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The effect of reheating conditions and chemical composition on δ ferrite content in austenitic stainless steel slabs

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The specimens, selected from different locations of as-cast 304 stainless steel slab, were annealed according to the temperature profiles, which simulated the industrial re-heating process before hot rolling. Annealing, following thermal cycles with maximum temperatures between 1230 and 1270 °C for a total time of 1 h, reduced the δ ferrite content, increased the size of the individual ferrite island and changed its shape to a more spherical one. An increase of annealing time to 1.5 h caused a drastic reduction in δ ferrite content and its further spheroidization. Moreover, the size of the individual ferrite islands was decreased. While after 1 h of annealing, the δ ferrite content depended on a particular thermal cycle and its maximum temperature; after a longer annealing time of 1.5 h, the δ ferrite content was very similar for all the thermal cycles applied. A statistical analysis of over 2200 industrial data, describing the slab chemical compositions and edge quality of the hot rolled plates was conducted. Generally, the alloying elements suppressing the δ ferrite formation improved the plate edge quality. In particular, carbon and nitrogen exerted the strongest influence, and the higher sum of both elements led to a lower probability of the edge cracking.

1. Introduction

The microstructure of 304 stainless steel, under equilibrium conditions, is composed entirely of austenite (γ). However, during the manufacturing process, e.g. continuous casting or welding, the cooling rates are usually high and some metastable δ ferrite can remain, due to an incomplete $\delta \rightarrow \gamma$ transformation [1]. The presence of δ ferrite degrades mechanical properties, and is detrimental for steel ductility during high-temperature processing. In particular, our results suggest a correlation between the content of δ ferrite and the edge cracking of hot-rolled plates [2]. A reduction of δ ferrite content in the austenitic matrix during hot processing is therefore of engineering interest.

Before hot rolling, the continuously casted steel slabs undergo thermal treatment while traveling through the re-heating furnace. The annealing, which affects phase composition, also improves hot ductility of the steel. According to experiments on the ingots of 304 stainless steel [3], the improvement gained by a long period of soaking at high temperatures of as-cast structures containing lingering ferrite areas, is not only caused by the dissolution of excess δ ferrite, but also by the changes in its morphology and distribution. The temperature distribution inside the industrial furnace is rarely uniform and, combined with a chemical segregation, causes the inhomogeneity of δ ferrite distribution inside the slab. As a result, the regions with an excessive content of δ ferrite will be prone to cracking during further hot processing.

This study focuses on the possibility of modifying by thermal treatment, the content and morphology of δ ferrite in as-cast stainless steel slabs. The laboratory tests were compared with a statistical analysis of the manufacturer data regarding the chemical composition, re-heating conditions and edge quality of the hot-rolled plates.

2. Experimental details

The specimens for investigation were cut from the slab of AISI 304 stainless steel after continuous casting. At first, three bars with the size of 125 × 33 × 33 mm were cut from both sides and the middle part of the slab with a cross-section of 130 × 12.5 cm, as shown in Fig. 1a. Then, the specimens having a size of 33 × 33 × 5 mm were cut from each bar, according to Fig. 1b. Heat treatment was performed in a laboratory furnace with programmed temperature changes versus time. In order to simulate the inhomogeneous distribution of the temperature inside the industrial furnace, the annealing of each specimen was conducted according to the individual thermal cycle, identical to the cycle, which different parts of the slabs undergo during the industrial process of slab re-heating. Thermal cycles with a maximum temperature between 1230 and 1271 °C were designed

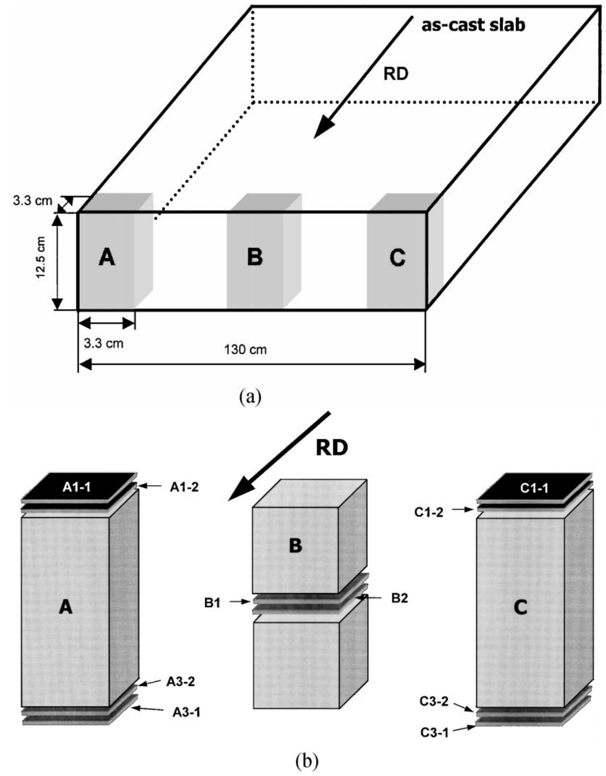


Figure 1 The schematic diagram illustrating the specimen selection from the slab: (a) location of the bars within the slab; (b) selection and marking of the specimens within the bars. Future rolling direction is indicated.

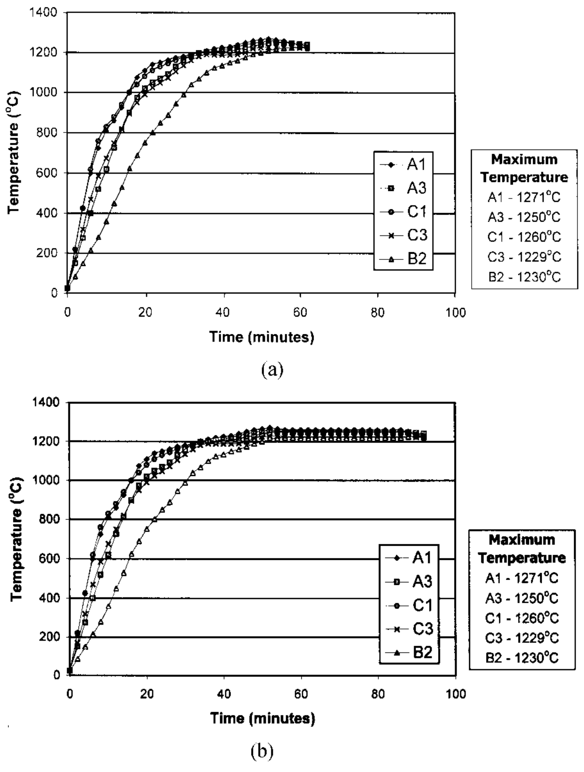


Figure 2 The thermal cycles for annealing in the laboratory furnace: (a) cycle with total time of 1 h; (b) cycle with total time of 1.5 h. The specimen names, heated according to the specific cycle are indicated.

on the basis of the industrial data for two total re-heating times of 1 h (exactly 62 min) and 1.5 h (exactly 92 min), with details shown in Fig. 2a and b.

The steel microstructure was analyzed using optical microscopy. To distinguish δ ferrite from the austenitic matrix, the polished sections were etched in a solution composed of 8 g NaOH and 80 ml H_2O. Electrolytic etching for 1 min at room temperature at a current density of 30 mA/cm^2 made the ferrite dark, while the austentic matrix remained unattacked. The chemical composition of the steel was analyzed using an emission spectrometer. In order to express the tendency for ferrite or austenite formation, the chromium and nickel equivalents were calculated, according to the following equations [4]:

$$\mathrm{Cr_{eq}} = \%\mathrm{Cr} + \%\mathrm{Mo} + 1.5x\%\mathrm{Si} + 0.5x\%\mathrm{Nb} \quad (1)$$

$$\mathrm{Ni_{eq}} = \%\mathrm{Ni} + 30x\%\mathrm{C} + 30x\%\mathrm{N} + 0.5x\%\mathrm{Mn} \quad (2)$$

A quantitative analysis of the ferrite content and morphology was conducted using an automatic image analyzer LECO 2000.

3. Results

3.1. Characterization of the as-cast microstructure

A typical microstructure of the steel slab after continuous casting is shown in Fig. 3. The dark phase represents δ ferrite, as was identified previously by transmission electron microscopy and electron diffraction analyses [5]. There are differences in morphology and distribution of δ ferrite within the slab. On the other hand, the morphology of δ ferrite in the vicinity of the slab surface can be classified according to Ref. [6] as vermicular or skeletal (Fig. 3a). On the other hand, δ ferrite located in the middle of the slab gathers in circular groups, which can be classified as an interlaced network structure (Fig. 3b). According to metallographical quantitative analysis, the volume fraction of δ ferrite was between 6 and 10% with the highest value in

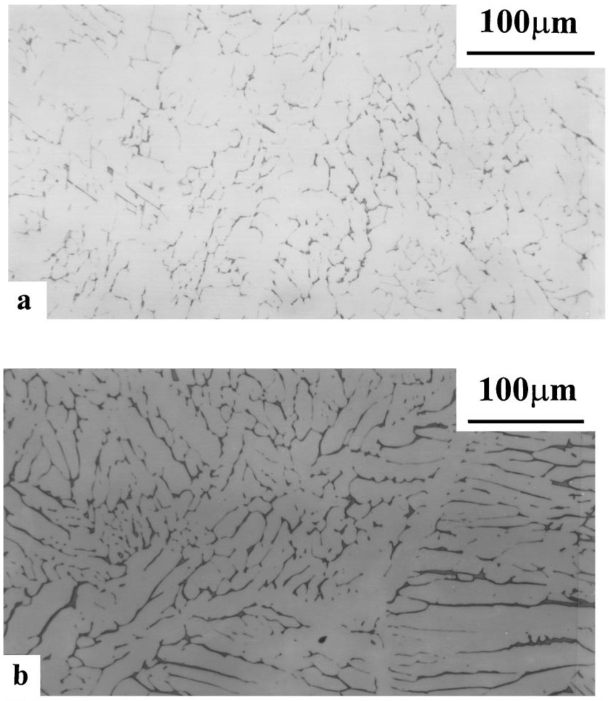


Figure 3 The microstructure of as-cast slab: (a) vicinity of the outer surface; (b) central region. A dark phase represents δ ferrite.

TABLE I

Specimen	C	Cr	P	S	Mn	Si	Mo	Ni	Cu	Co	Ti	N_2	Ni_{eq}/Cr_{eq}
A1	0.065	18.19	0.028	0.002	1.72	0.53	0.38	8.09	0.31	0.1	0.01	0.037	0.62174
A3	0.069	18.24	0.029	0.002	1.73	0.53	0.39	8.14	0.31	0.1	0.01	0.045	0.64273
B2	0.055	18.19	0.035	0.002	1.77	0.54	0.39	8.34	0.32	0.09	0.01	0.039	0.62112
C1	0.066	18.21	0.030	0.002	1.72	0.54	0.38	8.07	0.31	0.1	0.01	0.037	0.62268
C3	0.073	18.23	0.028	0.002	1.73	0.52	0.39	8.12	0.31	0.1	0.01	0.038	0.63634

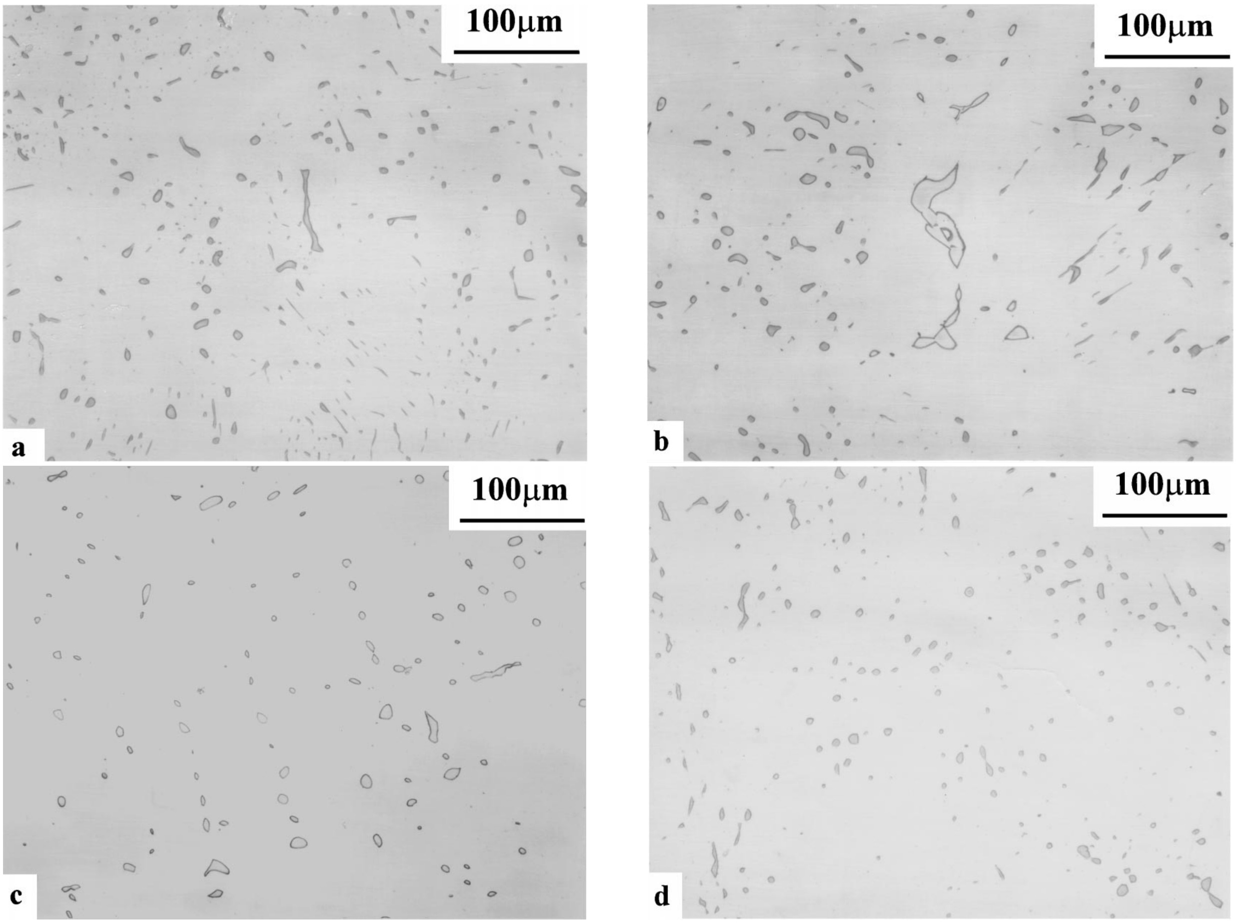


Figure 4 A typical morphology of δ ferrite after heat treatment: (a) surface region, 1 h cycle; (b) central region, 1 h cycle; (c) surface region, 1.5 h cycle; (d) central region, 1.5 h cycle.

the middle of the slab. Details for all the specimens examined will be compared later with the heat-treated conditions.

The chemical compositions of the different parts of the slab are shown in Table I. All the elements are within the ranges predicted by AISI for grade 304 [4]. In terms of the influence on δ ferrite formation, the most important differences are in the content of C and N. Although the range of the changes is rather narrow, the contents of C, N and the sum of both elements are lower in the middle of the slab (Table I). Similarly, the ratio of Ni to Cr equivalents, calculated according to the Equations 1 and 2 using the values listed in Table I, is lower in the middle of the slab.

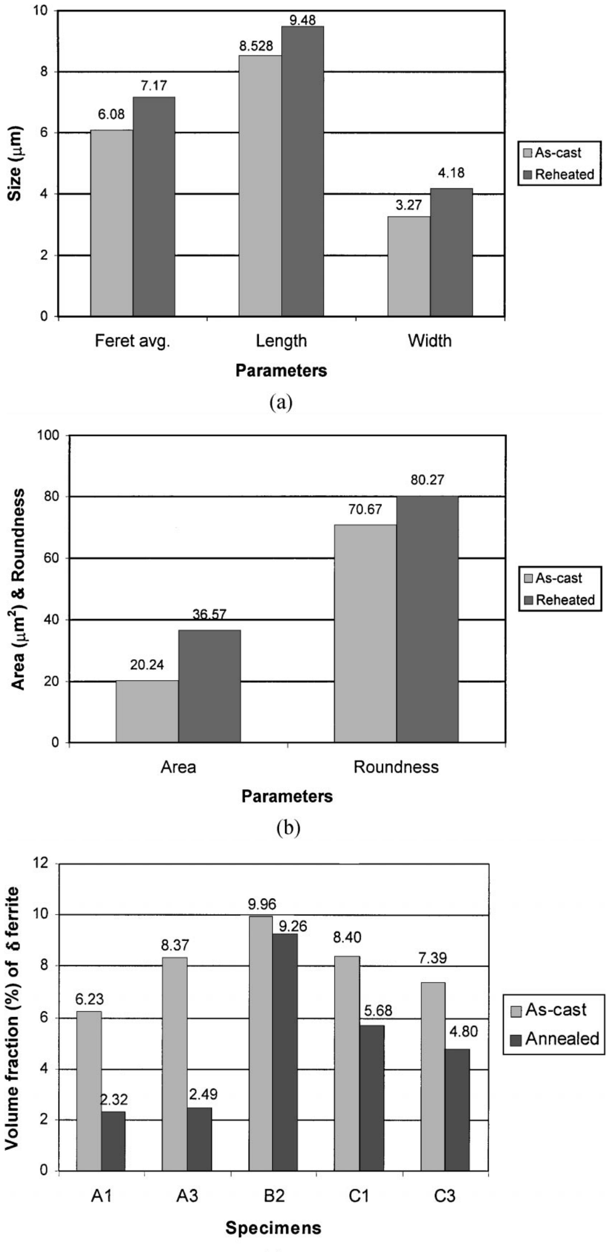
3.2. The steel microstructure after heat treatment

The heat treatment following the thermal cycles shown in Fig. 2a and b changed the slab microstructure. Some exemplary images, showing the morphology and distribution of δ ferrite are shown in Fig. 4a–d. A visual assessment of the slab microstructure suggests some differences both in the morphology and distribution of δ ferrite. In order to express these differences quantitatively, some stereological parameters describing the ferrite size, shape and volume fraction were measured using an image analyzer.

The annealing performed, according to the thermal cycles with a total time of 1 h, increased the size of individual precipitates of δ ferrite, expressed by length, width, Feret's diameter and the surface area (Fig. 5a and b). There was also a change in the shape of δ ferrite. This is expressed by roundness O, defined as follows [7, 8]:

$$O = 4\pi A \times 100/P^2 \tag{3}$$

where A is a particle area and P is its perimeter. For a perfect circle, the roundness is equal to 100. It is

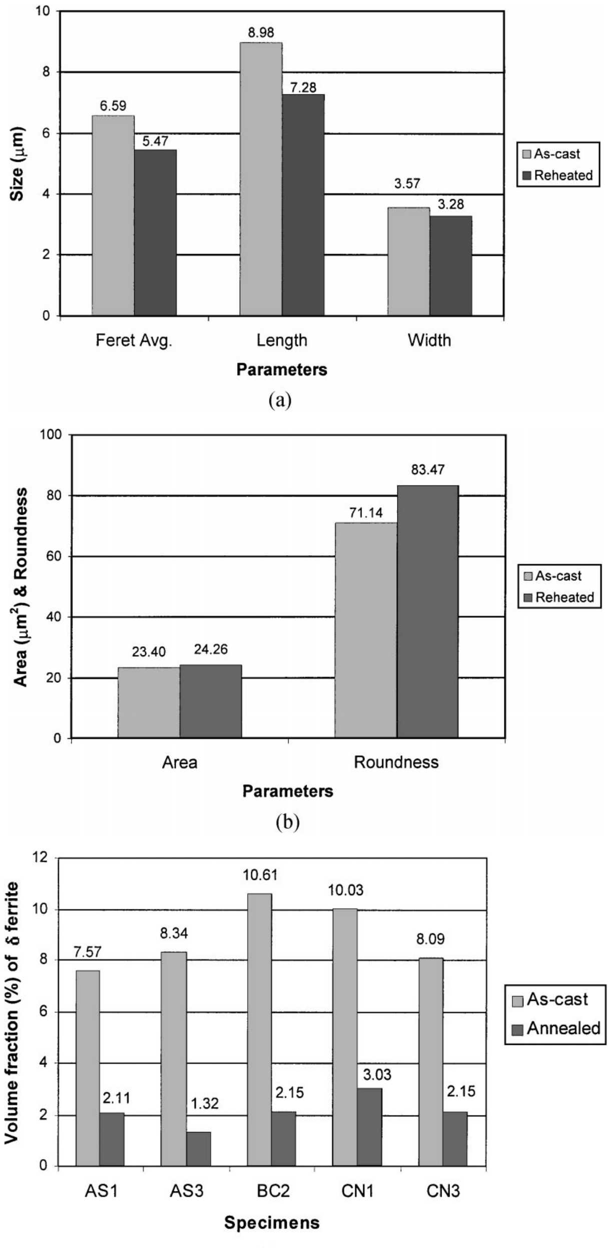


(a)

(b)

(c)

Figure 5 The results of the metallographical analysis of the content and morphology of δ ferrite after heat treatment following the cycles for 1 h: (a) (b) basic stereological parameters of individual δ ferrite precipitates in specimen C1; (c) volume fraction of δ ferrite for specimens cut from the different locations within the slab. The stereological parameters are defined in Ref. [7, 8].



(a)

(b)

(c)

Figure 6 The results of metallographical analysis of the morphology and content of δ ferrite after heat treatment following the cycles for 1.5 h: (a) (b) basic stereological parameters of individual δ ferrite precipitates in specimen C1; (c) volume fraction of δ ferrite for specimens cut from different locations within the slab. The stereological parameters are defined in Ref. [7, 8].

impossible to obtain exactly 100 with an image analyzer because of the square matrix created by the pixels. The best circle will usually have a roundness of approximately 95. An important result of the annealing was a reduction of the content of δ ferrite in all the specimens. This is shown in Fig. 5c, in comparison to the ferrite contents after casting. The smallest reduction of δ ferrite took place in the specimen representing the middle part of the slab. This part was characterized by the highest content of δ ferrite after casting.

The annealing performed according to the thermal cycles with a total time of 1.5 h reduced the ferrite's length, width and Feret's diameter while leaving their average area practically unchanged (Fig. 6a). The shape of δ ferrite was more spherical than after annealing for 1 h (Fig. 6b). These changes were accompanied by a drastic reduction in the volume fraction of δ ferrite in all parts of the slab (Fig. 6c).

3.3. The chemical composition of steel and edge cracking during hot rolling

The influence of steel's chemical composition on the edge quality after hot rolling was conducted using 2258 sets of data obtained from the industrial plant. The average content of chemical elements and their scattering were within the range predicted by the AISI. The edge quality of steel plates after hot rolling was characterized using the scale with numbers from 1, for the crack-free edge, to 8 for the ruptured edge with saw-pattern cracks with a depth over 13 cm from the plate edge. A detailed description of the manufacturer classification standard is presented in Table II.

TABLE II

Edge description	Code No.
Very good edge, no saw marks	1
Saw mark edge with length below 3 mm	2
Saw mark edge with length about 6 mm	3
Edge rupture with length below 12 mm	4
Edge rupture with length 12–127 mm, no saw marks	5
Edge rupture with length 12–127 mm, saw marks	6
Edge rupture with length above 127 mm, no saw marks	7
Edge rupture with length above 127 mm, saw marks	8

The alloying elements present in the steel, were divided into two groups in terms of their influence on ferrite formation. While the elements enhancing the formation of ferrite in steel are expressed by the chromium equivalent, the elements enhancing austenite are expressed by the nickel equivalent [4]. The dependence of the plate edge quality on the ratio of the chromium to nickel equivalents is shown in Fig. 7. The data were averaged for each edge quality number. It is clear that the elements reducing the amount of ferrite in steel have a beneficial influence on edge cracking. Of all the elements present in the steel, carbon and nitrogen exhibited the strongest influence on edge cracking. The highest sum of carbon and nitrogen prevented the edge cracking (Fig. 8).

Figure 7 The relationship between the average value of the ratio of the chromium to nickel equivalents, calculated according to the formulas 1 and 2, and the edge severity in steel plates. Results based on the analysis of 2258 operation data Numbers representing the edge severity are described in Table II.

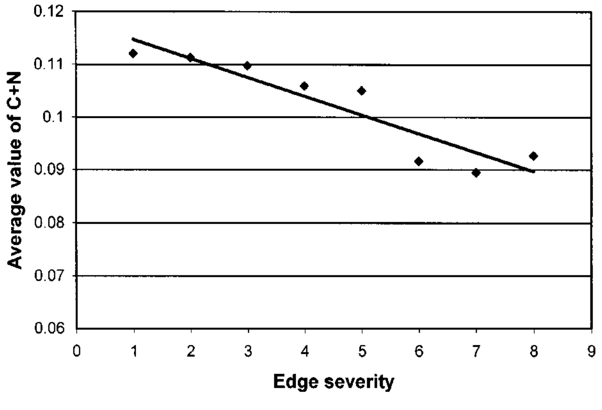


Figure 8 The relationship between the average value of the sum of carbon and nitrogen (C + N) and the edge severity in steel plates. Results based on the analysis of 2258 operation data.

4. Discussion

The content and distribution of δ ferrite in as-cast 304 steel slabs depends on cooling rates between the liquidus and solidus as well as within the solid phase, below peritectic transformation temperatures. While the first range determines the dendrite arm spacing, the latter controls the extent of the transformation to the austenite. The resultant content of δ ferrite in the steel slab is affected by the combined effect of dendrite arm spacing and diffusion time, although they have the opposite effect [6]. Our analysis of δ ferrite distribution within the slab indicates that there is 6.2 ÷ 10.0% of ferrite near the surface and of 10.0 ÷ 10.6% in the middle part (Figs 5c and 6c). This means that δ ferrite content is the lowest at the highest cooling rate and the highest at the lowest cooling rate. This is in agreement with Ref. 6, although there are some other investigations stating the increase of δ ferrite content with an increase in the cooling rate [9].

The content of δ ferrite in steel, before it enters the hot roll mill, is modified by pre-heating at temperatures above 1200 °C. Two factors have a significant impact on the ferrite level: nonuniform distribution of temperature within the furnace and the existing microstructural inhomogeneity. Direct temperature measurements, using thermocouples placed into the various slab regions, revealed the extent of these differences. First, the heating rate at the center of the slab and the maximum temperature it reached, are lower than in other parts of the slab. Moreover, the maximum temperature reached by the slab side marked as A1, is 42 °C higher than that reached by the opposite side C3. Comparing the temperature distribution (Fig. 2a) and δ ferrite content (Fig. 5c) one can see a correlation between these two parameters. Although there are some discrepancies in individual specimens, the general trend is that the higher energy supplied to the specimen during re-heating results in a larger reduction of δ ferrite content. This is clearly seen for an annealing time of 1.5 h. The differences between thermal cycles were not significant and the content of δ ferrite in all the specimens used to simulate different parts of the slab, was the same (Fig. 6c).

Annealing affects not only the δ ferrite content, but also its morphology, as summarized in Fig. 9. An increase in size, roundness and area of an individual ferrite island, is accompanied by reduction in volume fraction. This observation characterizes the steel after annealing following temperature profiles of the time period of 1 h, indicating that, in addition to the phase transformation of some ferrite to austenite and the ferrite spheroidization, some ferrite islands coalescend in this treatment (Fig. 9a and b). By contrast, the reduction in the size of the ferrite island after 1.5 h annealing suggests that the phase transformation of the existing δ ferrite to austenite is the dominating process (Fig. 9c). A comparison of the relatively small reduction of δ ferrite content, its nonuniform distribution dependant on the specific thermal cycle after 1 h annealing with a drastic reduction of the ferrite volume fraction for all the thermal cycles applied during 1.5 h treatment, supports the importance of the precise selection of annealing parameters in controlling the content of δ ferrite in 304 steel slabs.

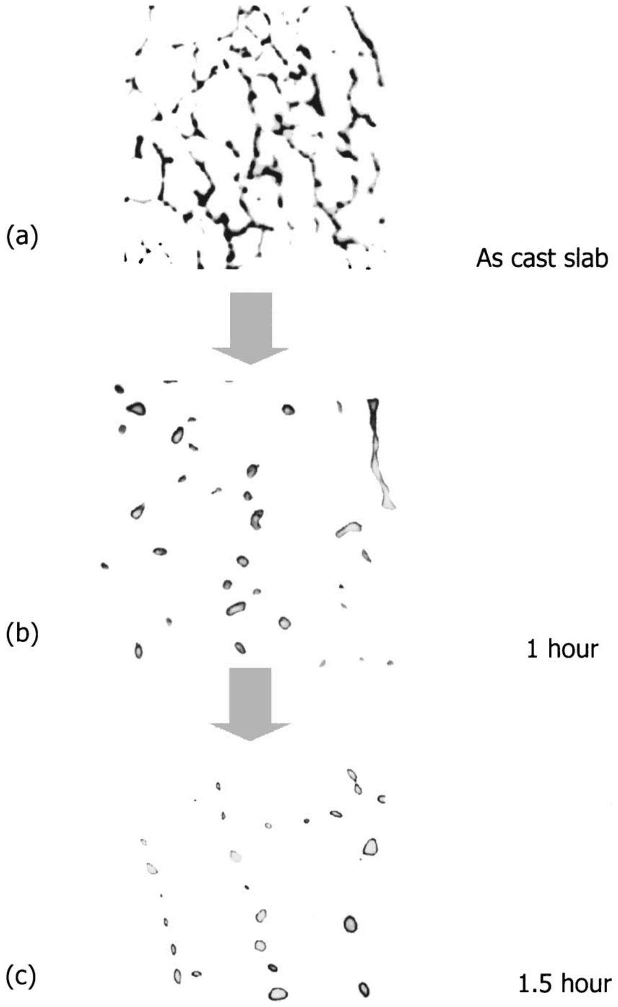


Figure 9 The morphological evolution of δ ferrite within the slab during the re-heating process: (a) as-cast morphology; (b) reduction of volume fraction, increase in the average size and roundness after 1 h of annealing; (c) reduction of volume fraction and the average size, and increase in the roundness after 1.5 h of annealing.

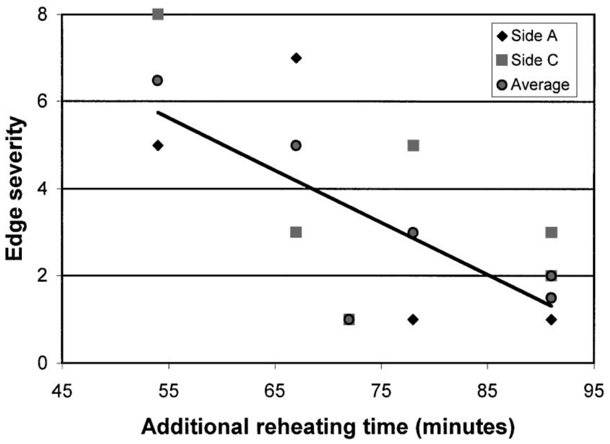


Figure 10 The relationship between the annealing time of the slab and the plate edge quality after hot rolling.

The role of steel chemistry in δ ferrite formation and edge cracking was investigated in our previous work [2, 5]. The statistical analysis of a large number of 2258 manufacturer's data presented in this study, supports the previous findings. It should be emphasized that the chemistry of all the specimens examined was strictly within the range provided by the AISI standard [4]. Within the narrow range of changes of $C + N$ between 0.009 and 0.113 (Fig. 8) or Cr/Ni equivalents between 1.545 and 1.597 (Fig. 7), the steel edge quality changes from a very good (number 1) to a very bad (number 8). An estimated δ ferrite content, based on the Schaeffler diagram [4] should change between 5 and 8%.

The above discussion shows that the metallurgical assessment of hot ductility of the steel by edge cracking requires the consideration of both the steel chemistry and the conditions of thermal pre-heating. While the slab chemistry predicts the low content of δ ferrite there is a respectively lower temperature and time range required to reach the appropriate ductility during hot rolling. However, there is possibly a second case when the slab chemical analysis predicts an excessive content of δ ferrite. Our results suggest that providing more energy to the slab by an appropriate increasing of the temperature, and/or re-heating time, may reduce the δ ferrite content and the possibility of edge cracking. In order to support the latter finding, the edge quality of 5 slabs is presented in Fig. 10. These slabs were additionally re-heated for time periods between 55 and 90 min as compared to the regular time. Although a number of data is limited, there is a clear improvement in the edge quality for longer heating times.

When tested in $\alpha + \gamma$ region, the two-phase structure of low-carbon steel is much less ductile than either of the component phases [10]. For austenitic stainless steel, the duplex structure is created in the presence of δ ferrite [1]. According to the hot torsion tests, the phase boundary cracking is responsible for early failure, where most of the deformation is concentrated in the stronger austenitic phase [11]. Therefore, assuming that the δ ferrite content is the key factor for hot ductility of the 304 steel, the precise control of the slab chemistry and the selection of re-heating parameters appropriate for that chemistry, will lead to improved hot ductility and prevention of edge cracking.

5. Conclusions

- As-cast slab of 304 stainless steel exhibited chemical and microstructural inhomogeneity. The lower content of carbon and the ratio of nickel to chromium equivalents in the middle part of the slab, were accompanied by a higher volume fraction of δ ferrite.
- The annealing of as-cast slab according to thermal cycles with a maximum temperature between 1230 and 1270 °C, affected the content, distribution and morphology of δ ferrite; however, the particular effect depended on the annealing time:

  A reduction of δ ferrite content, observed after annealing for 1 h was accompanied by an increase in the size of the individual ferrite island and a change of its shape to one more spherical.

  Annealing for a total time of 1.5 h caused a reduction in size of the individual ferrite island and its further spheroidization. The content of δ ferrite drastically decreased after 1.5 h-long annealing.

- The physical simulation of the re-heating process in the industrial furnace showed that the nonuniform distribution of temperature within the slab caused the inhomogeneity in δ ferrite distribution after annealing for 1 h. When the annealing time was increased to 1.5 h, the temperature distribution was not important.
- The statistical analysis of over 2200 manufacturer's data showed a correlation between the steel's chemical composition and edge cracking during hot rolling. Generally, the elements suppressing the formation of δ ferrite improved the edge quality. Of all the alloying elements present in the steel, the most effective were carbon and nitrogen: the higher the sum of carbon and nitrogen, the better the edge quality of the plates after hot rolling.

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