
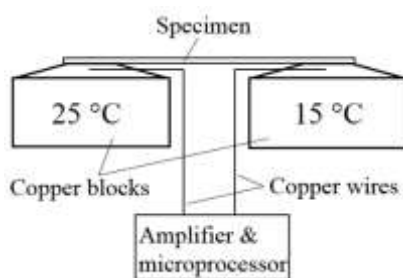

Figure 2. Basic assembly of the equipment used for measuring the thermoelectric power [4]

The methodology of Massardier et al. [7, 8] has been used for experimental investigation of nitride precipitation in hot rolled state. The method consists of an equalization treatment at 270 °C for 3 hours in order to set up the equilibrium carbon level in solid solution in each specimen. The cold rolling is intended to produce a high dislocation density, which allows the movement of all carbon and nitrogen atoms from solid solution (SS) to the vicinity of dislocations, see Fig. 3. The difference (ΔS_a) between the thermoelectric power before (S_ε) and after (S_a) the aging treatment is related to the amount of carbon and nitrogen atoms diffused to the dislocations. From that, using the Nordheim-Gorter law [9, 10], the amount of free nitrogen can be evaluated. The method is described in detail in Refs. [4-6].
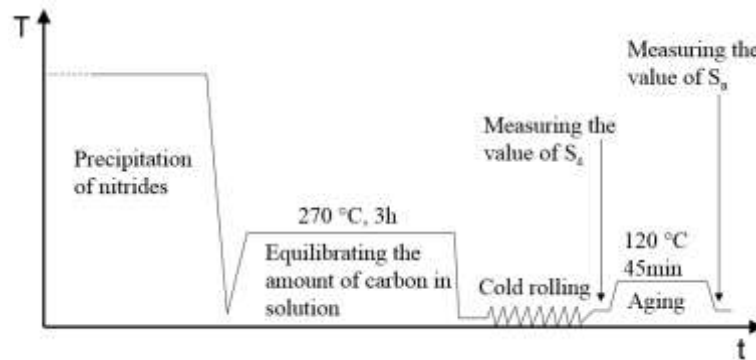
Figure 3. Methodology for determination the free nitrogen content [5]

The difference ΔS_a between S_a and S_ε carries the information about the amount of free interstitials at 270 °C [7]:

$$\Delta S_a = S_a - S_\varepsilon = K_C \cdot x_C + K_N \cdot x_N \tag{1}$$

where K_C and K_N are the Nordheim-Gorter coefficients for carbon and nitrogen (K_C=45 μV/K·wt%, K_N=24 μV/K·wt% [5,6]), x_C and x_N are the amount of carbon and nitrogen in solid solution (wt%). If all of the nitrogen are precipitated as nitrides (in case of high temperature and long treatment times), only the solute carbon determines the value of ΔS_a. So, firstly a long and large temperature treatment is necessary to determine the solubility of carbon in the given steel at 270 °C. After a long and high temperature treatment, no free nitrogen exist in the sample, so, the carbon in solid solution at 270 °C (called residual carbon) can be expressed as:

$$x_C = \Delta S_a / K_C \tag{2}$$

The value of x_C is forund to be 15±2 ppm by weight. Using this value the amount of free nitrogen in other samples can be calculated as:

$$x_N = \left(\Delta S_a - K_C \cdot x_C\right)/ K_N \tag{3}$$

3. Results and discussion

The free nitrogen distribution in transverse direction is shown in Fig. 4. It can be seen, that the different hot rolling thermal histories and the local position has a marginal effect on the free nitrogen content (and, of course, on the nitride precipitation process).
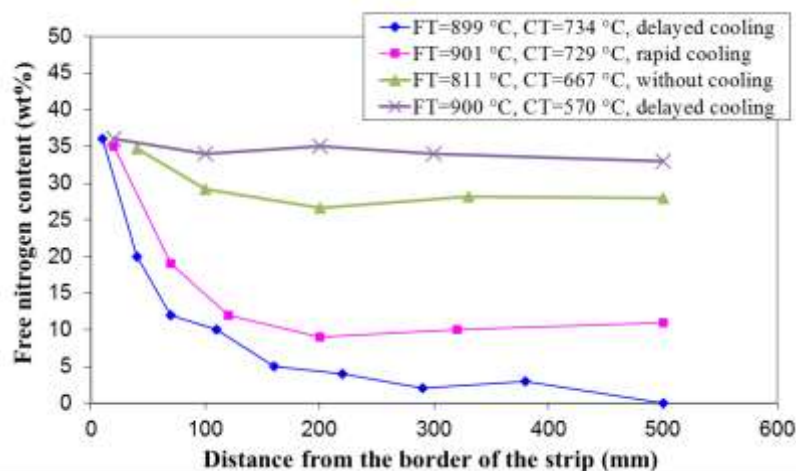

Figure 4. Distribution of free nitrogen content along the strip width

The major parameter, which influences the amount of nitride precipitation is the local position along the strip width. Quite close to the borders of the strip (<40 mm), the free nitrogen content is ranging between 34 and 36 ppm regardless to the hot rolling thermal history. The difference between the total nitrogen content (45 ppm) and this value is approx. 10 ppm (22% of the total nitrogen content). Since the cooling rate of the coil is the largest at this position, the nitride precipitation after coiling is negligible. This 10 ppm nitrogen is composed of two parts: a part of it was precipitated during hot rolling until the last finishing stand and another part is present as high stability precipitates (such as TiN) which were not dissolved during the reheating of the slabs. Titanium is present as a trace element in this steel (its content is lower than 10 ppm, and its more exact measurement with spectroscopy is difficult), but this amount is still large compared to the precipitated amount of free nitrogen (also ~ 10 ppm) at the borders of the strip. Considering a 10 ppm titanium level and taking the molar weights of titanium (M_{Ti}=47.9 g/mol) and nitrogen (M_N=14 g/mol), the nitrogen present as titanium-nitride ($x_{N, as TiN}$) can be estimated as:

$$x_{N, asTiN} \approx x_{N,f,b} \cdot M_N / M_{Ti} = 10ppm \cdot 14/47,9 \approx 3ppm \qquad (4)$$

where $x_{N,f,b}$ is the nitrogen present as precipitates at the border of the strip. Considering this simple calculation, the nitride precipitation process (altogether 10 ppm) until the beginning of strip coiling is composed of: 3ppm/45ppm≈ 6.7% could present as TiN, while 7ppm/45≈ 15.6% precipitates during hot rolling.

The free nitrogen content decreases strongly to the center of the strip, if the coiling temperature is high (670-730 °C). The large and the small CT at the borders of the strip decreases quickly after the coil is placed into the coil store and the time available and temperature are not large enough for aluminium (-chromium) nitride precipitation. In contrast to this, the regions of the strips located closer to the centerline cool much more slowly causing the gradual increase of the rate of nitride precipitation. When the strip is coiled at low temperature (CT=570 °C), negligible nitride precipitation (1-2 ppm nitrogen forms nitrides) takes place inside the coil.

The cooling method at the run-out table has a detectable effect of the nitride precipitation process. When the strip is cooled down quickly to the coiling temperature CT=730 °C just after the last stand of the finishing mill (rapid cooling), the free nitrogen content is found to be approx. 10 ppm in the centerline. When the strip is cooled down to the same temperature at the end of the run-out table (just before the coiler) the free nitrogen content is tending to zero at the centerline. The strip temperature in this case decreases gradually from FT=900 °C to an unknown value until it reaches the cooling unit at the end of the run-out table. During this travel of the strip (usually 6-8 s), 2-8 ppm nitrogen (the difference between the two lowest curves on Fig. 4.) precipitates.

With regard to the free nitrogen content of such mild steels, a simple observation connected to this topic should be mentioned. It is well known, that the free nitrogen content is in close relation with the elastic-plastic behavior of mild steels (Cottrell-theory [12]). Generally, it is believed, that the free nitrogen (and carbon) content of the steel is responsible for the discontinuous yielding phenomenon. The so-called coil break defects on low carbon steel strips are connected to the presence of pronounced yield point, they can form only if the steel exhibits discontinuous yielding [13-15]. Generally, according to the industrial investigations, coil break defects are not formed just at the borders of the strip where the free nitrogen content is high. This fact and other investigations [14, 15] suggest that the free interstitial content of a steel is not the individual reason of the discontinuous deformation.

4. Conclusions

On the basis of the experimental work outlined in this study, the following conclusions can be drawn:

1. The nitrogen distribution function in transverse direction of the hot rolled strip is determined by the coiling temperature and cooling method at the run-out table.
2. The free nitrogen content of the steel investigated is found to be 34-36 ppm (by weight) (approx. 22% of the total nitrogen of the steel) at the border of the strips regardless to the hot rolling temperature history.
3. The precipitated 22% nitrogen is composed of two parts: undissolved precipitates (in case of titanium-nitride it could not be more than 6-7%) and nitrides precipitated during hot rolling (in this case 15-16%).
4. From the 34-36 ppm level, the free nitrogen content decreases (while the amount of precipitated nitrides increases) gradually to the centerline of the strip. The decreasing rate depends on the coiling temperature and on the cooling method at the run-out table.
5. The strip cooling method cause a measurable difference in the free nitrogen distribution curves. When the strip is cooled quickly just after the hot rolling, the free nitrogen in the middle regions is 2-8 ppm larger than cooling at the front of the coiler.
6. The background of the uneven distribution of free nitrogen along the strip width is the uneven cooling of the coil after coiling. The difference between the border and the centerline became more pronounced, when the coiling temperature is high.

Acknowledgement

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