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Effects of accelerated cooling after rolling and of additions of Ni and Nb on the microstructure and mechanical behaviour of 0.1 wt% C steels

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ABSTRACT: *The study of the microstructure is necessary so as to investigate the effects of the parameters of interrupted accelerated cooling (IAC) on the mechanical behaviour of 0.1 wt % C, Ti or Nb + Ti microalloyed steels. The paper deals with larger variations of the cooling rate and of the finish cooling temperature than are usual in industrial practice. The work establishes: 1) the favourable effects of the highest cooling rate and for the Nb-free materials of a lower than usual finish cooling temperature, 2) the unsuitable character of a 0.4 wt% addition of Ni in IAC samples and 3) the chief advantage of a 0.0015 wt% microaddition of Nb. The metallurgical origins of these results are detailed.*

INTRODUCTION:

The stringent control of all the stages of the hot-rolling process (reheating, rolling and cooling) is necessary so as to reach the best mechanical behaviour for high strength low alloy (HSLA) steels by means of the single optimization of their microstructure. In particular, a combination of controlled-rolling and subsequent accelerated cooling can strengthen the steel while maintaining superior low-temperature toughness in spite of reduced contents of alloying additives or carbon in HSLA steels [1, 2]. This double goal requires the systematic study both of the effects of the numerous parameters of the thermomechanical process and of the steels chemical composition. In accordance with this presentation, the aim of this paper - concerned by the correlations between processing parameters, microstructure and mechanical properties of a 0.1 wt% structural steel- is twofold. It consists firstly in an investigation of the potentiality of large modifications of the cooling rate (CR) and of the finish cooling temperature (FCT) during interrupted accelerated cooling (IAC). Its second part deals with the effects of a 0.4 wt% addition of Ni and of 0.4 wt% Ni plus 0.015 wt% Nb additions.

MATERIALS AND EXPERIMENTAL PROCEDURES:

Steel A possess the 0.1 wt% C, 1.5 wt% Mn, 0.3 wt% Si, 0.025 wt% Al, 0.010 wt% Ti and 0.004 wt% N base composition. Alloys B and C are characterized respectively by additions of 0.4 wt% Ni and of 0.4 wt% Ni plus 0.015 wt% Nb. These steels were cast by continuous process. The materials' temperature was continuously registered during the thermomechanical treatment by means of a thermo-couple placed at the center of the plates. The 70 X 70 X 200 mm slabs were firstly reheated for 35 minutes at 1100°C before being controlled-rolled and finally cooled according to an interrupted accelerated (IAC) schedule. Controlled rolling was performed with a laboratory mill. The process comprises two subsequent stages. In the first one, the alloy was deformed over the 1040 to 1010 °C temperature interval, and therefore, in the recrystallization temperature range. The second phase of rolling was carried out at true strain of 1.6. Due to these thermomechanical parameters, at the end of rolling, steel C was totally unrecrystallized; whereas alloys A and B exhibited a residual deformation of 0.1 conforming with their 28% recrystallized state. These characteristics of recrystallization were computed by means of a model that gives the microstructure of austenite (grain size, residual deformation, level of recrystallization) at each stage of rolling. The 12 mm thick plates were then rapidly cooled by a series of water jets. At the onset of this accelerated cooling phase, the cooling rate of austenite (CR) was monitored at the 7, 15 or 25°C/s value by adjusting the water flow. This accelerated cooling was interrupted at a finish cooling temperature (FCT) comprised between 775°C and 200°C. The specimens were finally air cooled to room temperature. Aside from these IAC

samples, some plates were directly air cooled at 2°C/s to room temperature after controlled rolling (AC samples). For each rolling condition, 30 full size Charpy V and 3 tensile test specimens were machined in the transverse direction and at the midthickness of the plates. The 5 mm in diameter and 30 mm in gauge

length tensile samples were cut as close as possible to the 60 mm wide thermo-couple affected zone. The Charpy V notch is oriented perpendicularly to the plate surface. The tension tests were performed at room temperature and at the $1.5 \cdot 10^{-4} \text{ s}^{-1}$ strain rate. Moreover, three impact tests were systematically carried out at any test temperature. This paper gives the average values of the mechanical properties. Microstructural analysis was achieved by means of optical and thin foil transmission electron microscopy, with steels A and C in the AC and 7 and 25°C/s IAC conditions. With regard to optical metallography, it is worthwhile to note that, beside the usual 2% nital or picral reagents that reveal the whole microstructure, the Lepera reagent (1 volume. of 1% $\text{Na}_2\text{S}_2\text{O}_5$ aqueous solution + 1 volume of 2% nital) was proved very suitable so as to identify the martensitic second phases [3]. Moreover, the grain sizes of ferrite (α) were measured by the linear intercept method applied to more than 500 grains.

Figure 1: Variations of Y.S., T.S. and 50% FATT of steel C with the IAC parameters (CR and FCT).

steel C results (Figure 1). Figure 1 shows that IAC can be fruitful or not compared with the behaviour of the air cooled samples. At any CR value, the 0.2% offset yield stress (YS) or the ultimate tensile strength (TS) exhibit similar FCT dependencies which can be divided into three domains. At high FCT ($>T_1$), YS and TS increase both with the FCT reduction. At intermediate FCT, between T_1 and T_2 , the yield and tensile strength remain at constant values. At FCT between T_2 and T_3 , the YS exhibits a marked decrease contrary to TS that increases significantly with the FCT diminution. In consequence, whatever CR, the yield ratio (YS/TS) displays a marked decrease of quasi-constant magnitude over the $[T_2, T_3]$ temperature interval. Concomitantly with this variation, and as a general rule, the reduction of area at fracture decreases. In addition, and over the same $[T_2, T_3]$ domain, but except at the 25°C/s highest cooling rate, the toughness is highly impaired by the FCT reduction. This feature is the more marked, the lower CR. For instance, the 50% fracture appearance transition temperature (50% FATT) of steel C increases by $+70^{\circ}\text{C}$ at $\text{CR} = 7^{\circ}\text{C/s}$, but only by $+35^{\circ}\text{C}$ at $\text{CR} = 15^{\circ}\text{C/s}$, whereas, it remains equal to -115°C at $\text{CR} = 25^{\circ}\text{C/s}$.

Figure 2: The proof stress depends on the steels chemical composition ($\text{Cr} = 25^{\circ}\text{C/s}$).

RESULTS:

Mechanical behaviour: The evolutions of the steels mechanical properties with the IAC parameters (CR and FCT) are typified by the

Moreover, as illustrated by Figure 2 and table 1, the values of T1, T2 and T3 vary with CR and with the steel chemical composition.

Table 1: Effects of CR and of the steels' chemical composition on T1, T2 and T3.

Steel	A			B		C		
CR	7 °C/s	15 °C/s	25 °C/s	7 °C/s	25 °C/s	7 °C/s	15 °C/s	25 °C/s
T1 (°C)	520	520	420	560	420	640	620	580
T2 (°C)	440	-	-	460	280	500	480	440
T3 (°C)	-	-	-	370	-	370	350	330

Figure 3: Microstructures of the second phases of:

- a) type I (steel A, CR = 7°C/s, FCT = 680°C)*
- b) type II and III (steel C air cooled)*
- c) type III (steel C, CR = 7°C/s, FCT = 336°C).*

thickness and continuity of the bands decrease with the FCT reduction, and above all with an augmentation of CR. This diminution of the banded character of the microstructure entails a more homogeneous distribution of the second phases, and it is worth noting that this trend towards homogeneity is more

Microstructure: For any thermomechanical treatment, the steels microstructure is composed of second phases that are more or less randomly distributed in a ferritic matrix. As illustrated by Figure 3, three kinds of second phases, named I, II or III in this paper, are distinguished. The microstructure of the components I and III is rather simple corresponding respectively to pearlite and to a quasi-exclusive lath martensite. By way of contrast, the type II microstructure is highly polyphased. It seems of a medium quench kind. It comprises very predominant and highly dislocated ferritic grains, a significant volume fraction of small, equiaxed and rather undislocated grains of ferrite and a great number of intragranular carbides.

Air cooled samples: The AC samples contain bands of pearlite that are embedded in a matrix of ferrite with equiaxed grains. This kind of microstructure is observed in every parts of the plates, except for the midthickness of the steels B and C plates where the bands are of type III (steel B), and of types II and III (alloy C) (Fig. 3). The microprobe analysis of steel C connects this change of nature of the bands with their Mn and Ni contents. The central band contains more Mn and Ni (2.55 wt% Mn, 0.53 wt% Ni), than pearlitic bands (1.9 wt% Mn, 0.4 wt% Ni). Moreover, it should be noticed that in any steel, the Mn and/or Ni concentrations of the bands are always higher than those of the ferritic matrix. For instance, the chemical composition of the steel C ferritic matrix was checked close to the steel's nominal composition, i.e. 1.5 wt% Mn and 0.4 wt% Ni. In the same way, the Mn content at the midthickness of the steel A plate is close to 2.25 wt% compared with the mean 1.5 wt% of the alloy. Otherwise, the ferritic grain size of steels A and B is rather homogeneous and close respectively to 8.5 µm and to 7 µm. On the contrary, the $d\alpha$ value is less constant in steel C with an average of 5 µm.

IAC samples: The spatial repartition and kind of second phases depend both on the IAC parameters and on the steel chemical composition. As illustrated by Figure 3a, at the highest FCT, the microstructure remains of the banded kind found in AC samples. In contrast, at lower FCT, the

marked in alloy C than in steel A. The microstructure of the second phases vary simultaneously. The highest FCT gives rise to continuous bands of pearlite. At lower FCT between 600 and 500°C, the modifications of the microstructure of the plates depends both on the steel chemical composition and on CR. In the case of steel A rapidly cooled at CR=7°C/s, the bands located at the midthickness of the plates become of type II. In contrast, after an accelerated cooling with CR=25°C/s, steel A displays no more bands of type I, but only a thin band of type III in its midthickness; whereas steel C contains also no more bands of type I, but numerous bands of type II. At the lowest FCT, close to 300-350°C, and whatever the steel chemical composition, the second phases are very predominantly of type III. Moreover, and as already noted, these martensitic particles are the more finely distributed, the higher CR. In addition, the ferritic grains in close contact with these particles of martensite adopt most often a Widmanstätten morphology. This latter feature is the more marked, the higher CR and the higher the steel's hardenability. Otherwise, and as illustrated by table 2, the grain size of ferrite decreases markedly at the highest FCT values, and the more the higher CR. Together with this diminution, the ferritic grains become less and less equiaxed, and this modification increases both with CR and with the steel's hardenability.

Table 2: Effects of the steel's chemical composition and of the IAC parameters on the average value of $d\alpha$ measured in the midthickness of the plates.

Steel	A								C									
CR (°C/s)	AC	7°C/s				25°C/s				AC	7°C/s				25°C/s			
FCT (°C)	-	680	572	492	328	672	570	348	-	655	552	336	672	532	328			
dα (μm)	8.5	7	6.5	6.3	6	6.3	5.1	4.4	5	5	4.2	4.3	4	2.9	2.6			

DISCUSSION:

Effect of the thermomechanical treatment on the microstructure:

Grain size and morphology of ferrite: The ferritic grain size is governed by numerous parameters, i.e. the grain size ($d\gamma$) and the residual deformation ($D\gamma$) of austenite at the end of rolling, the cooling parameters and the effects of alloying elements on the $\gamma \rightarrow \alpha$ transformation temperature. In particular, according to the literature data [4], the grain size $d\alpha$ of the 0.05 to 0.25 wt%C, Mn and Nb microalloyed steels continuously cooled to room temperature at the CR α rate in ferrite from the finish transformation temperature is related to $d\gamma$ and $D\gamma$ by the phenomenological expression:

$$d\alpha = a \cdot CR_{\alpha}^{-b} \cdot d\gamma^c \quad (1)$$

$$\text{avec } a = 6,77 - 10(\text{wt}\%C) - (\text{wt}\%Mn)$$

$$b = 0,175$$

$$c = 0,4 - 0,25.D\gamma^{0,5}$$

According to equation (1), that indicates the favorable action of $D\gamma$ on ferrite nucleation [1], $d\alpha$ depends essentially on $D\gamma$ at high values of $D\gamma$. In agreement with this remark, the grain size of ferrite in the AC samples is smaller in steel C ($d\gamma = 34 \mu\text{m}$, $D\gamma = 1.6$) than in alloys A and B ($d\gamma = 12 \mu\text{m}$, $D\gamma = 0.1$). The previous values of $d\gamma$ and $D\gamma$ were calculated by means of the structural rolling model. Moreover, it is worth noting that the use of equation (1) is proved rather suitable in order to compute $d\alpha$ for steels A and C, but less convenient for steel B. The calculated value of $d\alpha$ is equal to 5.8 μm for steel C, and to 9.7 μm for alloys A and B. These estimates are rather close to the measurements in steel C (5 μm) and in alloy A (8.5 μm), but a little too high for steel B ($d\alpha = 7 \mu\text{m}$). This discrepancy originates from the loose fit of equation (1) to the case of steel B out of ignorance of the hardenability effect of Ni. Otherwise, and for the IAC samples, the smaller grain size of ferrite in alloy C than in alloy A originates from the same parameters, i.e. $D\gamma$ and the hardenability effects of Ni and Nb [5]. Moreover, $d\alpha$ varies with the modifications of the IAC parameters. The increase of CR entails a decrease of $d\alpha$ (table 2) because of the diminution of the temperature of the $\gamma \rightarrow \alpha$ transformation. Due to this temperature lowering, the driving forces of the ferritic transformation, and more especially of the nucleation of ferrite are exacerbated. In the same way, the FCT diminution brings about a further decrease of $d\alpha$ because of its slackening effect on the diffusion-controlled growth of ferrite. Regarding the morphology of ferrite in the IAC samples, it is less and less equiaxed with the decrease of the FCT and the increases of CR and of the chemical hardenability of the steel. This evolution is explained by the temperature of the ferritic transformation. At medium temperatures of transformation, the phenomenon originates from the asymmetry of the diffusion of carbon in austenite [6]. At the lowest temperatures, corresponding to the formation of Widmanstätten ferrite, the

peculiar morphology of ferrite is linked with the quantity of austenite (γ) that transforms. The quantity of transformable γ increases with CR and with the steel's hardenability, with the consequences both of greater amounts of latent heat of transformation and of carbon to evacuate from the transformed parts of the specimens. These scavenging phenomenae are promoted by the acicularity of Widmanstätten ferrite that enlarges the area of contact between ferrite and austenite.

Microstructure and distribution of the second phases: The banded microstructure arises both from the segregation of substitutional elements, here Mn and Ni, during solidification, and from the subsequent shaping by rolling of these segregated areas in form of pancakes parallel to the plates surfaces. Due to these phenomenae, during cooling the ferrite nucleation begins at first in the bands impoverished in γ -stabilizing elements. The low solubility of carbon in ferrite should therefore entail the scavenging of carbon atoms to the γ -stabilized areas. The level of carbon in these γ -stabilized zones is governed by its ease of diffusion.

The microstructure of the bands located in the midthickness of the air-cooled (AC) plates depends more definitely on the "rolling hardenability" of the materials. This hardenability in rolling is governed not only by the usual chemical hardenability of the steels, but also by $D\gamma$. For instance, the observation of bands of martensite in steel B, but of pearlitic bands in steel A, agree well both with the same $D\gamma = 0.1$ value for the two materials, and with the increase of the chemical hardenability of steel B due to its Ni-content. On the contrary, the lower hardenability of steel C, compared with that of steel B, displays a deformation rather than a chemical origin. The greater value of $D\gamma$ in steel C (1.6) than in steel B (0.1) accelerates the $\gamma \rightarrow \alpha$ transformation in alloy C. This effect originates from the slackening action of niobium on the recrystallization of austenite [1] that overcomes the slowing effect of the same element in solid solution on the nucleation of ferrite [5].

The microstructure of the IAC samples depends on the parameters of accelerated cooling that control the diffusion of carbon. The homogeneity of the distribution of the second phases is promoted by a high value of CR and by a low FCT temperature. The highest value of CR gives rise to the formation of a greater number of ferrite nuclei that are randomly located. Moreover, it is worth noting that this transformation occurs at rather low temperatures. Due to these low temperatures, the rejection of carbon occurs only at short distance from the primary ferrite nuclei with the outcome that the second phases, formed in place of these carbon enriched zones, will be more homogeneously distributed than in a specimen cooled at a lower rate. In contrast, at low CR values, the diffusion of carbon occurs over longer ranges to the Mn and Ni segregated bands that become therefore significantly enriched in carbon. In consequence, according on the one hand to the CR and FCT values, and on the other hand to their content in γ -stabilizing elements, the carbon enriched zones will transform by diffusion-controlled (pearlite), displacive (martensite) or semi-displacive (bainite) reactions.

Correlations between the microstructure and the mechanical properties:

Yield Strength: Whatever the thermomechanical history of the plates, the YS is equal to the stress necessary for the onset of plastic deformation of the softer phase, i.e. ferrite. This stress depends on the FCT according to three successive relationships defined over the temperature intervals bounded by the temperatures T_1 , T_2 and T_3 (see table 1). The thorough study of the steels A and C indicates however that the microstructural origin of these similar laws may differ with the material. Figure 4 shows that, at FCT

Figure 4: Hall-Petch relationship in steel A

higher than T_1 , the YS of both steels depends on d_{α} according to a Hall and Petch relationship. At lower FCT between T_1 and T_2 , the constancy of YS agrees again with the steady value of d_{α} . This predominant effect of the grain size of ferrite upon the YS remains indeed likely in steel C at any CR, and in steel A at CR = 7°C/s because of their equiaxed ferritic structure. On the contrary, and as shown by the point arrowed on figure 4, the Hall - Petch law is no longer verified by steel A, accelerated cooled down to $[T_1, T_2]$ at CR = 25°C/s. This disagreement seems to arise from the peculiar microstructure of steel A due to its very low value of T_1 after this thermomechanical treatment. This microstructure consists of a random distribution of second phases of the semi-displacive or displacive type in an acicular ferritic matrix. It can be guessed from this observation that thorough studies of the effects of the morphology of ferrite and of the distribution and microstructure of ferrite and of the distribution and microstructure of the second phases on YS should be of the greatest importance. Moreover, the decrease of T_1 with the increase of CR suggests the effectiveness of

Nb(CN) particles so as to stop the growth of the grains of ferrite at T₁ in steel C. Further studies of the precipitation phenomenae are however needed to ascertain this a priori astonishing hypothesis [7]. At FCT lower than T₂, YS of alloy C decreases drastically. This diminution occurs together with the disappearance of a yield point on the tensile curves, and together with the formation of martensite. These features arise because either of a too loose concentration of interstitial atoms in the dislocations cores, or of a too high density of mobile dislocations in ferrite.

Reduction of area at fracture, toughness and yield ratio: The variations of ductility and of toughness are correlated with the distribution and microstructure of the second phases that act as obstacles for the plastic deformation. These effects of the second phases are more prominent in impact than in tension testing, because of the high strain rates that reduce the magnitude of deformation of the softer ferritic matrix. Furthermore, and as a more general rule, the banded type of distribution of the second phases is harmful to toughness. This phenomenon arises from the strain partitioning effect that concentrates the deformation in the softer ferritic phase rather than in the stiffer second phases. This heterogeneous deformation entails stress concentrations in close contact with the interfaces of the second phases. Moreover, it is worth emphasizing that this stress raising effectiveness increases with the continuity of second phases because of the hindering of the strains accommodation. Similar observations have previously been reported for HSLA steels with regard to the mechanical behaviour of their intercritical heat affected zone in welding [8]. This explanation applies in particular for the improvement of the steel C toughness with the increase of CR at FCT lower than T₂ (see Figure 1). Furthermore, and in the same way, the increased deterioration of the steel C toughness with the FCT diminution below T₂ is likely to arise from the change of microstructure of the second phases with the consecutive enhancement of their stiffness. Otherwise, the same causes, i.e. the enhancement of the displacive character of the second phases, seem to bring about the significant decrease of the yield ratio of steel C that occurs at lower than T₂ FCT temperatures.

IAC benefits: Figures 1 and 2 prove the double advantage of interrupted accelerated cooling so as to improve the strength (YS and TS) without impairment of toughness, and to ensure a given mechanical behaviour in spite of a decrease of the content in alloying elements. The first benefit is evident for the three alloys. For instance, and as illustrated by figure 1, steel C exhibits at one and the same time a very good level of toughness characterized by a 50% FATT of -115°C and a 525 MPa yield strength, that is 65 MPa higher than that provided by controlled rolling. The second point of interest arises from the comparison between alloys A and B. This comparison indicates that after an IAC treatment defined by CR = 25°C/s and an FCT value close to 350°C, the yield strength of the Ni-free alloy A is equal to that of the Ni-bearing steel B (figure 2). Moreover, it should be noticed that this performance is observed together with a small improvement of toughness without Ni. Similar results are observed at CR = 7°C/s. On the contrary, the benefit of alloying is demonstrated, after any thermomechanical treatment, by the Nb-microalloyed steel C that exhibits higher combinations of yield strength and toughness than the Nb-free steels A and B.

CONCLUSIONS:

Interrupted accelerated cooling leads to higher combinations of strength and toughness than single controlled rolling. These combinations are optimal with the higher cooling rate and for intermediate FCT between T₁ and T₂. Moreover, it is worth emphasizing that the necessary FCT temperatures may be lower than in usual practice. With the present steels, the increases in strength due to IAC are able to establish the outmoded nature of a 0.4 wt % Ni alloying. These advantages of IAC arise both from the decrease of the ferrite grain size and from the more even distribution of the second phases. The microstructure of these second phases is governed by rolling hardenability (chemical composition and level of deformation of austenite) and by the cooling parameters. The thorough study of the effects of these second phases seems essential so as to understand the microstructural dependency of mechanical behaviour.

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