Hot Deformation Characterization of AISI316 and AISI304 Stainless Steels

D.K. Suker¹, T.A. Elbenawy¹,², A.H. Backar¹,³, H.A. Ghulman¹, M.W. Al-Hazmi¹
1- Mechanical Engineering Department, Umm Al-Qura University, Saudi Arabia
2- Design & Production Engineering Department, Ain Shams University, Egypt
3- Production Engineering Department, Alexandria University, Egypt
ahmedbackar@gmail.com – Phone: +966592244888
Mechanical Engineering Department, College of Engineering, Umm Al-Qura University, Makkah, Saudi Arabia

Abstract— Austenitic stainless steel types AISI304 and AISI316 are material characterised by their high ductility and toughness. In this paper, the deformation characteristics of AISI316 and AISI304 stainless steel are investigated in a temperature range of 850-1050 °C and strain rate range of 0.001-0.1 s⁻¹ using dilatometer hot compression tests. The tests aimed at producing hot processing map and TEM images of the alloys. Based on the deformation results, an empirical model is used to predict the behaviour of both alloys in terms of the stress/strain characteristics and the recrystallization dynamics. The developed model is considered to be a very good tool which can be used to predict the materials characteristics under predefined deformation conditions.

Index Term— AISI316 stainless steel, AISI304 stainless steel, hot deformation, dynamic recrystallization

1. INTRODUCTION

Austenitic stainless steels are frequently used in the construction of both nuclear and non-nuclear energy systems and will be subjected to elevated temperatures for long periods. Types AISI304 and AISI316 austenitic stainless steel are known for the high ductility and toughness (Wang et al 2012). Therefore, stainless steels (austenitic) have been tested in different ways, which includes the deformation characteristics. An important approach testing for steels is hot deformation, since it involves a complex microstructural change, which may optimize the mechanical properties of the finished product (Dehghan-Manshadi, 2010). In manufacturing process, defect can be formed during deformation, such defects can be avoided by studying the microstructural evolution during hot deformation process under different conditions, as reported by Tan et al. (2008) and Belyakov et al. (2000). Recently, different research works have been conducted on hot deformation behaviour of austenitic stainless steel, mainly were AISI304 (Kin and Yoo, 2001), and AISI316L steel (Mataya et al 2003). Many researchers have investigated the materials under a series of dynamic recrystallization conditions (DRX) for example 304 austenitic stainless steel (Dehghan-Manshadi et al. 2004, 2008).

In this study, an investigation of the hot deformation parameters effects (temperature, strain rate and strain) on the microstructural evolution of 316 and 304 austenitic stainless steel is conducted. In addition, the influences of the process on the DRX, and grain boundary character distributions were also discussed and compared based on the electron backscatter diffraction (EBSD) analysis.

2. EXPERIMENTAL WORK

The materials received have been analysed for their chemical compositions. The results are reported in Tables 1 for AISI316 and AISI304 respectively.

<table>
<thead>
<tr>
<th>AISI316 and AISI304 chemical compositions</th>
</tr>
</thead>
<tbody>
<tr>
<td>C %</td>
</tr>
<tr>
<td>AISI316</td>
</tr>
<tr>
<td>AISI304</td>
</tr>
</tbody>
</table>

The specimen was prepared as a rod to be used on the machine. The rod is of diameter 5 mm and length of 10 mm. The materials were heat treated for 1 hour at 1050 °C for steel and 500 °C for aluminium. The shape is shown in Figure 1a. Different locates were selected for testing the microstructure of the material before and after heat treatment as shown in Figure 1b.
The process of homogenisation is usually based on treating the materials for 1 hour under high temperature. The materials microstructure is tested before and after homogenisation as shown in Figures 2 and 3 for AISI316 and AISI306 respectively. The figures show the microstructure at different scales. The homogenisation process has made the grain uniformity and distribution more homogeneous.
3. Properties Modelling

3.1. Stress/Strain Characteristics

In hot deformation conditions, the flow stress/strain relationship for most alloys is based on the Zener–Hollomon parameter, \( Z = \dot{\varepsilon} \exp \frac{Q_{\text{def}}}{RT} \), where \( \dot{\varepsilon} \) is equivalent strain rate, \( Q_{\text{def}} \) is a constant activation energy for deformation, \( R \) is the gas constant and \( T \) is the absolute temperature. Many laboratory tests are used to develop empirical equations to model the flow stress at specific \( Z \) values which are usually constant. However, during industrial rolling, constant \( Z \) cannot be realized due to the continuous changes in temperature and strain rate.

Many researchers reported such results, such as MacDonald et al. (2000) who reported a series of tests on austenitic (AISI304) stainless steel channel columns. Equations were developed using Ramberg-Osgood curves to model the stress/strain obtained from the column and tension testing using different stresses. The fitted Ramberg-Osgood results have experienced significant error at strains exceeding the 0.2% total strain which was modified to the following equation:

\[
\varepsilon = \frac{\sigma}{E_0} + 0.002\left(\frac{\sigma}{\sigma_0}\right)^i + \left(\frac{\sigma}{\sigma_0}\right)^k
\]

where the constants \( i \), \( j \) and \( k \) are in the range of 2.5 to 6 which depends on the material thickness. The suggested equation has shown very good accuracy, however, it was limited to specific alloys and thicknesses. Furthermore, Olsson (2001) studied stainless steel plasticity models under different tests condition, namely uniaxially and biaxially loading conditions. The results which presents the stress/strain curves shown that the stress can be s straight line under large strain conditions. The proposed model is based on the true-stress versus engineering strain using the equations for a total strains up to 2%. The constant stress is chosen as the best fit to the measured stress/strain curve. It was not necessary to consider the true ultimate tensile strength (\( \sigma_u \)) at the ultimate total strain (\( \varepsilon_u \)).

Acknowledging the relationship between the true and engineering stresses,

\[
\sigma' = \sigma (1 + \varepsilon)
\]

and observing that \( d\sigma/d\varepsilon \to 0 \) for \( \sigma \to \sigma_u \), it is apparent that as \( \sigma \to \sigma_u \) the true stress vs engineering strain curve asymptotes to the line,

\[
\sigma' = \sigma (1+\varepsilon), \ u
\]

Observably, the gradient of this line and the intercept of the line with the stress axis both equal \( \sigma_u \). The gradient of the line is different from the gradient of the line through the true 2% proof stress and true tensile strength.

3.2. Recrystallization

The stress-strain curves obtained under DRX conditions display characteristic shapes. A considerable amount of strain softening is evident, which leads eventually to the establishment of a steady state. More particularly, the shapes of the curves change from the single peak to the multiple peak type as the strain rate is decreased. The equivalent effects of decreasing the strain rate and increasing the temperature have long been observed under hot working conditions; this permits a temperature corrected strain rate \( Z \), or Zener-Hollomon parameter (Zener and Hollomon, 1944), to be defined:

\[
Z = \dot{\varepsilon} \exp \left(\frac{Q}{RT}\right)
\]

where \( \dot{\varepsilon} \) is the strain rate, \( T \) the absolute temperature, \( R \) the gas constant, and \( Q \) an apparent activation energy.

The flow stress curve conversion from multiple peaks to single peak behaviour can be influenced by a critical Zener-Hollomon parameter \( Z_c \); when \( Z < Z_c \) (or \( Z > Z_c \)) the flow curve shows multiple peaks (or a single peak, respectively). However, in the case when several initial grain sizes are used
in the testing, the transition in the flow stress behaviour occurs over a range of \( Z \) values rather than at a particular \( Z_c \).

The influence of the Zener-Hollomon parameter on particular points of the DRX flow curve has been a matter of investigation. In particular, variations in the peak stress \( \sigma_p \) and steady state stress \( \sigma_s \) have been investigated at various temperatures \( T \), strain rates \( \dot{\varepsilon} \) and initial grain sizes \( d_0 \). Both have been suitably related to the Zener-Hollomon parameters, \( Z_{hw} \) and \( Z_s \), respectively, through power law equations:

\[
\sigma_p = B Z_{hw}^m \\
\sigma_s = C Z_s^m
\]

where \( B \) and \( C \) are material constants and \( m \) is the strain rate sensitivity (\( m \approx 0.1 \) to 0.2). The constant \( B \) is dependent on the initial grain size \( d_0 \), especially if the initial grains are coarse. The Zener-Hollomon parameters \( Z_{hw} \) and \( Z_s \) are associated with two different activation energies \( Q_{hw} \) and \( Q_s \), respectively. \( Q_{hw} \) is called the apparent activation energy for hot working and, on the other hand, \( Q_s \) is called the apparent activation energy for DRX. In 304 stainless steel, \( Q_{hw} \) ranges between 390 and 430 kJ/mol and \( Q_s \) between 290 and 310 kJ/mol (Ryan and McQueen, 1990).

4. MICROSTRUCTURE CHARACTERISATION

The batch of steels are commercially supplied as 72 mm square ingots. These were annealed for 1 hrs at 1200 °C to eliminate \( \delta \)-ferrite, then hot rolled to bars of 10 mm thickness and 50mm width. The final pass at 1050 °C and subsequent annealing at 1050 °C resulted in a microstructure free from \( \delta \)-ferrite, with a grain size of 27 μm.

EBSD imaging was performed on the materials after homogenisation to test the grain size and uniformity of the grains. The following sections show for the two steels (AISI316 and AISI304) EBSD images as whole and microscopy images at different locations.

4.1. AISI316 Stainless Steel

The homogenisation process was base on treating the materials being heat treated for 1 hour under 500 °C temperature. Figures 4 and 5 show the microstructure of AISI316 at different scales. The process has made the grain uniformity and distribution more homogeneous.
Fig. 5. AISI316 homogenisation

**4.2. AISI304 Stainless Steel**

Figures 6 and 7 show the microstructure of AISI304 at different scales. The homogenisation process has made the grain uniformity and distribution more homogeneous.
5. Experimental Deformation Results

The effects of different strain rates are studied in respect to two parameters variations, namely the flow stress and the temperatures, and from the effects on the kinetics of static recrystallisation, which are sensitive to the total stored energy and to the stored energy distribution in the microstructure. For these measurements, specimens were water quenched after deformation and cut into eight pieces, so that a full recrystallisation curve could be obtained from each specimen. Annealing of the pieces was carried out at 900 or 950 °C which were dependent on the deformation conditions. Specimens were then mechanically polished on faces that were at 0.21 of the specimen breadth, and were electroetched in 10% oxalic acid. Recrystallised grains were easily recognised by their smaller size and strain-free microstructure. The fraction recrystallised was determined by point counting over all the section, excluding the regions within 0.5 mm depth from each surface, using a step size of 0.1 mm.

5.1. AISI316 Stainless Steel

The results of deforming AISI316 at different temperatures and strain rates as shown in Table 3 and Figure 8, while the recrystalised grain size is shown in Figure 9.

<table>
<thead>
<tr>
<th>Initial size (mm)</th>
<th>Sample</th>
<th>AISI316 Post-deformation size (mm)</th>
<th>AISI304 Post-deformation size (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>850°C 0.001 s⁻¹</td>
<td>D=5.07, L=9.82</td>
<td>D=5.07, L=9.80</td>
<td></td>
</tr>
<tr>
<td>850°C 0.01 s⁻¹</td>
<td>D=5.05, L=9.82</td>
<td>D=5.06, L=9.83</td>
<td></td>
</tr>
<tr>
<td>850°C 0.1 s⁻¹</td>
<td>D=5.06, L=9.79</td>
<td>D=5.05, L=9.82</td>
<td></td>
</tr>
<tr>
<td>950°C 0.001 s⁻¹</td>
<td>D=5.03, L=9.85</td>
<td>D=5.05, L=9.83</td>
<td></td>
</tr>
<tr>
<td>950°C 0.01 s⁻¹</td>
<td>D=5.10, L=9.76</td>
<td>D=5.05, L=9.83</td>
<td></td>
</tr>
<tr>
<td>950°C 0.1 s⁻¹</td>
<td>D=5.08, L=9.79</td>
<td>D=5.05, L=9.83</td>
<td></td>
</tr>
<tr>
<td>1050°C 0.001 s⁻¹</td>
<td>D=5.04, L=9.84</td>
<td>D=5.08, L=9.75</td>
<td></td>
</tr>
<tr>
<td>1050°C 0.01 s⁻¹</td>
<td>D=5.11, L=9.73</td>
<td>D=5.10, L=9.72</td>
<td></td>
</tr>
<tr>
<td>1050°C 0.1 s⁻¹</td>
<td>D=5.15, L=9.70</td>
<td>D=5.11, L=9.71</td>
<td></td>
</tr>
</tbody>
</table>
5.2. **AISI304 Stainless Steel**

The results of deforming AISI304 at different strain rates and temperatures (Table 3) are shown in Figure 10, while the recrystallised grain size is shown in Figure 11.
6. MODELLING

In hot deformation conditions for most alloys, the flow stress at defined can be described using the Zener–Hollomon equation (Zener and Hollomon, 1944), \( Z = \dot{\varepsilon} \exp \frac{Q_{\text{def}}}{RT} \), where \( \dot{\varepsilon} \) is equivalent strain rate, \( Q_{\text{def}} \) is a constant activation energy for deformation, \( R \) is the gas constant and \( T \) is the absolute temperature. For most of tests done in the laboratory, a nominal constant \( Z \) conditions are usually used to obtain the constitutive equations. However, as indicated earlier, under industrial rolling conditions, there are major changes of strain rate and temperature, which lead to \( Z \) being variable.

Many researcher have proposed different models to describe the relationship between the strain/stress. Barbosa’s Model is one particular model which is based on estimating the stress at different stages (Barbosa, 1992). The flow curve behaviour can be described by a critical Zener-Hollomon parameter \( Z_c \): when \( Z < Z_c \) (or \( Z > Z_c \)) the flow curve displays multiple peaks (or a single peak, respectively). The recrystallization of AISI316 was also modelled by Barbosa (1992), which is presented in terms of the time for 50% recrystallization and the recrystallized grain size.
6.1. AISI316 Stainless Steel

The model has been utilised to model the AISI316 material which the following equations are obtained:

Activation Energy ($Q$) = 460 kJ/mol

Zener Holomon Constant = strain rate $Q/RT$

$R = 8.31$

$T = Temperature$ in Kelvin

$$\sigma = \sigma_0 + (\sigma_s - \sigma_0) \left[1 - \exp \left(-\frac{C}{\varepsilon} \right) \right]^{0.5}$$

$$\varepsilon_p = 8.0832 \ Z^{-0.045}$$

$$C' = -10 \ln \left\{ \frac{1}{[\sigma_{01} - \sigma_0] / \sigma_s - \sigma_0} \right\}^2$$

$$\sigma_0 = 0.0 \log(Z) - 0.0$$

$$\sigma_{01} = 1.4448 \ \log(Z) \ - \ 7.6389$$

$$\sigma_p = 5.4548 \log(Z) \ - \ 29.351$$

$$\sigma_{ss} = 5.0888 \ \log(Z) \ - \ 18.857$$

$$\sigma_{se} = 5.9351 \ \log(Z) \ - \ 40.874$$

$$\sigma' = 1.0 \left[ \sigma_0 + (\sigma_s - \sigma_0) \left[1 - \exp \left(-C' \varepsilon \right) \right]^{0.5} \right] \quad (\varepsilon < 0.7 \varepsilon_p)$$

$$\sigma = \sigma' - 1.0 \left[ \sigma_s - \sigma_{ss} \right] \left[1 - \exp \left(-0.49 \left( \frac{\varepsilon - 0.7 \varepsilon_p}{\varepsilon_p} \right)^{1.4} \right) \right] \quad (\varepsilon \geq 0.7 \varepsilon_p)$$

$$d_{rex} = 470 \ \ Z^{-0.1} \ \ d_0^{0.3} \ \ \varepsilon^{-1.0} \quad (\varepsilon < \varepsilon_c)$$

$$d_{rex} = 2650 \ \ Z^{-0.1} \quad (\varepsilon \geq \varepsilon_c)$$

$$\varepsilon_c = 0.18 \ d_0^{0.3}$$

The results are shown in Figure 12 for setting the parameters, Figure 13 for the flow stress curves, and Figure 14 for the recrystallised grain size.
Fig. 12. AISI316 parameters modelling

Fig. 13. AISI316 strain/stress modelling
6.2. AISI304 Stainless Steel

The model has been utilised to model the AISI30 material which the following equations are obtained:

Activation Energy \((Q)\) = 266 kJ/mol

Zener Holomon Constant = strain rate \(Q/RT\)

\[ R = 8.31 \]

\[ T = \text{Temperature in Kelvin} \]

\[ \sigma = \sigma_0 + (\sigma_{ss(c)} - \sigma_0)(1-\exp(-\epsilon/\epsilon_r))^{0.5} \]

\[ \epsilon_r = 4.0095 \ Z^{-0.064} \]

\[ C = -10\ln\left[1 - \left(\frac{\sigma_{01} - \sigma_0}{\sigma_{ss} - \sigma_0}\right)^2\right] \]

\[ \sigma_0 = 0.01\log(Z) - 0.0 \]

\[ \sigma_{01} = 1.2843 \log(Z) - 5.779 \]

\[ \sigma_p = 5.45481\log(Z) - 29.351 \]

\[ \sigma_{ss} = 12.591 \log(Z) - 40.629 \]

\[ \sigma_{ss} = 12.921 \log(Z) - 48.834 \]

\[ \sigma' = 1.0(\sigma_0 + (\sigma_e - \sigma_0)(1-\exp(-C\epsilon))^{0.5}) \quad (\epsilon < 0.7\epsilon_p) \]

\[ \sigma = \sigma' - 1.0(\sigma_e - \sigma_{ss}) \left\{ 1 - \exp\left[-0.49\left(\frac{\epsilon - 0.7\epsilon_p}{\epsilon_p}\right)^{1.4}\right] \right\} \quad (\epsilon \geq 0.7\epsilon_p) \]
\[ d_{\text{re}} = 23 \ Z^{-0.1} d_0^{0.13} \varepsilon^{-1.0} \] \quad (\varepsilon < \varepsilon_c)

\[ d_{\text{re}} = 132 \ Z^{-0.1} \] \quad (\varepsilon \geq \varepsilon_c)

\[ \varepsilon_c = 0.18 \ d_0^{0.3} \]

The results are shown in Figure 15 for setting the parameters, Figure 16 for the flow stress curves, and Figure 17 for the recrystallised grain size.
6. DISCUSSION

The engineering stress-strain curves of both 316 and 304 specimens are shown in Figures 13 and 16 respectively. It can be seen that 304 sample experience lower tensile mechanical properties compared to the 316 sample. For both specimens, the behaviour of the materials indicate that strain hardening occurs throughout the duration of the stress application. However, the amount of strain hardening for the observed increment in the stress decreases as stress increases.

The results show that the yield stress (σ) and Young’s modulus (E) are improved due to the grain refinement in comparison to the raw sample. In cold-work conditions, crystalline defects like dislocations and porosity will increase with the degree of deformation and decrease the mechanical properties. Furthermore, the sample size can affect the mechanical tests results. The combined effect of crystalline defects and the size of tested samples has an influence on the reduction of mechanical properties.
7. CONCLUSIONS
The investigation carried out in this study on the hot deformation behaviour and microstructural evolutions of the 316 and 304 austenitic stainless steel, it was observed that the static recrystallization curves have led to work hardening and recovery, but not dynamic recrystallization, generally conform to recrystallization kinetics. The power law tends to decrease for coarse initial grain sizes. This is attributed to non-uniform deformation across the grains in these materials. Furthermore, recrystallization times and recrystallized grain size decrease with decrease in initial grain size, with increase in strain and to a lesser extent with increase in strain rate and decrease in temperature of deformation. Recrystallization time also decreases rapidly with increase in deformation temperature. The effects of these variables on both recrystallization time and recrystallized grain size can be satisfactorily described by relatively simple parametric equations for the entire range of conditions studied experimentally. Finally, at high strain rate and temperature, the true stress/strain curves exhibited tiny serrations effects due to the interaction of the recrystallization and precipitation.

ACKNOWLEDGEMENTS
The authors acknowledge with gratitude King Abdulaziz City for Science and Technology (KACST), Saudi Arabia for the financial support (project no.: 10-ADV1247-10) and the Science and Technology Unit (STU) at Umm Al-Qura University for the logistic support throughout the project.

REFERENCES