Measurement of Dynamic Strain Aging in Pearlitic Steels by Tensile Test

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(Received on August 5, 2002; accepted in final form on October 30, 2002)

The mechanical behavior of a pearlitic steel was studied by means of tensile tests performed in the temperature interval from 298 to 773 K, at strain rates from 10^{-4} to 10^{-2} s^{-1} , aiming to analyze the role of cementite decomposition in the dynamic strain aging (DSA) phenomena occurring in these steels. Typical features of DSA were observed: serrated flow (the Portevin-LeChatelier–PLC effect), the presence of a maximum and a minimum in ultimate tensile strength and reduction of area *versus* temperature curves, respectively, and a less evident maximum in the yield strength *versus* temperature curve. Apparent activation energies were calculated based on the onset of the PLC effect, the maximum in ultimate tensile strength and the minimum in reduction of area. Results suggest that changes in mechanical properties associated with DSA in pearlitic steels are related to cementite decomposition. Differences on the phenomenological aspects regarding DSA in pearlitic and in low carbon steels are discussed, based on the kinetics of the process.

KEY WORDS: dynamic strain aging; Portevin-LeChatelier effect; pearlitic steels; cementite decomposition.

1. Introduction

Static strain aging in pearlitic steels has been the subject of various studies^{1–7)} due to its influence on the wire drawing process and on the resulting properties of the drawn wire. It seems well established that pearlitic steels present an additional strain aging mechanism besides the classic one, which operates in low carbon steels and is related to dislocation locking by N and C atoms dissolved in ferrite. This non-classic mechanism, known as the second stage of static strain aging, takes place at higher temperatures and causes more drastic changes in strength and ductility. The apparent activation energy for the second stage of static strain aging indicates that the phenomenon is associated with dislocation locking in ferrite–cementite interfaces by C atoms released during the decomposition of cementite, which follows plastic deformation in pearlitic steels.^{1,2,5)}

The strain-induced decomposition of cementite was first detected by Belous and Cherepin⁸⁾ in 1961. The amount of decomposed cementite in deformed Fe–C alloys was estimated by Gridnev *et al.*⁹⁾ using Mossbauer spectroscopy. Several studies, in which various experimental techniques were employed,^{10–14)} gave further evidence of cementite decomposition during plastic deformation of pearlite. More recently, Gavriljuk^{15,16)} gave important contributions for the understanding of the atomic mechanism responsible for this process.

Dynamic strain aging of pearlitic steels has been less explored than static strain aging, although important technological aspects associated with this phenomenon have been

reported in the literature, such as the stabilization of pre-stressed wire¹⁷⁾ and the decrease in fatigue life of this steel in strain-controlled fatigue.¹⁸⁾ Sudo and Yutori¹⁹⁾ studied the changes in mechanical properties with temperature, T , and strain rate, $\dot{\epsilon}$, of two pearlitic steels, one containing 120 ppm in weight of nitrogen in solid solution and the other containing none. In the investigated temperature interval, 293 to 573 K, typical manifestations of DSA were observed: serrated flow, maximum in tensile strength, σ_t , minimum in reduction of area, Z , and increased work hardening rate, but for the steel having no soluble nitrogen, the PLC effect was not detected. For the steel containing 120 ppm of soluble N, the observed PLC effect was attributed to dislocation locking by N atoms dissolved in ferrite, since the activation energy determined for the appearance of serrations on the stress–strain curves coincided with the activation energy for the diffusion of N in α -Fe. According to the authors, the maximum in σ_t and the minimum in Z , occurring in both steels, could not be attributed to dislocation-interstitial interaction, because the amount of dissolved carbon was very low (around 1 ppm in weight). They suggested that these effects were associated with the solubilization of C atoms during plastic straining, due to the decomposition of cementite.

Later, Yamada *et al.*²⁰⁾ showed the results of Sudo and Yutori¹⁹⁾ for the steel containing 120 ppm of soluble N in a plot of $\ln(\dot{\epsilon} \cdot T)$ *versus* T^{-1} and found activation energies of 75.2 and 117 kJ/mol for, respectively, the appearance and disappearance of serrated flow. Yamada *et al.*²⁰⁾ associated the PLC effect with the presence of soluble N and empha-

sized that, for the values of $\dot{\epsilon}$ and T in between the end of the PLC effect and the maximum in σ_p , the work hardening rate was high and the ductility was very low, irrespectively of the content of soluble nitrogen in the steel. However, the mechanism responsible for these effects was not discussed in their work.

The PLC effect and a maximum in the 10% flow stress versus temperature was also observed by Tsuzaki *et al.*,¹⁸⁾ in a pearlitic steel tested between 293 and 623 K, at strain rates of 3.3×10^{-4} and $3.3 \times 10^{-3} \text{ s}^{-1}$. The activation energies for these phenomena were not determined in their work and the observed DSA effects were associated with the interaction of carbon atoms in solid solution with dislocations in ferrite.

The present work was undertaken to provide a more complete description of the phenomenological and kinetic aspects of dynamic strain aging in pearlitic steel. The aim was to verify if cementite decomposition plays an important role in DSA phenomena taking place in pearlitic steels, as it does in static strain aging, as well as to compare the behavior of this steel with that generally observed in low carbon steels.

2. Experimental

The commercial grade pearlitic steel studied was received as a lead patented, 5.5 mm in diameter wire rod. Its chemical composition (in weight percent) was: 0.80% C, 0.76% Mn, 0.0015% P, 0.005% S, 0.25% Si, 0.019% Al, 0.0038% N. Tensile-test specimens 2.5 mm in diameter and with a gauge length of 25.0 mm were machined from wire rod samples spheroidized at 953 K for 24 h. After machining, the specimens were heated at 1153 K for 20 min and lead patented in laboratory conditions at 793 K for 10 min. According to Fisher,²¹⁾ the mean true interlamellar spacing, S_p , of pearlite formed at this temperature should be equal to 77 nm. Atomic force microscopy (AFM) images taken from the transverse section of patented test specimens confirmed that the laboratory heat treatment produced a fully pearlitic microstructure. Using a procedure described elsewhere,²²⁾ measurements of pearlite interlamellar spacing by AFM led to an average value of $S_p = 76 \pm 9 \text{ nm}$, in excellent agreement with the predicted value.

The tensile tests were performed in a servo-hydraulic MTS testing machine in a temperature range from 298 to 773 K, at strain rates of 10^{-4} , 10^{-3} and 10^{-2} s^{-1} . For the determination of the conditions of appearance and disappearance of serrated flow, an Instron testing machine, equipped with a heating and cooling chamber operating between 213 and 523 K was employed, since it is a "hard" machine, and thus more appropriate for the study of the PLC effect. The tests were performed between 348 and 478 K, at strain rates of 10^{-4} , 5×10^{-4} , 10^{-3} and $2 \times 10^{-3} \text{ s}^{-1}$. Temperature variations during testing in both machines did not exceed $\pm 1 \text{ K}$. The values of yield strength, σ_y , ultimate tensile strength, σ_t , and reduction in area at necking, Z , were determined as the average of at least three tensile tests performed at identical conditions. The σ_y value was taken as the stress associated with the discontinuous yield point when it was observed and as the 0.2% offset yield stress in the conditions for which yielding was continuous.

3. Results and Discussion

Stress-strain curves for the pearlitic steel tested in the servo-hydraulic machine at various temperatures and at a strain rate of 10^{-2} s^{-1} are shown in Fig. 1. The PLC effect was not observed under any testing condition, for the tests performed in this machine. It is interesting to note that Weidig *et al.*²³⁾ observed the PLC effect when testing, in the same machine, a low carbon steel containing about 20 ppm in wt. of N in solid solution. The amount of soluble N in pearlitic steels can be estimated by using reported data regarding the precipitation of AlN in patented steels.^{24,25)} Considering the total amount of Al and N in the steel studied, one finds that about 23 ppm in wt. of N is dissolved in ferrite. According to the literature,²⁶⁾ the PLC effect is displayed by low carbon steels containing amounts of soluble N as low as 10 ppm in wt. Although serrated flow was not observed on the stress-strain curves obtained with the MTS machine, this does not mean that the PLC effect is absent in pearlitic steels. For the estimated amount of soluble N, 23 ppm, the serrations amplitude was too weak to be observed with this relatively soft testing machine, as it will be shown later. Nevertheless, analysis of the curves depicted in Fig. 1 shows that the work hardening rate increases with temperature between 373 and 623 K. Compared with the behavior of low carbon steels in the temperature range of DSA,²⁶⁾ the increase in work hardening rate observed in the pearlitic steel is less evident.

The changes in yield stress, σ_y , and tensile strength, σ_t ,

Fig. 1. Nominal stress-strain curves for specimens tested at a strain rate of 10^{-2} s^{-1} at various temperatures.

Fig. 2. Changes in yield stress and tensile strength with temperature for the pearlitic steel investigated and for a low carbon steel, LCS.²³⁾

with temperature are shown in **Fig. 2** for the three strain rates considered. This figure also presents the change in σ_t with temperature for a low carbon steel²³⁾ containing a similar amount of N in solid solution and tested at a strain rate of 10^{-2} s^{-1} . The curves of the pearlitic steel shown maxima in σ_t occurring at increasing temperatures for increasing strain rates. The amplitude of these maxima decreases as the strain rate is increased. The maxima in σ_y are less pronounced than those in σ_t , but are influenced in the same way by the strain rate. For a given strain rate, the strength of the maximum in σ_t can be defined as the difference between this maximum ($\sigma_{t\max}$) and the minimum value of σ_t ($\sigma_{t\min}$) between room temperature and the temperature of $\sigma_{t\max}$. For a strain rate of 10^{-2} s^{-1} , the temperature at which the maximum in σ_t occurs is about 593 K, and the value of $\sigma_{t\max} - \sigma_{t\min}$ is approximately 100 MPa. In the low carbon steel, for the same strain rate, $\sigma_{t\max}$ occurs at a lower temperature, 548 K, and $\sigma_{t\max} - \sigma_{t\min}$ is equal to about 180 MPa.

The changes in the reduction of area, Z , with test temperatures for the three strain rates investigated are shown in **Fig. 3**. This figure also presents the results of the same low carbon steel shown in Fig. 2 at a strain rate of 10^{-2} s^{-1} . For the pearlitic steel, there is a small decrease in Z as the temperature increases between 373 and 548 K. For higher test temperatures, Z increases abruptly, attaining values in excess of 90%. The pearlitic steel displays a minimum in Z at 548 K, while in the low carbon steel this minimum occurs at about 473 K.

The mechanical behavior of the pearlitic steel at the temperatures and strain rates investigated indicates that the manifestations of DSA are similar to those found in low carbon steels. However, these manifestations take place at higher temperatures and with lower intensity than those occurring in low carbon steels.

Details of the stress-strain curves obtained in the Instron machine for temperatures between 348 and 473 K and a strain rate of 10^{-3} s^{-1} are shown in **Fig. 4**. Serrations of small intensity can be observed, for higher strains, starting between 348 and 438 K and disappearing almost completely at 443 K. At 448 K, one can observe the appearance of serrations of considerably larger amplitude, which are present since the start of plastic flow. A similar behavior was observed in the stress-strain curves obtained for the other strain rates, although for the strain rate of $2 \times 10^{-3} \text{ s}^{-1}$, the small amplitude and lower temperature serrations were not detected. The higher the strain rate, the higher the temperature at which the larger amplitude serrations appear and the smaller the serrations amplitude, characteristics that are also typical of the PLC effect in low carbon steels. In these steels, DSA phenomena has been associated with dislocation locking by N and C atoms dissolved in ferrite, the latter being less effective due to its lower solubility.²⁶⁻²⁸⁾

The serrated flow observed in the pearlitic steel at lower temperatures, between 398 and 438 K, is certainly associated with the presence of N and C atoms in solid solution in ferrite, since the temperature at which the serrations appear is close to that of the manifestation of the PLC effect in low carbon steels for strain rates of the order of 10^{-3} s^{-1} and the same amount of soluble nitrogen.²³⁾ However, the larger amplitude serrations, because of the higher temperature at which they start, cannot be associated with the same mech-

Fig. 3. Changes in reduction of area with temperature for the pearlitic steel investigated and for a low carbon steel, LCS.²³⁾

Fig. 4. Details of the nominal stress-strain curves at a strain rate of 10^{-3} s^{-1} at various temperatures.

anism.

According to the literature,²⁸⁻³⁰⁾ the minimum absolute temperature, T , associated with the occurrence of the PLC effect in low carbon steels is related to the strain rate, $\dot{\epsilon}$, by the equation:

$$\dot{\epsilon} = \frac{B\rho_m b l}{T} \exp\left(-\frac{Q}{RT}\right) \dots\dots\dots (1)$$

where B is a constant, ρ_m 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. Supposing that the term $B\rho_m b l$ remains constant when T and $\dot{\epsilon}$ vary, the value of the apparent activation energy, Q , can be determined from a $\ln(\dot{\epsilon} \cdot T)$ 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 solute atoms responsible for dislocation locking, N and C.^{26,28,29)} The absolute temperature at which the PLC effect ends, which coincides with the temperature corresponding to the maximum in σ_t ,^{28,30)} is also related to the strain rate by an expression similar to Eq. (1). The values of Q found in this case have been associated with the sum of the activation energy for diffusion of the solute responsible for dislocation locking and the solute-dislocation binding energy. This association is based on the hypothesis that, at this stage, the dislocations move dragging their solute atmos-

pheres.²⁸⁾

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^{26,28)} and are very close to the activation energies for diffusion of N and C in ferrite, respectively 76 and 84.1 kJ/mol. Typical values of the activation energy associated with the disappearance of serrated flow are 127.6²⁸⁾ and 134 kJ/mol,²⁶⁾ while those related to the maximum in tensile strength are 127.6 to 156.1 kJ/mol.^{28,23)} The lower values for the activation energies related to the end of the PLC effect and to the presence of the maximum in σ_t 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 127.6–79.5 kJ/mol, equal to 48.1 kJ/mol (0.50 eV). For C atoms, the value is 156.1–84.1 kJ/mol, or 72.0 kJ/mol (0.75 eV). These values are in good agreement with the classical solute–dislocation binding energies in ferrite, reported by Petarra and Beshers³¹⁾ for N atoms, 0.47 eV, and by Cochardt *et al.*³²⁾ for C atoms, 0.75 eV.

In this study, the serrated flow, the maximum in σ_t and the minimum in Z indicate that DSA is taking place. Thus, apparent activation energies can be estimated for these processes, as described before. **Figure 5** shows $\ln(\dot{\epsilon} \cdot T)$ vs. T^{-1} plots for the onset of the more intense PLC effect, the maximum in σ_t and the minimum in Z, and the values obtained from the slopes of these plots, which are, respectively, 119, 187 and 192 kJ/mol. The first of these values is considerably higher than the activation energies associated with dislocation locking by N and C atoms in solid solution in ferrite, 79.5 to 84.1 kJ/mol. The other two values are very close to each other and are higher than the activation energies found in low carbon steels for the end of the PLC effect and the occurrence of the maximum in σ_t (127.6 to 151.6 kJ/mol). These results suggest that the dynamic strain aging effects found in pearlitic steels should be controlled by a mechanism different from that operating in low carbon steels.

The apparent activation energy for the onset of the higher intensity PLC effect determined in the present work, 119 kJ/mol, is in the range of the activation energy for cementite decomposition, 114 to 125 kJ/mol.^{1,5)} The apparent activation energies for the maximum in σ_t and the minimum in Z, 187 and 192 kJ/mol, are very close to the sum of the activation energy for cementite decomposition and the C-dislocation binding energy, 72 kJ/mol.³²⁾ This sum leads to values between 186 and 197 kJ/mol. These results indicate that the DSA manifestations observed in the pearlitic steel should be controlled by dislocation locking due to C atoms released by the decomposition of cementite.

It is worth noting that Sudo and Yutori¹⁹⁾ determined the following equation relating the absolute temperature, T, to the strain rate, $\dot{\epsilon}$, for the minimum in reduction of area:

Fig. 5. $\ln(\dot{\epsilon} \cdot T)$ versus T^{-1} at the conditions concerning the onset of the PLC effect, the maximum in tensile strength, σ_t , and the minimum in reduction of area, Z.

$$\dot{\epsilon}T = A \exp\left(\frac{-181.0}{RT}\right) \dots\dots\dots(2)$$

were A is a constant. Comparison of Eqs. (1) and (2) indicates that 181 kJ/mol is the value of the apparent activation energy associated with the minimum in reduction of area found by these authors. The apparent activation energies found in the present work for the minimum in Z and the maximum in σ_t , 192 and 187 kJ/mol, respectively, are thus in agreement with this value of activation energy and with that obtained from their $\ln(\dot{\epsilon} \cdot T)$ versus T^{-1} plot for the maximum in σ_t , 176 kJ/mol.

As previously discussed, Sudo and Yutori¹⁹⁾ also observed, for a pearlitic steel without soluble nitrogen and carbon, a maximum in σ_t and a minimum in Z. The main aspect regarding these results is that DSA manifestations occur in pearlitic steels that initially have no interstitial C and N atoms. According to this observation, it should be considered that the PLC effect due to N in the pearlitic steel investigated, containing about 23 ppm N in solid solution, was weaker than that in low carbon steels and the other DSA manifestations associated to soluble nitrogen were too weak to be detected.

The results obtained in this work, based on apparent activation energy values, give support to the following description of the mechanisms of DSA in pearlitic steels:

- Upon straining, dislocations are stored in cell walls along ferrite–cementite interfaces.²⁾ Since the binding energy of C atoms to dislocations is higher than in Fe_3C , decomposition of cementite takes place at this stage,¹⁵⁾ releasing C atoms to dislocations in the cell walls adjoining Fe_3C lamellae, where a layer with high concentration of C atoms is formed;
- Carbon atoms migrate from these high concentration layers to neighboring dislocations in the ferrite lamellae, and lock them. Plastic flow requires a stress high enough to produce free dislocations at the ferrite side of the cell walls. The motion of these dislocations in ferrite occurs, then, at lower stress levels. The dislocations at the cell walls–ferrite interfaces are locked again by solute atmospheres and higher strain levels are required to produce free dislocations for further plastic flow. This process results in discontinuous plastic flow—the PLC effect, and

its apparent activation energy should thus be of the same order as that for cementite decomposition in pearlitic steels.

- Increasing the temperature, the mobility of C atoms increases so that at a sufficiently high temperature the dislocations move dragging their solute atmospheres. The PLC effect ends and the tensile strength displays a maximum. The apparent activation energy for this process should thus correspond to the sum of the activation energy for cementite decomposition and the C-dislocation binding energy.

This description does not exclude dislocation locking by N and C atoms dissolved in ferrite, which should occur at lower temperatures, as observed in low carbon steels. The fact that the amplitude of the lower temperature serrations is smaller in the pearlitic steel is probably associated with the same mechanisms that renders an yield point not as sharp as in low carbon steels: the high stress concentration present at ferrite–cementite interfaces, which favors the nucleation of a great number of free dislocations at lower applied stresses. This mechanism would also explain the smaller amplitude of the maximum in the σ_t versus temperature curve in the pearlitic steel.

4. Conclusions

(1) Pearlitic steels exhibit DSA phenomena that are similar to those displayed by low carbon steels. The changes in mechanical properties due to DSA in these steels take place at higher temperatures and are less intense than those observed in low carbon steels.

(2) Differently from low carbon steels, the DSA effects associated with dislocation locking by N atoms dissolved in ferrite, except for low intensity serrations in stress-strain curves, were not detected in the pearlitic steel investigated, containing about 23 ppm of soluble nitrogen.

(3) The values of the apparent activation energy related to the onset of the PLC effect, 119 kJ/mol, and to the maximum in ultimate tensile strength and the minimum in reduction in area versus temperature curves, 187 and 192 kJ/mol, respectively, suggest that DSA in pearlitic steels is controlled by cementite decomposition, as observed for the second stage of static strain aging in these steels.

Acknowledgments

The authors wish to acknowledge the financial support of the Fundacao de Amparo a Pesquisa do Estado de Minas Gerais–FAPEMIG, and of the Conselho Nacional de

Desenvolvimento Científico e Tecnológico–CNPq.

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