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Processing of ultra low carbon steels with mechanical properties adequate for automotive applications in the as-annealed condition

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Abstract

A series of ultra low carbon/Ti added steels were produced with the aim of evaluating the steelmaking route and processing conditions of slabs, in order to achieve mechanical properties on resulting annealed sheets adequate for automotive applications. Characterization of microstructure was carried out in the as-cast, deformed and annealed specimens by means of scanning and transmission electron microscope techniques. Slab specimens were hot rolled, coiled, cold rolled and annealed. From the resulting annealed sheets, evaluation of mechanical properties in terms of the percent of elongation and the normal anisotropy ratio was carried out on three experimental ultra low carbon/Ti added steels from heats of 230 t. From the resulting mechanical properties, it was noticed that the sheet steels achieved very high formability, especially for parts requiring good deep drawability, adequate for automotive applications. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Interstitial free steels; Ti stabilized; Normal anisotropy ratio; Mechanical properties

1. Introduction

Improvements in pellet workshop, direct reduction process, steelmaking practice, vacuum degassing, ladle refining treatment and continuous casting of slabs have led to the production of steels containing microalloying constituents which are controlled in parts per million.

With regards to alloy composition, there has been a great improvement in steelmaking in order to allow, for instance, production of steels with low interstitial elements — carbon (C) and nitrogen (N) and with low sulfur (S) and low phosphorus (P) contents [1]. The steelmaking improvements have allowed the production of ultraclean steels for automotive applications.

Vacuum degassed interstitial free (IF) sheet steels have very high formability, especially for parts requiring good deep drawability [2]. The high formability is achieved by lowering the interstitial element content to very low levels in steelmaking and by additions of stabilizing elements such as titanium (Ti) or niobium (Nb) which combine with the N and C not removed by the steelmaking process [2,3]. At the end, the chemical composition of IF steels must be adjusted in order to satisfy the different requirements that are put on modern sheet steels.

On the other hand, it has been reported [4], that the ferrite matrix of heavily cold rolled IF sheet steels recrystallizes during annealing to a polycrystalline structure with a strong (111) $<\bar{1}10>$ recrystallization texture, producing high values of the average normal anisotropy ratio, $\bar{r}$, which is associated with the high formability of ultra low carbon IF steels.

The deep drawing characteristics of IF sheet steels, in terms of the measured normal anisotropy ratio from a tensile test, are strongly dependent upon the development of strong {111} recrystallization texture during in line annealing [5]. Furthermore, during the process of removing interstitial elements by using microalloying elements, precipitates could be left in the matrix that may adversely affect the recrystallization process [6]. Ti left in excess [7] in the matrix is far less effective than Nb in retarding the recrystallization process, but this characteristic is considered a distinct advantage in the

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design and processing of Ti stabilized interstitial free steel for extra deep drawing grades.

The aim of this study is to present results of mechanical properties obtained in ultra clean/Ti stabilized annealed sheets in terms of its percentage of elongation and its average normal anisotropy ratio in order to evaluate the steelmaking route and processing conditions of slabs in order to obtain mechanical properties which would be adequate for producing parts which require good deep drawability.

2. Experimental

The steelmaking process to produce the experimental steels involved the use of 100% sponge iron with high metallization grade, which was fed into an electric arc furnace of 264-t capacity. During the melting of the sponge iron, 8.6 t of $Ca(OH)_2$ were added to it, with a total melting time of ~ 140 min. Afterwards, the 230 t of steel were poured into a ladle furnace with a tapping temperature of 1699°C and 100 kg of Al were added to the steel.

In addition, a vacuum degassing procedure was carried out for a period of 48 min, reaching a pressure of 0.5 Torr, during the first 5 min. Immediately after the vacuum degassing operation, additions of Al, CaF_2, $Ca(OH)_2$, FeSi, FeMn and FeTi were made, with an operation time of ~ 76 min. Throughout this period, argon was fed. After compositional adjustments, continuous casting of the steel was carried out, with the resulting slabs being 25 cm thick and 110 cm wide. The experimental heats were identified as U430/40133 (sample 1), U430/42229 (sample 2) and U430/30119 (sample 3), and their chemical compositions are shown in Table 1.

Table 1
Chemical composition of samples of slab (in wt.%)

Element	U430/30119 sample 1	U430/42229 sample 2	U430/40133 sample 3
C	0.004	0.005	0.005
Mn	0.098	0.085	0.11
Si	0.012	0.023	0.030
P	0.004	0.004	0.004
S	0.008	0.012	0.010
Al	0.031	0.042	0.071
Nb	0.001	0.003	0.000
V	0.001	0.000	0.001
Cu	0.01	0.010	0.015
Cr	0.000	0.000	0.000
Ni	0.012	0.008	0.013
Sn	0.001	0.000	0.001
N_2	0.003	0.0048	0.0044
Ti	0.061	0.075	0.069
B	0.000	0.003	0.000

Table 2
Dimensions of slab samples previous to the hot rolling operation

U451/30119 sample 1 (cm)	U430/42229 sample 2 (cm)	U430/40133 sample 3 (cm)
13.5 × 5.0 × 1.2	9.0 × 5.0 × 1.1	10.0 × 5.0 × 1.0
13.5 × 5.0 × 1.2	9.0 × 5.0 × 1.1	10.0 × 5.0 × 1.0

Slabs specimens with dimensions shown in Table 2, were re-heated in a resistance furnace under an argon atmosphere to a rolling start temperature of 1250°C (30 min), in order to carry out the hot rolling of slab specimens of ultraclean steel. For that operation, a hot rolling (Hilly) mill of 50-t capacity (15.24-cm roll diameter) was used. During the rolling operation, a roll velocity of 24 cm/s was employed and the specimens were hot rolled until $\approx 64\%$ of total reduction (Table 3) was achieved, finishing the hot rolling operation at 950°C. After hot rolling, a coiling operation was simulated by cooling the specimen down to 700°C and keeping it for 30 min at that temperature in a resistance furnace and then air cooling it.

In order to carry out the cold rolling operation a Fenn mill of 25-t capacity was used (with 12.5-cm roll diameter). During the cold rolling operation, a speed of 20 cm/s was employed and the specimens were lubricated by using a solution of 10 vol.% of natural oil in water. The deformed specimen achieved a final thickness of about 0.05 cm in 11 steps and $\approx 84\%$ of total reduction.

Finally, the specimens were annealed under an argon atmosphere at 800°C for 3 min. Specimens in all conditions were prepared by using normal metallographic procedures and etched with 2% Nital for microscopic observation and microanalysis. Tensile tests on annealed specimens were conducted on an Instron 1125 (10-t) test machine, with the tensile test specimens machined out according to ASTM E-8 standard for flat specimens. From the results obtained on tensile test specimens in the annealed condition, the normal anisotropy value was calculated at 0, 45 and 90° with respect to the rolling direction according to the following expression:

$$r = \ln (w/w_0)/\ln (t/t_0) \quad (1)$$

where r is the normal anisotropy ratio or the Lankford value, t_0 and w_0 are the initial thickness and initial width, respectively, and the values after deformation are denoted by t and w. The average normal anisotropy ratio ($\bar{r}$) was evaluated according to:

$$\bar{r} = (r_0 + 2r_{45} + r_{90})/4 \quad (2)$$

Texture measurements were carried out on a Siemens D5000 texture goniometer by using cobalt radiation. The pole figures were measured up to a tilt angle

Table 3
Hot rolling data of processed ultraclean steels[a]

I_t (cm)	1st pass (cm)	2nd pass (cm)	Δh_1	Δh_2	e_1	e_2	V_{def} 1st pass (1/s)	V_{def} 2nd pass (1/s)
U430/401331.0	0.650	0.34	0.35	0.31	0.431	0.648	6.331	9.987
U430/42229 1.1	0.715	0.38	0.38	0.34	0.431	0.632	6.036	9.522
U451/30119 1.2	0.780	0.41	0.42	0.37	0.431	0.643	5.779	9.116
							V_{ave} 6.049	V_{ave} 9.542

[a] I_t is the initial thickness of slab specimen, 1st pass is the thickness of specimens after the first hot rolling pass, 2nd pass is the thickness of specimen after the second hot rolling pass, V_{def} is the deformation velocity and V_{ave} is the average deformation velocity in each hot rolling pass.

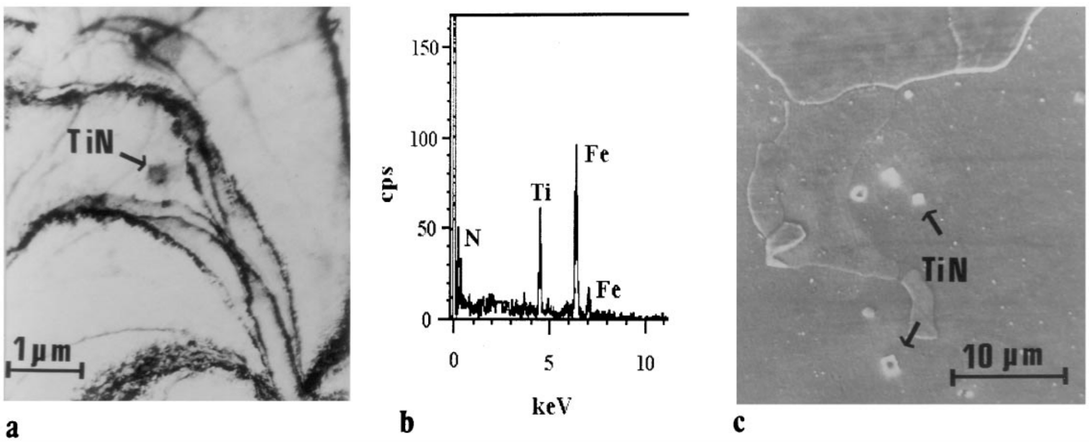

Fig. 1. TiN precipitates of cuboidal-like morphology observed in ultraclean steels in the as-cast condition: (a) TEM micrograph, (b) STEM microanalysis and (c) SEM micrograph.

$\chi = 80°$ with steps of $\Delta\chi = 2.5°$ in tilt direction and $\Delta\phi = 4°$ in azimuth direction.

3. Results and discussion

Microstructural characterization of specimens in the as-cast condition showed the presence of precipitates in ferrite matrix and ferrite grain boundaries. These precipitates were identified by means of scanning transmission electron microscopic (STEM) microanalyses as the cuboidal-like TiN (Fig. 1). After re-heating and hot rolling of slabs, precipitates of the TiS (Fig. 2) and $Ti_4C_2S_2$ (Fig. 3) type were also identified by STEM microanalyses in addition to TiN precipitates.

With regard to the identified precipitates, it has been reported [8,9] that in ultraclean Ti stabilized steels, several precipitates can be present such as TiN, TiS, $Ti_4C_2S_2$, MnS and TiC, and the presence of those precipitates can have a significant influence on microstructure and its subsequent mechanical properties. Although TiN particles are stable [10], they can contribute to the precipitation reactions which occur during the reheating and hot rolling operations by acting as nucleation sites for precipitates of the TiS and $Ti_4C_2S_2$ types, in agreement with several reports [11,12] and with this study. As mentioned above, TiN precipitates in ferrite matrix and ferrite grain boundaries were the only precipitates detected in the as-cast steels without detecting the presence of entrapped slag, oxides or non-metallic inclusions.

Fig. 4a and b shows a representative microstructure of the ultraclean steel in the as-cold rolled and annealed condition, respectively, obtaining in the fully recrystallized specimens an average grain size of 15 ± 2.3 μm. In addition to the above precipitates in the as-annealed specimens, the presence of TiC precipitates (Fig. 4c) was detected.

The Ti precipitation compounds in Ti-stabilized IF-steels were identified during processing as following: (i) TiN precipitates form during the addition of the FeTi ferroalloy to the liquid bath, being those precipitates present at ferrite matrix and grain boundaries with sizes in the range 0.5–2.0 mm; (ii) after reheating (1250°C) and hot rolling (1250–950°C) of slabs in the austenite region, the presence of TiS and $Ti_4C_2S_2$ compounds was detected; and (iii) TiC precipitates were identified after the coiling simulation (700°C) in the ferrite region. As reported [13], the precipitation of these compounds will affect the mechanical properties of the resulting annealed IF sheets. For instance [14], the size and dispersion of the precipitates in a Ti stabilized IF steel can interact with the recrystallization and evolution of texture. A dense dispersion of fine precipitates was corre-

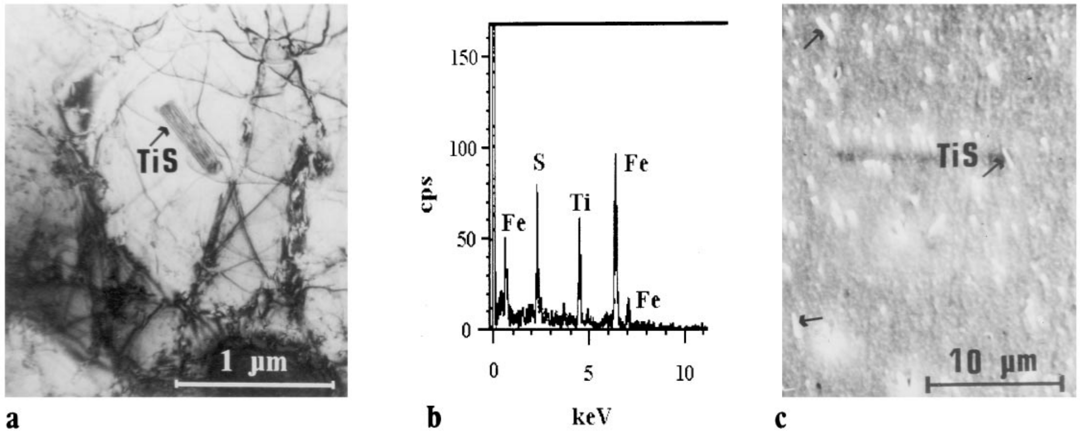

Fig. 2. TiS precipitates of elongated morphology observed in ultraclean steels in the as-hot rolled condition: (a) TEM micrograph, (b) STEM microanalysis and (c) SEM micrograph.

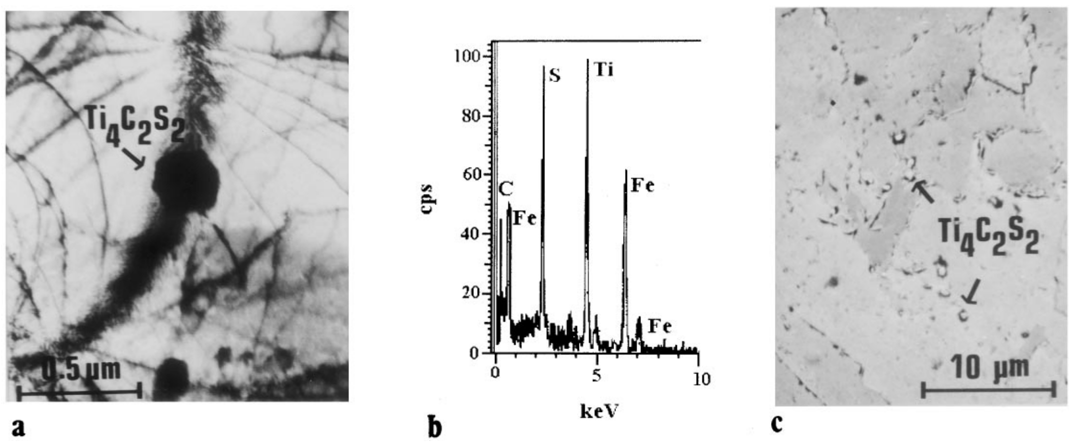

Fig. 3. $Ti_4C_2S_2$ precipitates of hexagonal-like morphology observed in ultraclean steels in the as-hot rolled condition: (a) TEM micrograph, (b) STEM microanalysis and (c) SEM micrograph.

lated with the low intensity of {111} texture, the particle pinning effect on the boundary mobility being the dominant mechanism in inhibiting the {111} texture. So, in order to develop a strong {111} recrystallization texture during in line annealing, the removal of interstitial elements as a sparse dispersion of coarse precipitates will be necessary in order to promote high values of the average normal anisotropy ratio, it also being advisable to use a high coiling temperature in order to coarsen the ultrafine precipitates that form in ferrite. In addition, it is known that a cold-rolled steel sheet with excellent formability can be produced by the use of interstitial free steels stabilized by the addition of Ti and/or Nb. These steels develop a strong {111} recrystallization texture which results in high r-value and good deep drawability: for that purpose it is necessary to carry out proper control (in addition to the steelmaking process) of the rolling conditions, coiling temperatures as well as cold rolling reductions and annealing practices. Texture analysis of resulting annealed sheet steels is shown in Fig. 5, where a standard texture for recrystallized ultra low carbon steels (800°C, 180 s) with $<111>$ // ND fiber was obtained, which means that most of the crystallites have {111} planes parallel to the surface of the specimens. This was recognized by the fact that the maxima in all the orientation distribution function (ODF) sections appear at $\Phi = 55°$ and $\varphi_2 = 45°$. There are nevertheless differences between the annealed specimens: the values of the peaks in the ODF decrease from specimen 3 to specimen 1. The maximum ODF values were 7.1, 7.2 and 8.0 for specimens 3, 2 and 1, respectively.

In order to evaluate the steelmaking process, thermomechanical processing, coiling simulation, mechanical processing and annealing of steels, the quantification of mechanical properties was carried out in annealed specimens (800°C), in terms of 0.2% yield strength (0.2% YS), ultimate tensile strength (UTS) and percent of elongation (El%), in specimens machined out at 0, 45 and 90° with respect to the rolling direction. In addition, from the results obtained on tensile test specimens in the annealed condition, the normal anisotropy ratio (Lankford value) was calculated at 0, 45 and 90° with respect to the rolling direction, and from those

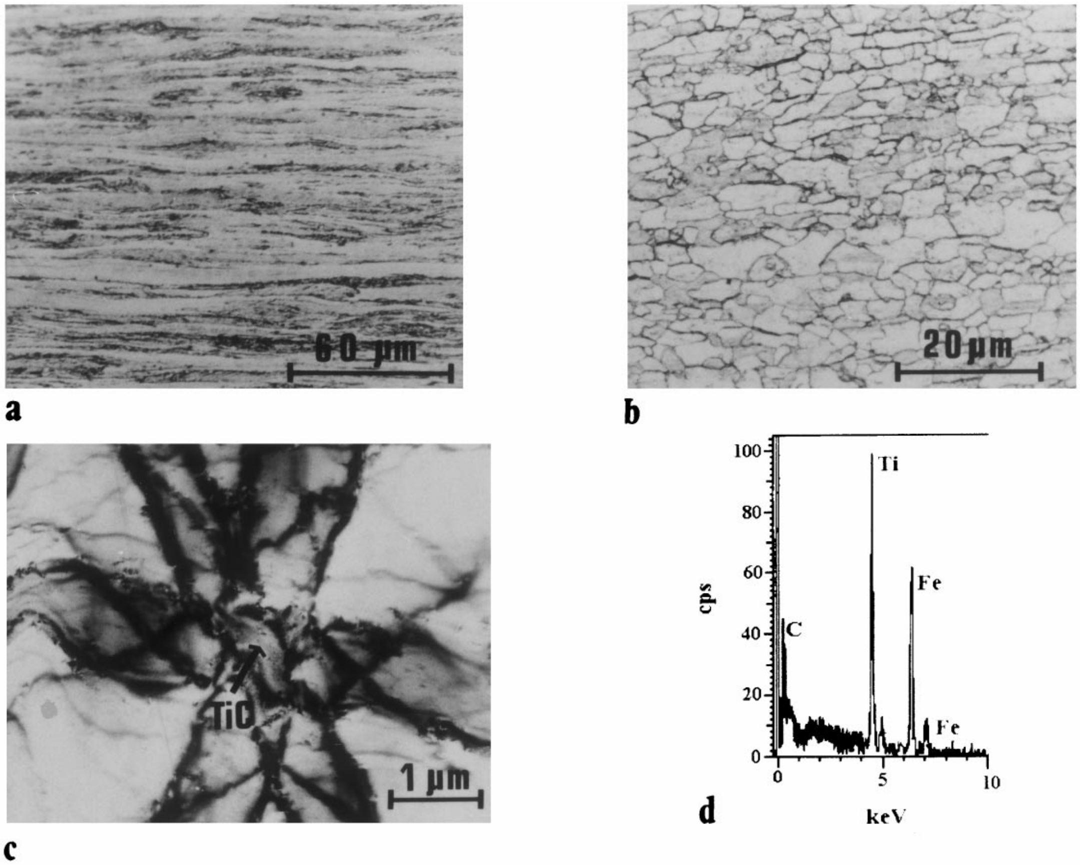

Fig. 4. Representative micrographs of ultraclean steels: (a) in as-cold rolled condition, (b) annealed condition, (c) TiC precipitates in the as-cold rolled specimen and (d) STEM microanalysis.

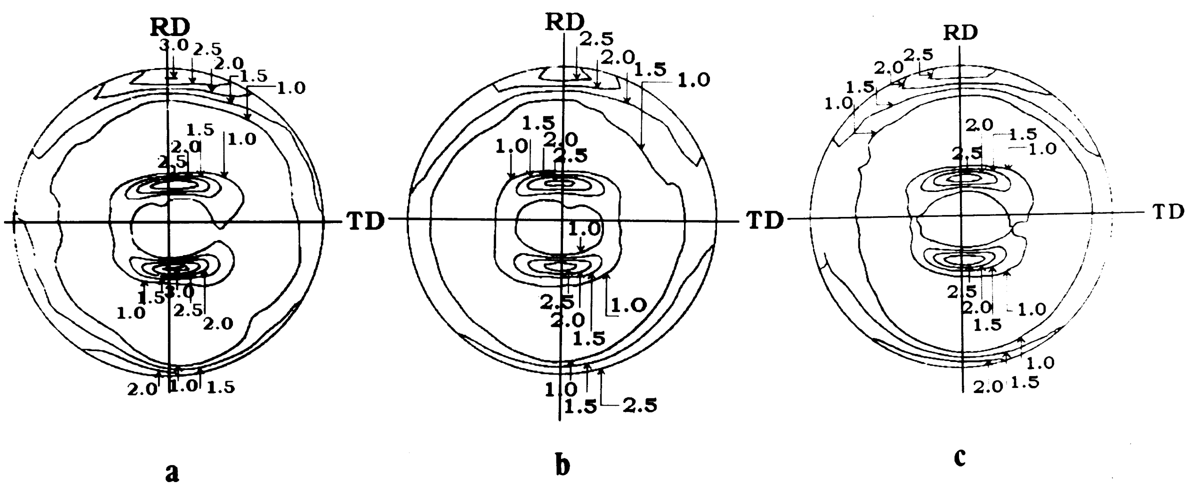

Fig. 5. Computed (110) pole figures of ultraclean steels in annealed sheets: (a) sample 1, (b) sample 2 and (c) sample 3.

values, the average normal anisotropy ratio was calculated, with the results summarized in Table 4.

According to Takechi [2], the reduction of the C contents significantly improves the mechanical properties of steel sheets in terms of the $\bar{r}$ value, and also Ti additions bind interstitial solute atoms such as C and N, dramatically improving the $\bar{r}$ value [15]. For instance the critical amount of Ti; Ti*% (effective Ti%) necessary for obtaining an excellent $\bar{r}$ value, can be expressed according to the following expression:

$$\text{Ti}^* = \text{Ti}(\%) - [4\text{C}(\%) + 3.43\text{N}(\%)] \quad (\text{in wt.\%}) \qquad (3)$$

From that equation and the C and N contents present in our experimental steels, Ti* is for specimen 1, 2 and 3 equal to 0.034, 0.038 and 0.034%, respectively, which under specific processing conditions (as those

Table 4
Mechanical properties and the normal anisotropy ratio for annealed sheets at 800°C per 3 min

Mechanical properties	0° to the rolling direction			45° to the rolling direction			90° to the rolling direction		
	Specimen								
	1	2	3	1	2	3	1	2	3
0.2%YS (MPa)	153	138	167	145	142	156	137	140	153
UTS (MPa)	295	254	306	311	298	295	288	293	306
El (%)	55.6	61.7	56.7	75.3	72.0	62.3	61.2	64.5	68.4
r	2.07	2.19	2.03	2.01	2.05	2.00	2.34	2.28	2.07
$\bar{r}$	2.10 (sample 1), 2.14 (sample 2) and 2.02 (sample 3)								

Table 5
Classification of super formable sheets

Properties	Commercial quality	Drawing quality	Deep drawing quality	Extra deep drawing quality
El (%)	36.9–42.2	40.0–45.0	42.7–47.5	46.1–50.5
$\bar{r}$	1.0–1.41	1.23–1.51	1.42–1.84	1.73–2.12

presented above) can guarantee [15] $\bar{r}$ values ≥ 2. From Table 4, it is observed that the processed steels reached $\bar{r}$ values of 2.10, 2.14 and 2.02 for specimens 1, 2 and 3, respectively, and elongation values $> 55\%$. Those values agree with the work of Takechi [2], in which he concluded that Ti addition is adequate for binding nitrogen from the point of view of maintaining ductility of products. In addition, cold rolled sheet steels have been classified by their average plastic strain ratio ($\bar{r}$) and total elongation (El) in four grades as is shown in Table 5: due to tough customer requirements, a new grade is now demanded, whose formability value and total elongation will be higher than 2.0 and 50%, respectively. To achieve these mechanical properties, it is recommended that the cold rolled sheets are annealed to temperatures at or over 800°C. In the present study, the annealed steel sheets showed a total value of elongation, obtained in tested specimens at 0, 45 and 90°, higher than 50% and an $\bar{r}$ value between 2.02 and 2.14, making our experimental steels for suitable for extra deep drawing applications. The influence of annealing temperature on the $\bar{r}$ value has been discussed [16] in terms of the volume fraction of γ transformed during annealing in the temperature range 775–975°C. This is mentioned because ultra low carbon steels have a narrow $\alpha+\gamma$ temperature and $\alpha \rightarrow \gamma$ transformation finishes in a brief period, and the $\bar{r}$ value increases as the temperature increases, achieving a maximum close to the Ac_3 temperature. After that, the $\bar{r}$ value decreases abruptly when annealing is carried out in the γ region. The values of $\bar{r}$ achieved in this study are similar to those reported by Yoshinaga et al. [17] when the specimens were annealed at 800°C, suggesting that for the achieved chemical composition of steels, together with the thermomechanical, coiling and mechanical processing conditions, it is advisable to anneal the sheets at temperatures in the range between 800 and 850°C. At temperatures higher than 850°C, it was observed that properties in terms of the Lankford value and elongation decreased.

4. Conclusion

From the results presented above, the experimental interstial free steels obtained by the electric arc furnace, ladle vacuum degassing, ladle treatment and continuous casting route, and processed by using a hot rolling temperature of 1250°C, and finishing the hot rolling at 950°C, coiled at 730°C, cold rolled and annealed at 800°C showed: (a) that TiN precipitates in ferrite matrix and grain boundaries in the as-cast condition; (b) after reheating (1250°C) and hot rolling (1250–950°C) of slabs in the austenite region, the presence of TiS and $Ti_4C_2S_2$ compounds were detected, as were TiC precipitates in the ferrite region after the coiling simulation; (c) texture analysis of the recrystallized sheets (800°C, 180 s) showed that most of the crystallites have {111} planes parallel to the surface of the specimens; and (d) the thermomechanically processed steels reached $\bar{r}$ values > 2.0 and elongation values $> 55\%$, making the experimental steels suitable for extra deep drawing applications.

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