Fine-Grained Structure Formation in Austenitic Stainless Steel under Multiple Deformation at 0.5 $T_m$

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Fine-Grained structure formation taking place in a 304 type austenitic stainless steel was studied in multiple compression at a temperature of 873 K (0.5$T_m$) under a strain rate of about $10^{-3}$ s$^{-1}$. The integrating flow curve shows a maximum in flow stress at strains around 1.5 followed by a minor strain softening at cumulative strains beyond 3. The structural changes during deformation can be characterized by the evolution of elongated subgrains with their boundaries as dense dislocation walls at low to moderate strains. These subgrains become more equiaxed and the subboundary misorientations gradually increase with strain, finally leading to the development of fine-grained structure with misorientations beyond 20° and an average grain size of about 300 nm. Some of the newly evolved grains contain much lower dislocation densities in their interiors than those evolved at early strains. The mechanisms of such structural development as well as the relationship between microstructures and mechanical properties are discussed in detail.

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1. Introduction

Very fine-grained structures in metals and alloys have some advantages for the improved mechanical properties and so have aroused great interest among researches in materials science. The studies on grain refinement under thermomechanical processing are of specific practical importance, since the fine grain evolution can be achieved by plastic deformation and recrystallization in many metallic materials which do not show polymorphism. New grain formation taking place during hot deformation is usually associated with operation of dynamic recrystallization (DRX). The dynamic grain size sensitively depends on deformation conditions, therefore, ultrafine grains should be evolved by decrease in deformation temperature.$^{1,2}$ On the other hand, the strain required for the initiation of DRX drastically increases with decreasing temperature. The studies on structural changes taking place at high strains are often difficult because of limited workability of most metallic materials at low-to-moderate temperatures. Recently, several techniques have been proposed to provide very high plastic strains, including torsion under high pressure, equal-channel angular pressing, multiple forging, etc.$^{3-13}$ Multiple forging seems to be a simple method, because neither high working nor special costly equipment are required.

It has been shown in recent works on severe cold and warm deformations$^{3,4,9-10}$ that the structural changes are characterized by the formation of strain-induced dislocation subboundaries, such as dense dislocation walls and microbands. These subboundaries having high angle misorientations can rapidly transform to usual grain boundaries at high strains.$^{11}$ The new grain formation, therefore, results from a strain-induced continuous reaction assisted by dynamic recovery, which is sometimes called as continuous DRX$^{11,12,16}$ However, the characteristics of fine grains evolved through severe deformation and the interrelation between mechanical behaviors and strain-induced microstructures have not been fairly clarified.

The aim of the present work is to study the warm deformation behavior and the structural mechanisms responsible for the evolution of fine grains in a 304 type austenitic stainless steel. The samples were deformed to high strains above 6 in multiple interrupted compression at 873 K (0.5$T_m$), which is considered as a kind of multiple forging.$^{16}$ The dynamic mechanisms of new grain evolution as well as the relationship between the developed microstructures and the mechanical properties at room temperature are discussed in detail.

2. Experimental Procedure

A 304 type austenitic stainless steel (C:0.058, Si:0.7, Mn:0.95, P:0.029, S:0.008, Ni:8.35, Cr:18.09, Cu:0.15, Mo:0.13, all in mass%, and the balance Fe) with an initial grain size of about 25 μm was used as the samples. For multiple compression, the samples were machined in a rectangular shape with the starting dimension of 9.8 mm × 8.0 mm × 6.5 mm. Such ratio of about 1.5 × 1.22 × 1 was maintained the same during subsequent compressions to a strain of 0.4 in each pass. Multiple compression tests were carried out with consequent changing of the loading direction in 90° through three of mutually perpendicular axes (i.e. x to y to z · · · ) from pass to pass. The samples were compressed at 873 K (0.5$T_m$) in vacuum under a strain rate of about 10$^{-3}$ s$^{-1}$ with boron nitride powder as a lubricant. The deformed samples were quenched in water, slightly ground to a rectangular shape, and then reheated to 873 K within 0.6 to 0.8 ks in each deformation pass. Some of deformed specimens were polished and then recompressed to a strain of 0.1 for observations of deformation relief.

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Metallographic analyses were carried out using an optical microscope and a JEM-2000FX transmission electron microscope (TEM) operated at 200 kV. Microstructural, topographical and TEM observations were carried out on the sections parallel to the compression axis. Misorientations of strain-induced (sub)grain boundaries were studied using a conventional Kikuchi-line technique\(^{17}\) with accuracy within one degree. The subboundary misorientations were determined from at least four typical and different areas of thin foils prepared from two deformed specimens. All of the visible boundaries between subgrains was studied in the areas including ten-to-fifteen grains. Total number of the analyzed boundaries was 80 ± 20 at each strain. The lattice curvatures developed inside new grains were studied by analysis of a component for the rotation vector lying in the plane of electron diffraction pattern within 0.1° accuracy.

3. Results

3.1 Warm deformation behavior

Figure 1(a) shows the stress ($\sigma$) vs cumulative strain ($\Sigma \varepsilon$) curves for sixteen passes of compression to a total strain of 6.4. The flow stresses rapidly increase in about three times of the first compression and approach a saturation value in subsequent deformation passes. Strain hardening taking place in each pass becomes quite small after the third compression. The integrated flow curve shows a maximum and an apparent steady-state flow at cumulative strains of around 1.5 followed by a minor work softening at higher strains. Such a shape of the integrated flow curve can be roughly similar to that appearing under operation of dynamic recovery. It is remarkable in Fig. 1 that the yield stresses at reloading are quite different from that for an annealed state in the first pass, and roughly similar to the stresses immediately before unloading after the second pass. Such behavior may suggest that any static annealing effect hardly took place at $0.5T_m$ for a period of reheating time under interrupted tests (i.e., 0.6 to 0.8 ks).

The relationship between the room temperature hardness ($H_v$) for the samples compressed and the cumulative strain is depicted in Fig. 1(b). It can be seen in Fig. 1(b) that the strain dependence of $H_v$ is almost similar to that of flow stresses at strains up to 3 under warm deformation. Under high strains from 3 to 6.4, $H_v$ shows slight hardening, while the warm flow stresses continuously decrease. These results will be discussed in the subsequent sections.

A typical deformation relief appearing at an early warm deformation is represented in Fig. 2(a). It can be seen that there are fine and wavy slip lines and also some grain boundaries evolved, as indicated by arrow heads. The appearance of grain boundaries suggests the operation of grain boundary sliding and/or shearing taking place along them even at a relatively low temperature of 0.5$T_m$. It should also be noted that some boundaries give high contrast image, whereas the others are not clearly visible. This may suggest that the sliding takes place inhomogeneously at each grain boundary, which can bring about various local strain gradients along them. In order to study the deformation relief at high strains, a sample previously deformed to 6.4 was quenched, polished and then deformed again to an additional strain of 0.1. Figure 2(b) shows the deformation relief on the surface of a highly deformed sample. This relief is quite different from the ones appearing at early deformation (Fig. 2(a)) and looks like a powdered aggregate with a granule size below 1 µm. This suggests that a very fine grained structure may be evolved under severe deformation.

![Fig. 1](image1.png)  
**Fig. 1** (a) True stress ($\sigma$) vs cumulative strain ($\Sigma \varepsilon$) curves for 304 stainless steel under multiple compression at 873 K and at a strain rate of about $10^{-3}$ s$^{-1}$. (b) Effect of the cumulative warm deformation on the room temperature hardness for the samples tested in (a).

![Fig. 2](image2.png)  
**Fig. 2** Deformation relief for 304 stainless steel strained to (a) $\varepsilon = 0.1$ and (b) $\Sigma \varepsilon = 6.5$ at 873 K. The sample in (b) was pre-deformed to a total strain of 6.4, unloaded, repolished, and then deformed again to $\varepsilon = 0.1$. 
3.2 Structural changes during multiple warm deformation

Typical microstructures evolved under warm multiple deformation to various strains are shown in Fig. 3. Early deformation leads to the evolution of pancake shaped grains with their irregular boundaries, as shown in Fig. 3(a). Further multiple deformation to moderate strains above 1 brings about more distinguishable effect on microstructure, that is serrated grain/twin boundaries and very fine grains developed near the boundaries. The average grain size is about 5 μm in Fig. 3(b), which is one fifth of the initial size. With further deformation to high strain, the microstructural analysis becomes more complicated because of high density irregular grain boundaries and these grain structures cannot be clearly revealed under light microscopy. It should be noted in Fig. 3(c), however, that a lot of very fine grains with size of below 1 μm is evolved by severe warm deformation to 3.2.

Figures 4 and 5 present the substructures evolved at an early stage of rapid work hardening. The first compression to a strain of 0.4 leads to the development of highly dislocated substructures, in which most dislocations are homogeneously arranged in rich dislocation layers (Fig. 4(a)). The misorientations between such layers are 1–3°. The second compression brings about the evolution of dense dislocation walls (DDWs) with low-to-middle angle misorientations, which cross the rich dislocation layers (Fig. 4(b)). Another example of the substructures evolved near an initial grain boundary is shown in Fig. 5. It can be clearly seen here that the subboundaries with medium-to-high angle misorientations are frequently formed near the initial grain boundary. They give a sharp contrast on TEM image, which is typical of ordinary grain boundaries. This suggests that local lattice rotations can progressively take place along grain boundaries, leading to the formation of dislocation subboundaries with high misorientations. These subboundaries increase the average subboundary misorientation up to around 6° at a cumulative strain of 0.8.

Further multiple deformation to a total strain of 1.6 evolves the recognizable substructure structures, as shown in Figs. 6 and 7. Figure 6 shows high density dislocation substructures developed near a triple junction of original grain boundaries. These subgrain boundaries have medium-to-high angle misorientations up to 17.2°. Furthermore, highly misoriented substructures are formed in a form of chains of elongated subgrains in the vicinity of initial grain boundaries, as presented in Fig. 7. It can be stressed here that some of these subgrains contain quite few dislocations in their interiors and their neutron boundaries have medium-to-high angle misorientations. The volume fraction of such substructures is about 30 ~ 40% after multiple straining to 1.6. The average size of
Fig. 5  (a) Strain-induced subboundaries developed in vicinity of a grain boundary (GB) in 304 stainless steel during warm multiple deformation to $\Sigma e = 0.8$. The numbers indicate the misorientation in degrees. (b) Medium angle dislocation subboundaries evolved near the serrated grain boundary. (c) and (d) Subboundaries with low-to-middle angle misorientations developed in grain interiors.

Fig. 6  Typical substructures with high density dislocations evolved near a triple point of grain boundaries in 304 stainless steel deformed to a strain of 1.6. Initial grain boundaries (GB) are covered by second phase precipitations.

Fig. 7  Elongated subgrains formed near an initial grain boundary (GB) in 304 stainless steel deformed to $\Sigma e = 1.6$. Note here that some subgrains contain quite few dislocations in their interiors and their sharp boundaries have medium to high misorientations.

these (sub)grains is about 0.3 $\mu$m in the longitudinal direction and 0.13 $\mu$m in the transverse one.

Upon further multiple deformation to high cumulative strains, the volume fraction of these highly misoriented substructures increase substantially, leading to the full development of well defined (sub)grains in initial grain interiors. The typical substructure evolved at a total strain of 3.2 is presented in Fig. 8. The elongated subgrains become more equiaxed due to increase in the transverse size and their boundaries with high misorientations become sharp. It can be noted here that the second phase precipitations, as indicated by arrow heads
in Fig. 8, may roughly mark the original position of initial grain boundaries. The latters cannot be exactly recognized because of the development of many fine grains with high-angle boundaries at $\Sigma \varepsilon = 3.2$. Figure 9 shows the TEM micrographs of the sample deformed to a cumulative strain of 6.4. The equiaxed fine-grains with an average grain size of about 0.3 $\mu$m are homogeneously developed. It is remarkable in Fig. 9 that dislocation densities in each (sub)grain are clearly lower at $\Sigma \varepsilon = 6.4$ than those evolved at preceding strains; and also the secondary precipitations, located along the original boundaries (Fig. 8), are randomly distributed in the new grains.

Figure 10 represents changes in the distribution of misorientation for strain-induced subgrain and/or grain boundaries with warm multiple deformation. The average misorientations continuously increase with the accumulated strain. The distribution of misorientations shows a bimodal distribution with two peaks corresponding to low and high angles at strains of 1.6 and 3.2. With further deformation to 6.4 the fraction of low-angle misorientations rapidly decreases, while that of high-angle ones increases. This directly connects with the evolution of a new fine-grained structure with high-angle grain boundaries at $\Sigma \varepsilon = 6.4$.

4. Discussion

4.1 Microstructural changes with warm multiple deformation

The flow stresses under multiple warm compression
demonstrate very strong work hardening and a steady-state-like flow in the strain range of 1 to 3, followed by a minor work softening with further deformation. Change in the average dislocation densities with warm multiple deformation is represented in Fig. 11. This result roughly corresponds to the $\sigma - \Sigma \varepsilon$ curve at 873 K (Fig. 1); namely, early deformation to a strain of about 1 results in an increase of dislocation densities to a maximum, which can bring about a rapid work hardening and a stress peak. Then the dislocation densities smoothly decrease and approach a value of about $2 \times 10^{15}$ m$^{-2}$ at high cumulative strains, where gradual work softening takes place. It is generally agreed that the formation of subboundaries as DDWs due to dislocation rearrangement and annihilation is always accompanied by a reduction of strain energy.\textsuperscript{18,19}

Figure 12 shows changes in the average subgrain/grain size ($d$) in the longitudinal and transverse directions and the average boundary misorientation ($\theta$) with cumulative strain during warm deformation. It is clearly seen in Figs. 11 and 12 that the decrease in dislocation densities corresponds to the evolution of fine grains with high angle boundaries in initial grain interiors at moderate strains above 1.6. The longitudinal subgrain sizes of about 0.3 $\mu$m hardly depend on strain, while the transverse ones continuously increase with multiple deformation. This leads to the development of more equiaxed grains with high-angle boundaries at high strains.

It should be added that an increase in room temperature hardness at $\Sigma \varepsilon = 6.4$ (see Fig. 1) can result from the formation of fine-grained structure due to Hall-Petch relationship. The work softening at 873 K as well as the hardness decrease after a peak can result from the decrease in average dislocation densities (Fig. 11). On the other hand, the strain-induced dislocation subboundaries increase their misorientations to above 20° at high strains, leading to the almost full evolution of new fine-grained structure. Then an effect of grain size strengthening due to such a grain refinement should clearly appear at room temperature, while this effect usually decreases with increasing temperature and may approach zero at high temperatures.

**4.2 Internal stresses in strain-induced subgrains**

It should be noted in Figs. 4 and 9 that the fine-grained structures evolved at high strains contain relatively lower dislocation densities comparing to the substructures evolved at early deformation. Such decreases in dislocation densities can result from the operation of dynamic and static recovery during interrupted compression. It is interesting to note in Fig. 7 that dislocations are relatively free in some subgrains evolved even at lower strains as 1.6. A material with such high density dislocations heterogeneously distributed should be considered as a composite of the dislocation subboundaries and the subgrain interiors as hard and soft regions on subgrain scale, respectively.\textsuperscript{18-20} The dislocation densities in subgrain interiors, therefore, is determined by the actual stresses in the soft regions, which are less than an applied stress by internal back stresses.

It has been shown that fine grain evolution under a severe plastic deformation is frequently accompanied by the development of high internal stresses.\textsuperscript{7,13,21} Strain-induced grain boundaries in very fine-grained materials can produce long-range stress fields because of their non-equilibrium state, in addition to the high density lattice dislocations evolved at relatively low temperatures. To study the elastic lattice curvatures occurring at high strains, some fine grains with quite low dislocation densities were chosen, as shown in Fig. 13. The misorientation component evaluated in the horizontal plane between points marked as “A” and “B” in Fig. 13 is about 0.3°. This elastic distortion may be associated with the grain boundaries because of quite few dislocations in the grain interior. Let us consider here one of the possible mechanisms for high lattice distortions evolved under a severe deformation.
Plastic deformation at relatively low temperatures leads to the accumulation of so-called grain boundary dislocations not only at initial grain boundaries, but also at strain-induced subboundaries because of any small differences in slip systems operating in neighboring subgrains. The grain boundary sliding taking place even at a warm deformation, as can be seen in Fig. 2, may also result in the evolution of high lattice distortions due to accumulation of dislocations at grain boundary ledges and junctions. The sources of high elastic strains, therefore, may be generally considered as superdislocations (or disclinations) placed at corners of grains and subgrains. The Burgers vector of such superdislocation may be presented by a sum of Burgers vectors of grain boundary dislocations accumulated at high strains.

Assuming that grain boundary dislocations are inhomogeneously distributed along the subboundaries, the internal stresses developed in subgrain interiors can be considered in terms of a junction distortion network. Figure 14 shows a simple example for the interaction between a moving edge dislocation and distortion sources developed at grain corners due to excess grain boundary dislocations. The internal stresses created by the joint distortions are analogous to those produced by wedge disclinations with rotation vectors of \(\omega_i\) placed at a distance \(d\), as shown schematically in Fig. 14(a). The back force acting on a dislocation per unit length with Burgers vector \(b\) moving towards grain boundary can be evaluated by the following relation:

\[
F = \frac{bG}{2\pi(1-\nu)} \sum \frac{\omega_i x_i y_i}{x_i^2 + y_i^2}
\]

where \(G\) and \(\nu\) are the shear modulus and Poisson’s ratio, respectively. The interaction force depends on the relative distance of the dislocation from disclinations, i.e. \(x_i\) and \(y_i\) in eq. (1). Assuming the same power for disclinations, i.e. \(\omega_i = \omega\), the force distribution inside a square area in Fig. 14(a) is shown in Fig. 14(b) in units of \(Gb\omega b / 2\pi(1-\nu)\). The present model allows one to analyze the interaction between a dislocation and the non-equilibrium boundary in a very simplified case. Considering the square area in Fig. 14 as a cross section of subgrain, one can readily see that a maximum interaction appears at the central part of the subgrain. \(G = 58500\,\text{MPa}\) and \(\nu = 0.27\) for 304 stainless steel at 873 K. The maximal back shear stress evaluated is \(\tau = (F/b) \approx 130-260\,\text{MPa}\) for \(\omega = 0.005-0.01\). This \(\omega\) corresponds to 0.3°–0.6° which is close to the value in Fig. 13. It is concluded that such high internal stresses are comparable with the flow stresses of \(\sigma \approx 650\,\text{MPa}\) at 0.5\(\tau_m\) (Fig. 1(a)) and so may be responsible to the decrease in dislocation densities in new grain interiors.

### 4.3 Fine grained structure evolved under warm deformation

The room temperature hardness for the warm deformed samples closely parallels to the deformation behavior at 873 K, as can be seen in Fig. 1. It has been shown in the previous work on the same stainless steel that the flow stresses which become above 400 MPa hardly depend on strain rate and temperature and so are in the region of athermal deformation. Under such conditions, any diffusion mechanism assisting the dynamic structural changes becomes difficult in a similar way to the cold deformation. Warm multiple deformation to moderate strains above 1 leads to the formation of dislocation substructures, such as cells and DDWs. It should be noted that grain boundary sliding takes place inhomogeneously and so brings about a strain gradient along grain boundaries. The subboundaries with middle angle misorientations evolved near grain boundaries, such as those in

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Fig. 13 A fine grain with low dislocation density in 304 stainless steel warm multiple deformed to \(\Delta t = 3.2\). The misorientation between “A” and “B” is above 0.3°. This indicates some high internal stresses evolved within such grains.

Fig. 14 Interaction between an edge dislocation and subboundaries with junction distortions. (a) Schematic drawing for mutual arrangement of a dislocation moving towards the right subboundary and (b) the back force distribution acting on a unit element of dislocation.
Fig. 5, therefore, should be considered as geometrically necessary boundaries. The number of such subboundaries and their misorientations increase with further straining. The evolution of subgrain misorientations is due to the increase in the number of lattice dislocations evolved by usual intrinsic slip and the absorption of accommodation (or grain boundary) dislocations that originate from strain incompatibility between subgrains. The accumulation of grain boundary dislocations can lead to evolution of high internal stresses and promote sliding along the strain-induced subboundaries, which should be accompanied with subgrain rotations.

It is clearly seen in Fig. 12(b) that the misorientation of strain-induced subboundaries linearly increases with strain and approaches a typical value of conventional grain boundaries at strains above 3.0. This result for 304 stainless steel is quite similar to that for a pure copper deformed at 0.35T_m, as shown in Fig. 12(b). In the strain range from 0.5 to 3.0, the average misorientation (θ) can be approximated by a linear function of cumulative strain (Σε) as follows:

$$\theta = \alpha (\Sigma \varepsilon - \varepsilon_0)$$  

(2)

where \(\alpha = 7^\circ\) and \(\varepsilon_0 = 0.2\) are obtained in Fig. 12(b). The slope of 7° is roughly close to 4° and 12° reported for a duplex stainless steel and an aluminum, respectively. The \(\varepsilon_0\) is usually 0.2 to 0.4 and it is considered as a critical strain where geometrically necessary subboundaries are produced by lattice rotations. The average value of \(\theta\) for random disorientation of polycrystalline grains, 40.7°, can be considered as an upper limit of average boundary misorientations. The saturation value at \(\Sigma \varepsilon = 6.4\) in Fig. 12(b), however, is much lower than 40.7°. This suggests that such strain-induced grain structure should always contain low angle dislocation subboundaries, which continuously evolve and disappear during multiple deformation irrespective of strain accumulation.

It should be noted that dynamic and static recovery processes taking place during interrupted deformation at 873 K can play an important role in the evolution of new grains. The new grains developed at high strains become more equiaxed as compared to the preceding subgrains at moderate strains (Fig. 12(a)). Such increasing subgrain size during deformation may result from minor local migration of strain-induced subboundaries, which in turn should be affected by recovery processes through dislocation rearrangement occurring in subboundaries. The phenomenon of new grain evolution described above is similar to continuous and/or rotation DRX taking place in some materials under hot deformation. It is concluded that the fine grain formation discussed above can result from a kind of strain-induced continuous reaction during deformation, that is essentially similar to continuous DRX, although the role of dynamic recovery becomes less effective at a relatively low temperature 0.5T_m.

5. Conclusions

Warm deformation behavior and strain induced grain evolution in 304 type austenitic stainless steel were studied in multipass compression at a temperature of 873 K (0.5T_m). The main results can be summarized as follows:

1. The integrating curve for the flow stresses shows an apparent steady-state flow at moderate strains followed by a small softening at high strains above 3. The strain dependence of room temperature hardness is quite similar in appearance to that of warm flow stresses at strains below 3, and, in contrast, the hardness increases at high strains up to 6.4. This difference at high strains can result from grain size strengthening effect due to a fine grain evolution.

2. The structural changes are characterized by the evolution of elongated subgrains with their dense dislocation boundaries at low to moderate strains. The subgrains become more equiaxed and the misorientations between them gradually increase with increase in cumulative strain, finally leading to the full development of new fine-grained structure with an average grain size of about 300 nm.

3. The fine grain formation at high strains can result from a kind of strain-induced continuous reactions taking place during deformation, that is essentially similar to continuous dynamic recrystallization.

4. The warm multiple compression may provide a potential thermomechanical processing for producing a very fine-grained microstructure in metallic materials.

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