1. Introduction

In steels alloyed with Mo, Mn and Cr or microalloyed with Nb, Ti and V the high temperature strength related to dynamic strain aging has been attributed to the phenomenon known as interaction solid-solution hardening, ISSH, due to the dynamic interaction of interstitials-substitutional solute dipoles and dislocations. The ISSH effect displaces DSA manifestations to higher temperatures than those displayed by mild carbon steels, while in this steels DSA happening in a temperature range of 100 to 400°C and strain rates of $10^{-4}$ to $10^{-1}$ s$^{-1}$. This steel showed a ferrite and pearlite microstructure. DSA manifestations are less intense than those observed for low carbon steels and they take place at higher temperatures. The secondary precipitation behavior of the steel was also investigated. The hardness of samples heat treated at 100 to 600°C displayed a maximum at 400°C. Samples treated at this temperature and tensile tested at 600°C didn’t showed a higher yield strength than the untreated specimens, indicating that secondary precipitation does not contribute to its high temperature strength. Results obtained here indicated that DSA in the structural steel might be an important mechanism responsible for its fire resistance. The empirical activation energies related to the appearance of serrations on the stress–strain curves and to the maxima on the variation of tensile strength with temperature or disappearance of serrations suggested that the high temperature strengthening associated with DSA in this steel is the dynamic interaction of interstitial-substitutional solute dipoles and dislocations.

KEY WORDS: dynamic strain aging; Portevin–LeChatelier effect; fire resistant steel; structural steels.

2. Experimental

The influence of dynamic strain aging (DSA) on the high temperature strength of a structural steel microalloyed with Mo and Nb was investigated by means of tensile tests performed at temperatures ranging from 25 to 600°C and strain rates of $10^{-4}$ to $10^{-1}$ s$^{-1}$. This steel showed a ferrite and pearlite microstructure. DSA manifestations are less intense than those observed for low carbon steels and they take place at higher temperatures. The secondary precipitation behavior of the steel was also investigated. The hardness of samples heat treated at 100 to 600°C displayed a maximum at 400°C. Samples treated at this temperature and tensile tested at 600°C didn’t showed a higher yield strength than the untreated specimens, indicating that secondary precipitation does not contribute to its high temperature strength. Results obtained here indicated that DSA in the structural steel might be an important mechanism responsible for its fire resistance. The empirical activation energies related to the appearance of serrations on the stress–strain curves and to the maxima on the variation of tensile strength with temperature or disappearance of serrations suggested that the high temperature strengthening associated with DSA in this steel is the dynamic interaction of interstitial-substitutional solute dipoles and dislocations.

Table 1. Chemical composition of steel (mass%).

| C  | Mn | Mo | Cr | Si | P  | S  | Nb | Al | N  |
|----|----|----|----|----|----|----|----|----|----|
| 0.092 | 0.80 | 0.19 | 0.10 | 1.21 | 0.038 | 0.0045 | 0.020 | 0.018 | 0.0052 |
and with a gauge length of 27.0 mm were machined from the plate with their axes along the rolling direction.

The tensile tests were performed in a servo hydraulic MTS testing machine in a temperature range from 25 to 600°C, at strain rates of $10^{-4}$, $10^{-3}$, $3.5 \times 10^{-2}$, $10^{-2}$ and $10^{-1}$ s$^{-1}$. Samples were held for 5 min at temperature before testing. The heating system used kept temperature variations during testing stable enough to not exceed ±2°C throughout and a negligible temperature gradient along the length of the sample. The values of yield strength, $\sigma_y$, ultimate tensile strength, $\sigma_t$, and total elongation, $\varepsilon_t$, were determined as the average of at least three tensile tests performed at identical conditions. The relative error in the values of stress and elongation was lower than 3%.

Vickers hardness of specimens as taken at transverse section of the plates and heat treated from 100 to 400°C for 30 min. A load of 50 kgf was used.

3. Results and Discussion

The examination of the microstructure of the steel showed that it consisted of ferrite and pearlite. The volume fractions and ferrite grain sizes are presented in Table 2.

Stress–strain curves for the structural steel tested at various temperatures and at a strain rate of $10^{-4}$ and $10^{-1}$ s$^{-1}$ are shown in Fig. 1. The Portevin–LeChatelier effect (PLC) was present at a temperature interval of 80 to 275°C and 175 to 375°C for the two strain rate, respectively. The same serrations was observed for tests performed at the three other strain rate. The temperature at the onset and offset of the serrations was observed to increase for increasing strain rate, however the degree of serration decrease for increasing strain rate. Figure 1(a) shows also that the work hardening rate increases with temperature between 150 to 350°C.

For the five strain rate employed here the value of the yield stress, $\sigma_y$, at 600°C was higher than 67% of its value specified at room temperature for this class of commercial steel, 325 MPa, as required from specifications for a structural steel with fire resistant properties. The changes in yield stress, $\sigma_y$, and tensile strength, $\sigma_t$, with temperature are shown in Fig. 2 for four of the five strain rates considered. The curves of the steel show maxima in $\sigma_t$ occurring at increasing temperatures for increasing strain rates, Table 3. The amplitude of these maxima decreases as the strain rate is increased. The $\sigma_t$ curves displays plateau in the temperature interval at which the maxima in $\sigma_t$ were observed for the five strain rates considered.

The changes in the total elongation, $\varepsilon_t$, with test temperature for four of the five strain rates investigated are shown in Fig. 3.

For the five strain rate, $\varepsilon_t$ decreased with increase in temperature, showing a minimum at a temperature range of 110 to 270°C and then it increased with raising temperatures. The temperature where the elongation showed the minimum and maximum increased with increasing strain rate, Table 3.

The presence of a PLC effect, of maxima and minima in the tensile strength and elongation curves versus temperature, respectively, of a plateau in the yield stress curves versus temperature as well as of a presence of increments in the work hardening rates with increasing temperature are

| Table 2. Volume fractions (fv) of ferrite and pearlite and ferrite grain size (GS). |
|----------------------|----------------------|----------------------|
| fv ferrite (%)       | fv pearlite (%)      | GS (μm)             |
| 81 ± 2               | 19.0 ± 0.2           | 6.7 ± 0.3            |

| Table 3. Variation of minima elongation and maxima tensile strength temperature with strain rate. |
|-----------------------------------------------|-----------------|-----------------|
| Strain Rate (s$^{-1}$) | Temperature Minima Elongation (°C) | Temperature Maxima Tensile Strength (°C) |
|------------------------|-----------------------------|---------------------------------------|
| $10^{-1}$             | 260                         | 375                                   |
| $10^{-2}$             | 230                         | 350                                   |
| $10^{-3}$             | 200                         | 300                                   |
| $10^{-4}$             | 110                         | 275                                   |
all indications that the behavior of this structural steel at the temperatures and strain rates investigated, regarding the observations of DSA, is similar to those found in low carbon steels.\(^1\)–\(^3\) However, these observations take place at higher temperatures and with lower intensity than those occurring in low carbon steels.\(^6\)–\(^7\) For a given strain rate the strength of the maximum in \(\sigma\), can be defined as the difference between this maximum \((\sigma_{\text{max}})\) and the minimum value of \(\sigma\), \((\sigma_{\text{min}})\) between room temperature and the temperature of \(\sigma_{\text{max}}\). For a strain rate of \(10^{-2}\ \text{s}^{-1}\), the temperature at which the maximum in \(\sigma\) occurs in the structural steel microalloying with Mo and Nb is about 350°C, and the value of \(\sigma_{\text{max}} - \sigma_{\text{min}}\) is approximately 80 MPa. In low carbon steels containing 10 ppm and 20 ppm N in solid solution, for the same strain rate, \(\sigma_{\text{max}}\) occurs at about 280°C and \(\sigma_{\text{max}} - \sigma_{\text{min}}\) are 120 and 180 MPa, respectively.\(^8\) The amount of soluble nitrogen in the steel, containing 0.0052% of total nitrogen is probably not lower than 10 ppm, considering the processing conditions. This lesser pronounced manifestation of DSA in steels microalloyed with niobium and or vanadium was already reported.\(^3\)–\(^9\)

According to the literature,\(^10\)–\(^11\) the minimum absolute temperature, \(T\), associated with the occurrence of the PLC effect in low carbon steels is related to the strain rate, \(\dot{\varepsilon}\), by the equation:

\[
\dot{\varepsilon} = \frac{B \rho_b l}{T} \exp\left(\frac{-Q}{RT}\right) \text{..........................(1)}
\]

where \(B\) is a constant, \(\rho_b\) is the density of mobile dislocation, \(l\) is the average distance traveled by dislocations between penetrable obstacles, \(b\) is the Burgers vector, \(R\) is the universal gas constant and \(Q\) is the activation energy of the process. Assuming that the term \(B \rho_b l\) remains constant when \(T\) and \(\dot{\varepsilon}\) vary, the value of the apparent activation energy, \(Q\), can be determined from a \(\ln(\dot{\varepsilon} \cdot T)\) vs. \(T^{-1}\) plot. The values usually found for this apparent activation energy are of the order of the activation energies for diffusion of the interstitial atoms responsible for dislocation locking, N and C.\(^3\)–\(^10\) The absolute temperature at which the PLC effect ends, which coincides with the temperature corresponding to the maximum in \(\sigma\),\(^10\) is also related to the strain rate by an expression similar to Eq. (1). The values of \(Q\) found in this case \((Q')\) have been associated with the sum of the activation energy for diffusion of the interstitial responsible for dislocation locking and the interstitial-dislocation binding energy. This association is based on the hypothesis that, at this stage, dislocations move dragging their solute atmospheres.\(^10\)

The values reported for the activation energy for the start of serrated flow in low carbon steels range from 79.5 to 84.1 kJ/mol\(^10\)–\(^11\) and are close to the activation energies for diffusion of N and C in ferrite, respectively, 76.1 kJ/mol and 84.1 kJ/mol.\(^12\) Typical values of the activation energy associated with the disappearance of serrated flow are 127.6 kJ/mol\(^10\) and 134 kJ/mol,\(^11\) while those related to the maximum in tensile strength are 127.6 to 156.1 kJ/mol.\(^3\)–\(^10\) The lower values for the activation energies related to the end of the PLC effect and to the presence of the maximum in \(\sigma\) have been attributed to dislocation locking by nitrogen atoms, while the higher values have been associated to dislocation locking due to carbon atoms. Intermediate values found for these energies reflect the combined effect of C and N. Thus, considering the minimum values of these activation energies, the binding energy of N atoms to dislocations in ferrite can be estimated, while the carbon-dislocation binding energy is estimated using the maximum value of these activation energies. In the case of N, the binding energy should be 127.6 kJ/mol – 79.5 kJ/mol, equal to 48.1 kJ/mol (0.50 eV). For C atoms, the value is 156.1 kJ/mol – 84.1 kJ/mol, equal to 72.0 kJ/mol (0.75 eV). These values are in good agreement with the classical solute-dislocation binding energies in ferrite for N atoms, 0.47 eV,\(^13\) and for C atoms, 0.75 eV.\(^14\)

In this study, the serrated flow, the maximum in \(\sigma\), and the minimum in \(\varepsilon\), indicate that DSA was taking place. Thus, apparent activation energies can be estimated for these processes, as described before. Figure 4 shows \(\ln(\dot{\varepsilon} \cdot T)\) vs. \(T^{-1}\) plots for the onset of the PLC effect and the maximum in \(\sigma\), and the values obtained from the slopes of these plots, which are, respectively, 97 ± 4 kJ/mol (\(Q\)), and 203 ± 1 kJ/mol (\(Q'\)). The first of these values is considerably higher than the activation energies associated for diffusion of N and C atoms in solid solution in ferrite, 79.5 to 84.1 kJ/mol. The value for the maximum in \(\sigma\) is higher than the activation energies found in low carbon steels for the end of the PLC effect and the occurrence of the maximum in \(\sigma\) (127.6 to 156.1 kJ/mol). These results suggest then that the dynamic strain aging effects found in this structural steel should be controlled by a mechanism in
some aspects different from that operating in mild carbon steels.

As already mentioned, in steels containing Mn, Mo, Cr, Ti and Nb, interstitial N and C atom are in close association with substitutional forming interstitial-substitutional dipoles that interact strongly with dislocations over a large temperature range, the effect of interaction solid solution hardening, ISSH.\(^2\) If the interaction energy between interstitials and dislocations is higher than the interaction energy between interstitials and substitutionals the high temperature strengthening will be due to a displacement of the DSA phenomena similar to those displayed for low carbon steels. However, DSA takes place at higher temperatures and is less intense than that observed in low carbon steels.

The difference in the values of activation energies for the onset of the PLC effect and for the occurrence of the maxima in \(\sigma_t\), 203 kJ/mol—97 kJ/mol, is 106 kJ/mol (1.1 eV), higher than the values of the N-dislocation and C-dislocation binding energies, 0.47 and 0.75 eV, respectively,\(^1,2\) indicating that DSA in this steel is related to locking of dislocations by atmospheres of N and C atoms whose mobility is reduced due the interstitial-substitutional interaction as discussed for the effect of Mn on the strain aging due to N and C in a Nb microalloyed steel.\(^3\)

Figure 5 shows the changes in hardness with temperature for the untreated and treated samples. The maximum in hardness observed at 400°C is probably associated with secondary precipitation of Mo carbides. According to Lenk et al.\(^6\) the precipitation of Mo\(_2\)C is a significant contribution to high temperature resistance in steels.

The analysis of Table 4 indicates that secondary precipitation has a small effect on the yield stress at 25°C. On the other hand, this effect has no influence on the yield stress at 600°C. At such high temperatures the carbides precipitated at 400°C are not effective in keeping resistance because of Ostwald Ripening.

### 4. Conclusions

1. The structural steel considered in this work presents DSA phenomena similar to those displayed for low carbon steels. However, DSA takes place at higher temperatures and is less intense than that observed in low carbon steels.

2. The values of the apparent activation energy related to the onset of the PLC effect, 97 kJ/mol, and to the maximum in ultimate tensile strength versus temperature curves, 203 kJ/mol, indicate that, in this structural steel, the mobility of dislocations atmospheres, which controls DSA, is affected by substitutional solutes such as Mn, Cr, Mo and Nb.

3. The ISSH effect present in the steel studied here is a contribution to the fire resistance properties of the steel through the DSA phenomenon and in the same way it contributes to creep resistance of heat resistant steels.

4. Secondary precipitation in the structural steel studied here does not contribute to its fire resistance.

5. The structural steel containing Mo and Nb studied here have their yield strength at 600°C as 67% of the specified value at room temperature, thus, it’s a fire resistant steel.

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### Table 4. Yield stress and tensile strength of the untreated and treated at 400°C for 30 min structural steel samples, strain rate \(10^{-3}\) s\(^{-1}\).

| Test temperature (°C) | \(\sigma_t\) (MPa) | \(\sigma_t\) (MPa) | \(\sigma_{(total)}\) (MPa) | \(\sigma_{(total)}\) (MPa) |
|----------------------|------------------|------------------|------------------------|------------------------|
| 25                   | 475 ± 2          | 603 ± 4          | 492 ± 10                | 609 ± 9                |
| 600                  | 288 ± 2          | 294 ± 4          | 282 ± 16                | 287 ± 11               |

Fig. 5. Variation in hardness with temperature of samples submitted to a 30 min heat treatment.