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Mechanical Properties of Ultra Clean Low C/Cr Stabilized Annealed Sheets

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Abstract. The microstructural and mechanical properties of an ultra-clean low carbon steel sheet with 0.035% Cr have been evaluated. The isothermal recrystallization kinetics at 800 °C is analyzed by using the Kolomogorov-Johnson-Mehl-Avrami (KJMA) equation. The obtained results indicate that Cr retards the recrystallization kinetics owing to the presence of fine particles of chromium carbides. The annealing process produces a fully recrystallized microstructure. The main texture components in the specimens are: {111}<112>, {112}<110> and {111}<110>. These are related with the Lankford value and the elongation. The obtained results suggest that the formability of this steel is excellent.

Introduction

One of the most important achievements in the steel industry has been the development of microalloyed steels, followed by the appearance of ultra-low C (ULC) and interstitial free (IF) steels [1,2]. Modern sheet steels for high deep drawing applications are based on the fabrication of steels with the following gradual improvements: (i) Tighter control of the composition (in parts per million) and stabilization of the interstitial elements C and N, (ii) decreasing the content of residual elements and improved cleanliness by elimination and control of almost all the impurity elements (S, P), (iii) increased uniformity of properties with the development of a favorable texture and (iv) improved mechanical properties by controlling of grain size [3, 4]. These improvements were applied in the Mexican steel industry for the development of ULC steels with C and N contents around 0.01 wt. % for automotive applications [3].

ULC sheet steels have high values of normal anisotropy (r-value) and non-aging properties adequate for deep-drawing applications [4,5]. This drawability is achieved by reducing the content of interstitial elements to ultra low levels and with the addition of small quantities of stabilizing elements such as Ti and Nb [4, 6]. The high formability of ULC steels is associated with the effects of the cold rolled α phase and the production of a polycrystalline structure with a strong (111)<110> recrystallization texture during annealing, that gives high values of the normal (r) and average ($\bar{r}$) plastic anisotropy ratio [5,7]. Recent developments [8-10] show that the stabilization of carbon in these steels occurs by the formation of $(Ti,Nb)_4C_2S_2$ in addition to the traditional MCN (M=Ti, Nb). The stabilization of C by the formation of either $(Ti, Nb)_4C_2S_2$ or MCN (M=Ti, Nb) depends on the overall chemical composition of the steel. It can affect the final microstructure and the texture. The precipitation of fine particles of the type (Ti,Nb)C, in hot rolled steel sheets is important to promote the formation of the **{111}**<uvw> recrystallization texture [11]. The aim of this work is to report results of an investigation on an ultra-clean steel stabilized with Cr in terms of its mechanical properties in the as-annealed condition, in order to evaluate its performance for possible applications in the automobile industry as a deep drawing sheet.

Experimental Procedure

The experimental steel used was produced with 100 % sponge iron in an electric arc furnace, vacuum degassed, ladle treated and continuously cast to obtain ingots of about 200 mm thickness and 962 mm width. The slabs were re-heated at 1100 °C in argon to a rolling start temperature of 1250 °C, finishing the hot rolling operation at 870 °C to get 2.6 mm in thickness (91 % total reduction) and coiled at 600 °C. After coiling, the plates were cold rolled at room temperature, reaching a total reduction of 73 % to get 0.7 mm in thickness. The cold rolled coils were batch annealed at 670 °C for 20 h under N_2-H_2 atmosphere and then continuously annealed at 700, 750 and 800 °C. Microstructure characterization of the steel plates was carried out using a 440 Stereoscan scanning electron microscope using a point counting technique in order to evaluate the progression of the recrystallization (ASTM E112-828). Scanning-transmission electron microscopy (STEM) observations and microanalysis were performed in a 1200 Jeol transmission electron microscope. Flat tensile tests (ASTM E-8-98) of fully annealed specimens were made with an Instron 1125 (15 tons) test machine. These tests were made with specimens oriented at 0°, 45° and 90° with respect to the rolling direction. The results were used to calculate the normal (r) and the average $(\bar{r})$ anisotropy ratio values for the annealed specimens. Texture measurements of the steel sheets in the hot rolled, cold rolled and annealed conditions were carried out by X-ray diffraction with a Siemens D5000 texture goniometer and Co radiation. The pole figures were measured up to a tilt angle $\chi = 80°$, with steps of $\Delta\chi = 2.5°$ in the tilt direction and $\Delta\Phi = 5°$ in the azimuth direction.

Results and Discussion.

The experimental Cr stabilized ultra-clean C steel used in this work contained 0.010 % C, 0.20 % Mn, 0.011 % P, 0.002 % S, 0.050 % Al, 0.025 % Si, 0.004 % Ni, 0.035 % Cr, 0.004 % N. The purpose of this design was to achieve good deep drawability and the following minimum mechanical properties: 170 MPa of 0.2 % Yield Strength ($YS_{0.2\%}$), 345 MPa of Ultimate Tensile Strength (UTS), 46 % elongation, a strain hardening exponent of $n > 0.21$ and an average plastic deformation ratio of $(\bar{r}) \geq 2$.

Figure 1 shows the microstructures in the steel samples obtained after different processing conditions: re-heated, hot rolled and coiled (Fig. 1a), cold rolled (Fig. 1b) and annealed at 800 °C (Fig. 1c). The observed microstructures consist mainly of ferrite grains with different morphologies depending on the thermomechanical process.

Fig. 1a shows a matrix of α-Fe grains with few subgrains formed. Small round precipitates (indicated by arrows) are present along the subgrain boundaries and within the α-matrix. These precipitates were analyzed by STEM and reported in a previous work [12]. They appear to be mainly carbides of the type $Cr_{23}C_6$, Cr_3C_2 and Fe_3C that are aligned during the cold rolling process. This is shown in Fig. 1b indicated by arrows, where a fine-banded microstructure of mixed ferrite grain is observed. The mixed ferrite grain structure is quite likely inherited from a mixed austenite grain size that would result if the austenite had partially recrystallized between rolling passes, and would persist during subsequent rolling [13]. A partially recrystallized microstructure that consists of recrystallized grains with no equiaxial grains is obtained after annealing at 800 °C (Fig. 1c). The formation of this microstructure is due to the presence of the mixed microstructure that was formed during the cold rolling. It has been related with the later stages of the annealing process [14].

The precipitates appeared in the ferrite matrix and in the grain boundaries were identified by STEM. Selected area diffraction (SAD) is taken to determine their crystallographic characteristics. Fig. 2 shows the STEM images that confirm the microstructures observed in the optical and SEM microscopes in the different processing stages of the experimental ULC-Cr/stabilized steel. The precipitates found are located inside and along the grain boundaries of the α-ferrite in the re-heated, hot-rolled and coiled condition. It is evident that there is a precipitate free zone in the microstructure (Fig. 2a). The microstructure of the cold rolled specimens is shown in figure 2b. This figure shows

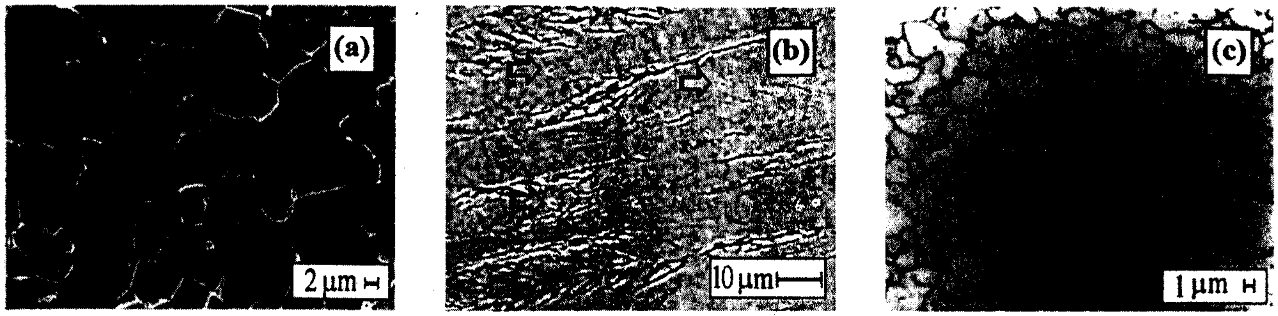

Figure 1. Microstructure of the specimens after different processing conditions. (a) Re-heated hot rolled and coiled, (b) cold rolled and (c) annealed at 800 °C.

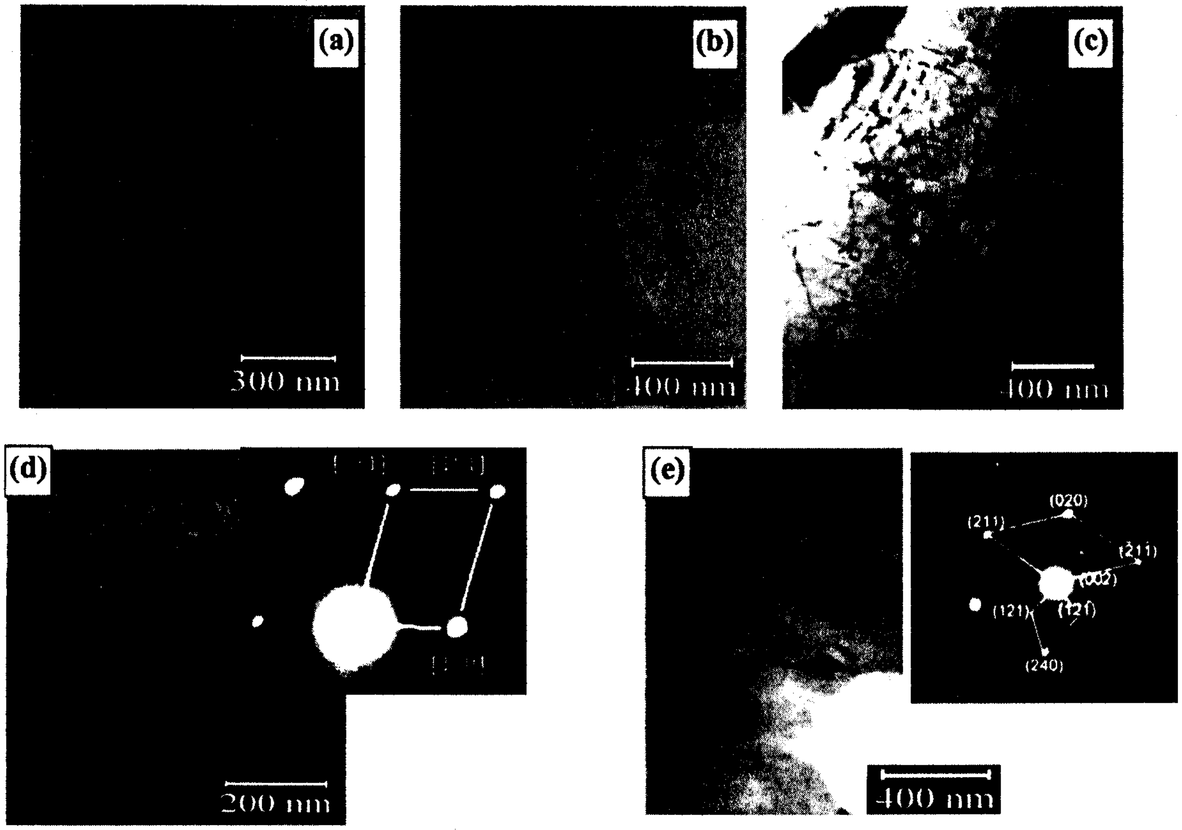

Figure 2. Thin foil electron micrographs showing different aspects of the precipitates obtained during the different thermo-mechanical stages, (a) re-heated, hot rolled and coiled, (b) cold rolled and (c) annealed at 800 °C, (d) Cr_3C_2 carbides and (e) Fe_3C carbides obtained during annealing at 800 °C.

that the original α-grains are elongated and that they have deformation bands and a dislocation cell structure. The formation of the precipitates is associated with dislocations. Figures 2d and 2e show SAD patterns of the precipitates. The majority of the larger precipitates are identified as Cr_2C, and $Cr_{23}C_6$ [12] and the smaller ones that had an average size of 1-1.5 µm are mainly Fe_3C and Cr_3C_2. These types of chromium carbides are usually reported in steels with lower chromium contents [13]. By comparison with the hot-rolled and coiled samples, the annealed samples (Fig.2c) have a higher precipitate density (Fig.2a). This may indicate that there is a decrease of dissolved C when the Cr carbides are formed.

The recrystallization kinetics is shown in Fig. 3. The results are for annealing at 700 °C, 750 °C and 800 °C after a 73 % rolling reduction at room temperature. The recrystallization becomes almost two orders of magnitude slower as the temperature decreased from 800 °C to 700 °C. At 800 °C, a 100 % recrystallized microstructure is obtained in approximately 150 s. The incubation time for the onset of the recrystallization is longer at lower annealing temperatures. It has been mentioned that the effect of temperature on the precipitation and recrystallization is rather complex because it involves solute-drag [14], precipitate-pinning [15,16], and supersaturation effects [17] among others. Thus, the precipitates mainly formed by chromium carbides such as $Cr_{23}C_6$ and Cr_3C_2 induce the retardation of the recrystallization kinetics process.

The recrystallization kinetics of ferrite is generally described by an Avrami equation. The recrystallyzed fraction X(t) at time t is given by $X(t) = 1- exp(-bt^n)$, where b and n are constants. The values of n were calculated by plotting $\log \{ln[1/1-x)]\} = \log b - n \log t$. Fig. 3b shows the curves obtained as a function of time. In all cases two stages are observed. For ULC steels stabilized with Ti [18] the annealing process also proceeds in two defined stages. The first stage corresponds to the early stages of the annealing process. It is related with values of $n > 1$. The value of n increases as the temperature increases (n = 2.4 for 700 °C, 3.17 for 750 °C and 3.4 for 800 °C). The second stage is related to the later stages of annealing. It is characterized by values of $n < 1$. The lower n values are related to an anisotropic nucleation behavior, with different nucleation rate referred to a site saturated nucleation at grain boundary edges [19, 20]. Also, the lower n values have been related with the shape of non-equiaxial recrystallized grains [7].

Fig. 4 shows the textures of the steels that were cold rolled and annealed at different temperatures. The texture of the cold-rolled steel is shown in Fig. 4a. The texture is preferentially in the systems {111}<011> and {111}<112>, which are very close to the γ fiber. It has been reported [21] that the γ fiber texture induces the nucleation of the recrystallized grains in the same {111} texture during the annealing process. However, in this work when the sheets were continuously annealed at 700 °C, the recrystallization process was less homogeneous, producing a gradual consumption of the γ fiber texture. Fig. 4b shows that the recrystallized texture components were {112} <110> and {554} <225>. The family planes {554} are very close to the {111} planes. This texture induces a lower deep-drawability for the steel.

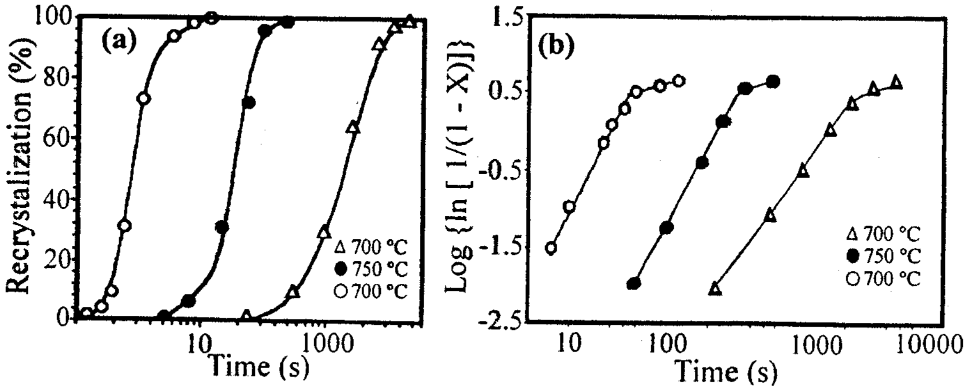

Figure 3.
(a) Recrystallization curves and b) plot of $log\{ln(1/1\text{-}x)\}$ versus time for the low C/Cr stabilized steel cold rolled 73%, cooled and annealed at 700, 750 and 800 °C.

The steel sheet annealed continuously at 800 °C shows a texture in the system {111}<110> (Fig. 4c). It is mainly a γ fiber texture that was formed due to the recrystallized grains that nucleated and grew in the vicinity of the grain boundaries of the cold deformed grains that had a strong γ texture. It is evident that increasing the annealing temperature, a {111} texture tends to develop. This texture improves significantly the deep-drawability characteristics of the steel.

The mechanical properties of the Cr stabilized steel in the anneal condition are summarized in Table 1, including the target properties and the properties of an ULC Ti-stabilized steel [18]. From the tensile test results of the samples that were measured at 0°, 45° and 90° from the rolling direction, the average Lankford value ($\bar{r}$) was determined. The coils annealed at 800 °C have a higher average

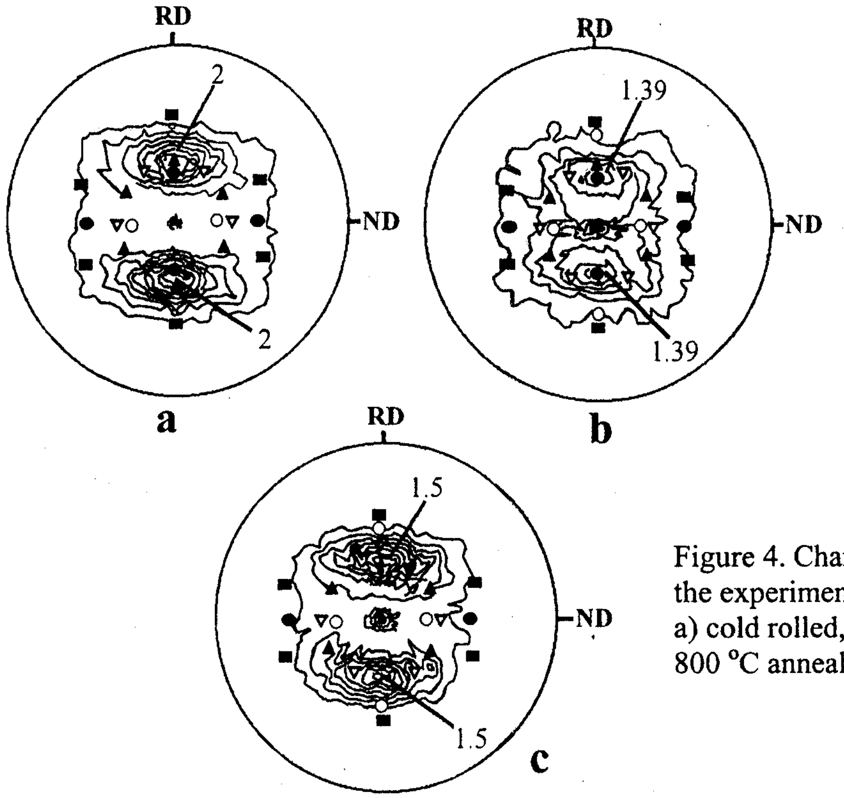

Figure 4. Changes in {110} pole figures for the experimental low C/Cr stabilized steel a) cold rolled, b) 700 °C annealed and c) 800 °C annealed.

The mechanical properties of the Cr stabilized steel in the anneal condition are summarized in Table 1, including the target properties and the properties of an ULC Ti-stabilized steel [18]. From the tensile test results of the samples that were measured at 0°, 45° and 90° from the rolling direction, the average Lankford value $(\bar{r})$ was determined. The coils annealed at 800 °C have a higher average Lankford value than the coils batch annealed a 670 °C, and than the coils that were annealed continuously at 700 °C and 750 °C. The average Lankford value of the Ti stabilized steel annealed at 800 °C is also smaller [18]. Regarding the batch annealed coils, their properties and $(\bar{r})$ values are comparable to the values for the Ti/stabilized steel, but inferior than the Cr-stabilized steel annealed continuously at 800 °C.

Table 1. Mechanical properties for the steel annealed at different temperatures*.

Properties → Specimen ↓	$YS_{0.2\%}$ [MPa]	UTS [MPa]	El [%]	$(\bar{r})$	n
Required properties	170	345	46	≥ 2	≥0.21
Annealed 800 °C/ Ti [15]	145±16	311±12	55.6±1.2	2.08±0.05	-
Batch annealed 670°C/Cr	169±17	318±20	43.8±2.8	1.98±0.03	0.24±0.01
Annealed 700 °C / Cr	181±23	304±18	47.0±3.7	1.35±0.09	0.32±0.02
Annealed 750 °C / Cr	234±13	378±9	50.3±1.0	1.62±0.07	0.31±0.05
Annealed 800 °C/ Cr	242±6	378±17	51.3±0.8	2.21±0.05	0.30±0.03

n: strain-hardening exponent. *Each value is average of 20 measurements

Takechi [4] pointed out that cold-rolled sheet steels are classified in four quality grades depending on their average Lankford value ($\bar{r}$) and total elongation. The Lankford values for the different steel grades range from 1.0 to 2.1 and from 40 to 50 % elongation. New Super Extra Deep Drawing Quality steel (SEDDQ, Table 2) has ($\bar{r}$) and total elongation higher than 2 and 50%, respectively [4]. Finally, the ($\bar{r}$) values for the low C/Cr stabilized steel annealed at 800 °C, indicate that a fully recrystallized condition is related with a texture that improves the deep-drawability.

Conclusions.

- A sparse dispersion of fine and coarse precipitates retards the recrystallization kinetics of Cr stabilized low C steel.
- The steel annealed at 800 °C has higher ($\bar{r}$)-values and higher intensity of the {111} component.
- Low C/Cr stabilized steel with improved deep drawability was obtained using these processing conditions.

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