Thermoelectric Power Study of Nitride Precipitation and Recrystallization in Continuously-heated Low Carbon Al-killed Steels

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Abstract: This paper deals with the metallurgical phenomena occurring in aluminium-killed low carbon steels during industrial batch annealing process. The formability of these steels is strongly influenced by the nitride precipitation – recrystallization interaction during the batch annealing stage of the production technology. The accurate precipitation kinetics of nitrides is not clearly described yet because of the difficult evaluation of precipitated fraction of nitrides in cold rolled state steels. The aim of this study is to present a methodology for measuring the precipitated nitride fraction in cold rolled state, moreover to investigate the precipitation – recrystallization sequence during batch annealing process. Another purpose of this study is to give the conditions of the development of good deep-drawable microstructure. The nitride precipitation process in cold rolled and annealed state is measured using a special thermoelectric power test based methodology. On the basis of the experimental work, it is concluded that good formable microstructure develops if the precipitated nitride fraction reaches at least ~40% at the beginning of the recrystallization. This condition can be satisfied if the heating rate is held between 30 and 45 °C/h during the industrial batch annealing process.

Keywords: Al-killed low carbon steel; nitride precipitation; thermoelectric power

1 Introduction

1.1 Basics of Industrial Processing of Al-killed Low Carbon Steels

Aluminium killed low carbon steels are widely used for deep-drawing purposes due to their excellent formability. These grades of steels 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…0.02 Cr, 0.01…0.03 Cu, and low amount of Ti, Mo, Ni, Nb. In the manufacturing process of these steels it is essential to control the nitrogen content in liquid as well as in solid state [1]. It is recommended to avoid alloying with Ti and Nb. In this case, there is no possibility to form stable TiN particles, moreover the NbC particles will not increase the yield strength. Additionally, it is advantageous, if the Mn content of the steel is held around 0.17…0.2 w.t. %. Steels with larger Mn content exhibit lower deep-drawability. Some other important condition exists for producing good formable sheet, which can be satisfied with the proper thermomechanical processing of the steel. The time-temperature diagram of the production technology is shown in Fig. 1.

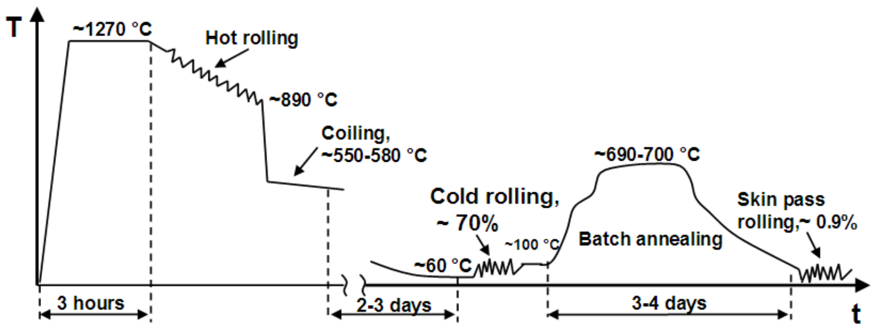

Figure 1
Production technology of Al-killed low carbon steels for deep-drawing purposes [1]

The production technology starts with the reheating of the slab to 1270 °C. It is followed by hot rolling in the austenite region with 890 °C average finishing temperature. At this point the strip is cooled down with water sprays to the coiling temperature. In order to keep the nitrogen in solid solution, the cooling rate after hot rolling must be fast enough and the coiling temperature must be low. Therefore the usual temperature range is 550-580 °C for coiling. The hot strip coil cools very slowly, with about 15-30 °C/hours cooling rate, depending on the position in the coil. The next step in the production technology is the cold rolling to around 70% thickness reduction, and it is followed by 3-4 day long batch annealing. According to the practical experience, if the free nitrogen precipitates in the form of aluminium and complex nitrides during the heating-up stage of the batch annealing process, then a good deep drawable microstructure develops [2]. Since the nitrides forming in cold rolled sheets are not only aluminium-nitrides but complex nitrides [3], moreover they can transform into other nitrides during heating [4], the term “nitride” will be used in this study. There are two advantageous effects of nitrides in the cold rolled sheet [5]:

1) If small, disperse nitride precipitates are present in the vicinity of dislocations and grain boundaries, the so-called pancake type microstructure develops, which decreases the sensitivity of the sheet to thinning.

2) During the recrystallization process, if small nitride precipitations are present in the cold rolled microstructure, then a good deep-drawable {111} texture will form.

It is not entirely clarified either complete or partial nitride precipitation should occur before the recrystallization starts. A possible change in the heating rate during the heating up stage of the batch annealing process largely influences the formability of the end product. The heating rate could be different even due to the large dimensions of the coil, so, in an indirect manner the formability and the mechanical properties could change significantly along the length of the strip. The knowledge of the nitride precipitation process in cold rolled state would be advantageous for the optimization of the batch annealing process.

The nitride precipitation in steels can be measured using different methods (chemical dissolution techniques [6], internal friction [4], resistivity [5], and numerous other methods [3]), but most of them provide inappropriate results if small, disperse nitride precipitations are present [3]. It was demonstrated that the measurement of thermoelectric power can be successfully applied to follow the nitride precipitation even in the case of small precipitates. Therefore in this study a special thermoelectric power test was used for determining the precipitated nitride fractions.

1.2 The Thermoelectric Power of Metals

The measurement of thermoelectric power (TEP) is a powerful method to quantify the microstructural changes occurring in metals. It can be used for investigating the effect of cold working [7], recovery and recrystallization [8], precipitation and dissolution processes [9-11]. It can provide the quantitative evaluation of free interstitial content of steels [12], so, in an indirect manner nitride- and carbide precipitation processes can be evaluated. The general set-up for measuring thermoelectric power of materials is shown in Figure 2 [4, 9].

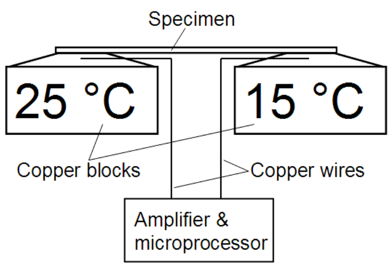

Figure 2

General set-up of a thermoelectric power measuring device

The sample under investigation is placed between two blocks made of high specific thermal capacity metal (usually copper blocks are used). The temperature of one of the blocks is held at 25 °C, whilst the temperature of the other one is cooled to 15 °C. Because of the temperature difference (ΔT=10 °C) between the two blocks, a temperature gradient develops in the specimen and in this way a thermoelectric voltage (U) is arising between the two copper blocks. This voltage is measured using a low noise nanovoltmeter. The ratio of the voltage (U) generated and the temperature difference (ΔT) is called thermoelectric power (TEP) and denoted by S [11]:

$$S = U / \Delta T \ . \tag{1}$$

The thermoelectric power strongly depends on the microstructural state of the given material. It is generally accepted that the larger is the distortion of the crystal lattice the smaller is the numerical value of the thermoelectric power [4, 7-9]. Thus, cold working, dissolution of precipitations into the matrix and cold working decrease the value of TEP, whilst recovery, recrystallization and precipitation of elements from solid solution increase it. It is necessary to know the effect of the amount of elements in solid solution on TEP. The change in thermoelectric power (ΔS) due to alloying elements in solid solution is described by the Nordheim-Gorter law [13]:

$$\Delta S = \sum K_{\mathrm{i}} \cdot x_{\mathrm{ss,i}} \ , \tag{2}$$

where K_i is a coefficient linking the amount of element i in solid solution ($x_{ss,i}$) with the change ΔS in TEP. Coefficients K_i for some elements are given in Table 1 [4].

Table 1
Values of coefficients K_i in Nordheim-Gorter law for different elements [4]

Element	C	N	Al	Cu	Mn
K_i μV/(K·wt%)	-45	-24	-30	-2	-3

The evaluation of amount of precipitated nitrogen in steels is based either on the measurement of the amount of precipitated nitrides or on the estimation of the amount of free nitrogen [3]. Using the thermoelectric power method, the latter can be applied. The precipitation kinetics in hot rolled (in low dislocation density) material can be followed by a special TEP methodology, which will be outlined in the next section.

1.3 Measurement of Free Nitrogen Content in Hot Rolled State

Massardier et al. [4] have developed a thermoelectric power based methodology, by which the free nitrogen content and thus the amount of precipitated nitrides can be quantified in soft (low dislocation density) material. Their methodology is based on the aging phenomenon occurring in steels. During aging, free interstitials are moving from solid solution to the vicinity of dislocations and grain boundaries, where they cause less distortion in the crystal lattice, so aging also changes the thermoelectric power. On the basis of this phenomenon, the free interstitial content (C and N) can be accurately (within 2-3 ppm precision) measured. In this way, the amount of nitride or carbide precipitations can be evaluated on the basis of the change of the free interstitial content. It was also verified by comparison of thermoelectric power and internal friction measurements that the effect of free nitrogen and carbon on thermoelectric power can be separated [4]. The principle of this methodology is illustrated in Figure 3. The aim of the methodology was to evaluate the precipitated nitride fraction after heat treatment of steel specimens at temperatures T_p=600…700 °C for different times t_p.

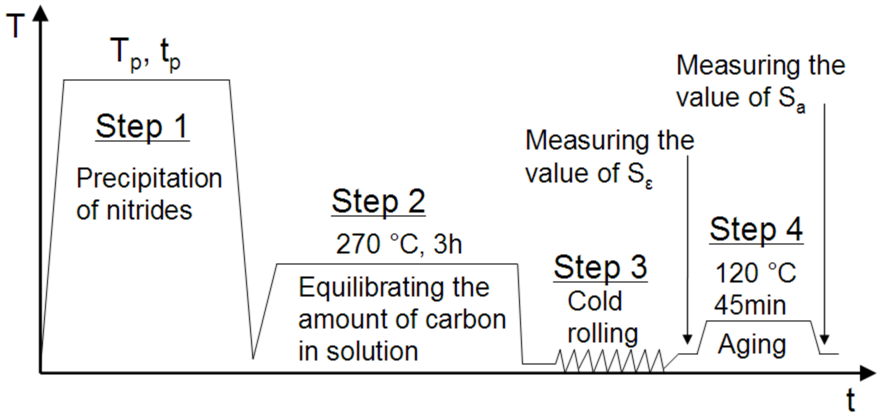

Figure 3

Method for measuring the nitride precipitation kinetics in hot rolled state [4]

It is obvious that after heat treatment at temperature T_p for any time t_p the amount of dissolved carbon and precipitated nitrogen can be quite different in each specimen. In order to ensure equal amount of carbon in solid solution for each sample, a low temperature treatment at 270 °C for 3 hours and water quenching was performed (Step 2). After this operation only a little amount of carbon (it is called residual carbon) remains in interstitial state (usually about 10-15 ppm) and according to the earlier studies [4, 12] this amount seems to be equal in all samples regardless to the prior treatment temperature (T_p) and time (t_p).

It is not possible of forming iron-nitride at this low temperature (270 °C) if the total nitrogen content of the steel is smaller than 0.01 w.t. % [14]. Moreover the precipitation of aluminium- and other nitrides is also unlikely because of the low diffusion rate of substitutional elements [3]. Another role of this treatment is to precipitate as much carbon from solid solution as possible, and ensure a reference state for all samples. The water quenching after the treatment at 270 °C results in a supersaturated solid solution of carbon and free nitrogen in steel. The next steps are designated to make visible the dissolved nitrogen and carbon. In the third step cold rolling has been followed by 75% reduction in thickness, this causes a large dislocation density microstructure. In this state the thermoelectric power of the samples denoted by S_ε was measured. The fourth step is an aging at 120 °C for 45 minutes, which causes the elimination of carbon and nitrogen from solid solution and transfers the interstitials to the vicinity of dislocations. It was proved that all of the nitrogen can be eliminated from solid solution by aging if the dislocation density is large enough [4]. In order to trap all of the free nitrogen in the vicinity of dislocations during aging, the cold reduction in Step 3 should be larger than 50%. If the nitrogen and carbon atoms are completely eliminated from solid solution, then they are invisible by TEP [4, 12]. After the aging operation the thermoelectric power of the specimens denoted by S_a was measured. The difference between the S_ε and S_a ($\Delta S_a = S_a - S_\varepsilon$) gives information about the free interstitial content of the steel. Using the Nordheim-Gorter law [13], the difference ΔS_a can be expressed as:

$$\Delta S_a = S_a - S_\varepsilon = K_C \cdot x_C + K_N \cdot x_N . \tag{3}$$

If all of the nitrogen has been precipitated (after treatment at high T_p temperature for long time), only the dissolved carbon atoms move from solid solution to the vicinity of dislocations. This means that only the so-called residual carbon contributes to the value of ΔS_a. In this case the amount of dissolved carbon at 270 °C can be calculated as:

$$x_C = \Delta S_a / K_C . \tag{4}$$

If the nitrogen is not completely precipitated as nitrides, the amount of free nitrogen can be expressed as:

$$x_N = \left(\Delta S_a - K_C \cdot x_C\right) / K_N . \tag{5}$$

Based on the above concept the precipitated fraction (Y) of nitrides can be evaluated as:

$$Y = 1 - x_t / x_M , \tag{6}$$

where x_t is the amount of free nitrogen at treatment time t_p, and x_M is the maximal free nitrogen content of the steel.

The aim of this study is to present a methodology for measuring the precipitated nitride fraction during continuous heating of the cold rolled strip, and to determine the general conditions for producing good formable sheets. Another purpose of this study is to present the effect of the degree of overlapping between the nitride precipitation and recrystallization on the cold formability.

2 Experimental

2.1 Material

The material investigated in this study is an aluminium killed low carbon steel having the following composition (in w.t. %): 0.044 C, 0.261 Mn, 0.009 Si, 0.008 P, 0.009 S, 0.028 Cu, 0.018 Cr, 0.015 Ni, 0.031 Al, 0.006 N, 0.001 Ti. The industrial processing of the steel started with the reheating of the slab to 1270 °C, which was followed by hot rolling to 4 mm thickness. The average finish temperature of hot rolling was 883 °C, whilst the average coiling temperature was 565 °C. After that the hot rolled strip was cold rolled to 1 mm thickness. Due to the low coiling temperature, most of the nitrogen content of the steel is expected to be in solid solution [1].

The free nitrogen content of the steel was measured in hot rolled state according to the concept presented in Fig. 3 and a value of 49 ppm was obtained.

2.2 Evaluation of Free Nitrogen Content in Cold Rolled and Partially Recrystallized State

To measure the free nitrogen in cold rolled or partially recrystallized state the methodology of Massardier et al. has been modified. The modified methodology is demonstrated in Figure 5. During the batch annealing process, 20-60 °C/hour heating rate is usual [1, 2]; therefore the first step of the modified methodology is heating up the cold rolled specimens with constant heating rate (20, 55 and 120 °C/h) up to temperature $T_i = 50…700$ °C. Then specimens were cooled down from every applied T_i temperatures with 10 °C/min cooling rate. After cooling down, the thermoelectric power of the specimens denoted by S_T was determined.

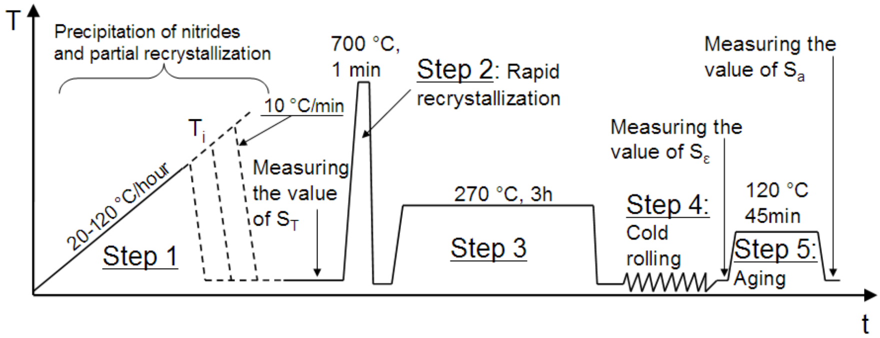

Figure 5

Method for measuring the nitride precipitation kinetics in cold rolled or partially recrystallized state

The specimens are either in cold rolled or in partially recrystallized state after the first treatment. In this state almost all of the free nitrogen and carbon in the steel is located in the vicinity of dislocations, so, a heat treatment at 270 °C would be ineffective for the amount of interstitials in solid solution.

Therefore an additional step, a heat treatment at 700 °C for 1 min was inserted in order to lower the dislocation density of the specimens. During this step, the recrystallization finishes completely, but the precipitated fraction of nitrides increases only with negligible amount. This fact is proved by measurement of free nitrogen content of the steel in hot and in cold rolled state. Approximately the same results were obtained in both microstructural states; it means that the rapid recrystallization (700 °C, 1 min) does not influence significantly the free nitrogen content of the steel. After the rapid recrystallization treatment, the specimens have a similar dislocation density as the hot rolled specimens after isothermal treatment at temperature T_p for time t_p (see Fig. 4). Therefore from this point the evaluation of free nitrogen content of the steel is exactly the same as in hot rolled state (see Fig. 4). The third step of the modified methodology is a heat treatment at 270 °C for 3 h, which was followed by a second cold rolling and aging (the fourth and fifth steps). The thermoelectric power of the specimens was measured before and after the aging treatment, and the free nitrogen content of the steel was determined by the same way as it was outlined in Section 1.3. The method described in this study provides the evaluation of the amount of precipitated nitrides 1-2 ppm precision, which can be advantageously used for estimating the uncertainty in various mathematical models [15].

2.3 Investigation of recrystallization process

In order to investigate the recrystallization process, the Vickers-hardness of the specimens was measured at room temperature after Step 1 (see Fig. 5) using a load of 70.63 N (7.2 kg). The hardness was measured in the midthickness of the sheet.

After the metallographic preparation of the samples, the recrystallized fraction has been also determined using the conventional point-counting method in the centre of the sheet.

2.4 Testing of Formability of Heat-treated Sheets

The formability is characterized with the plastic strain ratio (r-value) estimated from tensile tests and using Erichsen cupping tests. The r-value was measured on tensile test specimens prepared perpendicular to the rolling direction according to ISO 10113:2006 [16]. Square-shaped pieces were prepared to investigate their formability with Erichsen tests according to ISO 20482:2003 [17]. The cold rolled samples were heated with heating rate 20, 30, 55, 85 and 120 °C/h to 690 °C and held at this temperature for six hours. After cooling, three Erichsen and three tensile tests were performed on each group of specimens.

3 Results and Discussion

3.1 The Change of Hardness during Continuous Heating

Diagrams in Figure 6 show the change of hardness against temperature after heat treatment with different heating rates and final heating temperatures.

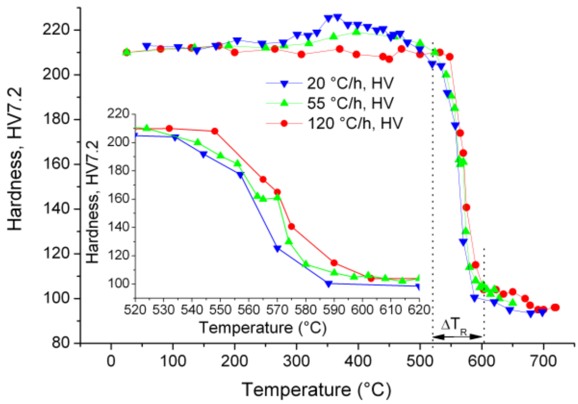

Figure 6
The hardness of cold rolled samples heated with different heating rate

As it can be seen in Figure 6, the heating rate influences the change of the hardness. At 20 and 55 °C/hour heating rate, the hardness increases in the temperature interval T=300…450 °C. The hardness change in this temperature interval is found to be 14 (± 2) and 7 (± 2) HV7.2 at heating rate 20 and 55 °C/h, respectively. In spite of that, no hardness change was detected at heating rate 120 °C/h between 300 and 450 °C. The hardness increase could be explained with the nitride precipitation process, which can occur at low heating rates (20-55 °/h), but it is not able to proceed significantly at large (120 °C/h) heating rate. This phenomenon will be explained in Section 3.3.

In the temperature interval (ΔT_R=525-600 °C) recrystallization occurs at all heating rate. The temperature range of recrystallization is shown in Fig. 6 and denoted by ΔT_R, whilst the start temperature of recrystallization is denoted by T_R. The shift of the temperature at 50 % recrystallized fraction due to the heating rate change from 20 to 120 °C/h is approximately 15 °C. The recrystallized fraction was measured by the generally used point-counting method after metallographic preparation of partially recrystallized samples. The relation between the hardness and recrystallized fraction is shown in Fig. 7.

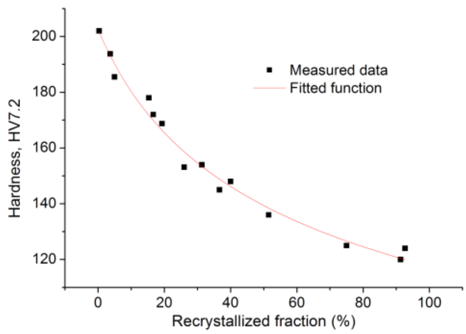

Figure 7
The relation between hardness and recrystallized fraction

In order to establish a mathematical relation between hardness and recrystallized fraction, a simple power function was fitted on the measured data:

$$HV = 212 - 12.2 \cdot X^{0.45} \tag{7}$$

where X is the recrystallized fraction. At the initiation of the recrystallization process (X=0) 212 HV7.2 hardness was measured, which equals to the hardness of the cold rolled sheet.

3.2 The Change of Thermoelectric Power during Continuous Heating

The thermoelectric power of specimens after Step 1 (see Fig. 5) denoted by S_T was measured for each sample. The change of TEP and hardness are plotted together against temperature in Fig. 8.

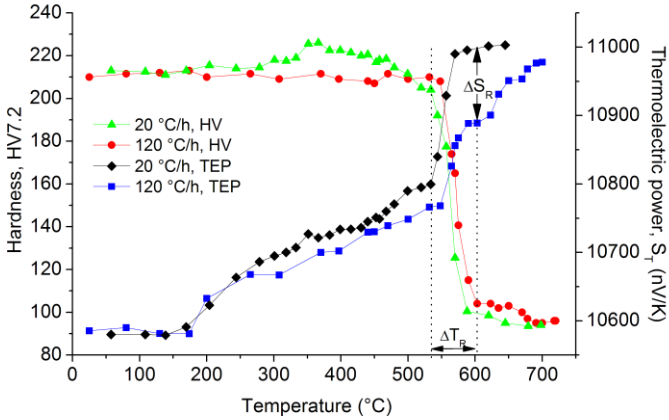

Figure 8

The relation between hardness and recrystallized fraction

No hardness and TEP change was detected below 200 °C. In temperature range 200…525 °C the TEP of specimens increases, but the change is more intensive at 20 °C/h than at 120 °C/h. In the temperature interval of recrystallization (ΔT_R=525-600 °C) the TEP significantly increases. At the end of the recrystallization process, a TEP difference denoted by ΔS_R (≈110 nV/K) appears between the specimens heated at 20 and 120 °C/h. This difference is attributed to the fact that at 20 °C/h heating rate much more metallurgical processes occur, which decrease more intensively the distortion of the crystal lattice. As it will be presented in the next section, the nitride precipitation process also contributes to difference ΔS_R. In the temperature range 600…700 °C (after recrystallization), the TEP of specimens heated at 120 °C/h increases, whilst at heating rate 20 °C/h no TEP change was obtained. The hardness of specimens after recrystallization is found to be a little bit larger (~ 105 HV7.2) at 120 °C/h than at 20 °C/h. The increase in hardness and TEP in temperature interval 600…700 °C is also attributed to the effect of nitride precipitation process.

3.3 The Change of Free Nitrogen Content of the Steel

The free nitrogen content of the specimens was measured according to the method outlined in Section 2.2 and demonstrated in Fig. 5. The change of the free nitrogen content of the steel is shown in Fig. 9.

In Fig 9, the temperature interval of recrystallization (ΔT_R) also indicated. The amount of free nitrogen at low temperatures (100-200 °C) equals the amount of it in hot rolled state (~ 49 ppm). This means that the rapid recrystallization operation (Step 2, 700 °C, 1 min, see Fig. 5) does not change the significantly the amount nitrogen in solid solution.

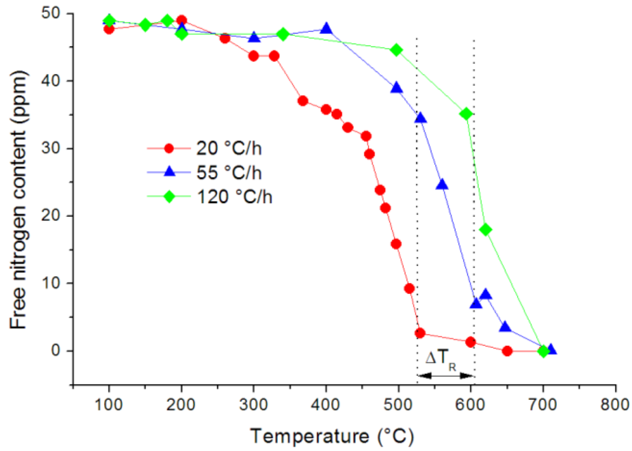

Figure 9
The change of free nitrogen content in continuously heated specimens

The nitride precipitation process precedes the recrystallization at 20 °C/h heating rate. At 55 °C/h, the precipitation and recrystallization occur concurrently, and a slight retardation in the recrystallisation can be also observed between 560 and 575 °C (See Fig. 6). At 120 °C/h the nitride precipitation proceeds mainly after the recrystallization finished. The difference ΔS_R in TEP after recrystallization (see Fig. 8) can be explained by the amount of free nitrogen and substitutionals in solid solution. After recrystallization, at heating rate 120 °C/h much more nitrogen (~ 30 ppm) is remained in solid solution than at 20 °C/h heating rate. Due to the crystal lattice distortion caused by the solute atoms, a lower TEP was measured at 120 °C/h heating rate after recrystallization. This solute nitrogen precipitates in the temperature interval 600-700 °C at heating rate 120 °C/h, which causes a TEP and a slight (7 ± 2 HV7.2) hardness increase (see Fig. 8).

For the better understanding, the change of free nitrogen content of the steel is plotted together with the hardness change for the experiments performed at 20 °C/h heating rate in Fig. 10.

At heating rate 20 °C/h the nitrogen precipitates mainly in cold rolled state in the temperature interval 250…520 °C. Between 250 and 450 °C, the third part of the free nitrogen precipitates as nitrides, this causes a slight hardness increase (~14 ± 2 HV7.2). At temperatures larger than 450 °C, the recovery, recrystallization and restoration of microstructure lower the hardness, and the effect of precipitates disappears.

In order to study the relation between the formability and recrystallization – precipitation sequence, some additional measurements were performed. The free nitrogen content of the steel was measured at the beginning of the recrystallization (at T_R = 525 °C) at heating rates 20, 30, 55, 85 and 120 °C/h.

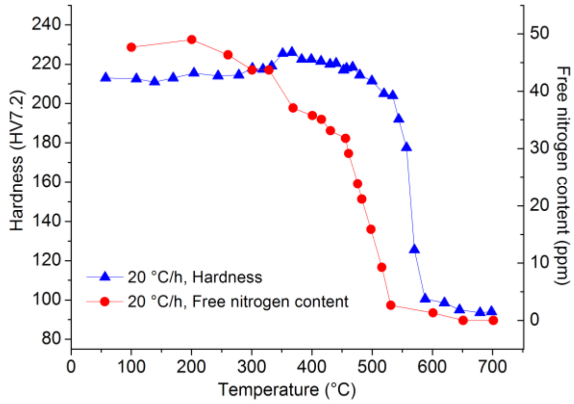

Figure 10

The change of free nitrogen content in continuously heated specimens

3.4 Formability test Results

The formability of heat treated sheets is characterized by the r-value determined using tensile tests and by the so-called cupping indices measured applying the conventional Erichsen-test. The results are presented in Fig. 11.

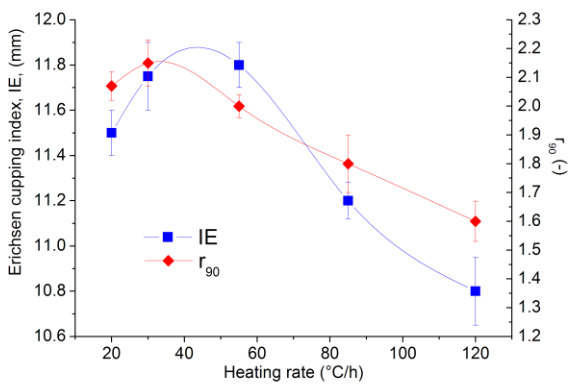

Figure 11

The change of the formability with heating rate during batch annealing

The formability reaches its maximum between heating rate 30 and 45 °C/h. The cupping indices measured at 30 and 55 °C/h heating rate are found to be almost the same (11.75 and 11.8). The correspondence between the nitride precipitation – recrystallization sequence and the optimal formability can be explained with the aid of Fig. 12. In this figure the precipitated nitride fraction was plotted against the heating rate at the beginning of recrystallization.

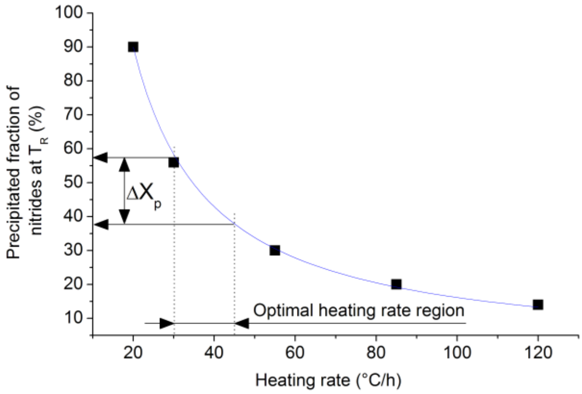

Figure 12
Effect of heating rate on the precipitated fraction of nitrides at the start temperature (T_R) of recrystallization

The precipitated nitride fraction at the beginning of the recrystallization depends on the heating rate. As it is revealed in Fig. 12, in the interval of the optimal heating rate (30-45 °C/h) the precipitated fraction of nitrides is ranging between 38 and 58%. According to the references [1, 3], the nitride precipitation can occur before or during the recrystallization. From the results presented in this section, it can be concluded that for ensuring good formability, the nitride precipitation should reach at least ~ 40% extent in the cold rolled sheet before the recrystallization process begins. On the basis of this observation, the industrial batch annealing process can be optimized by the adjustment of the heating rate during the heating up stage of the process.

Conclusions

The relationship between the formability and nitride precipitation – recrystallization sequence was investigated on 75% cold rolled low carbon aluminium killed steel. On the basis of the experiments, the following conclusions can be drawn:

1) The recrystallization process of 75% cold rolled sheet occurs in the temperature range 525-600 °C at heating rates between 20 and 120 °C/h. The shift of the recrystallization temperature is found to be 15 °C at 50% recrystallized fraction due to the heating rate change from 20 to 120 °C/h.

2) During the annealing treatment of the cold rolled strip, the nitride precipitation process occurs before the recrystallization at heating rate 20°C/h, concurrently the recrystallization at heating rate 55 °C/h and after the recrystallization at heating rate 120 °C/h.

3) The precipitation of 15 ppm nitrogen during heating up with 20 °C/h of the cold rolled steel results in a slight (~14 ± 2 HV7.2) hardness increase. The

possible further hardness increase due to the precipitation of nitrides is eliminated by the recovery and recrystallization.

4) The formability of cold rolled and annealed sheets reaches its maximum, if 30…45 °C/h heating rate is applied. In this case, r_{90}=2.1 and IE=11.7…11.8 mm was measured.

5) In order to obtain an optimal formability, the precipitated nitride fraction should reach ~38…58% at the beginning of the recrystallization.

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