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Identification of the parameters controlling the grain refinement of ultra-rapidly annealed low carbon Al-killed steels

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ABSTRACT

Ultra-rapid annealing (URA) cycles were used in order to refine the microstructure of cold-rolled low carbon Al-killed steel sheets. It was shown that the degree of grain refinement is controlled by the parameters (heating and cooling rates, annealing temperature) of the URA cycles and by the size, distribution and number of iron carbides formed during the coiling of these steels. In order to obtain a strong refinement, four conditions have to be fulfilled: (i) the initial microstructure of the steels before annealing has to be composed of numerous iron carbides finely distributed in the ferritic matrix, (ii) intercritical annealing leading to the formation of about 80% of austenite has to be performed, (iii) the heating rate (R_H) has to be higher than 500 °C/s and (iv) the cooling has to be rapid between 800 and 650 °C. From a microstructural point of view, the grain refinement is the result of three phenomena: (i) the inhibition of the grain growth of the primary ferrite grains by the iron carbides (below A_{C1}) and by the austenite nuclei resulting from these carbides (above A_{C1}), (ii) the development of the austenite grains above A_{C1} and (iii) the $\gamma \rightarrow \alpha$ transformation during cooling.

1. Introduction

Grain refinement of steels is an important issue for the improvement of the strength and toughness of these materials without addition of costly alloying elements. During the last 10 years, novel process techniques (frequently involving severe plastic deformation) have been developed to refine the microstructure of steels. In recent publications [1–4], it has been suggested that rapid transformation annealing (RTA) cycles may be used to obtain substantial grain refinement of low carbon steels. These cycles that can be performed either in the intercritical domain (between A_{C1} and A_{C3}) or in the austenitic domain combine rapid heating to the annealing temperature, a short holding time and rapid cooling. As can be seen in Table 1, these cycles differ from conventional continuous annealing cycles by the values of the parameters (heating rate (R_H), annealing temperature (T_a), annealing time (t_a), cooling rate (R_C)) involved in the annealing cycles.

According to [3,4], the degree of grain refinement of low carbon steels resulting from the use of RTA cycles is generally higher for austenitic annealing and is greatly influenced by the interaction that may occur in the intercritical domain between the recrystallisation and the ferrite-to-austenite phase transformation. In particular, the grain diameter of low carbon steels has been shown to decrease when this interaction becomes stronger. In the case of conventional low carbon Al-killed steels without any microalloying, Lesch et al. [3] did not observe significant grain refinement after RTA cycles (a minimum mean grain size of 6.5 μm was obtained) and they attributed this result to the fact that the aforementioned interaction is not important for this type of steels.

In this context, the aim of the present paper is to bring some insight into the possibilities of refining the grain size of low carbon Al-killed steels using ultra-rapid annealing (URA) cycles. For this study, the effect of the parameters of URA cycles and of the initial state of the investigated steels on grain refinement were considered, taking into account the fact that the initial state of the steels considered in this study is highly dependent on their coiling temperature. Namely, this process parameter is likely to influence: (i) nitrogen precipitation in the form of AlN nitrides and (ii) the size and the distribution of the cementite that forms during the coiling stage [5]. A high coiling temperature (≈700 °C) is generally associated with total nitrogen precipitation and with the formation of coarse pearlitic and/or intergranular cementite. On the contrary, a low coiling temperature (≈600 °C) generally leads to an absence of nitrogen precipitation and to the formation of fine cementite in the steel.

From an industrial point of view, it has long been established that the deep-drawability of continuously annealed low carbon Al-killed steels is of good quality only when these steels undergo a coiling at high temperature before annealing [5,6]. In these

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Table 1
Comparison of the heating rates (R_H), annealing temperatures (T_a), annealing times (t_a) and cooling rates (R_C) involved in CA or URA cycles.

	R_H (°C/s)	T_a (°C)	t_a (s)	R_C (°C/s)
Continuous annealing (CA)	10–50	700–800	30	10–100
Ultra-rapid annealing (URA)	100–1000	800–1000	0–5	100–300

conditions, the {1 1 1} ⟨u v w⟩ texture component, which promotes a high value of the mean plastic ratio (r_m), may develop. In contrast, if the steel was coiled at low temperature before continuous annealing, the fine iron carbides formed during the coiling stage may dissolve during the heating stage of the annealing, leading to the presence of carbon in solution during recrystallisation. This phenomenon is generally recognized as being detrimental to the development of the {1 1 1} ⟨u v w⟩ texture component [6]. Hence, the deep-drawability of low carbon Al-killed steels coiled at low temperature is less satisfactory. These steels are thus generally used in applications requiring high strength combined with acceptable ductility. As a consequence, grain refinement of these steels thanks to the use of URA cycles could improve their strength.

2. Materials and experimental procedure

Most of the work presented in this paper was conducted on a low carbon Al-killed steel produced industrially. It was coiled at low temperature (600 °C) before cold-rolling. It will hereafter be referred to as $LC_{(LCT)}$. Its chemical composition is given in Table 2. However, in order to investigate the effect of the initial state of the steel on the possibilities of grain refinement, URA cycles were also performed: (i) on a steel with a chemical composition close to that of the $LC_{(LCT)}$ steel and that underwent a coiling at high temperature (700 °C) (hereafter referred to as $LC_{(HCT)}$) and (ii) on a medium carbon Al-killed steel, hereafter referred to as $MC_{(LCT)}$, which differs from the $LC_{(LCT)}$ steel mainly by its total carbon content.

In all cases, the steels investigated in this work were reheated at high temperature, hot-rolled in the austenitic domain, coiled at high (700 °C) or low (580–600 °C) temperature and cold-rolled with a 75% reduction ratio leading to sheets 0.6 mm thick. During cold-rolling, the pearlitic and intergranular cementite formed during coiling was broken and aligned along the rolling direction (RD). Fig. 1 shows the initial microstructure of the investigated steels after cold-rolling or after cold-rolling followed by an annealing treatment performed at 700 °C (below A_{C1}). After such annealing treatment, the size, number and distribution of the iron carbides originating from the cementite that formed during the coiling stage can be easily detected on the micrographs. For the $LC_{(LCT)}$ steel, the initial microstructure is composed of fine iron carbides which are quite uniformly distributed in the iron matrix. These carbides were aligned along the rolling direction during the cold-rolling of the steel. In the case of the $LC_{(HCT)}$ steel, the carbides are much coarser and their distribution is not uniform. Lastly, in the case of the $MC_{(LCT)}$ steel, fine iron carbides aligned along the rolling direction are present as in the case of the $LC_{(LCT)}$ steel but they are much more numerous.

The different steels were ultra-rapidly annealed in a simulator based on induction heating and on cooling through inert gas jets on the steel sheets. This simulator, described in [7], was specifically built for the study. It enables steel sheets (60 mm wide by 100 mm long) to be treated with a precise control (±5 °C) of the temperatures thanks to the use of type K-thermocouples. Ultra-rapid annealing cycles were conducted in this simulator. These cycles consisted in: (i) rapid heating with a heating rate (R_H) within the range [25–1000 °C/s], (ii) holding time (t_a) at the annealing temperature (T_a) with $0\,s < t_a < 5\,s$ and $700\,°C < T_a < 1060\,°C$ and (iii) rapid cooling with a cooling rate (R_C) within the range [25–250 °C/s]. The parameters of the URA cycles were varied in order to determine their impact on grain refinement.

It has to be noted that the maximum cooling rate of the parameter study (i.e. 250 °C/s) was imposed by the simulator for steel sheets 0.6 mm thick. This is why a Gleeble 3500 thermo-mechanical simulator was also employed in this work when URA cycles requiring a high cooling rate of the order of 1000 °C/s had to be performed (as in the case of the studies presented in Sections 3 and 4.1). In this case, the rapid cooling rate was obtained by a water-quench.

The microstructure of the ultra-rapidly annealed samples was characterized: (i) through optical microscopy in order to determine the mean grain size of the steels, to follow the evolution of the morphology of the iron carbides initially present before annealing and to determine the austenite fraction formed during intercritical annealings and (ii) through thermoelectric power (TEP) measurements in order to evaluate the carbon and nitrogen content in solution after annealing. A linear intercept method was used to measure the mean grain sizes and no correction factor was applied. Furthermore, the carbon and nitrogen contents in solution in the ferritic matrix were assessed using a methodology based on TEP measurements and described in [8].

Moreover, the mechanical properties of the annealed samples associated with the $LC_{(LCT)}$ steel were measured through Vickers microhardness and through tensile tests. All the tests were carried out after annealing followed by an overageing treatment of 3 min at 350 °C performed in a salt bath. This overageing treatment is commonly used industrially to limit the ageing of continuously annealed low carbon steels. In the present work, this treatment was employed to precipitate the carbon remaining in solution in the steel after annealing and to reach a residual carbon content in solution identical for all the samples. Indeed, the objective of the present work was to identify the sole effect of the grain size on tensile properties.

For each investigated state, two tension specimens with a uniform gage section (5 mm wide by 30 mm long, 0.6 mm thick) were prepared from the sheets with their length parallel to the rolling direction (RD) and were tested at room temperature with a deformation rate of 0,1% per second. For specific studies, tensile tests were also performed on specimens with their length at 45° or 90° from the rolling direction.

Table 2
Chemical composition of the low and medium carbon Al-killed steels investigated in this work (in 10^{-3} wt.%).

Steel	C	N	Mn	Al	Cr	CT[a] (°C)
$LC_{(LCT)}$ $LC_{(HCT)}$	60	5	325	50	23	600 700
$MC_{(LCT)}$	140	3.3	425	40	25	580

[a] CT = coiling temperature; LCT = low coiling temperature; HCT = high coiling temperature.

3. Preliminary work: effect of the heating rate on recrystallisation and on the ferrite-to-austenite phase transformation of the $LC_{(LCT)}$ steel

Preliminary work was conducted in order to highlight the effect of the heating rate on the recrystallisation phenomena taking place in the $LC_{(LCT)}$ steel during the heating stage. For this study, the steel was heated at varying heating rates from 20 to 1000 °C/s to different peak temperatures (ranging from 650 to 800 °C with an