Tensile Deformation and Work Hardening Behaviour of AISI 431 Martensitic Stainless Steel at Elevated Temperatures

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Abstract: Tensile deformation and fracture behaviour of AISI 431 martensitic stainless steel, over the temperature range of 300-823 K, has been examined. Yield and ultimate tensile strength values decreased gradually from room temperature to intermediate temperatures, followed by a rapid decrease at high temperatures. At the intermediate temperatures (523-673 K), the steel exhibited jerky/serrated flow and anomalous variations in terms of a plateau in flow stress/strength and work hardening parameters and minima in ductility. These manifestations were identified to be the signatures of dynamic strain ageing, operating at these temperatures. At high temperatures, a rapid decrease in flow stress/strength values and increase in ductility with increasing temperature indicated the dominance of dynamic recovery. The fracture remained transgranular ductile at all temperatures. Work hardening relations that best described the flow behaviour of AISI 431 steel were identified. Variations of the work hardening parameters with temperature were consistent with the variations exhibited by strength and ductility values.

Keywords: AISI 431, Martensitic steel, Tensile properties, Dynamic Strain Ageing

1 Introduction

AISI 431 steel is a 16Cr-2Ni martensitic stainless steel which exhibits excellent strength, toughness, and resistance to stress corrosion and oxidation up to 863 K [1, 2]. Hence these steels find application in chemical and power industries, and as compressor blades in marine and modern aircraft engines. These grades of martensitic stainless steel are used in quenched and tempered condition. Typically the martensitic stainless steels are hardened by heating to the austenitising temperature range of 1198-1338 K, followed by air cooling or oil quenching. In austenitised condition, the steel exhibits high strength and hardness values, but with low ductility and toughness values. In order to obtain useful engineering properties, it is usual practice to employ a tempering treatment to these steel. On quenching 16Cr-2Ni steel from the austenitising temperature results in the formation of complex microstructure consisting of inherited δ-ferrite, martensite and retained austenite with few undissolved M23C6 carbides [3, 4]. The chemical composition in terms of the balance between Cr- and Ni-equivalents critically influence the resulting volume fraction of the above phases. Lower tempering temperatures of 573 K resulted in the transformation of retained austenite at lath boundaries to Fe3C, whereas at higher tempering temperatures of above 673 K, Fe3C progressively dissolves and M7C3 type carbides were observed to be precipitating. At tempering temperatures above 773 K, M23C6 type carbides were replaced by M13C6. From the tempering studies on 16Cr-2Ni steel, Balan et al. [3] reported that the hardness values decreased on increasing tempering at 573 K from the as quenched values and exhibited a secondary hardening for the tempering temperatures of 673-773 K. Based on the transmission electron microscopy (TEM) observations and selected area diffraction pattern (SADP) analysis, the secondary hardening observed at 673 K was attributed to precipitation of fine austenite particles [3]. In 17Cr-2Ni steel, Liu Ning et al. [4] observed that ultra-high austenitising temperature resulted in presence of many δ-ferrite net-
works and few twinned martensite and hence did not improve the impact toughness of the steel. Tempering at 823 K resulted in lower impact energies due to temper embrittlement caused by segregation of phosphorus, chromium and nickel to prior austenite grain boundaries [4]. In Type 431 welds, it was reported that presence of nickel lowers \( A_c1 \) and hence in order to avoid re-austenitisation during tempering, it was suggested to have employed double tempering treatment at temperatures below \( A_c1 \) [5].

While considering material for component or structural applications, the tensile, creep and fatigue properties are of critical importance. Evaluation of high temperature tensile properties is a prerequisite mechanical characterization towards the performance of material at elevated temperatures. Room temperature tensile strength for AISI 431 steel is specified to vary between 800 and 1300 MPa depending upon the tempering temperature, with ductility values greater than 20% [6]. In the present study, the influence of temperature on the tensile deformation and work hardening behaviour of AISI 431 martensitic stainless steel has been examined in the quenched and double tempered condition. The role of dynamic strain ageing manifested at the intermediate temperatures, and the dominance of dynamic recovery at high temperatures affecting tensile properties are discussed.

### 2 Experimental

AISI 431 steel plates of chemical composition conforming to that specified in ASTM standards [2] were obtained in quenched and double tempered condition. Chemical composition of the steel in weight percentages of the elements is given in Table 1. The material was subjected to quench hardening and double tempering treatment. Austenitising was carried out at 1283±10 K for 30 minutes, followed by quenching in oil. The hardened alloy was given a tempering treatment at 923±10 K followed by quenching in oil and again re-tempered at the same temperature and quenched in oil. Specimen blanks of 12 mm diameter and 60 mm length were cut along the rolling direction of the plate. Button head cylindrical specimens of 26 mm gauge length and 4 mm gauge diameter were machined from the specimen blanks. Tensile tests were carried out in air using a floor model Instron 1195 universal testing machine (UTM). The UTM was equipped with a three-zone temperature control furnace for precision temperature control and a stepped-load suppression unit for fine load measurement. Tests were performed in the temperature range of 300-823 K employing a nominal strain rate of \( 3\times10^{-3} \) s\(^{-1} \). Few additional tensile tests were also performed at the nominal strain rates of \( 3\times10^{-4} \), \( 3\times10^{-5} \) and \( 3\times10^{-6} \) s\(^{-1} \) at selected temperatures to evaluate the occurrence of dynamic strain ageing. Load-elongation curves for all the test conditions were recorded using a data acquisition system attached to the tensile test system. Since no strain gauge was employed, the crosshead displacement was taken as the specimen extension and slope of the initial linear elastic portion of the load–elongation data is contributed by the specimen, machine frame, and load-train assembly. True stress (\( \sigma \))–true plastic strain (\( \varepsilon \)) data were evaluated from the load–elongation data using a computer program up to the maximum load values corresponding to the onset of necking. The temperature during the tests was controlled within ± 2 K. A strain resolution of \( 7.5\times10^{-4} \) and a stress resolution of 0.80 MPa were obtained on the \( \sigma–\varepsilon \) data.

### 3 Constitutive Description Tensile Flow Curve

Plastic flow behaviour in terms of true stress (\( \sigma \))–true plastic strain (\( \varepsilon \)) plot for several metals and alloys in the uniform elongation regime had been well described using several empirical relations proposed in the literature. Hollomon relation [7] is the most widely used and is given as,

\[
\sigma = K_1 \varepsilon^\eta_1,
\]

Where \( K_1 \) is the strength coefficient and \( \eta_1 \) is the strain hardening exponent. However, for materials exhibiting varied yield strength and yet having similar strain hardening behavior or in the case of materials exhibiting similar yield strength with different work hardening behavior, the yield and strain hardening cannot be described by a single relation, as in the case of Hollomon relationship [8]. Hence, introducing an additional term to the above power law relation in order to accommodate the mechanical history was considered. The positive deviation observed at
the low strains due to yielding was accounted with an additional stress term \( \sigma_0 \), as in the case of Ludwik relation [9],

\[
\sigma = \sigma_0 + K_L e^{n_L},
\]

(2)
or with an additional strain term \( e_0 \) to account the pre-strain left in the material, as in the case of Swift relation [10],

\[
\sigma = K_S (e_0 + e)^{n_S},
\]

(3)

where, \( K_L \) and \( K_S \) are the strength coefficient for Ludwik and Swift equations, respectively. \( n_L \) and \( n_S \) are the strain hardening exponents for Ludwik and Swift equations, respectively. Ludwigson [11] measured the positive stress deviations at low strains from the Hollomon relation and observed that the deviation stress values decreased exponentially with increasing strains and hence proposed the following relation,

\[
\sigma = K_1 e^{n_1} + \exp(K_2 + n_2 e),
\]

(4)

where \( K_1 \) and \( n_1 \) are the same as in Hollomon equation and, \( K_2 \) and \( n_2 \) are additional constants.

For the materials exhibiting saturation in stress values at strains close to the onset of instability, Voce [12] proposed a flow relation as,

\[
\sigma = \sigma_S - (\sigma_S - \sigma_I) \exp(n_V e),
\]

(5)

where \( \sigma_I \) and \( \sigma_S \) are the initial and saturation stresses, respectively, and \( n_V \) is a constant.

The above described constitutive relations were applied to the experimental true stress-true plastic strain data of AISI 431 steel using Levenberg-Marquardt least square method, with the unknown constants of the relation as free parameters. The goodness of the fit was indicated by the low \( \chi^2 \) value, the sum of the square of deviation of the calculated stress values from the experimental stress values. The lowest \( \chi^2 \) value indicated the high degree of goodness of fit.

4 Results & Discussions

4.1 Tensile Properties

Figure 1 shows the typical tensile curve in terms of variations in engineering stress with engineering strain in the temperature range of 300–823 K at the nominal strain rate of 3×10^{-3} s^{-1}. Typically, the influence of temperature on the engineering tensile curve is observed as a systematic decrease in stress values with increasing temperature which is pronounced more significantly at engineering strains above the elastic limit. In Figure 1, the elongation to fracture was found to decrease with increasing temperatures up to 723 K and exhibited an increasing trend thereafter. However, the engineering strain to the onset of plastic instability, marked by the peak in engineering stress (Figure 1) was found to decrease systematically with increasing temperature. Figures 2 and 3 show the variation of yield strength and ultimate tensile strength, respectively, with increasing temperatures for the AISI 431 steel. The yield and ultimate tensile strength values decreased from room temperature values up to 523 K with increasing temperature. At the intermediate temperatures of 523 to 673 K, the strength values exhibited a plateau, followed by a rapid decrease in strength values at temperatures above 673 K. As shown in Figure 4, variation in uniform elongation values with temperature also exhibited a trend simi-
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4.2 Tensile Fracture Behaviour

At all temperatures examined, the fracture mode was observed to be transgranular ductile. As shown in Figure 7a, the low magnification images of the fractured surfaces exhibited star-like appearance for the specimen tested at room temperature (300 K). Figures 7b and 7c show the fracture surface for specimen tested at intermediate (623 K) and high temperature (823 K), respectively, which reveal typical cup and cone fracture. With increasing temperature pronounced necking was observed (Figures 7a, 7b and 7c). It was also observed that coalescence of microvoids leads to the formation of dimples which governed the mode of fracture at all temperatures. At 300 K along with ductile dimples, large amount of splitting of martensitic lath boundaries which result in chisel tip ap-
appearance are seen in Figure 7d. However, with increasing temperature, chisel tip appearances reduce significantly and the fracture was dominated mainly by the presence of equiaxed dimples (Figure 7e). Increase in size of equiaxed dimples with increasing temperature indicates the decreasing number of microvoid nucleation and dominance of growth process at high temperatures as shown for 823 K in Figure 7f.

The transgranular mode of fracture observed in the tensile deformation of AISI 431 steel for wide range of temperatures compares well with those reported for P9 and P91 ferritic-martensitic steels [13, 14]. Presence of secondary precipitates in the steel may also influence the chisel tip appearances caused by split in the martensitic lath boundaries. In thermally aged P9 steel, it had been shown that because of increased formation and growth of brittle Laves phase (Fe$_2$Mo) at longer ageing durations, a large number of chisel tip appearance were observed [13]. Intergranular cracking cannot be the possible reason for the reduced strength values exhibited at elevated temperatures because of the observed transgranular fracture in the AISI 431 steel. Hence the observed rapid decrease in strength values at elevated temperatures is rationalised due to the effect of dynamic recovery. Also, the observed decrease in flow stress/strength values and increase in the ductility values at high temperatures can be attributed to the dominance of dynamic recovery at these temperatures (Figures 2-6).

4.3 Influence of Dynamic Strain Ageing

Anomalous variations were observed at the intermediate temperatures of 523-673 K, in terms of a plateau in yield
strength, ultimate tensile strength, and uniform elongation values, and minima in ductility values. These anomalous variations could be attributed to the influence of Dynamic Strain Ageing (DSA) operating at these temperatures and strain rate. DSA, also known as Portevin Le-Chatelier effect, is caused by the dynamic interaction between solute atoms diffusing under the thermal activation and the mobile dislocations, driven by the imposed strain rate, during plastic deformation [15, 16]. This phenomenon had been reported in 2.25Cr-1Mo [17], 9Cr-1Mo [18, 19], modified 9Cr-1Mo steels [20] and 316 SS [21]. In order to confirm the occurrence of DSA in the AISI 431 steel, few additional tests in the temperature range of 523-673 K were carried out employing strain rates ranging from $3 \times 10^{-6}$ to $3 \times 10^{-3}$ s$^{-1}$.

AISI 431 steel exhibited serrated/jerky flow in the engineering tensile stress-strain curve, which is one of the manifestations of DSA. Serrations due to DSA had been classified into various types, identified as type A, B, C, D and E [15, 16]. Type A serrations are locking serrations, characterized by an abrupt rise in stress value above the mean values, followed by a drop to below the general level of the stress-strain curve. Type B serrations are oscillations about the general level of the stress-strain curve that occur in quick succession due to discontinuous band propagation arising due to DSA. Type C serrations are considered to be due to the unlocking of dislocations leading to stress drops below the general level of the stress-strain curve. Type D serrations are plateaus in the flow curve due to band propagation, with no work hardening. Type A serrations change over to type E serrations at high strains as irregularities, with no work hardening during band propagation.

Figure 8 depicts the typical serrations/jerky flow observed at different temperatures and strain rates in the AISI 431 steel. As evident from the figure (Figure 8), the observed serrations could be identified to be Type D or mild irregularities. The occurrence of type D serrations results from negligible or small work hardening following a rapid initial increase in stress (Figure 8). The observed serrations were mild or irregular and the number of load drops or, the number of events of serrations in AISI 431 steel was observed to be significantly low compared to those obtained for P9, P91 and 316 steels [18–21]. These indicate reduced activity of serrated flow. Table 2 lists the temperature and strain rates under which the appearance of serrated/jerky flow was noticed in the tensile stress-strain curve. As shown in Figure 9, the steel exhibited negative strain rate sensitivity in terms of decreasing ultimate tensile strength values with increasing strain rate, which is one of the important manifestations of DSA. It may be noticed that though serrations were not observed for the applied nominal strain rates of $3 \times 10^{-4}$ and $3 \times 10^{-3}$ s$^{-1}$ at 523 K, the influence of DSA is reflected in the decrease in ultimate tensile strength values with increasing strain rate. The average macroscopic strain rate sensitivity was estimated to be $-6.97 \times 10^{-3}$ at ultimate tensile strength for 523 K.

A plot of appearances of serrations/jerky flow in a $\log(\dot{\varepsilon}) \text{ vs } 1000/T$ space is shown in Figure 10. The slope of the boundaries separating the serrated flow regime from

### Table 2: Temperatures and strain rates at which serrations were observed at for AISI 431 martensitic stainless steel.

| Temperature | Strain Rate |
|-------------|-------------|
| 423 K       | $3 \times 10^{-3}$ s$^{-1}$ | X | – | X | – |
| 473 K       | $3 \times 10^{-4}$ s$^{-1}$ | X | X | X | – |
| 523 K       | $3 \times 10^{-5}$ s$^{-1}$ | X | X | O | X |
| 573 K       | $3 \times 10^{-6}$ s$^{-1}$ | – | – | O | – |
| 623 K       | – | – | – | – | X |
| 673 K       | – | – | – | – | O |

- : Test not carried out
X : No serration
O : Serrated flow / Irregularity
Figure 9: AISI 431 martensitic stainless steel exhibited negative strain rate sensitivity in terms of decreasing ultimate tensile strength values with increasing nominal strain rate at 523 K.

Figure 10: Arrhenius plot of Log\[strain rate\] vs (1000/Temperature) for the appearance of serrations/jerky flow in the tensile curve of AISI 431 martensitic stainless steel.

A smooth flow can be used to evaluate the average activation energy, \( Q \) for serrated flow. The activation energy is estimated as

\[
Q = \text{slope} \times 1000 \times R \times 2.303,
\]

where \( R \) is the universal gas constant [22, 23]. In the log(e) -1000/T plot (Figure 10), the serrated flow was found to be well encompassed by two mean empirical lines (a) and (b) which well-represent the onset and disappearance of serrated flow respectively. The values of activation energy for the onset of serrated flow as \( Q = 96 \text{kJ mol}^{-1} \) and for the disappearance of serrated flow as \( Q = 144 \text{kJ mol}^{-1} \) have been obtained from the slopes of lines (a) and (b), respectively.

The activation energy of 96 kJ mol\(^{-1}\) observed for the onset of serrated flow process in the present investigation (Figure 10) compares favourably with the reported values of 76-103 kJ mol\(^{-1}\) for the diffusion of interstitial solutes such as carbon in BCC iron and steels [24–26]. The obtained activation energy of 144 kJ mol\(^{-1}\) for the disappearance of serrated flow in the AISI 431 steel, is in agreement with the reported values in the range of 128-167 kJ mol\(^{-1}\) for the disappearance of serrations towards the end of serrated flow temperature regime in low carbon steels [25, 26]. From their studies on 2.25Cr-1Mo steel, Hayes et al. [27] have also reported the activation energy for the disappearance of serrations to be between 130-155 kJ mol\(^{-1}\). In 2.25Cr-1Mo, P9 and P91 ferritic steels, based on the values of activation energy, locking of mobile dislocations by diffusing carbon atoms were identified to be the source of serrations [14, 18–20]. From the activation energy values obtained for the onset of serrations in the present study, it is suggested that the occurrence of dynamic strain ageing can be attributed to the diffusion of interstitial carbon in the AISI 431 steel. Whereas, the higher activation energy values obtained for the disappearance of serrations is attributed to the sum for the activation energy for the diffusion of interstitial solute and the binding energy of the solute to dislocation [25]. DSA causes an increased rate of dislocation multiplication and delay in recovery of dislocation structure, and promotes an increased propensity towards a uniform distribution of dislocations rather than cell structures in many metals and alloys [28, 29, 33]. The dislocation density in the DSA regime was also observed to be higher, compared to that observed at room temperature. Keh et al. [25] reported higher dislocation density in 0.35% C steel in the DSA regime. In low carbon martensitic steel, uniform dislocation distribution characterized by linear arrays of screw dislocations were observed in the strain rate range of serrated flow [33]. A significant increase in dislocation density in the DSA temperature regime than those at room and high temperatures was reported for P91 steel [34]. Morris [31] pointed out that the tendency to produce a non-cellular array of dislocations increases with an increase in the intensity of dynamic strain ageing. These investigations indicate reduced dynamic recovery in the DSA regime arising from the diffusion of solutes, which affects the rate of dynamic recovery by pinning of dislocations and thereby preventing screw dislocation to cross slip due to its reduced mobility.

4.4 Tensile Flow Behaviour

Figure 11 shows tensile flow behaviour of AISI 431 martensitic SS, as the variations in true stress with true plastic strain, within the uniform plastic deformation region, for
the temperature range of 300-823 K. The double logarithmic plot of $\sigma - \varepsilon$ data exhibited a curvilinear behaviour with positive stress deviations at lower strains from the extrapolated linear $\sigma - \varepsilon$ data at high strains for the room and intermediate temperatures. The extent of positive stress deviation at low strains decreases with increasing temperature. At the higher temperatures, $\sigma - \varepsilon$ plots exhibited a linear for the entire uniform plastic deformation regime with marginal negative stress deviations at the higher stresses, indicating saturation in stress values at strains close to the onset of instability. The flow stress values corresponding to respective true strain values decrease systematically with increasing temperature. The decrement in flow stress values with increasing temperature in the intermediate temperature range of 373 to 623 K was observed to be insignificant, whereas at high temperatures in the range 673-823 K a rapid decrease in flow stress was observed. Besides the flow stress values, the true uniform plastic strain values were observed to systematically decrease with increasing temperature from 300 to 823 K.

Various flow relations proposed in the literature such as Hollomon [7], Ludvik [9], Swift [10], Ludwigson [11] and Voce [12] were employed to describe the tensile flow behaviour of AISI 431 martensitic stainless steel in the temperature range of 300-823 K. Applicability of various flow relationships in describing the observed $\sigma - \varepsilon$ data is demonstrated at 300 K in Figure 12. Table 3 gives the $\chi^2$ value obtained for various flow relationships in describing the observed $\sigma - \varepsilon$ curve of the AISI 431 steel at different temperatures. Ludwigson relation was found to best describe the flow behaviour in the temperature range of 300-773 K, with the lowest $\chi^2$ values which indicated the goodness of the fit. At the high temperatures of 773 and 823 K, Hollomon
Figure 13: Variation of Ludwigson / Hollomon work hardening parameter \( K_1 \) with temperature for the AISI 431 steel.

Figure 14: Variation of Ludwigson / Hollomon work hardening parameter \( n_1 \) with temperature for the AISI 431 steel.

Figure 15: Variation of Ludwigson work hardening parameter \( K_2 \) with temperature for the AISI 431 steel.

Figure 16: Variation of Ludwigson work hardening parameter \( n_2 \) with temperature for the AISI 431 steel.

4.5 Variations of Work Hardening Parameters with Temperature

Figures 13-16 show the variations of work hardening parameters \( K_1 \), \( n_1 \), \( K_2 \) and \( n_2 \) of the Ludwigson equation obtained for fitting stress-strain data with the temperature at the strain rate of \( 3 \times 10^{-3} \text{ s}^{-1} \). Figures 17 and 18 show the variation of transition strain, \( \varepsilon_{tr} \), and transition stress, \( \sigma_{tr} \), respectively, with temperature for the type 431 steel. The variation of \( K_1 \) and \( K_2 \) with temperature exhibited a gradual decrease to a plateau in values from room temperature to intermediate temperatures followed by a rapid decrease in the value at high temperatures (Figure 13 and 15). The variation of \( n_1 \) with temperature exhibited a gradual increase to a peak value at the intermediate temperature, followed by a rapid decrease at high temperatures (Figures 14). The additional constant in the Ludwig-
Detailed investigation on tensile deformation, fracture and work hardening behaviour of AISI 431 martensitic stainless steel exhibited distinct intermediate and high temperature regimes in the variations of tensile strength values, tensile ductility and work hardening parameters. The fracture mode remained transgranular for the range of temperatures examined. Anomalous variations in terms of peaks in flow stress/strength values, ductility minima and serrated/jerky flow at the intermediate temperatures indicate the manifestation of dynamic strain ageing occurring at these temperatures. Measurement of activation energy suggested that diffusion of interstitial carbon is responsible for DSA in the steel. At high temperatures, rapid decrease in flow stress/strength values and work hardening rate, and an increase in ductility with the increase in temperature indicated the dominance of dynamic recovery.

5 Conclusions
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