Study of the thermomechanical behavior of API 5L X80 steel micro-alloyed with Nb-Ti through hot torsion tests

Estudo do comportamento termomecânico do aço API 5L X80 microligado ao Nb-Ti através de ensaios de torção a quente

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Abstract

The controlled rolling of high strength low alloy steel has been widely used in the production of large diameter pipes for the oil and gas industry. The thermo-mechanical control process (TMCP) is used to maximize grain refinement and achieve both higher strength and toughness in the steels. In this sense, API 5L X80 micro-alloyed Nb-Ti steel was submitted to hot torsion tests to evaluate its hot deformation behavior. The critical temperatures (Tnr, Ar3 and Ar1) were obtained by testing with multiple strains in continuous cooling. From the flow stress curves generated in the isothermal tests, it was possible to obtain a critical strain that starts dynamic recrystallization, the peak strain for temperatures ranging from 1150ºC to 850ºC, and the strain rates at 0.2; 0.4 and 0.8s^{-1}. The results showed that peak and critical stress values increase as the temperature decreases and strain rate increases.

Keywords: Hot torsion test, API 5L X80 steel, dynamic recrystallization (DRX), flow stress curves.

1. Introduction

Steels produced by controlled rolling are of great importance for the metallurgy due to their excellent performance at low production costs. These steels have many applications in the production of oil and gas pipelines.

Austenite hardening in the finishing rolling produces very high values for the rolling forces (GORNI, *et al*., 2009). An alternative to this situation is the use of steels with a relatively high niobium content, usually between 0.08 and 0.11%, which increases the non-recrystallization temperature. This rolling type is known as High Temperature Processing (HTP). One of the main benefits of the HTP alloy project is the processing capacity at temperatures higher than the conventionally controlled rolling, which allows a reduction in the rolling forces (SICILIANO, *et al*., 2008).

The objective of this study was to find the critical processing temperatures of API 5L X80 steel microalloyed Nb-Ti and evaluate its thermomechanical behavior during hot deformation. For this purpose, hot torsion with multiples strains in continuous cooling and isothermal tests were done.

The value of Tnr can be determined by the chemical composition in Equation 1 proposed by Borato, Barbosa and Santos, from the results obtained in hot torsion tests with multiple strains (BORATO *et al*, 1988; MACCAGNO *et al*, 1994):

(1) $$Tnr\left(°C\right) = 897 + 464.C + \left(6445.Nb - 644\sqrt{Nb}\right) + \left(732.V - 230.\sqrt{V}\right) + 890.Ti + 363.Al - 357.Si$$

The value of Ar3 can be obtained from Equation 2 developed by Ouchi (MACCAGNO *et al*, 1994):

(2) $$Ar3\left(°C\right) = 910 - 310.C - 80.Mn - 20.Cu - 15.Cr - 55.Ni - 80.Mo + 0.35(t - 8)$$

The critical temperatures were obtained through multiple strains in continuous cooling tests, and isothermal tests produced the flow stress curves, whereby it was possible to analyze the thermomechanical behavior of the material. The dynamic recrystallization (DRX) and dynamic recovery were verified.

The Dynamic recrystallization is an important mechanism for controlling the microstructure during hot deformation. This softening mechanism has an important part in reducing the flow stress, and austenite grain size. It is also useful for controlling the mechanical properties during processing. The prediction of the critical condition for the beginning of the DRX is of great importance for industrial processes modeling (SARMENTO and EVANS, 1992), (SICILIANO and JONAS, 2000), (SHABAN and EGHBALI, 2010).

2. Materials and methods

The material used in the test was API 5L X80 steel (with high Nb) produced in a Hot Strip Mill at ArcelorMittal Tubarão. The chemical composition (% in weight) was C < 0.10; Si < 0.30; Mn < 1.70; P < 0.018; S < 0.005; Al < 0.050; N < 0.0100; Nb ~0.09; Ti < 0.030 and Ca < 0.0050.

The torsion specimens were performed on a plate made from a coil of 15.88 x 1500 x 500 mm, with a length of 20 mm and diameter of 5 mm. The tests were done in an INSTRON model 55MT horizontal hot torsion machine, installed in the mechanical conformation laboratory at the Instituto Federal do Espírito Santo (IFES). Specimens were enclosed in a quartz tube with a continuous, inert Argon gas to avoid steel oxidation during induction heating. The temperature was monitored by a type K thermocouple that was inserted in a hole at the end of the specimen located immediately after the useful section. The test results corresponded to the measured values of the torque and angle that are converted into equivalent stress and equivalent strain, respectively.

For the multiple strain under continuous cooling test, the specimen was heated to a soaking temperature of 1240ºC at a heating rate of 3°C/s, and maintained at this temperature for 3 minutes. Then the specimen was cooled at a cooling rate of 1°C/s with a constant strain of 0.2 every 30 seconds at a strain rate of $0.2s^{-1}$ from 1170ºC until 600°C.

For the isothermal test, the specimens were heated to a soaking temperature of 1240ºC at a heating rate of 3°C/s, and maintained at this temperature for 3 minutes. Then the specimens were cooled at a rate of 1°C/s up to the test temperature and maintained at that temperature for 1 minute, to eliminate thermal gradients before the onset of deformation. Tests were carried out at 1150ºC; 1100°C; 1000°C; 950°C and 850°C for the same strain rate of $0.2s^{-1}$ and the maximum strain used during the test was 3. The tests were also carried out at 1150ºC and strain rate of $0.2s^{-1}$, $0.4s^{-1}$ and $0.8s^{-1}$ with the same maximum strain.

3. Results and discussions

3.1 Test with multiple strains in continuous cooling

From these tests, the critical temperatures, Tnr, Ar3 and Ar1, were obtained. In Figure (1a), it is possible to see the result of the curves of true stress x true strain obtained through the hot torsion tests. As the stress increases, the temperature decreases until 750ºC. For temperatures below 1050ºC, starting from the 5th pass, there is an accentuated flow stress increase, which characterizes a change of the recrystallization area to a hardening area; in other words, areas where there is no recrystallization. Soon afterwards, with temperature decrease, the flow stress falls after 750ºC showing ferrite emergence, which is the start of the intercritical area ($\gamma + \alpha$) or Ar3 of the Fe_3C diagram. Finally, the flow stress increases again starting with the temperature of 690ºC, showing the cementite emergence, which is the beginning of the ferrite-cementite area or Ar1. This temperature is known as the transformation final temperature $\gamma \rightarrow \alpha$.

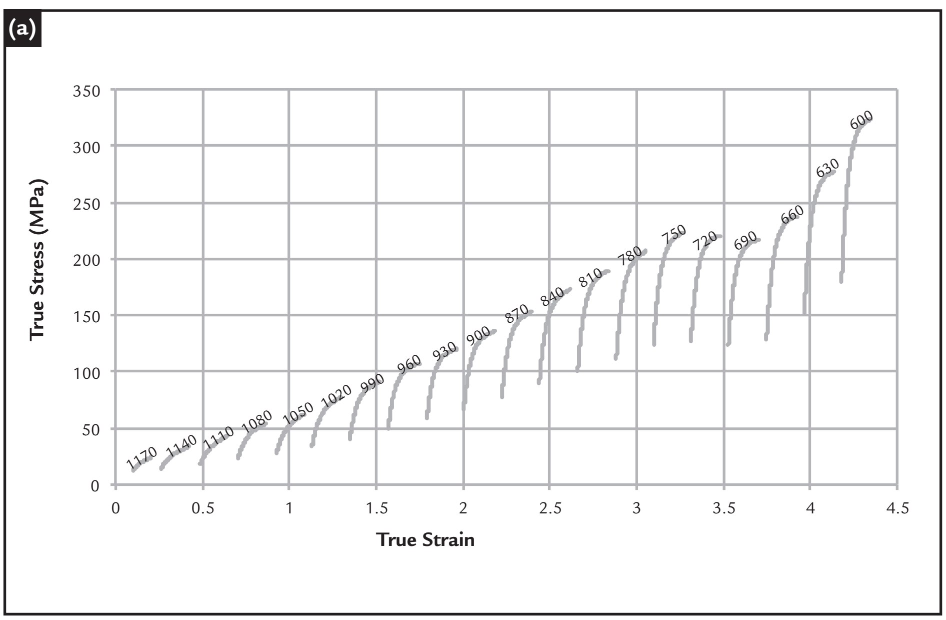

Fig. 1
Curves generated in the hot torsion test with multiple strains in continuous cooling for a API 5L X80 steel. Strain rate of $0.2s^{-1}$, deformation of 0.2 in each pass. The temperatures (°C) to which the strains were subjected are above each curve.
(a) True Stress versus True Strain.
(b) Mean Flow Stress versus 1000x1/T.

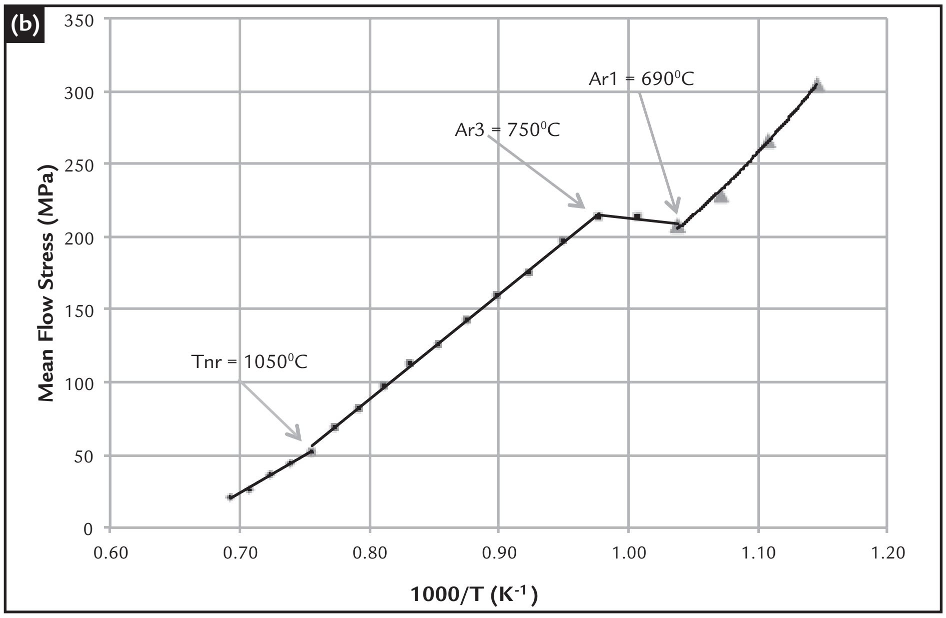

Fig. 1
Curves generated in the hot torsion test with multiple strains in continuous cooling for a API 5L X80 steel. Strain rate of $0.2s^{-1}$, deformation of 0.2 in each pass. The temperatures (°C) to which the strains were subjected are above each curve.
(b) Mean Flow Stress versus 1000x1/T.

For more accurate determination of the Tnr value, the Mean Flow Stress (MFS) was calculated. This is defined as the area below the stress curve in relation to the plastic deformation as shown in the Equation 3 (SICILIANO and JONAS, 2000), (SOLHJOO and EBRAHIMI, 2010):

$$MFS = \sigma = \frac{1}{\varepsilon_2 \;\; \varepsilon_1} \int_{\varepsilon_1}^{\varepsilon_2} \sigma d\varepsilon \quad (3)$$

Hence, a graph of the Mean Flow Stress (MFS) for each curve in function of the inverse temperature in Kelvin was obtained. In Graph (1b), there is a straight line verified at high temperatures, where austenite recrystallization takes place and another straight line at lower temperatures, with a larger inclination than the first, where austenite recrystallization doesn't take place. Tnr is defined in the intersection of these two straight lines (SICILIANO and JONAS, 2000), (SOLHJOO and EBRAHIMI, 2010). As illustrated in this graph, Tnr is confirmed at 1050°C. Also, Ar3 and Ar1 values can also be determined by the change in the curve behavior pattern at lower temperatures. The values obtained for the temperatures Ar1, Ar3 and Tnr were 690, 750 and 1050°C, respectively.

Equation 1 for Tnr calculation could not be used, as the Nb (~0.09%) content in the steel was above the maximum for the equation's validity (<0.05%).

In the HULKA and GRAY experiment for a low carbon steel with Nb (0.09 / 0.10%) content similar to this work, the resulting Tnr value was 1060°C (HULKA and GRAY, 2001). The variation between Tnr values of the present work and of previous experiment is approximately 1%.

Equation 2 provided a value of 747°C at the beginning for temperature of the phase transformation phase $\gamma \rightarrow \alpha$ (Ar3). There is a 0.4% difference between values obtained by equation 2 (747°C) and the torsion test (750°C).

3.2 Isothermal tests

Through the isothermal test, it was possible to plot the true flow stress curves versus true strain, where the peak stress (σ_p) and steady state stress (σ_{ss}) are identified. The flow stress curves are represented in two graphs: Figure 2 displays the tests with different temperatures and rates of constant strain, and Figure 3 shows different strains rates and constant temperature tests.

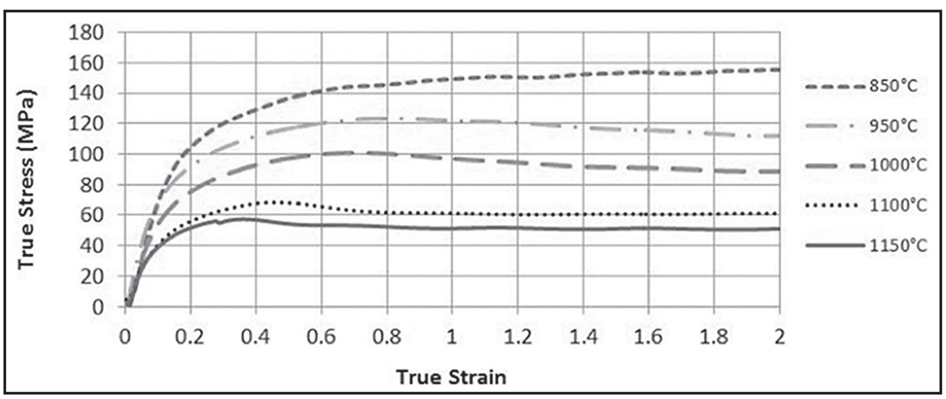

Fig. 2
True Stress Curves versus True Strain obtained by hot torsion tests for API 5L X80 steel at different temperatures and a constant strain rate of $0.2s^{-1}$.

In Figure 2, there is an increase in peak stress as the temperature decreases, presenting a similarity to another study with hot torsion testing (BARCELOS, 2013). This behavior is expected, since the density and distribution of dislocations, as well as energy stored in the deformation process, are factors that depend directly on the temperature during metal deformation (PADILHA and SICILIANO, 2005). As temperature decreases the dislocation mobility decreases, generating a flow stress increase and a displacement of the curves to the left, which shows that there is a greater hardening of the material.

At the 1150°C and 1100°C temperatures, the flow stress curves show a typical feature for material that recrystallizes dynamically, in other words, initially the flow stress increases with the strain until a maximum value and soon afterwards its value decreases to a steady state stress. This behavior was expected because these temperatures are above the Tnr (1050°C). For temperatures of 1000°C, 950°C and 850°C the curves have a typical feature of material that recovers dynamically; that is, a larger hardening occurs at the start of deformation reaching peak stress. In lower temperatures, the hardening is greater because metal recovery becomes slower.

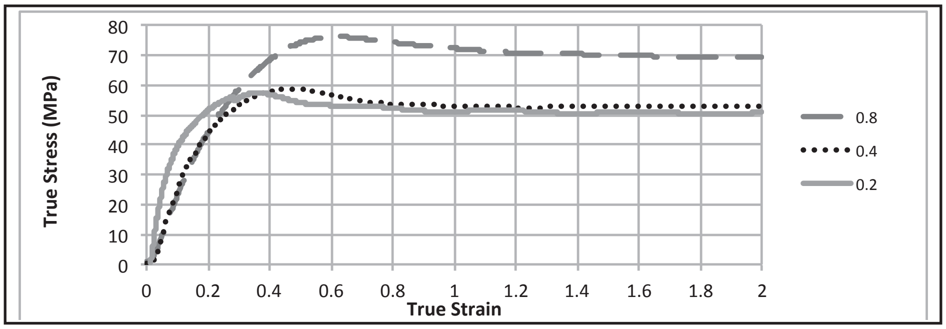

Fig. 3
True Stress Curves versus True Strain obtained by hot torsion test for API 5L X80 steel with different strain rates at a temperature of 1150°C.

In Figure 3, the three resulting curves from tests with different strains rates and 1150°C temperature show a shape typical of material that recrystallizes dynamically. This behavior was expected because the test temperature is above Tnr (1050°C). The greater strain rates show that the flow stress versus strain curves tend to move upward and to the right. Consequently, the greater the applied strain rate, the greater the steel peak stress. For dynamic recrystallization to occur, the flow stress curves have to reach certain critical values; the ones which can be found through the Strain Hardening Rate (θ) x True Stress (σ). The strain hardening rate (θ) is defined by the derived stress in relation to strain (dσ/dε). The inflection point of this curve represents the point at which the stress curve versus strain changes behavior, given that only dynamic recovery occurs (PADILHA and SICILIAN, 2005). In the curve θ x σ, at the point where the strain hardening rate is equal to zero corresponds to the peak stress (σ_p), and the inflection point of the curve indicates the critical stress (σ_c) at which dynamic recrystallization begins (PADILHA and SICILIAN, 2005; SHABAN and EGHBALI, 2010).

In Figure (4a), the strain hardening rate (θ) versus true stress (σ) curves are shown for tests carried out with a strain rate of $0.2s^{-1}$ for deformation temperatures above Tnr and in (4b) for different strain rates and a temperature of 1150°C. In Figure (4a), the strain hardening rate decreases as the stress increases initially in a linear way and soon afterwards in an approximately parabolic way, as expected. This behavior change of the curves is due to dislocation accumulation and the appearance of subgrains. In the parabolic sector of the curves, an inflection point is observed that corresponds to critical stress (σ_c) for the start of the dynamic recrystallization. To determine the critical strain (ε_c), the critical stress value (σ_c) moves towards the flow stress curve versus strain and meets the critical deformation (ε_c). Still in Figure (4a), the strain hardening rate (θ) decreases until a maximum stress value that corresponds to the peak stress (σ_p), where the strain hardening rate (θ) is equal to zero.

In Figure (4b), there was a peak strain rate ($0.8s^{-1}$) and the inflection point was not observed. This behavior can be explained by the high strain rate, which tends to generate more dislocations in a short space of time, hindering the perception of the inflection point in the curve. The recrystallization influence is clearer with smaller strain rates (0.2 and $0.4s^{-1}$).

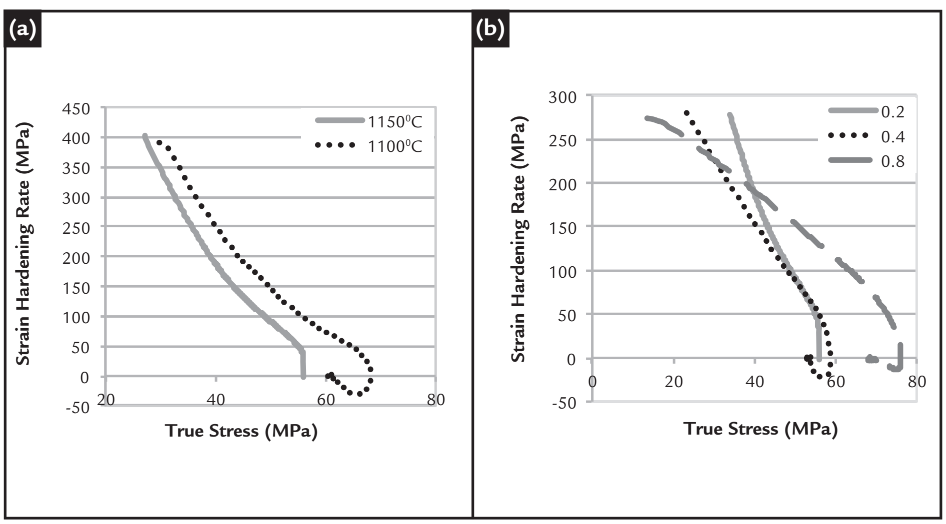

Fig. 4
Strain Hardening Rate Variation (θ) versus True Stress for API 5L X80 steel:
(a) for different temperatures at a constant strain rate 0.2s-1.
(b) for different strain rates and a constant temperature of 1150°C.

For a better visualization of the critical stress (σ_c), we can calculate the derivative of the strain hardening rate in function of the stress ($d\theta/d\sigma$), and plot the graph ($d\theta/d\sigma$ x σ_c), as can be seen in Figure 5. The maximum point of the curve represents the critical stress (σ_c), which is used to determine the critical strain (εc) corresponding to the flow stress curve ($\sigma x \varepsilon$) (SHABAN and EGHBALI, 2010).

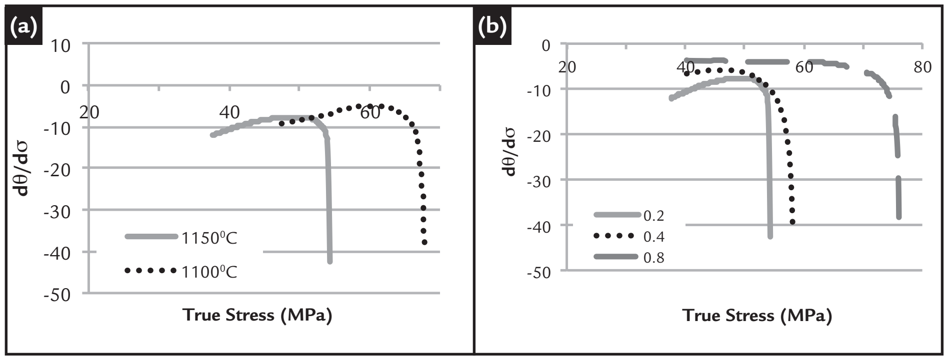

Fig. 5
Derived hardening rate ($d\theta/d\sigma$) versus the true stress (σ) curves for API 5L X80 steel:
(a) at different temperatures and a constant strain rate of 0.2s-1.
(b) with different strains and a constant temperature of 1150°C.

Using the curve details in Figures 2 to 5, Table 1 was elaborated with thermo-mechanical parameters, such as peak stress (σ_p), critical stress (σ_c), peak strain (ε_p), critical strain (ε_c) and steady state stress (σ_{ss}).

Temperature (°C)	$\dot{\varepsilon}$ (s^{-1})	σ_p (MPa)	σ_c (MPa)	ε_p	ε_c	$\varepsilon_c/\varepsilon_p$	σ_{ss} (MPa)
1150	0.2	57.4	48.5	0.36	0.17	0.47	50.5
1150	0.4	58.8	49.8	0.46	0.23	0.50	53.1
1150	0.8	76.2	64.1	0.62	0.35	0.57	68.3
1100	0.2	68.3	59.7	0.45	0.24	0.53	61.1
1000	0.2	100.9	-	0.70	-	-	88.6
950	0.2	123.4	-	0.80	-	-	107.0
850	0.2	155.6	-	1.99	-	-	155.6

Table 1
Values obtained in the isothermal tests at different temperatures and different strains rates.

According to Table 1 the values of peak and critical stress increase as the temperature decreases and the strain rate increases. For lower carbon steels, the $\varepsilon_c/\varepsilon_p$ relationship is 0.83 and for niobium steels that value can reach 0.5 (PADILHA and SICILIANO, 2005). For temperatures above Tnr, the value for the $\varepsilon_c/\varepsilon_p$ relationship is in agreement with literature.

4. Conclusions

Based on the results from the hot torsion tests, we can conclude that:

1. The critical temperatures of the API 5L X80 steel microalloyed Nb-Ti were Tnr = 1050ºC, Ar3 = 750ºC and Ar1 = 690ºC that present values close to those found in literature.

2. As expected, in the isothermal tests the values of the peak stress and critical stress increase as the temperature decreases and as the strain rate increases, and values in relation to $\varepsilon_c/\varepsilon_p$ were close to 0.5. The curves generated in the tests show a similar behavior to other steel studies.

3. In the isothermal tests for temperatures above Tnr (1050ºC), the main restoration mechanism is dynamic recrystallization. This can be confirmed through the shape of the flow stress curve. Below this temperature, the curves showed a slower recovery, characteristic of dynamic recovery.

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