Dynamic Recrystallization under Hot Deformation of a PH Stainless Steel

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Abstract
Dynamic recrystallization, DRX, behaviour of a precipitation hardened, PH, stainless steel was studied in connection with microstructural developments in a compression test. The experimental results showed that the dominant mechanism of softening is DRX, but at high strain rates and low temperatures, i.e., high Zener-Holman parameter, Z, work hardening and dynamic recovery, DRV, produced a pancaked structure. When Z values decreased, the flow curves displayed DRX in two ways: single peak behavior observed at low Z values and multiple peak behavior at the lowest ones. In addition, the peak strain, εp, necessary for DRX is also determined as a function of peak stress, σp, and Z. According to the observed and calculated data, at low σp values, εp is directly proportional to σp, while in higher σp values, εp rarely depends on it. Besides, εp exponentially depends on Z.

Keywords: Dynamic recrystallization; Hot compression; PH stainless steel.

Introduction
Precipitation hardened, PH, stainless steels have become increasingly important in a variety of applications. The most important of these properties are ease of fabrication, high strength, relatively good ductility, and good corrosion resistance. One of the ways of acquiring the best combination of these properties is to select the microstructure, which in turn depends on thermomechanical history as well as on chemical composition. An optimization of the thermomechanical process can be achieved through an understanding of the entire forming process and the metallurgical variables affecting the microstructural features occurring during deformation operations carried out at high temperatures. Most applications of these alloys are in the chemical process equipment, petrochemical, aerospace industry, and medical tools.

Hotworking behaviour of these alloys is generally reflected on flow curves which are a direct consequence of microstructural changes: the generation of dislocations, work hardening, WH, the rearrangement of dislocations, their self-annihilation, and their absorption by grain boundaries, DRV, the nucleation and growth of new grains, DRX. The latter is one of the most important softening mechanisms at high temperatures, which occurs after a critical strain, εc.

This is a characteristic of low and medium stacking fault energy, SFE, materials e.g., γ-iron, the austenitic stainless steels, and copper. After εc has been attained, the flow stress goes through a maximum which is associated with the development of DRX up to a steady state regime. This can be considered as an equilibrium state between the softening and hardening mechanisms.

Several DRX mechanisms have been proposed by many researchers. The bulging of the initial grain boundaries during hotworking has shown a mechanism of dynamic nucleation. This mechanism can explain the strain softening under DRX. However, the details of new grain formation are still not clear in connection with separation from the parent grains followed by the evolution of equiaxed grains. Another possible mechanism associated with DRV in high density dislocation substructure is the transformation of low angle to high angle grain boundaries with increase in strain. These mechanisms do not seem to cover a wide range of materials and deformation conditions.

The optimum processing condition is defined as the temperature and strain rate combination at which the highest efficiency is achieved, and both mechanical and thermodynamic stability criteria are satisfied. This optimum condition provides the most stable microstructure and material flow in metalforming processes.

The aim of the present work is to evaluate the DRX behavior of a PH stainless steel in compression test at various temperatures and strain rates. The effect of deformation conditions on the operating mechanisms is also studied with microstructural changes.
Experimental procedure

The specimens for compression tests, with 15 mm length and 10 mm diameter, were cut from ingot of 150 mm diameter. The chemical composition of the PH stainless steel is given in Table 1. Compression experiments were carried out in a computer controlled servo-hydraulic Instron machine equipped with an infrared furnace. The samples were deformed in argon atmosphere. Before the compression tests, samples were homogenized at 1220 °C for 30 min and quenched in water. The specimens were pre-heated at 1150 °C for 15 min and cooled to testing temperature at a rate of 2° C/s. All samples were kept at the test temperature for 2 min prior to loading in order to homogenize their temperatures. Tests were performed at temperatures of 850-1150°C and strain rates of $10^2$-$10^3$ s$^{-1}$.

Immediately after each compression test, the specimens were quenched in water in order to retain the deformed microstructure. The specimens were sectioned longitudinally, and the microstructures were examined by an optical microscope. For revealing austenite grain boundaries, specimens were etched with the solution of HF 20 cc + HNO$_3$ 10 cc + Glycerin 20 cc.

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>Ti</th>
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<tbody>
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<td>0.02</td>
<td>10.21</td>
<td>10.11</td>
<td>5.06</td>
<td>1.96</td>
<td>1.05</td>
</tr>
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</table>

Table 1. Chemical composition of the steel investigated (wt%).

Results and discussion

The analysis of stress-strain curves:

A series of typical stress-strain curves ($\sigma$-$\varepsilon$) of the tested steel is shown in Figure 1. The specimens were compressed at various temperatures from 850-1150 °C under a strain rate of about $10^2$-$10^3$ s$^{-1}$. The flow curves showed three types of behavior: flow hardening, flow softening and steady state flow. At the highest strain rate, $10^3$ s$^{-1}$, and at the temperature of 950°C, some dynamic recovery and the onset of dynamic recrystallization through necklacing occurred. On the other hand, the lowering of work hardening rate after peak stress was due to the continued flow softening. Figure 1a. As the temperature was decreased at a fixed strain rate, a steady state flow behavior was obtained with delay. This trend of increased conventional DRX through polygonization which was followed by nucleation of strain-free grains within the prior deformed grains, were more pronounced at the strain rate of $10^2$ s$^{-1}$. Such a behavior has been shown to be typical for DRX taking place under hot deformation.$^{58}$

Figure 2 shows plots of Log ($\sigma_p$) versus 1/T for various values of strain rates, $\dot{\varepsilon}$. As can be seen, by decreasing the temperature, the peak stress is increased. Peak stress versus strain rate was plotted in log-log scale and is shown in Figure 3. The slope of the plots gives the strain rate sensitivity "m", which is shown to be dependent on both temperature and strain rate. Higher values of m represent more uniform deformation. The m values are higher at lower strain rates and higher temperatures. This is expected since lower strain rates allow more time for deformation accommodation and higher temperatures make plastic deformation easier.
Fig. 1. Stress–strain curves for deformed samples at strain rates of (a) $10^{-3}$ s$^{-1}$, (b) $10^0$ s$^{-1}$, (c) $10^1$ s$^{-1}$, (d) $10^2$ s$^{-1}$, (e) $10^3$ s$^{-1}$ and several temperatures.

Fig. 2. Relationship between peak stress and temperatures for deformed samples at various strain rates.

Fig. 3. Relationship between peak stress and strain rate for deformed samples at various temperatures.

The evaluation of deformed microstructure:
The microstructures of deformed samples are shown as a function of strain rate and temperature at 0.7 strains in Figure 4 and Figure 5. At the temperature of $850 \, ^\circ C$ and strain rate of $10^3$ s$^{-1}$, dynamic recovery, occurred in a pancaked microstructure, and then the onset of dynamic recrystallization through necking took place. The fine grains nucleated at prior grain boundaries and twin boundaries continued to provide deformation accommodation, Figure 4a. As the temperature was increased to $950 \, ^\circ C$ at the same strain rate, and after a rise and drop in stress, a steady state flow behavior was observed. Closer examination of Figure 4b reveals that the nucleation of strain-free grains, conventional DRX, generally occur in heavily deformed grains. At this strain rate, the deformation accommodation through necking alone was not fast enough, and the accommodation was much more dependent on dislocation activity and polygonization.

In this case, the rate of softening compensated the rate of hardening, and steady state flow was formed. This trend of increased conventional DRX through polygonization followed by nucleation of strain-free grains within the prior deformed grains, was more evident at $1050 \, ^\circ C$ temperature, Figure 4c. Evidence of enhanced conventional recrystallization and grain growth was observed with increasing temperature such as $1150 \, ^\circ C$ and $10^3$ s$^{-1}$, Figure 4d. In precipitation strengthened structures, the dislocations are pinned by carbides and necking is the main mechanism of recrystallization, which is different from the solution strengthened alloys.

Typical microstructures developed under $1050 \, ^\circ C$ at various strain rates are shown in Figure 5. It is clearly seen that the features of evolved microstructures are sensitive to change of strain rate which can result from the transient behaviors from low to high strain rates.

This would explain why during cyclic dynamic recrystallization at lowest strain rate of $10^{-3}$ s$^{-1}$, the final grain size is larger than the grain size of the deformed sample at strain rate of $10^3$ s$^{-1}$, relatively few nuclei grew unhindered, Figure 5a. While dynamic recrystallization of multi peak promotes a finer grain size, large numbers of nuclei grow mutually limiting grain boundary mobility. By increasing strain rate, the size of grains nearly becomes equal, Figure 5b and Figure 5c. Increasing the strain rate further leads to the reduction of both the dynamic grain size and volume fraction of new grains, Figure 5c. It can be concluded that low temperature or high strain rate leads to the evolution of pancaked grain structure accompanied with minor serration of initial grain boundaries, Figure 5d.

Effect of dynamic recrystallization on flow softening:
During the present work, attempts were made to model the flow softening caused by DRX. This would enable to correlate the $\varepsilon_p$ to $\sigma_p$ and $Z$. The relationship between the peak strain, $\varepsilon_p$ and the peak flow stress, $\sigma_p$, is shown in Figure 6. The $\varepsilon_p$ is directly proportional to the $\sigma_p$ with a factor of about $2.3*10^{-3}$ MPa$^{-1}$ in the region of $\sigma_p<370$ MPa, while
the $\varepsilon_p$ hardly depends on the $\sigma_p$ in the region of $\sigma_p > 370$ MPa and $\varepsilon_p = 0.65$.

At low stress and strains, little recrystallization takes place. This occurs at high temperatures and low strain rates. On the other hand, when temperature is lower or the strain rates are high, the necessary stress-strains for DRX will noticeably increase. At high stresses, the effects of stress on critical strain decreases. In this study, at stresses and strains respectively higher than 370 MPa, and 0.65, no additional effect on strain was observed.

The kinetics and critical conditions of DRX taking place under hot deformation are sensitive to temperature and strain rate as well as the initial microstructures \(^\text{15,16}\). The deformation conditions are usually expressed in terms of temperature compensated strain rate \(^\text{15}\).

\[
Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = A (\sinh (\alpha \sigma_p))^n \tag{1}
\]

Where, $Z$ is the Zener-Hollomon parameter, $\dot{\varepsilon}$ is strain rate, $Q$ is an activation energy, $R$ is the gas constant, 8.31 J (mol K\(^{-1}\)), $T$ is absolute temperature, $A$, $\alpha$ and $n$ are constants, and $\sigma_p$ is the peak stress. With regard to the activation energy for deformation, in the region of hot deformation it is possible to calculate it by using the following equation \(^\text{16}\):

\[
Q = 2.3 R \left[ \frac{\partial \log \dot{\varepsilon}}{\partial \log \sigma_p} \right] \left[ \frac{\partial \log \sigma_p}{\partial (1/T)} \right] \dot{\varepsilon} \tag{2}
\]

The activation energy employed in this work was 432 kJ mol\(^{-1}\). This value corresponds to the typical activation energies reported \(^\text{2}\) for austenitic stainless steels, 400-500 kJ mol\(^{-1}\).

Fig. 4. Effect of temperature on microstructures developed in deformed samples to strain rate of $10^4$ s\(^{-1}\) and strains above 0.7: (a) 850 °C, (b) 950 °C (c) 1050 °C (d) 1150 °C.

Fig. 5. Effect of strain rate on the microstructures developed in deformed samples to temperature of 1050 °C and strains above 0.7. (a) $10^4$ s\(^{-1}\), (b) $10^5$ s\(^{-1}\), (c) $10^6$ s\(^{-1}\), (d) $10^7$ s\(^{-1}\), (e) $10^8$ s\(^{-1}\).
The critical strain $\varepsilon_p$ for the onset of dynamic recrystallization is usually used to describe this softening mechanism. Although it is recognized that dynamic recrystallization starts at a value close to 70% of the strain, $\varepsilon_p$ associated with the maximum stress, most authors associate the critical strain with the easy determination, if the initial grain size is kept constant:

$$\varepsilon_p = KZ^m$$ (3)

Where the parameters $K$ and $m$ depend on the alloy composition. In the present study, the critical strain was determined for every flow curve in which a peak stress was clearly apparent. Figure 7 shows how well the present data fit the above expression. The value 0.138 for $m$ in Eq. (3) is within the range of 0.12 to 0.14 reported by Roberts for stainless steels. The amount of $K$ is measured as $3.52 \times 10^{-4}$.

Conclusions

1- The main softening mechanism in the investigated steel during hot deformation is dynamic recrystallization, which is revealed in flow curves as single peaks and multiple peaks at low $Z$ values.

2- At the highest $Z$ value, only dynamic recovery and work hardening take place, and a pancake structure is formed.

3- In the region of flow stresses lower than 370 MPa, the deformation behaviors are typical for hot working and are accompanied by DRX. The peak strain, $\varepsilon_p$, where peak stress, $\sigma_p$, appears increases with $\sigma_p$.

4- According to $\sigma$-$\varepsilon$ results and the associated microstructures, a relationship between these parameters was established which allowed the prediction of $\varepsilon_p$ for the occurrence of DRX, by the known amounts of $\sigma_p$ and $Z$.

References