SOFTENING BEHAVIOR OF A TITANIUM ADDED ULTRA-LOW CARBON STEEL AFTER HOT AND WARM DEFORMATION*

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Abstract

In this work, the mechanical and metallurgical behavior of titanium added ultra-low carbon (ULC-Ti) steel under hot and warm working conditions was studied by means of dilatometer compression tests. Double compression schedules were performed at temperatures between 700°C and 1000°C, inter-pass times of 0.5-100 s, and deformations of 0.5 and 0.3 in the first and second stages, respectively, with strain rate of 1 s^{-1}. The analysis of the flow curves obtained allowed the evaluation of the softening characteristics of the steel. The softening and microstructure features indicate different restoration mechanisms depending on the temperature range. Static recrystallization with rapid microstructure restoration is observed at temperatures within the austenitic and intercritical field. Strain induced boundary motion and recovery mechanisms apparently control the restoration process in the ferrite region. In general, flow stress curves showed dynamic recovery behavior. Only the deformation in the intercritical region produced a peak in the flow stress curves. Isothermal holding tests associated with single hit compression indicated the presence of dynamic strain induced transformation, accelerating the softening kinetics at the intercritical region.

Keywords: Double compression; Softening mechanisms, Warm deformation; Dynamic transformation; Recrystallization.

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1 INTRODUCTION

Ultra-low carbon steels were developed as an alternative to conventional steels, aiming to improve the formability of flat products. The constant tendency of exposed panels with increasingly complex geometries makes this steel widely used in the present automotive construction.

Normally the processing of IF steels in a hot strip mill is performed in the austenite region. Due the ultra-low carbon content, phase transformation temperatures are relatively high, when compare with conventional steels. This feature in some cases can cause finishing rolling in the intercritical region in the last stands. This practice can generate operational instabilities, quality deviations and production interruptions [1,2].

An alternative to these reported issues is the ferritic or warm rolling. In addition to improving the flatness of the coils produced and reducing operational instabilities, warm rolling makes it possible to obtain new microstructural parameters in accordance with the quality requirements. It is possible to obtain larger grain sizes, resulting in lower loads in cold rolling. One can even maximize the crystallographic texture favorable to the material's drawability, in some cases it is possible to suppress the cold rolling of the material. The lower rolling temperatures make it possible to reduce the reheating temperature of the slabs, reducing the fuel consumption as well as the scale generation in the reheating furnace [1].

It is known that recovery and recrystallization are the main restoration mechanisms occurring during hot deformation. Each material phase has crystallographic characteristics that can change the predominance of a softening mechanism [3,4]. Warm rolled steels typically can present static and dynamic recovery, since the ferrite phase has a high stacking fault energy [1]. The deformed microstructure from ferritic rolling is therefore usually composed of an arrangement of dislocations due the recovery phenomenon. Some studies have also reported the occurrence of dynamic recrystallization (DRX) in ferrite. An apparently discontinuous dynamic recrystallization was demonstrated in studies with pure iron and with IF steels [1]. The authors identified a clear transition between dynamic recovery (DRC) and DRX as a function of temperature and strain rate [1].

In this direction, this work aims to extend the technical knowledge concerning the process of hot and warm deformation of a titanium added ultra-low carbon steel and the restoration mechanisms during the rolling process, giving subsides to the development of the industrial warm rolling practice, with the potential for cost reductions and new products manufacturing routes.

For this purpose, all the tests were performed in a Bähr DIL805 Dilatometer. The specimens were submitted to soaking, followed by deformations at temperatures above, between and below the phase transformation temperatures. For the study of the softening mechanisms double compression tests were applied, varying the temperature and time between the passes associated with microstructural analysis.

2 MATERIAL AND METHODS

The study was performed on a Ti added ultra-low carbon (ULC-Ti) steel, produced at Usiminas' Hot Strip Mill line. The chemical composition of the steel is given in Table 1.

Table 1. Chemical composition (% weight)

C	Mn	Al	P	S	Ti	Si
0.001	0.096	0.042	0.011	0.008	0.061	0.020

A sample was obtained from the hot rolled coil, with a thickness of 6 mm. To perform the dilatometric tests, solid cylindrical specimens with 5 mm diameter and 10 mm height were machined. The specimens were machined at the center of the sample thickness, with the length positioned at 90° with the rolling direction.

Preliminary phase transformation tests were performed in order to map the austenite to ferrite transformation temperatures on cooling. Then, double compression tests were executed in hot and warm regions. After soaking, the specimens were cooled to the deformation temperature, where the first compressive deformation was performed. After unloading, the deformed test specimen was held at the test temperature for a certain time between passes (Δt), and then received a second compressive deformation, as shown in Figure 1. The times between the passes (Δt) used were: 0.5, 1, 2, 5, 10, 20, 50 and 100 s. The deformation temperatures were 700, 750, 800, 850, 900, 950 and 1000°C. The strain at the 1st (ε_1) and 2nd (ε_2) passes was 0.5 and 0.3, respectively, with strain rate of 1 s^{-1}. The analysis of the flow curves obtained allowed the evaluation of the softening characteristics of the steel.

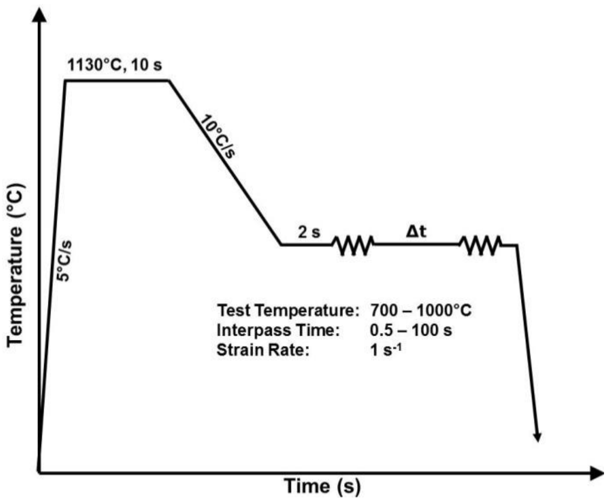


Figure 1. Schematic diagram of the schedule employed in the double compression tests.

The experimental data obtained in the double compression tests allowed the determination of the degree of softening of the steel as a function of the deformation temperature and time between the passes. In this study the offset method was used [5], which is based on the change of the yield stress between the first and second passes. The softening volume fraction (X_{off}) was calculated by the equation 1, where σ_1 and σ_2 are the yield stresses (0,2% offset strain) of the first and second compression, respectively, and σ_m is the maximum flow stress at the first compression.

$$X_{off} = \frac{\sigma_m - \sigma_2}{\sigma_m - \sigma_1} \times 100\ \% \qquad (1)$$

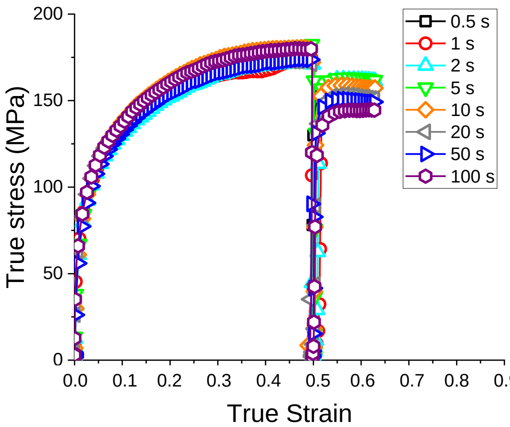
For the study of the microstructural evolution during the double compression tests in each temperature (700 to 1000°C), the thermomechanical cycle was interrupted at intermediate times of the tests, followed by rapid cooling. It was also evaluated the phase transformation effect on the flow stress curves, at temperatures in the intercritical region and in its vicinity. These tests consisted in soaking the specimen at 1130°C for 10 s, followed by cooling (10°C/s) to three different temperatures (950, 900 and 850°C) and keeping the specimen at each temperature for 600 s. Complementing this investigation, simple compression tests were carried out at different points along the holding time. Thus, a true strain of 0.5 was applied after isothermal holding times of 2, 10, 60 and 600 s at 900°C.

3 RESULTS AND DISCUSSION

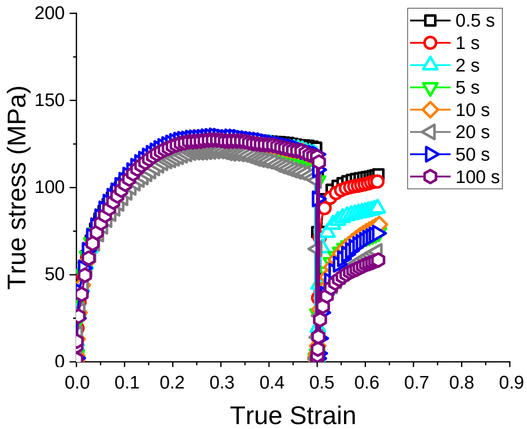
Preliminary dilatometer analysis indicated the intercritical region between 950°C and 850°C during cooling. This information was used to direct the deformations in three different phase regions: austenitic, intercritical and ferritic.

3.1 Flow Curves

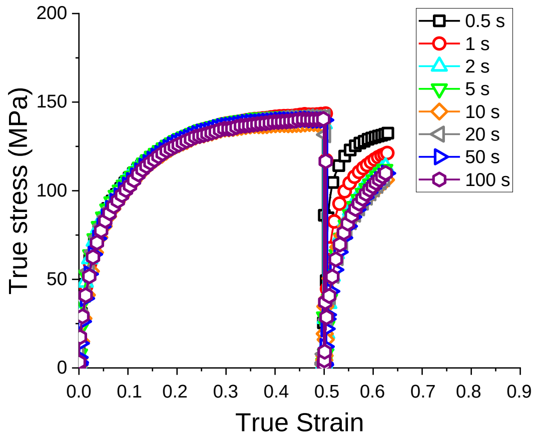
Figure 2 shows examples of the flow curves obtained by double compression for the temperatures of 750°C (ferrite region), 900°C (intercritical region) and 1000°C (austenitic region), with the abscissa being the true strain accumulated. The well-known friction influence in the flow stress curves when dilatometer tests are carried out without lubrication [5] was observed, starting from accumulated true strain of 0.65, and therefore suppressed from the results. It can be seen that generally the yield stress of the second deformation decreases with the increase of interpass time. As expected, the flow stress decreased with the increase of deformation temperature, except for 900°C, which showed an abnormal behavior, with very low stress values in second deformation, and dynamic softening in the first pass, characterized by the presence of a peak flow stress. This behavior was associated to dynamic strain induced transformation, further discussed in item 3.3. Except for this variation, the flow stress levels obtained were close to the values found in similar researches [6,7].



(a)



(b)



(c)

Figure 2. Strain-Stress flow curves for different interpass times and temperatures (a) 750°C (b) 900°C (c) 1000°C.

3.2 Softening Behavior Analysis

Figure 3 presents the evolution of the softening volume fraction curves for each temperature, where it is possible to observe the effect of time between the passes and temperature on the softening characteristics. As expected, increasing the time between the passes enabled the progress of the static softening mechanisms in the steel, gradually increasing the softening volume.

It is known that recrystallization generally follows a sigmoidal kinetics, developing rapidly after a short period of initial slower nucleation [8]. At temperatures of 800°C to 1000°C the curves showed a trend of a sigmoidal shape. At temperatures of 900°C and 1000°C the time to begin the recrystallization was very low, with the softening process trigged very fast, even immediately after the deformation. At temperatures of 750°C and 700°C, the slope of the softening curves was less pronounced, which is generally associated with the predominance of the recovery softening process [4].

Intercritical temperature of 900°C showed abnormal softening fractions, with the yield stress in the second deformation lower than the original state (X_{off}>100%). This unexpected behavior was further investigated on item 3.3.

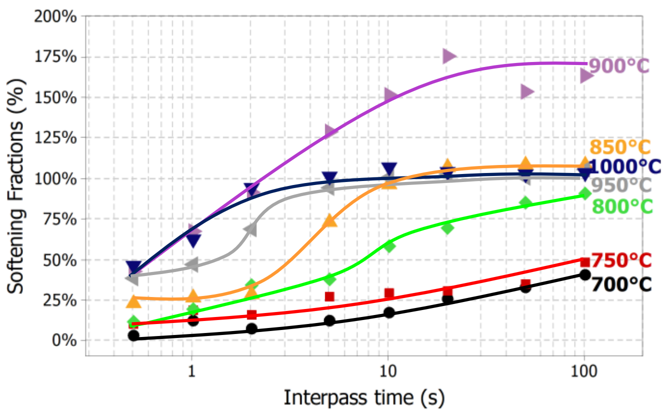


Figure 3. Softening fractions in function of interpass times under different temperatures.

In order to aid in the understanding of the evolution of the softening at different temperatures and times between passes, microstructural analysis was carried out through rapid cooling at certain points along the thermomechanical cycle. Figure 4 shows the microstructures analyzed with test temperatures of 800°C, 900°C and 950°C. These three temperatures show typical microstructural evolution observed during the tests in ferrite, intercritical and austenite regions, respectively. As expected, it can be seen that as the holding time after the first deformation increases, the microstructure evolves towards a state of lower deformation for all cases. This behavior was observed for all analyzed temperatures.

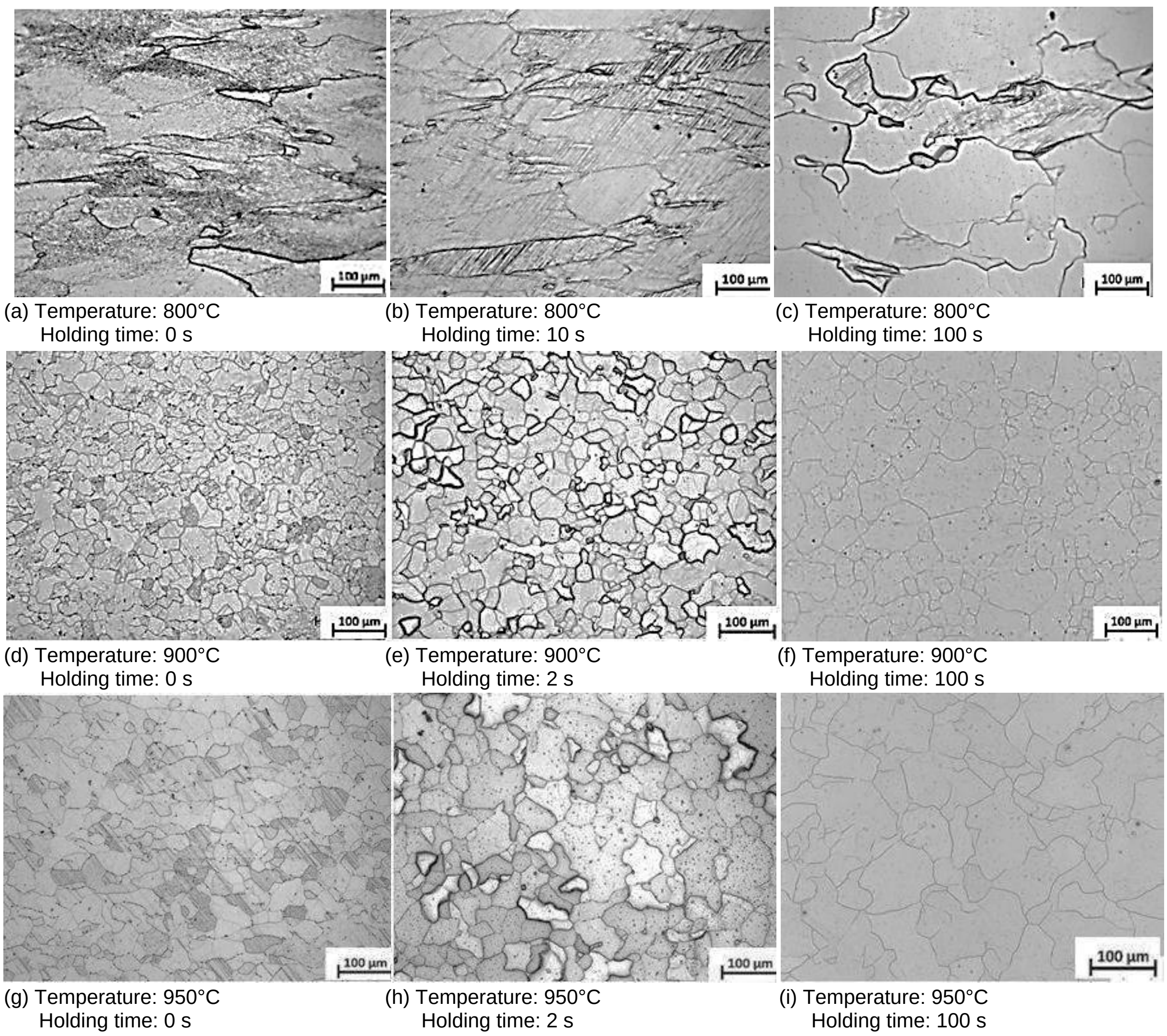


Figure 4. Microstructural aspect in different temperatures and different holding times after deformation.

In the fully ferritic temperature of 800°C, immediately after the pass (Fig. 4a) the grains are deformed and elongated. Moving to 10 s after the pass (Fig. 4b), there was an evolution of softening, probably due to the static recovery process, associated with the occurrence of strain induced grain boundary motion (SIBM) mechanism [4]. Figure 4b is a typical example of SIBM, where areas free of deformation advance towards the deformed region. By increasing the holding time after pass to 100 s (Fig. 4c), probably this mechanism was magnified, where the microstructure presents an increase in the volume fraction of grains free of deformation, which contributed to achieve the softening volume fraction of almost 100% (Fig. 3).

In the intercritical region, the microstructure was completely recrystallized after the deformation (Fig. 4d), with small equiaxed grains free of deformation, corroborating with the fast kinetics of recrystallization in these temperatures, verified by the analysis of the softening fractions. But as the holding time increased, very short grain sizes were seen, actually smaller than at the austenitic temperatures with the same time after pass.

For the austenite region (950°C), the microstructure presents a typical recrystallization evolution. In the first stage, immediately after deformation, it was observed newly formed recrystallized grains (Fig 4g). Advancing to the intermediate times (Fig. 4h) there was a growth of the recrystallized grains.

For holding time of 100 s after deformation, (Fig. 4i), it can be observed a complete restoration of the microstructure. These characteristics confirm the predominance of the static recrystallization phenomenon in the analyzed austenite region.

It is possible to model the softening fraction curves obtained experimentally, for softened fractions lower than 100%, following an Avrami type equation (Eq. 2) [8]:

$$X = 100\left\{1 - \exp\left[-B\left(\frac{t}{t_{50}}\right)^{n}\right]\right\} \quad (2)$$

Where “B” and “n” are steel constants. A linear dependence of the softening fraction with time is obtained by taking twice the natural logarithm of the term 100/(100-X) versus the time natural logarithm, as shown in Figure 5. This analysis covered the experiments with temperatures from 800°C until 1000°C, and with softening fractions lower than 100%. Temperatures of 700°C and 750°C were excluded from this analysis, due to its maximum softening fraction being lower than 50%.

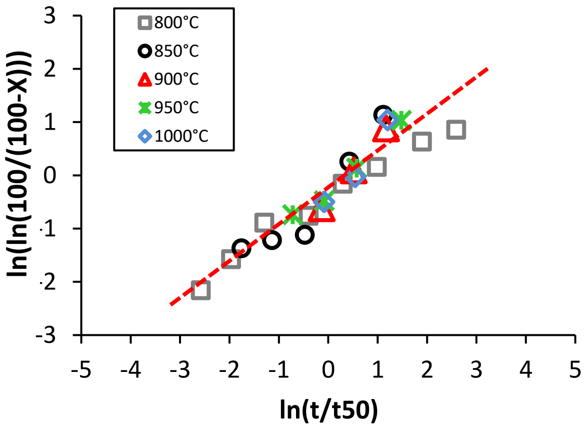


Figure 5. Dependency between Ln[100/(100-X)] and Ln(t/t_{50}) for different test temperatures.

In general the obtained results showed a good fit with the Avrami model, as can be seen at Figure 5. The inclination of these curves correspond the exponent “n” of equation 2. Thus, it was calculated the Avrami exponent “n” for each tested condition, as well as the coefficient of determination “R^2” associated to the data fitting to the linear regression model proposed. The results are exhibit on Table 2, and as expected they show the high dependency of the softening kinetics with the tested temperature.

Table 2. Fitting parameters obtained by linear regression for each test temperature.

Temperature °C	Avrami Exponent n	Coefficient of Determination R^2
800	0.566	0.974
850	0.722	0.803
900	1.151	0.999
950	0.827	0.975
1000	1.200	0.957

The lower Avrami exponent value obtained in the temperature of 800°C emphasizes the low speed of softening observed in the ferritic region. For the temperature of 850°C, besides the low value of "n", the coefficient of determination was low, due the non-linearity of the softening rate obtained in this temperature. There was a shift in the behavior from low to high softening rate, explained by the microstructure evolution analysis. This behavior was associated with the occurrence of an additional softening mechanism as the time between passes increased at 850°C, as already discussed. Once again, the results obtained in 900°C tests showed softening speed higher than expected. Finally the tests performed in temperatures higher than 900°C corroborated the high softening rate in the austenitic field.

One of the most widely used expression to calculate the parameter t_{50} is exhibit in equation 3 [9].

$$t_{0.5} = A.\varepsilon^{p}.\dot{\varepsilon}^{q}.\exp\left(\frac{Q_{rex}}{R.T}\right) \qquad (3)$$

Where A, p and q are material dependent constants, ε true strain, $\dot{\varepsilon}$ strain rate, Qrex the apparent activation energy for recrystallization (kJ/mol), T absolute temperature in Kelvin, and R the gas constant (J/mol.K). Taking the natural logarithm of previous equation, it was obtained Equation 4, where there is a linear dependency between temperature and 50% softening volume fraction.

$$\ln t_{0,5} = \ln A + p \ln \varepsilon + q \ln \dot{\varepsilon} + \frac{Q_{rex}}{RT} \qquad (4)$$

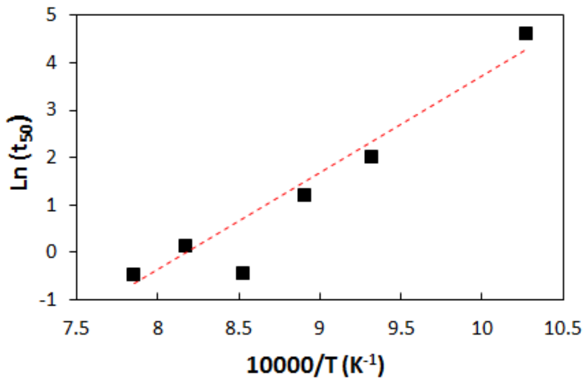


Figure 6. Dependency of 50% softening time with deformation temperature.

In general, the studied steel followed the linear dependency of equation 4, in temperatures between 800°C and 1000°C, as show in the Figure 6. Once again, the exception is found in intercritical temperature of 900°C (corresponding to 8.5 in abscissa, Fig. 6) due its high softening kinetics, with value of $\ln(t_{50})$ much lower than expected.

3.3 Intercritical Softening

During this investigation, it was presented several evidences of a softening higher than expected for the intercritical temperature of 900°C. Microstructure analysis could not explain this behavior, as the grain sizes were actually smaller than for the austenitic temperatures with similar time after pass. Aiming to identify the mechanism responsible for this behavior, it was performed isothermal holding test of 600 s at temperatures of 950°C, 900°C and 850°C. The results of this analysis is displayed in Figure 7, where the relative change in length of the specimen over the holding time is presented.

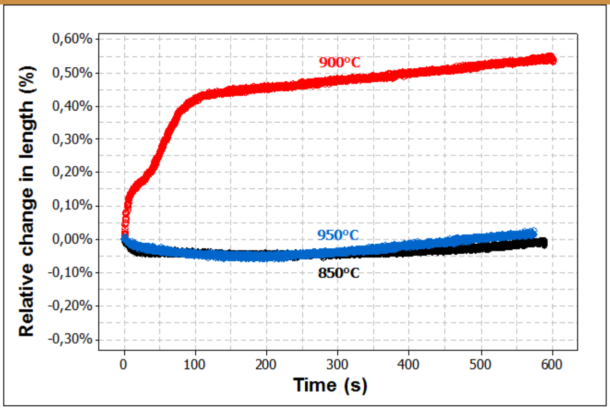


Figure 7. Dilatometric curves of isothermal holding tests at different temperatures.

When cooling was interrupted at temperatures of 850°C and 950°C there was a small initial volumetric contraction of the steel, but over time it was reached an equilibrium level with a near zero variation. In opposite way, at 900°C the material showed a sudden increase in volume with high kinetics, indicating a considerable transformation of austenite to ferrite over time. These characteristics demonstrate that the studied ULC-Ti steel has a great tendency to transform austenite into ferrite in short period of time when submitted to the temperature of 900°C.

At high temperatures the ferrite has lower resistance to deformation than austenite, a characteristic related to its crystallographic nature (greater dislocation mobility in ferrite associated with its high stacking fault energy) [4]. To consolidate the results, four additional tests of simple compression with a true deformation of 0.5 at 1 s^{-1} were performed after isothermal holding times of 2, 10, 60 and 600 s at 900°C. The results of these tests are shown at Figure 8.

As expected, the compression tests for holding times with different volume fractions of ferrite at 900°C showed variation on the flow curves shapes (Fig. 8).

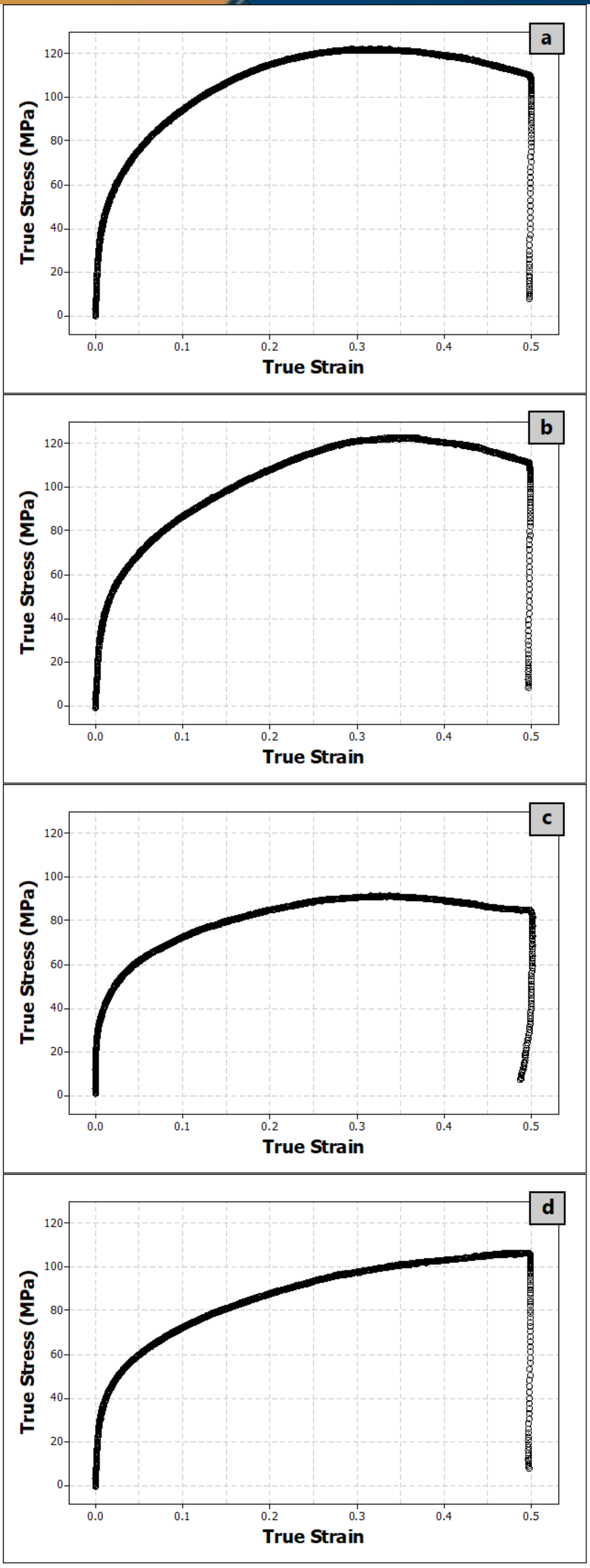


Figure 8. Stress-strain curves obtained at different isothermal holding times at 900°C: (a) 2 s, (b) 10 s, (c) 60 s and (d) 600 s.

Using the 0.2% offset method for calculating the yield stress the following values were found: 63.3 MPa, 57.6 MPa, 52 MPa and 50 MPa, for curves (a) to (d), respectively. A gradual decrease in the yield stress is observed as the holding time at the temperature of 900°C is increased. This effect is associated with a higher ferrite volume fraction formation over the holding time. Thus, in the double compression tests at 900°C, probably there was an increase in the ferrite volume fraction between passes, decreasing the material yield stress, which explains the excessive static softening observed. In this case, the presence of deformation probably increased this effect by accelerating the kinetics of ferrite formation.

An associated analysis of Figures 7 and 8 shows that the behavior of the flow stress suffers significant changes with the different phase fractions present in each test. It can be seen the presence of peak stress at deformation values near 0.3, with a subsequent sharp decrease of the stress in the flow curves (a), (b) and (c) of Figure 8 (holding times of 2, 10 and 60 s, respectively). In other way, curve (d) (600 s) does not show such behavior. As the ferrite volume fraction increased, the dynamic softening became gradually less pronounced. When the holding time was sufficiently high, 600 s, with almost no austenite volume fraction, there was no occurrence of dynamic softening (Fig. curve (d)). These characteristics are strong indications of the presence of dynamic strain induced transformation (DSIT) in the tests at 900°C, where the dynamic formation of ferrite decreased the flow stress observed during the deformation. It is known that ferrite nucleation can occur continuously during deformation in the intercritical region and even at temperatures slightly above Ar_3 [10]. In these cases, the deformation locally increases the free energy and consequently the number of potential sites for nucleation of the new phase, causing an acceleration of phase transformation process [11,12,13].

4 CONCLUSIONS

Double compression tests associated with metallographic analyzes allowed the evaluation of the softening evolution at different temperatures and times between passes. Static recrystallization governs the microstructure restoration in the austenitic field. Recrystallization is easily present in this steel at temperatures between 900°C and 1000°C. The softening fraction curves in this region showed high recrystallization kinetics, with short incubation period. Also immediately after deformation new nucleated equiaxed grains were observed. At 850°C, recrystallized grains were observed only after long holding times after deformation. At temperatures between 700°C and 800°C, the microstructure showed evidences of advancement of the ferrite grains free of deformation consuming the grains of higher free energy, indicating the presence of the strain induced boundary motion mechanism (SIBM). In the ferritic field, recovery and SIBM mechanisms governed the microstructural restoration, with low restoration kinetics.

In general, the flow curves of the material exhibit dynamic recovery behavior, with stabilization of the flow stress in a steady state. An exception was found for compression at the intercritical temperature of 900°C, where a dynamic softening with decrease of flow stress after a peak of stress. This effect was associated with the presence of dynamic strain induced transformation (DSIT).

According to this investigation, in order to obtain an equiaxed microstructure after warm deformation of the ULC-Ti steel analyzed, it is recommended to deform at high ferritic temperatures (800-850°C), since the steel in these temperatures showed the higher softening rate in the ferritic region analyzed, and can even undergo recrystallization with enough holding time.

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