Original Article

High temperature work hardening stages, dynamic strain aging and related dislocation structure in tensile deformed AISI 301 stainless steel

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1. Introduction

1.1. Work hardening stages

Polycrystalline metallic materials that undergo plastic deformation due to an applied load usually exhibit a convex-shaped stress–strain (σ–ε) curve, also referred as a "parabolic", which could be adjusted to a well-known Ludvik–Hollomon power law equation [1]. A linear interpretation of this power law can be obtained by plotting \(\frac{d\sigma}{d\varepsilon}\) versus \(\varepsilon\) in double logarithmic scales:

\[
\log\left(\frac{d\sigma}{d\varepsilon}\right) = \log kn + (n - 1) \log \varepsilon
\]

The exponent \(n\) is normally \(<1\), in many cases close to 0.5, which explain the term “parabolic” hardening for a cold worked polycrystalline plastic deformation [1].
Earlier works of Crussard and Jaoul [2–4], using plots of Eq. (1) for aluminum, revealed consistently two straight lines indicating that the work hardening of metals could be associated with two stages. This was also found for pure iron [5]. The second stage would always have a lower value of \( n \) corresponding to a marked decrease in the work hardening rate. The proposed mechanisms for a second stage formation was dynamic recovery based on dislocation annihilation as well as the development of subgrains through polygonization in the case of high stacking fault metals [6]. Eq. (1) was also earlier applied for AISI 302 and 316 stainless steels [7] at room temperature and below. Two stages of work hardening were found, but the second, non-linear, was associated with martensitic transformation.

Recently, Christopher and Choudhary [8–11] investigated work hardening stages in AISI 316L stainless steel using the \( \frac{d\sigma}{d\varepsilon} \) versus \( \sigma \) experimental curves for different temperature regimes. The authors found three stages based on the shape of their curves. In principle, these first two stages could be similar to those based on Eq (1). As for the third stage, Christopher and Choudhary [8], detected it as an inverted “parabolic” hardening at higher stresses. The critical parameters assessed from the \( \frac{d\sigma}{d\varepsilon} \) versus \( \sigma \) plots at higher temperatures (600–750 °C) exhibited dominance of dynamic recovery. However, Christopher and Choudhary [8–11] did not directly observe actual dislocation structures that were based on densities (\( \rho \) ) evaluated by the classic relationship [6]:

\[
\sigma = \sigma_0 + k\rho^{1/2}
\]

where \( \sigma \) and \( \sigma_0 \) are stress values and \( k \) is a constant.

1.2. Dynamic strain aging

The evolution of high temperature dislocation structures associated with work hardening stages in austenitic stainless steels is still a matter of discussion. These steels present a low stacking fault energy (SFE), which limits the movement of dislocations toward formation of consolidated cell structures. Moreover, the interaction of solute atoms with dislocations causes dynamic strain aging (DSA) [8], which might also affect the development of dislocation structures. Indeed, DSA has, since past decades, been reported in stainless steels [8–16]. It was found to be a relevant phenomenon in the range of 250–600 °C, which coincides with that of AISI 316L operating vessel and piping systems of liquid metal cooled fast breeder nuclear reactor [14,15]. In this temperature range, stainless steels display anomalous evolution of strength and ductility both for tensile [12] and fatigue [13–16] conditions. Karlsen et al. [12] indicated that there is also a strong effect of strain rate. By reducing the strain rate, the range of DSA broadens and brings it to lower temperatures, while an increase in strain rate might suppress DSA.

1.3. Dislocation structures

Another point of doubt is the complex 3D morphology of dislocation arrangement, specially the cell structures, in stainless steels deformed at high temperatures. Transmission electron microscopy (TEM) works [12–16] indicated that the changes in the mechanical properties of AISI 316 steel during DSA are associated with a more planar dislocation structure rather than a cellular one. TEM observations by Karlsen et al. [12] of tensile-tested AISI 316 NG steel at 200, 288 and 400 °C revealed planar dislocation structures. This was considered an evidence of DSA, which would affect the deformation behavior by restricting cross-slip and, therefore, promoting strain localization. Dislocation cellular structures, however, were not shown in TEM images of Karlsen et al. [12] at 288 and 400 °C. This result was surprising since earlier investigations on AISI 316 and 304 steels tensile-tested in the temperature interval of DSA, without mentioning this phenomenon, revealed well developed cell structures [17–20]. It is also worth mentioning that in fatigue-tested AISI 316L steel, disoriented cell structures were shown at 600 °C [16]. Furthermore, cells near grain boundaries were reported at 500 °C [16] and incipient cells shown between sharp bands at 400 °C [13,14].

One of the first investigations on the dislocation structure of an austenitic stainless steel was carried out in AISI 316 by Michel et al. [17] at temperatures between 21 and 816 °C. They presented the evolution of tangle arrangement of dislocations to cell structures and mentioned that distinct structures could be correlated with work hardening stages. However these stages were not quantitatively characterized. Two years later, the dislocation structure of tensile-deformed 304L steel was investigated at 400 and 600 °C [18]. It was then found that a regular equiaxed cell structure, similar to that reported in AISI 316 [17], represents only one 2D aspect of a quite inhomogeneous 3D spatial dislocation distribution. In fact, the equiaxed cell structure was mostly found in grains in which the applied stress acted along a direction close to \(<100>\) [18]. These grains showed a \{100\} diffraction pattern at TEM zero degree tilt. By contrast, the majority of other orientations exhibited a large variety of structures often dominated by planar-banded dislocations. Cell structure formation was also the subject of investigation in AISI 304 at medium and high temperatures [19,20]. Subgrains, however, were observed only at 700 °C or above. In particular, the work of Almeida et al. [20] reported on serrations, work hardening stages and related dislocation substructures. They found that distinct mechanisms and atomic species are involved with DSA due to its wide interval of temperatures.

1.4. Carbide precipitation

A characteristic microstructural event in relatively high carbon austenitic stainless steels is carbide precipitation, especially Cr\(_2\)C\(_6\), which might occur at temperatures from 500 to 800 °C [21–23]. This precipitation is well-known to be responsible for the sensitization effect associated with corrosion. However, carbide precipitation demands an aging time, which is normally longer than that of a common (quasi-static) tensile test. Indeed, investigations on the microstructure of high temperature tensile tested austenitic stainless steels [8–19] did not emphasized the importance of Cr\(_2\)C\(_6\) on the developed substructures. Almeida et al. [20] proposed that the extinction of DSA at higher temperatures, above 600 °C, was due to a surge in carbide precipitation owing to the increase in both mobility and self-diffusion.
The present work investigates the simplest (lower SFE) AISI 301 austenitic stainless steel, tensile tested at 500 and 600 °C, and the corresponding work hardening stages as well as DSA and related (100) dislocation cell structures.

2. Experimental procedure

The type AISI 301 austenitic stainless steel was supplied by Sandvik firm as a 10 mm in diameter rolled bar. The steel composition was found to contain 0.16% C, 17.6% Cr, 7.7% Ni, 1.36% Mn and 0.8% Si as well as small amounts of P and S, within the specifications for this steel.

Tensile specimens were machined with 4 mm gage diameter and 24 mm gage length as per ASTM standard. These specimens were first solution treated at 1100 °C for 1 h under vacuum. Tensile tests were carried out in a model 1125 Instron machine at a constant cross-head speed, corresponding to a strain rate of $3.5 \times 10^{-5}$ s$^{-1}$. Test temperatures were 500 and 600 °C with specimens inside the vacuum chamber of the Instron furnace. These temperatures were selected to be below, 500 °C, and above, 600 °C, the reference annealing temperature of about 560 °C [17].

Samples for transmission electron microscopy (TEM) observation were cut from specimens deformed up to a desired degree of plastic deformation by spark erosion. Discs of these samples were electropolished at 3 V in a 6% perchloric acid ethanolic solution until convenient thickness (first hole) was obtained for imaging by TEM using a Jeol microscope operating at 200 kV.

The methodology used to calculate the dislocation density was based in the classical technique proposed by Han [24] and refined by Hirsch et al. [25]. In this technique the dislocation density, $\rho$, is given by:

$$\rho = \frac{2N}{lt}$$  \hfill (3)

where $N$ is the number of intersections that dislocation lines make with a standard grid, $l$, the total length of lines in a certain area covered by the grid and $t$ is the thin foil thickness. The determination of $t$ was done by electron energy loss spectroscopy, EELS, using its own software, according to

$$t = \lambda_p \ln \left( \frac{1}{I_0} \right)$$  \hfill (4)

where $\lambda_p$ is the extinction length, $t$ the plasmon peak intensity and $I_0$ the zero loss peak (ZLP) intensity. In order to construct the grid and perform the intersection counting the stereological image program J/Fiji-1.48q was used. Due to relatively low precision in measurements of fuzzy tangles, dislocation density values are given within one order of magnitude.

The methodology applied to calculate the dislocation cell size was similar to the intercept method, conventionally used to estimate grain size. In this method, straight lines are drawn through several dislocation cell images. The cells intersected by each line are counted. The line length is then divided by an average number of intersected cells, taken over all line segments. The average cell size is found by dividing this result by the image magnification.

Fig. 1a shows typical load-elongation tensile curves for the AISI 301 steel. In these curves one should note the plastic flow instabilities in the form of serrations, at 500 and 600 °C, that are characteristic evidence of dynamic strain aging (DSA) effects. This phenomenon was also observed in AISI 316L steel between 250 and 600 °C [8–16]. Curves at 25 and 700 °C are also presented in Fig. 1a only as references, with no serrations. These two curves that failed to show DSA effects will not be further investigated.

Load-elongation curves in Fig. 1a were digitally transformed in corresponding stress–plastic strain ($\sigma$ vs. $\varepsilon_p$) curves that allowed the interpretation by computer of each curve with the conversion into log $da/d\varepsilon_p$ versus log $\varepsilon$ based on Eq. (1). Fig. 1b presents, for 500 and 600 °C, their logarithmic curves displaced in the vertical log $da/d\varepsilon_p$ scale to avoid superposition. It is important to notice that the curves in Fig. 1b, as expected, adjust well to straight lines starting from the conventional 0.2% yield deformation. Both initial straight lines in Fig. 1b display a transition point in going from a relatively low slope, first stage, to a higher slope, second stage, up to fracture around 35%. The two branches in each curve are associated
Fig. 2 – Dislocation structures in AISI 301 steel tensile tested at 500 °C up to plastic deformation of: (a) 2%; (b) 22%, transition point between first and second work hardening stages, and (c) 29% fracture strain.

with work hardening stages with different values of \( n_1 \) and \( n_2 \), obtained from Eq. (1), as well as distinct transition points between stages. For 500 °C, \( n_1 = 0.73 \) and \( n_2 = -0.08 \) with a transition strain of 22%. For 600 °C, \( n_1 = 0.71 \) and \( n_2 = -0.11 \) with a transition strain of 15%.

One should notice that the work hardening exponents for each stage (first and second) in both temperatures are very similar. The first stage with a value slightly higher than 0.7, corresponds to a typical “parabolic” hardening while the second stage with negative value of \( n \) close to zero is a faster decreasing rate. Mathematically the second stage would be better adjusted by a hyperbolic type of equation [26]. Another point of discussion regarding Fig. 1b is the fact that only two stages were found, using Eq. (1), in contrast with the three stages observed by Christopher and Choudhary [8] using \( d \varepsilon /d \varepsilon \) versus \( \varepsilon \) graphs. Indeed, the experimental points in the second stage for both 500 and 600 °C in Fig. 1b display a relatively large dispersion. This could be interpreted as a possible third stage. In other words, another straight line with different slope could eventually be drawn through the last points. As further discussed, the observed dislocation structures cannot be differentiated to justify a clear transition between the second stage and a probable third stage.

Fig. 2 shows for 500 °C the typical dislocation structures observed in grains with \{100\} diffraction pattern. These grains were selected in specimens deformed only to 2% of plastic strain, Fig. 2a, as well as grains deformed to the transition point at 22% of plastic strain, Fig. 2b, and deformed to fracture at 29% of final plastic strain, Fig. 2c. It can also be seen that at \( \varepsilon_p = 2\% \), Fig. 2a, incipient cells begin to form amid tangles of dislocations. At the transition point, a well developed cell structure is observed, Fig. 2b, with an average cell size of 0.40 ± 0.16 µm. The cell walls possess a dislocation density of about \( 10^{15} \text{ m}^{-2} \). At fracture, the same cell structure is maintained, Fig. 2c, with a slight tendency of cell alignment along the tension axis. In fact the average cell size was found as 0.43 ± 0.14 µm and a wall density of about \( 10^{15} \text{ m}^{-2} \). These cellular dislocation structures at 500 °C, a typical temperature for DSA in austenitic stainless steels, contradict the proposition of Karlsen et al. [12] of planar structure as opposed to cellular structure. The apparent reason for the misleading association of planar only dislocation structure with DSA [12,16] is probably the fact that grains other than those with \{100\} diffraction pattern [18] were observed by TEM.

Fig. 3 displays, for 600 °C, the dislocation structure observed in grain with \{100\} diffraction pattern. Tensile specimens were deformed \( \varepsilon_p = 5\% \) in Fig. 3a; deformed up to the transition point \( \varepsilon_p = 15\% \) in Fig. 3b; and deformed to fracture at 32% of final plastic strain, Fig. 3c. Only incipient cells are observed in the beginning of first stages, \( \varepsilon_p = 5\% \) in Fig. 3a. At the transition point from first to second stage, \( \varepsilon_p = 15\% \), partially consolidated cells with average size of 0.73 ± 0.22 µm are seen. At fracture, \( \varepsilon_p = 32\% \), the cell structure is well consolidated with a tendency to smaller cell size, 0.36 ± 0.12 µm. The same aforementioned argument for 500 °C applies for the 600 °C tensile-tested samples, still under DSA regime.
One important comment is that TEM observations with different diffraction patterns, as previously indicated [18], failed to show equiaxed cell structures like those in Fig. 2b and c as well as Fig. 3b and c. As two examples, planar-banded dislocation structures were observed in grains with \{111\} diffraction pattern as illustrated in Fig. 4. This could explain why Karlsen et al. [12] did not observe cell structures at 288 and 400 °C. Actually, in order to investigate cell structures, one should consistently observe grains with \{100\} only diffraction pattern, as in Figs. 2 and 3.

The results in Figs. 1–3 indicate that AISI 301 steel plastically deformed in tension at 500 and 600 °C produced cell structures at the transition point between the first and second work hardening stages. Both temperatures correspond to conditions at which DSA is still occurring, as seen in Fig. 1a by the instabilities in the flow curves. At 500 °C the cell structure is well consolidated and remains unchanged up to fracture. On the contrary, at 600 °C the cell structure does not appear to be well consolidated with relatively large cell size and lower density of cell walls of about \(n_1 = 10^{14} \text{ m}^{-2}\). The negative values of \(n_1\) for the second stage in both temperatures corroborate the generally proposed mechanism of dynamic recovery responsible for the fast decrease in work hardening rate of the second stage, Fig. 1b. However, pinning of dislocation by solute atoms associated with DSA restricts the formation of sub-boundaries. This mechanism can also explain the consolidation of cell structure and the decrease in cell size after the transition point, Fig. 3b and c, at 600 °C.

Regarding the dislocation structures in the second stage at 600 °C, no subgrains or evidence of sub-boundaries were observed by TEM, as previously indicated by Michel et al. [17]. In spite of this indication, a close look at their 650 °C dislocation structure failed to reveal subgrains. Only above 700 °C, Michel et al. [17] images exhibited definite sub-boundaries. This is consistent with the previous results of Almeida et al. [19,20]. It is then proposed that in the DSA range for stainless steels, including AISI 301, around 250–600 °C [8], dynamic recovery occurs by dislocation annihilation but not by sub-boundaries formation. Consequently, only cell structures are observed in grains with \{100\} diffraction pattern. Moreover, carbide precipitation does not seem to be effective during tensile tests conducted at 500 and 600 °C under \(3.5 \times 10^{-5} \text{ s}^{-1}\) strain rate.

4. Conclusions

- The work hardening rate of AISI 301 austenitic stainless steel tensile deformed under strain rate of \(3.5 \times 10^{-5} \text{ s}^{-1}\) at 500 and 600 °C, the upper limit of dynamic strain aging (DSA), displayed two stages with similar hardening exponents of about \(n_1 \sim 0.7\) for the first and a negative \(n_2 \sim -0.1\) for the second stage.
The dislocation structure at 500 °C showed, in grains with (100) diffraction pattern, well consolidated cells at the transition point of εp = 22% plastic strain from the first to the second stage. A cell size of about 0.4 μm with dislocation density ρ ~ 10^{15} m^{-2} in the cell walls are maintained up to fracture.

The structure at 600 °C consisted of partially consolidated cells at the transition of εp = 15%. Relatively large cell size of ~0.7 μm and lower wall density of ρ ~ 10^{14} m^{-2}, which evolved to well consolidated ~0.4 μm cells with ρ ~ 10^{15} m^{-2} in their walls, were observed.

At both investigated temperatures, 500 and 600 °C, TEM images in grains with other diffraction patterns such as (111) display planar-banded dislocation structures without cells.

The evidence of rapid decrease in the work hardening rate for the second stage in association with its negative exponent is in agreement with a generally proposed mechanism of dynamic recovery. This is associated with dislocation annihilation without evidence of sub-boundary formation, probably due to DSA effects at both investigated temperatures.

Conflicts of interest

The authors declare no conflicts of interest.

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