EFFECT OF CENTRAL SEGREGATION IN THE CONTINUOUSLY CAST SLAB OF A HTP STEEL OVER AUSTENITE RECRYSTALLIZATION KINETICS AND ITS CRITICAL TEMPERATURES*

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Abstract

The objective of this study was to determine the effects of an eventual slab central macro-segregation in microalloyed steels with low Mn content (0.80%) and high Nb content (0.08 to 0.10%) on the kinetics of recrystallization of austenite during its rolling, on its critical temperatures, that is, of non-recrystallization (T_{nr}), start A_{r3} and finish A_{r1} of ferrite transformation and its response to a simulated industrial thermomechanical processing. The determination of the austenite recrystallization kinetics was made through isothermal double compression tests using a dilatometer, while the critical temperatures were determined through hot torsion tests in a Gleeble® 3800. It was found that austenite recrystallization kinetics, A_{r3} and A_{r1} temperatures did not present significant changes for the samples extracted both at ¼ of the thickness and at the core of the plate, indicating that, as expected, the central segregation in this type of steel was not intense to the point of affecting this phenomenon. The T_{nr} values determined for the samples extracted in the core of the plate were, on average, 30°C lower than those obtained from specimens extracted at ¼ thickness, which suggests that a significant portion of the carbonitrides present in the core of the slab are eutectic and coarse, not having completely dissolved during the reheating of the specimens. This occurred despite the fact that the austenitization temperature adopted in this treatment theoretically leads to the total dissolution of Nb, as seems to have occurred in the case of specimens extracted at ¼ of thickness, where carbonitrides have a finer size.

Keywords: Niobium Microalloyed Steels; Austenite Recrystallization; Central Segregation; Critical Temperatures

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1 INTRODUCTION

With the introduction on the market of steels with low Mn content and higher Nb content high temperature processed steel (HTP steel), it is expected that the level of segregation will be reduced and consequently the optimization of the performance of heavy plates produced with these steels for "Sour Service".

In the heavy plates controlled rolling with high Mn the mechanisms of grain refinement and precipitation of Nb carbonitrides are already known. For steels with low Mn content and high Nb, the knowledge of these mechanisms, during the heavy plates rolling and subsequent accelerated cooling, it is still not well established. The knowledge of the kinetics of precipitation of carbonitrides (NbC) in deformed austenite can be extremely useful for the design of thermomechanical schemes of microalloyed steels. Therefore, it is of great importance the study of the behavior of this steel during the heavy plates rolling related to the strain induced precipitation of Nb carbonitrides and its effect on the mechanisms of softening and the temperature of non-recrystallization.

This work has the objective of studying the precipitation of Nb (C, N) in the austenite after the hot deformation of a steel with low Mn content and 0.10% Nb (HTP) and verify the influence of the central macro-segregation of plates in the precipitation of Nb (C, N) and in the heavy plates rolling.

To study the precipitation during hot rolling in the laboratory, several authors have used the application of double compression isothermal tests, varying the time between pass, and the technique of stress-relaxation [1-5]. Hong et al [1] determined, by double compression isothermal test, start and finish times of precipitation for one Nb-microalloyed steels and another for Nb and Ti and concluded that the precipitation start time for Nb steel is lower than for Nb-Ti steel. A study of two steels, 0.6C-20Mn-1.5Al, was carried out, one of them without Nb and the other containing 0.11% of Nb, also using the double compression technique but by torsion tests. These authors concluded that at temperatures above 1100°C, the recrystallization kinetics were similar for both steels and, at temperatures below 1000°C, strain induced precipitation was observed resulting in significant delay in the softening only for Nb steel.

In order to design thermomechanical schemes of controlled rolling of microalloyed steels, it is important to study the metallurgical behavior that occur during the hot rolling process of these steels, because relevant changes occur in the microstructure besides the phase transformations. With the determination of critical temperatures, such as non-recrystallization temperature (T_{nr}) and austenite-ferrite (A_{r3}) transformation start and their effects, it is possible to adjust the processing parameters such as: rolling temperature, strain rate, strain, time between pass and cooling rate. T_{nr} and A_{r3} can be calculated from the chemical composition of steels, according to equations 1 [6, 7] and 2 [8] which were established from multiple correlations of experimental results:

$$T_{nr} = 887 + 464C + \left(6445Nb - 644\sqrt{Nb}\right) + \\ + \left(732V - 230\sqrt{V}\right) + 890Ti + 363Al - 357Si \qquad (1)$$

$$A_{r3} = 910 - 310C - 80Mn - 20Cu - 15Cr - 55Ni - \\ - 80Mo + 0.35(t-8) \qquad (2)$$

Where t, in Equation 2, is the thickness of the rolled plate and is considered between 8 and 30 mm.

These equations have been widely used, but their application is limited to the alloying elements contents of the steels that were used in their determination. For example, they do not apply to high Niobium contents such as HTP steel studied in the present project. With hot torsion tests with multiple deformation in continuous cooling it is possible to determine the critical processing temperatures by applying the method of Boratto et al [6, 7].

2 MATERIAL AND METHODS

Samples were drawn from blank with 34 mm of thickness, after as rolled from steckel mill of a low Mn and high Nb HTP steel with chemical composition shown in Table 1.

Table 1: Chemical composition of HTP steel (wt. %)

C	0,039
Mn	0,54
Si	0,21
Cu	0,27
Ni	0,15
P	0,008
S	0,001
Ca	0,0025
Nb	0,090
Al	0,029
Cr	0,47
Ti	0,012
N	0,005

For the isothermal double compression tests in the dilatometer, the samples were drawn with the axis longitudinally to the rolling at ¼ of the thickness and in the core of the thickness and for simulations by hot torsion tests in the Gleeble® simulator, model 3800, specimens were machined in the direction with axis longitudinal to rolling.

2.1 Precipitation of Nb (C,N) in austenite after hot deformation

2.1.1 Isothermal double compression tests

The methodology used to study the precipitation during hot rolling was the application of isothermal double compression test, varying the time between passes. The softening between passes can be calculated by flow stress curves using equation 3:

$$X_{SRX} = \frac{(\sigma_{m1} - \sigma_{e2})}{(\sigma_{m1} - \sigma_{e1})} \qquad (3)$$

where:
X_{SRX} is the softening fraction;

σ_{m1} is the maximum stress in the first deformation;

σ_{e2} is the yield strength of the second deformation;

σ_{e1} is the yield strength of the first deformation.

For the isothermal double compression test, cylindrical specimens measuring 5 mm in diameter and 10 mm in length, with the axis parallel to the rolling direction, were machined at the ¼ plate thickness and center of thickness positions. The tests were carried out in a TA Instruments Dilatometer, model DIL805T/D, following the scheme shown in figure 1. Flow stress curves were obtained for the two deformations and calculations of the softening fraction between passes were made using equation 3.

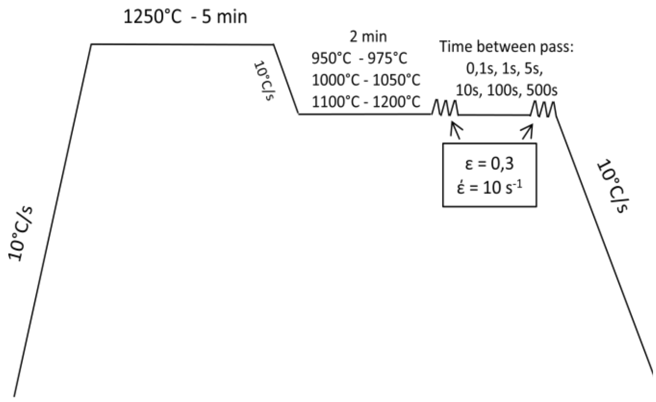

Figure 1: Scheme of isothermal double compression tests

2.1.2 Analysis of the precipitates by transmission electron microscopy

To obtain samples for the analysis of the precipitates by transmission electron microscopy (TEM) three tests of double hit compression at the temperature of 950°C were interrupted, followed by quenching to room temperature, in the following stages:
- Before the first deformation
- 10s after the first deformation;
- 100s after the first deformation.

The samples were prepared using the thin foil technique to analyze the precipitates formed in the transmission electron microscope. The thin foil were examined in the TEM JEOL 2100 PLUS® operating at 200KV and the chemical composition of the precipitates was determined using the Oxford® EDS X-Max 80 SD using an SDD type detector.

2.2 Determination of critical temperatures

In order to determine the critical temperatures (T_{nr}, A_{r1}, A_{r3}), the torsion tests were carried out in the Gleeble® simulator, model 3800, with multiple deformations in continuous cooling. Heating rate of 10°C/s was used until the solubilization temperature was 1250°C and soak time of 5 minutes. After the solubilization, the test of multiple deformations during the continuous cooling with rate of 1°C/s and application of equivalent deformations of 0,2, strain rate of $1s^{-1}$, was carried out from 1220ºC, in intervals of 30°C.

3 RESULTS AND DISCUSSION

3.1 Precipitation of Nb (C,N) in austenite after hot deformation

3.1.1 Isothermal double compression tests

Figure 2 shows examples of flow stress curves of the isothermal double compression test assays in samples drawn at ¼ of the thickness for the time between passes of 10s and figure 3 for the time between passes of 100s.
The yield strength was determined through the flow stress curves of Figures 2 and 3 by the 0.2 true deformation method. The softening between passes can be determined using equation 3.

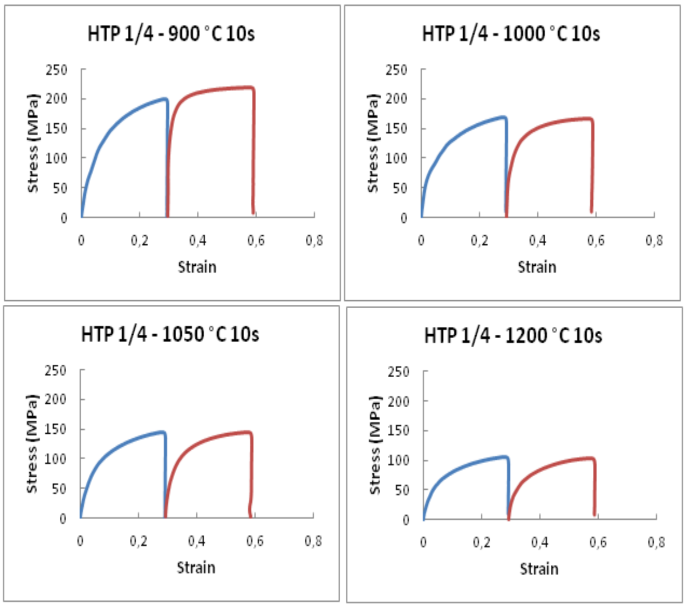

Figure 2: Flow stress curves of double hit compression isothermal tests of HTP steel, time between passes of 10s, samples taken at ¼ of the thickness.

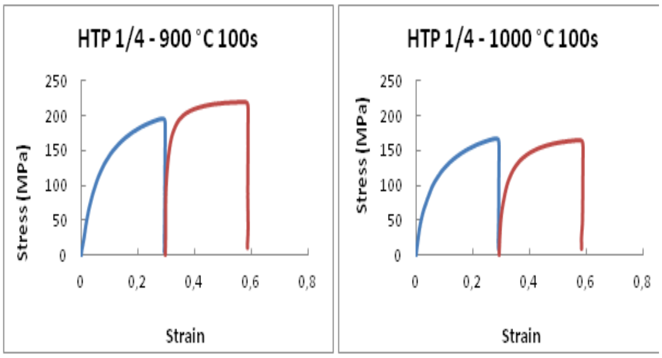

Figure 2: Flow stress curves of double hit compression isothermal tests of HTP steel, time between passes of 100s, samples taken at ¼ of the thickness.

Figure 4 shows the results of the softening between passes for the tests carried out on HTP steel specimens at ¼ of the thickness. The results show that for temperatures of 1100 and 1200°C, occurred total softening (recrystallization) for the time between passes of only 10s occurred, indicating the absence of precipitation. For this reason, temperatures below 1100°C were chosen for the studies of the precipitation of niobium carbides during the hot rolling of heavy plates. At the temperature of 1050°C, the curve is characteristic of static recrystallization, with total softening after 100s of time between passes. At a temperature of 1000°C, a deviation in the recrystallization curve, caused by the beginning of precipitation, is observed in the time of 10s. The precipitation continues, evidenced by a threshold in the softening curve up to the time of 100s and even for the time between passes of 500s, the softening was not total.

It can also be seen in figure 4 that deviation in the softening curve occurred for the time of 10s at the temperature of 975°C and that the softening fraction was only 0.5 in the time between passes of 500s as a result of precipitation of carbides of niobium. At temperatures of 900 and 950°C it can be considered that no softening has occurred by static recrystallization for any of the times used, provided that the softening fraction of 0.2 is due to static recovery. The temperatures of 950°C and 1000°C were chosen to obtain samples for the characterization of niobium carbides.

In Figure 5, the results of the tests performed on HTP steel specimens at the core of the thickness are presented. The softening fraction curves show that, in the same way as occurred in the specimens at ¼ of the thickness, precipitation occurred in times between passes equal to or greater than 10s for temperatures below 1050 ° C.

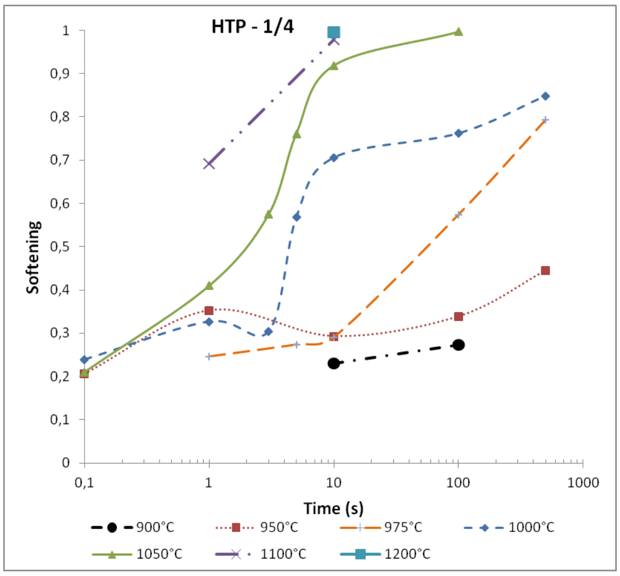

Figure 3: Curves of softening fraction, as a function of the time between passes, obtained from the isothermal double compression tests on HTP steel specimens at ¼ of the thickness.

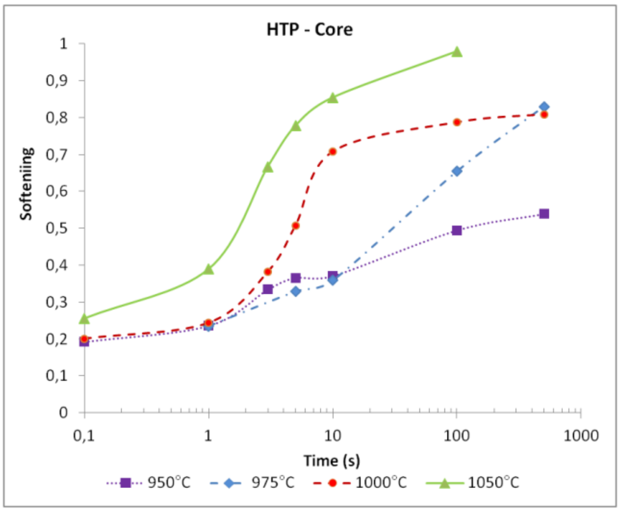

Figure 5: Curves of softening fraction, as a function of the time between passes, obtained from the isothermal double compression tests on HTP steel specimens at core of the thickness.

3.1.2 - Analysis of the precipitates by transmission electron microscopy

In the transmission electron microscope (TEM) analyzes the precipitates were identified by EDS analysis as being of NbC and (Ti, Nb)C. Figure 6 summarizes examples of precipitates of these carbides in the HTP steel observed in the thin foil on bright-field (BF) mode by scanning transmission electron mode (STEM). This figure also shows the results of EDS measured on some precipitates. The STEM-BF images, and the EDS data synthesize the results obtained for these HTP steel samples for which the average size of the precipitates reduces with post-deformation time while the Nb concentration increases.

Figure 7 shows the EDS elemental analysis of the precipitates in Figure 6 indicated by acronyms EDS1, EDS2 and EDS3. The composition of the precipitates analyzed by EDS shows rich in Nb content, and small amount of Ti. The Ti/(Ti+Nb) ratio values were found to be 0.67, 0.59 and 0 for the HTP 950ºC @ quenching, HTP 950ºC @ 10 s, and (c) 100 s, respectively.

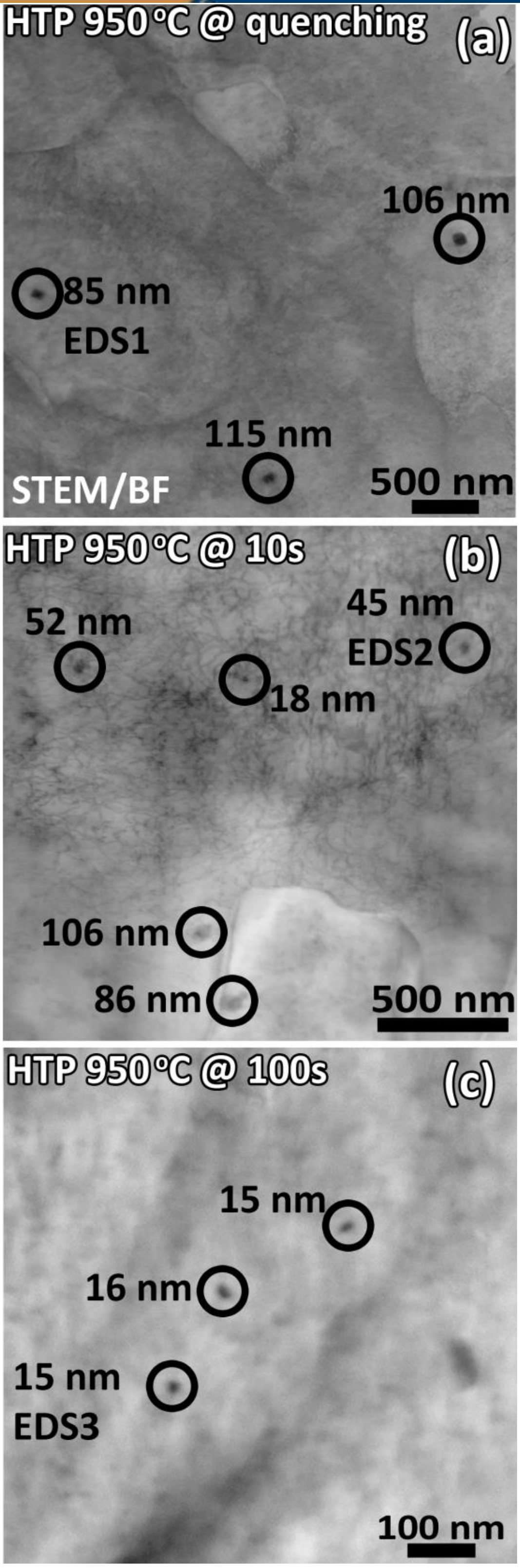

Figure 6: STEM BF images of HTP steel (a) HTP 950ºC @ quenching, (b) HTP 950ºC @ 10 s, and (c) 100 s. Also, this figure shows nanoparticles seen in the HTP steel in which EDS spectrums were acquired in attempt to determine their chemical composition.

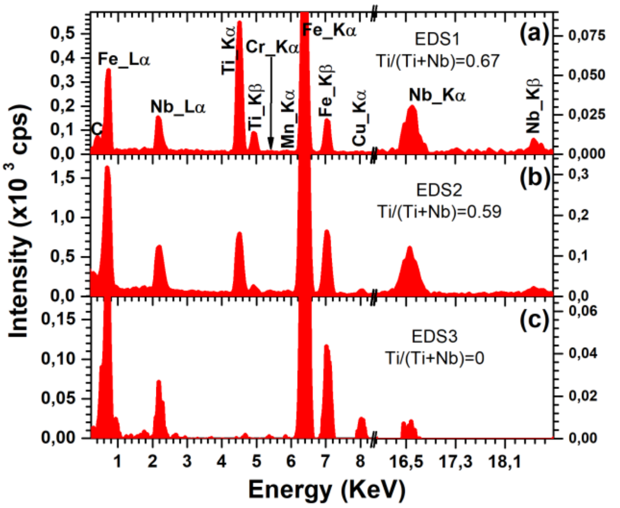

Figure 7: EDS results of the precipitates indicated in the Figure 6.

TEM images of the samples prepared by thin foil method shows in detail the morphology of the steel with the (Nb,Ti)C precipitates indicated by the yellow arrows are shown in Figure 8. The precipitates sizes were found to be in the range of 10 to 87 nm. TEM results also revealed the size dependence of the precipitates with the time before quenching.

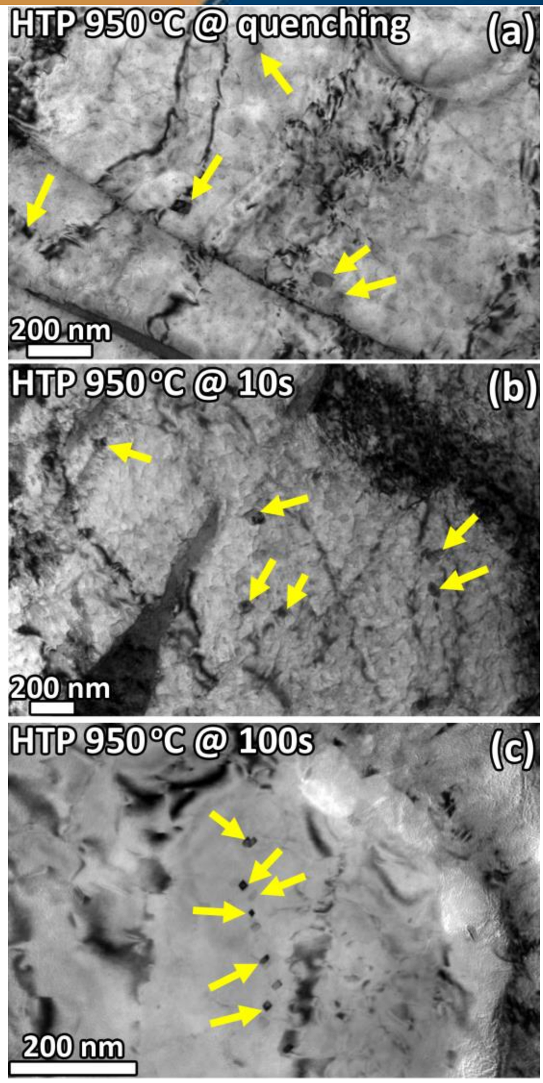

Figure 8: BF-TEM images of steel (a) HTP 950ºC @ quenching, (b) HTP 950ºC @ 10 s, and (c) 100 s.

3.2 Determination of critical temperatures

Figure 8 shows the flow stress curves obtained from hot torsion tests with multiple deformations in continuous cooling, samples in the core and ¼ of the thickness for HTP steel used for the determination of critical temperatures.

Figures 9 and 10 show the results of mean flow stress versus 1000/T (°K), showing T_{nr}, A_{r3} and A_{r1}, for HTP steel, in samples at the core and ¼ of the thickness, respectively. When the non-recrystallization temperatures in the core and ¼ of the thickness are compared, a reduction of 30°C of ¼ to the core can be observed. This can be explained by the fact that, although niobium contents are higher in the core, most carbonitrides of larger Nb and Ti are also formed in the core of the plate and do not dissolve completely during reheating for hot rolling. Therefore, the content of niobium dissolved in the austenite may have been higher in the ¼ position relative to the core, causing the increase of T_{nr}.

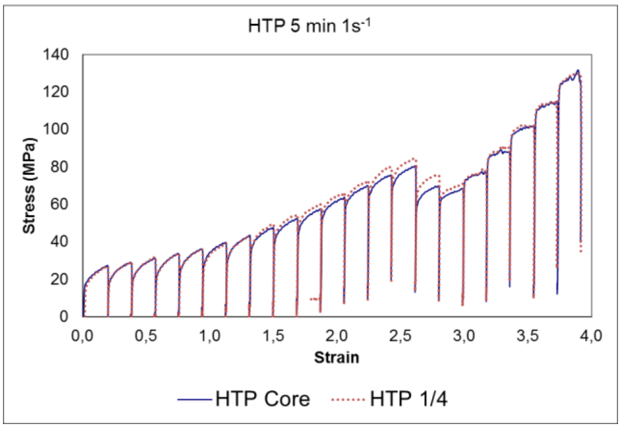

Figure 4: Stress-strain curves obtained after hot torsion tests with multiple deformations in continuous cooling for HTP steel in samples from the core and ¼ of the thickness.

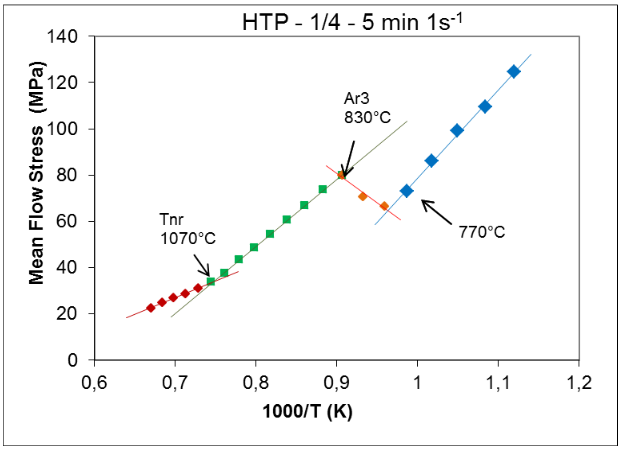

Figure 5: Mean Flow Stress versus 1000/T(°K), showing Tnr, Ar3 e Ar1 for HTP steel at the ¼ of thickness.

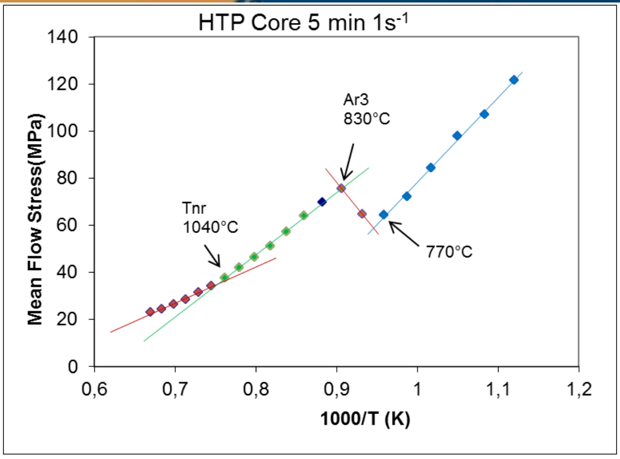

Figure 6: Mean Flow Stress versus 1000/T(°K), showing Tnr, Ar3 e Ar1 for HTP steel at the core of thickness.

4 CONCLUSIONS

• The results of transmission microscopy analysis in post-deformation times of 10s for HTP steel deformed at 950°C, the presence of small precipitates (<25 nm) in Nb was observed, indicating that the start time of the strain induced precipitation is below 10s at these temperatures.

• For the post-deformation time of 100s, it has been observed in TEM analyzes, for HTP steel, that the number of small (<25 nm) and Nb-rich precipitates increases with respect to the time of 10s, at the same time as it occurs a reduction in the number of precipitates in the range between 20 and 60 nm rich in Ti.

• The precipitates analysis by TEM is in agreement with the softening curves of the isothermal double compression tests of the HTP steel at 950°C, in which it was observed that no recrystallization occurred for any of the times between passes at these temperatures, evidencing that the precipitation of the Niobium carbides were started at times less than 10s.

• In the results of the isothermal double compression tests, no significant difference was observed in the HTP steel softening curves between the specimens at ¼ of the thickness and in the core.

• When the non-recrystallization temperatures in the core and ¼ of the thickness were compared, a reduction of 30°C of ¼ to the core of the thickness was observed.

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