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Dynamic Recrystallization of Austenitic Stainless Steel under Multiple Peak Flow Behaviours

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Abstract
The hot working behaviour of a Fe-0.02C-8.2Ni-18.5Cr (mass%) austenitic stainless steel was studied at high temperatures and low strain rates. These conditions led to a multiple peak stress-strain curve, where the second peak had a much larger strain and stress than the first peak. The first peak showed evidence of some grain refinement, indicative of conventional dynamic recrystallization. However, after this there was significant grain coarsening, similar to abnormal grain growth, with the formation of substructure within the grains. The strain to the first peak in the multi peak tests was much lower strain than expected from extrapolation of the single peak data.

Keywords: Stainless steel; dynamic recrystallization; EBSD; grain coarsening; multiple peaks.
1. Introduction

Dynamic recrystallization (DRX) is one of the most important restoration mechanisms during hot deformation of steels in their austenitic condition. The occurrence of DRX usually results in a single peak in the flow curve of deformed material. However, a transition from single peak to multiple peak behaviour has been reported under certain deformation conditions (very low strain rate and high temperature) and/or initial microstructures (small grain sizes) \(^{1-3}\). Despite several studies of DRX in austenite exhibiting multiple peaks \(^{2, 4-11}\), many details remain unclear. The critical conditions for transition from single to multiple peaks, the microstructural development and also grain size evaluation under multiple peaks behaviour, are examples of issues that have not been completely resolved.

Two different criteria have been proposed to characterize the transition from single to multiple peak flow curves. According to the first criterion, proposed by Luton and Sellars \(^6, 7\), the transition from multiple to single peak behaviour is associated with the ratio of the peak strain, \(\varepsilon_p\), to the recrystallization strain, \(\varepsilon_x\), (the strain equivalent to the time for a large fraction of recrystallization to occur). According to this model, when the peak strain is larger than the recrystallization strain (i.e. \(\varepsilon_p > \varepsilon_x\)), the flow curve will show multiple peaks. In contrast, the single peak behaviour appears when \(\varepsilon_p < \varepsilon_x\). In a different study using a similar approach, the peak strain was replaced by the critical strain to initiate DRX, \(\varepsilon_c\), \(^{12}\). However, it has been proposed that this criterion is mostly applicable for torsion tests and is unlikely for compression and tensile tests \(^1\).

In the second criterion, proposed by Sakai and his co-workers \(^1, 13, 14\), each type of flow curve represents a specific mechanism of recrystallization. According to this
model, the single peak flow curve reflects a growth controlled mechanism (i.e. grain refinement) and usually the initial grain size \(d_0\) is greater than twice the steady state DRX grain size \(d_{ss}\). In contrast, the multi peak flow curve is associated with a nucleation control and impingement mechanism (i.e. grain coarsening) and also \(d_0 < 2d_{ss}\). The single peak behaviour is usually associated with dynamic recrystallization based on the formation of a necklace structure. However, the necklace mechanism cannot take place during grain coarsening and the growth of each new grain is stopped by grain boundary impingement with other recrystallizing grains \(^1\). Several different studies \(^2, 15\) have been shown that this criterion in not always valid for all materials and the Luton and Sellars model can predict the flow curve behaviour more accurately.

Due to the importance of grain refinement in industrial hot processes, the critical conditions which cause a transition from single to multiple peaks, has a practical importance. The aim of this study was to evaluate the critical deformation condition for transition from single to multiple peaks and also characterize the microstructure evolution under multiple peak behaviours. The microstructure analysis was supported by crystal coordinate maps to study the texture change during dynamic recrystallization.

2. Experimental procedures

A 304 austenitic stainless steel with chemical composition (mass\%) of Fe-0.02%C-1.6%Mn-8.2%Ni-18.5%Cr-0.8%Cu was used in this study. Torsion samples with a gauge length of 20 mm and a diameter of 6.7 mm were machined from hot rolled bars. The detailed specification of test equipments and torsion samples have been described elsewhere \(^16\). Hot torsion tests were carried out according to the schedule illustrated
in Fig. 1. A roughing process at 1200 °C was used to achieve a homogenized microstructure with an average grain size of ∼35 µm. The samples, then, were deformed at this temperature to different strains at strain rates of 0.001 and 0.01 s⁻¹, or cooled to 1100 °C before deformation at the same strain rates. The deformed samples were quenched immediately after hot deformation.

Metallographic observations were performed on tangential sections at a depth of ∼100 µm below the gauge surface. The microstructures of the mechanically polished surfaces were examined by optical microscopy (after electroetching in Nitric acid) and also by Electron Back Scattered Diffraction (EBSD) under an accelerating voltage of 20 kV, a working distance of 25 mm from the gun and an aperture size of 60 µm. The EBSD maps were analysed using HKL technology channel 5.

The average grain sizes were measured using the linear intercept method and their distribution was plotted based on a geometric scale with spacing of \sqrt[10]{10}$, and a total of 15 intervals.

3. Results and discussion

Our previous studies on AISI 304 stainless steel have been showed that at a constant strain rate of 0.01 s⁻¹, increasing the deformation temperature above 1100 °C, resulted in faint second peaks in the flow curves. The more obvious multiple peak flow curves occurred when the strain rate was decreased and/or the temperature increased (Fig. 2). However, at a strain rate of 0.01 s⁻¹ and a temperature of 1100 °C, a very typical shape of DRX flow curve (i.e. a sharp peak followed by a long steady state region) was observed. In all other deformation conditions studied here, multiple peaks were observed in the flow curves. A characteristic feature of the multiple peaks in Fig. 2 is that at a very low strain rate of 0.001 s⁻¹, the stress of the second peak is
higher than the first peak. This indicates a significant work hardening after the first DRX cycle. The exact reason for such increase in the peak stress will be discussed later using the results from the microstructure characterization.

To study the flow curve behaviour under different deformation conditions, the peak strain, $\varepsilon_p$, (the first peak for multiple peaks conditions) was measured over a wide range of deformation conditions ($T= 650-1200 \, ^\circ C$ and $\dot{\varepsilon} = 0.001-10 \, s^{-1}$) and plotted as a function of Zener-Hollomon parameter (Fig. 3). This Figure showed a slope change at a critical Z value of $Z_c = 5 \times 10^{12} \, s^{-1}$. The flow curves shape showed multiple and single peaks for deformation under Z values lower and higher than $Z_c$, respectively. This break may suggest a significant change in the restoration mechanism of austenite. The breakdown of the power law behaviour implicit in this type of plot could also be another reason for this slope change. However, as here we are dealing with very low stresses, the power low breakdown is negligible.

To understand the microstructure characteristic of material deformed under a Z value lower than $Z_c$, the microstructures and substructures of samples deformed at $1200 \, ^\circ C$ and a strain rate of $0.001 \, s^{-1}$ were analysed by optical microscopy and EBSD mapping, respectively. The initial microstructure after the roughing process (Fig. 4a) consisted of equiaxed grains with a large fraction of twin boundaries and an average grain size of $35 \, \mu m$. EBSD analysis confirmed that more than 60% of the high angle grain boundaries in this microstructure have twin crystallography, indicating the important role of twining during hot deformation and recrystallization of this material.

After deformation to a low strain of 0.12, which is smaller than the first peak, no significant difference was observed in the microstructure (Fig. 4b). However, it
seems that a limited number of new small DRX grain were formed predominantly at triple junctions. Also, EBSD analysis showed that some twin boundaries lost their twin crystallography and changed to high angle grain boundaries.

As deformation increased and reached the first peak strain (i.e. $\varepsilon = 0.2$), more new grains were formed in the microstructure (Fig. 4c). At the same time, some of the initial grains coarsened and, therefore, an inhomogeneous grain size distribution developed. Overall, the average grain size showed a slight decrease. Further deformation to the end of the first DRX cycle, where a minimum appeared after the peak in the stress strain curve (i.e. $\varepsilon = 0.35$), led to a remarkable change in the microstructure (Fig. 4d). Here it consisted of very coarse grains, some grains close to the initial grain size (or even larger) with very few of the fine grains. It appears that the coarse grains have consumed the fine grains and decreased the frequency of these fine grains.

When the strain is increased to 0.5, the coarse grains in the previous microstructure grew and abnormal growth appeared to be occurring for some grains (Fig. 4e). Therefore, deformation is now leading to grain growth rather than grain refinement, as also reported by others\(^1, 3\) during deformation under multiple peak conditions. However, an interesting result of the present work is the limited grain refinement at the first peak (although it is accompanied by coarsening of some selected grains) and then coarsening of grains with further deformation.

The sample deformed to the second peak strain ($\varepsilon = 0.6$) showed the formation of a very limited number of new small DRX grains mostly on the triple junctions (arrows in Fig. 4f) accompanied by more abnormal grain growth. At $\varepsilon = 1.0$, a microstructure
with very large grains was produced (Fig. 4g) and further deformation showed no major change in the microstructure (Fig. 4h).

As is clear from Fig. 4, deformation under this condition caused heterogeneity in the microstructure development and also the microstructure is very different to the single peak behaviour 19). New DRX grains were formed only on a few grain boundaries and other grain boundaries were not serrated and also not covered by DRX grains, but continued their migration (grain growth). The mean grain size change, confirms these observations with an oscillation was observed during the deformation (Fig. 5). After the first peak, a slight decrease in the average grain size was observed compared to the initial microstructure. This indicates the progress of the conventional DRX (i.e. grain refinement), which is observed at least in some parts of the microstructure (Fig. 4). With an increase in the strain beyond the first peak, significant grain coarsening occurred in the microstructure. This grain coarsening increased slightly with further deformation and reached a steady state at higher strains.

The grain size distribution graphs (Fig. 6) more easily show the inhomogeneity in the microstructure of the multiple peak samples. This figure shows that while the initial microstructure has a normal distribution of grains (with an average of 35 µm), deformation caused expansion in the grain size distribution. This expansion was increased by increasing strain and the peak also moved to the larger grain sizes. However, at a very high strain of 2.0, a significant fraction of large grains with an average size of 80 µm appeared in the graph (Fig. 6), indicating remarkable grain coarsening.

The heterogeneity in the DRX microstructure could also cause an in-homogeneity in the strain distribution during further deformation. While the deformation of small
grains will result in sliding or serration of their boundaries, followed by formation of new DRX grains, the deformation of larger grains led to an increasing dislocation density and also formation of deformation features within the grains. This increase in the dislocation density inside the large grains can be considered as the main reason for the second peak being higher than the first peak in the stress-strain curve (Fig. 2). However, due to very high temperature and low strain rate, the dislocations inside these grains can move quickly to form low/high angle grain boundaries or sink in the existing grain boundaries. This high temperature also increases the migration rate of grain boundaries resulted in grain coarsening.

From the above observations, the main difference between DRX in the present study (i.e. with multiple peaks) and the single peak behaviour (e.g. 19, 20) was the major change in the DRX mechanism from grain coarsening to grain refinement. This change in the DRX mechanism caused a major slope change in the peak strain in Fig. 2. From this it appears that in the multiple peak behaviour the first peak strain was lower than expected by extrapolation of higher Z values (broken line in Fig. 3). The exact meaning of this is unclear, but it is important to remember that peak represents the point where the rate of work hardening balances the rate of softening from DRX. As the rate of work hardening is linked to Z it suggests that the rate of flow softening and hence the rate of dynamic recrystallization is faster than expected.

The crystal coordinate maps (Fig. 7) more clearly show the microstructure evolution; particularly the substructure within the grains. These maps show the significant change in the texture of this structure with increasing strain. While the initial microstructure showed a strong texture, it weakened with increasing strain to the first peak. Increasing the strain beyond the first peak, led to another strong texture
(possibly stronger than the initial microstructure) at the end of the first DRX cycle (Fig. 7d). The strong texture suggests that recrystallization has progressed by a continuous type of recrystallization (i.e. grain growth), rather than the nucleation and growth of many new grains which would tend to have a more random texture in austenite. It seems that beyond this point the texture becomes weaker and weaker, due to an increase in the deformation of the large grains without any significant fresh recrystallization.

Another interesting issue of microstructure development during hot deformation under multiple peak behaviour is the evolution of substructure, which is very different to the single peak behaviour. Under single peak behaviour, it has been shown that the frequency of low angle grain boundaries (LAGB’s) increases with increasing strain to the peak and then decreases continuously with increasing strain, due to the progress of DRX. However, under the present condition (i.e. multiple peaks) the frequency of LAGB’s slightly increased with increasing strain to the first peak and then a large increase was observed at the second peak (Fig. 8). This major change can be explained by the microstructure and flow curves. During the first DRX cycle (i.e. ε ≤ 0.35), where deformation caused a limited grain refinement, the generated dislocations have a high migration rate due to the high temperature, and can easily move to the grain boundaries. By the increasing strain beyond that point, where there is large grain coarsening (Fig. 4), the generated dislocations need to move a longer distance to reach the grain boundaries. Therefore, deformation will cause an increase in the dislocation density within the coarse grains, resulting in an increase in the flow stress (Fig. 2). While DRX is stagnant, dynamic recovery (i.e. formation of LAGB’s) is occurring, which results in the increase in LAGB frequency (clear evidence of a cell structure within the large grains can be seen in Figs. 7g and h). However, when
the dislocation density increases to a critical value, a new cycle of DRX can start. The second peak in the flow curve is the result of this new cycle of DRX. As is clear in Fig. 2, the strain associated with the second peak is almost three times larger than the first peak strain, indicating the much slower DRX kinetics.

4. Conclusions

Multiple peak behaviour was observed in a 304 austenitic stainless steel during hot deformation under any $Z$ lower than a critical value of $5 \times 10^{12}$ s$^{-1}$. The first stress peak of dynamic recrystallization below this critical $Z$ value occurred at lower strain than expected from extrapolation of the single peak data. Conventional dynamic recrystallization (i.e. grain refinement) and migration of high angle grain boundaries (i.e. grain coarsening) are shown to be the dominant phenomenon for the first and second peaks, respectively. This caused an oscillation in the mean grain size as a function of strain, with a minimum value at the strain for the first peak. A significant change in the texture was also observed during deformation, with a strong texture at the end of the first dynamic recrystallization cycle and a very weak texture at the fracture strain.
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Fig. 1  Schematic diagram of hot torsion tests

Fig. 2  Flow curves of samples deformed under different deformation conditions

Fig. 3  Peak strain as a function of Zener-Hollomon parameter
Fig. 4 Optical micrograph of samples deformed at 1200 °C and a strain rate of 0.001 s\(^{-1}\) to different strains of a) 0, b) 0.12, c) 0.2, d) 0.35, e) 0.5, f) 0.6, g) 1.0 and h) 2.0

Fig. 5 Average grain size as a function of strain (T = 1200 °C and \(\varepsilon = 0.001 \text{ s}^{-1}\))
Fig. 6  Grain size distribution of samples deformed to different strains (T = 1200 °C and $\dot{\varepsilon} = 0.001\text{s}^{-1}$)
**Fig. 7** Crystal coordinates maps of samples deformed at 1200 °C and a strain rate of 0.001s\(^{-1}\) to different strains of a) Zero, b) 0.12, C) 0.2, d) 0.35, e) 0.5, f) 0.6, g) 1.0 and h) 2.0

**Fig. 8** Misorientation distribution of samples deformed at different strains (T = 1200 °C and \(\dot{\varepsilon} = 0.001 \text{ s}^{-1}\).