Continuous recrystallization in austenitic stainless steel after large strain deformation

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Abstract

Static restoration mechanisms operating during annealing were studied in a 304 steel with strain-induced submicron grain structures. The initial microstructure with an average grain size of about 0.3 μm was developed by large strain deformation at 873 K. Early annealing leads to a full relaxation of high internal stresses associated with non-equilibrium strain-induced grain boundaries, while their boundary misorientations and the average grain size barely change. Further annealing results in a transient recrystallization followed by a normal grain growth. The average grain boundary misorientation increases up to around 45° in the former and becomes constant in the latter. This is associated with the change in the grain boundary misorientation distribution from a characteristic strain-induced one to a near random distribution corresponding to a fully recrystallized state. The annealing processes operating in the strain-induced fine grains take place homogeneously in the whole matrix and can be called continuous recrystallization.

Keywords: Annealing; Transmission electron microscopy (TEM); Austenitic steels; Microstructure; Recrystallization

1. Introduction

Among materials scientists, there is continual interest in utilizing ultra fine-grained metals and alloys as engineering materials. This is dictated by some benefit combination of mechanical properties for the sub-micron grained structural products [1–3]. Recently, submicrocrystalline structures have been shown to be produced in various metallic materials by the redundant strain deformation at relatively low temperatures [4–6]. The authors have carried out detailed examinations of the structural changes leading to the evolution of ultra fine strain-induced grains using warm multiple and multi-axial compressions with changing the compression axis [7–9]. Multi-axial deformations promote the rapid formation of many intersecting dislocation boundaries in different directions because of the variation in the slip systems operating from pass to pass. A gradual rise in misorientations across the strain-induced subboundaries with

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increasing strain finally leads to the evolution of ultra fine grains with high-angle boundaries.

Materials processed by large strain deformation are characterized by high internal stresses, which are associated with both high dislocation densities and non-equilibrium grain boundaries [3,6,10–12]. It is particularly important to study the annealing mechanisms operating in the ultra fine-grained metallic materials during heating. Most research work has mainly focussed on the thermal stability of the fine-grained structures [3,5,6,13]. Materials with a high density of strain-induced high-angle grain boundaries [5,13,14] have been suggested as being essentially resistant to discontinuous recrystallization (namely a primary recrystallization) regardless of an apparently high stored energy, and only the grain growth can operate frequently in such structures. However, high internal distortions existing in the strain-induced ultra fine grains may result in a specific annealing behavior under subsequent thermo-mechanical treatment. Further experimental studies are necessary for a more comprehensive understanding of the annealing behavior of such submicrocrystalline materials.

The aim of the present work is to study the annealing behavior of a 304-type stainless steel with an ultra fine-grained microstructure, which was developed by warm multi-axial deformation. Specific attention is given to the detailed consideration of the static restoration mechanisms operating in the ultra fine-grained structures and especially of continuous and discontinuous recrystallization.

2. Experimental procedure

A 304 type austenitic stainless steel (0.058%C, 0.7%Si, 0.95%Mn, 0.029%P, 0.008%S, 8.35%Ni, 18.09%Cr, 0.15%Cu, 0.13%Mo (all in wt%) and the balance Fe) was used as the starting material. The fine-grained microstructure with an average grain size of D_o = 0.3 μm was produced by multiple and multi-axial compressions to a total strain of 6.4 at a strain rate of around 10^{-3} s^{-1} at a temperature (T) of 873 K. The details of the processing are described elsewhere [8]. The samples multiple deformed at 873 K were annealed for various periods of time in air using a muffle furnace at temperatures from 873 to 1173 K. The rectangular specimens for annealing had a dimension of about 5.0 × 4.2 × 3.5 mm.

The annealed samples were quenched in water and were cut parallel to the final compression axis for hardness measurements and microstructural observations. The Vickers hardness of the annealed samples was evaluated with a load of 3 N. The annealed microstructures were studied with optical and transmission electron microscopy (TEM) techniques. The TEM observations were carried out using a JEOL JEM-2010F operating at 200 kV. The average grain size was measured by the mean linear intercept method, and the twins were omitted from measurements. The misorientations on the (sub)grain boundaries were studied using a conventional Kikuchi-line technique in TEM, and a total of about 60 ~ 80 boundaries was analyzed in each sample. The internal elastic distortions, which are associated with the strain-induced grain boundaries, were analyzed by measuring the local misorientations within at least ten individual dislocation-free grains per point [12]. Assuming that the elastic strain changes linearly with respect to the specimen thickness, the elastic strain can be roughly evaluated using the equation, $\varepsilon = (0.5\psi t_1)/t_2$, where ψ is the local misorientation angle between the two regions in a grain. They are spaced at a distance of t_2, and t_1 is the foil thickness.

3. Results

3.1. Effect of annealing temperature

Typical optical and TEM microstructures that evolved after annealing at various temperatures are shown in Figs. 1 and 2, respectively. Fig. 2(a) represents also the as-strained microstructure that developed in steel severely deformed at 873 K. This microstructure consists of almost equiaxed grains with a mean grain size of 0.3 μm. Annealed microstructures depend strongly on the reheating temperature. Annealing at 973 K does not lead to any rapid changes in the microstructure. After annealing at 973 K for 0.45 ks (Fig. 1(a)), the microstructure is still very fine to be clearly ana-

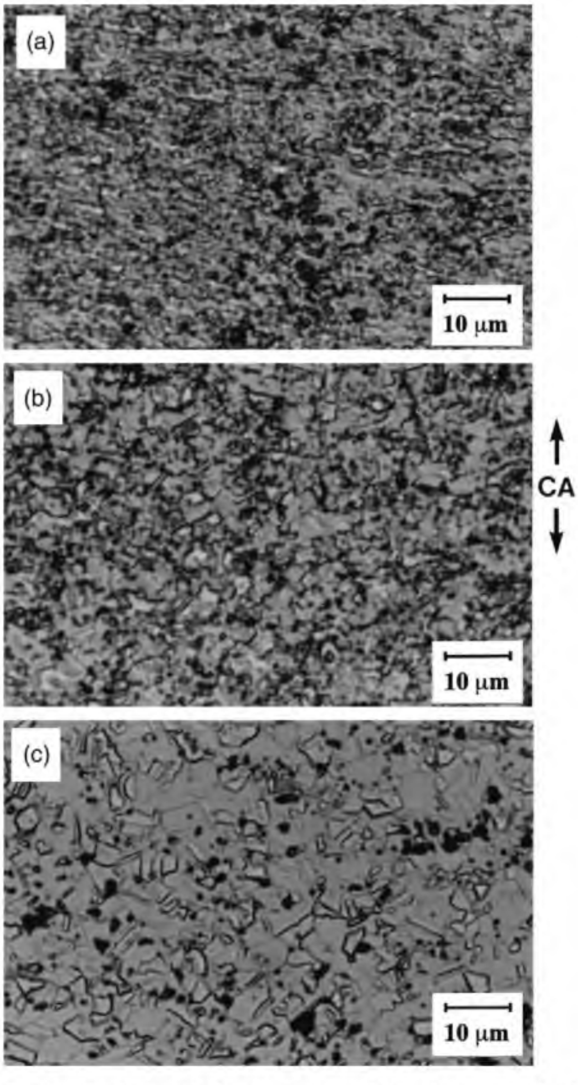

Fig. 1. Typical optical micrographs of the ultra-fine-grained 304 stainless steel samples annealed at (a) 973 K for 0.45 ks, (b) 973 K for 1.8 ks, and (c) 1073 K for 0.45 ks. "CA" indicates the final compression axis direction.

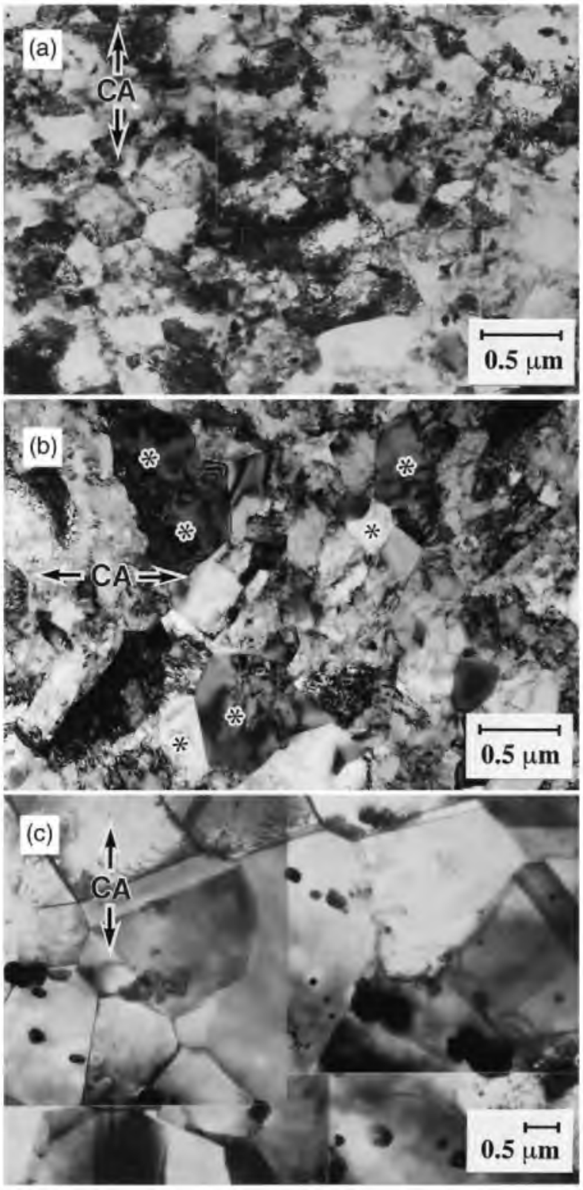

Fig. 2. Typical fine-grained microstructures evolved in the 304 stainless steel (a) after multiple deformation to a large strain of 6.4 at 873 K and then annealed for 0.45 ks (b) at 973 K and (c) at 1073 K. The asterisks (*) indicate the grains containing quite a few interior dislocations.

lyzed by optical microscopy. Referring to Fig. 2(b), this microstruture can be characterized by a mixed one consisting of fine grains with high and low dislocation densities in their interiors. The grains containing quite few dislocations, indicated by the star marks (*) in Fig. 2(b), may be considered as potential nuclei for recrystallizing grains, which can grow and consume the surrounding areas with high-density dislocations. After annealing at 1073 K, the strain-induced fine grains are almost replaced with relatively coarse ones with low-density dislocations and smooth grain boundaries (Figs. 1(c) and 2(c)). It should be noted that the microstructure evolves quite homogeneously upon annealing, as suggested by Fig. 1. The microstructure includes some second phase precipitations, which are probably carbides and/or sigma-phases. However, the total volume fraction of the second phase particles does not exceed 5%, and the sizes are relatively large. We can assume, therefore, that the second phase precipitations do not affect the annealing behavior of the fine-grained samples seriously (at least in the range of the grain sizes studied).

Fig. 3 summarizes the temperature effect on the microstructure, the hardness, and the internal stress of the ultra fine-grained steel during the isochronal annealing for about 0.45 ks. Two temperature regions divided at around 1000 K can be distinguished. Upon annealing at $T < 1000$ K, the hardness decreases slightly, and the average grain size does not change remarkably, compared to the as-processed state, by increasing the annealing temperature. However, the internal stress measured in dislocation-free grains is reduced considerably indicating that a recovery takes place rapidly at the strain-induced grain boundaries. In contrast, annealing at higher temperatures, i.e. $T > 1000$ K, results in a rapid grain coarsening and a considerable softening. There are almost zero internal residual stresses in this temperature region.

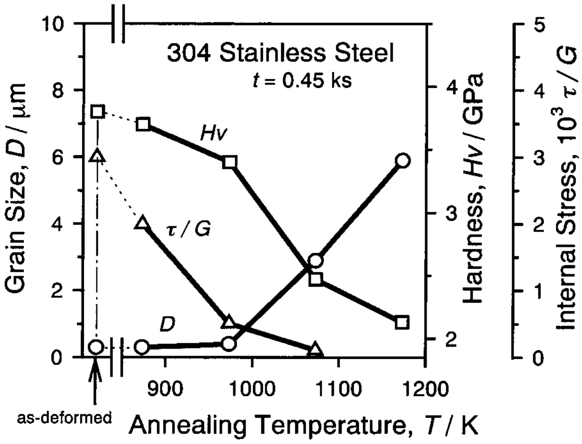

Fig. 3. Effect of annealing temperature on the grain size (D), the hardness (Hv) and the internal stress normalized by the shear modulus (τ/G) for the fine-grained 304 stainless steel. The annealing time was 0.45 ks.

3.2. Isothermal annealing

The restoration mechanisms operating in the ultra fine-grained steel at 973 K were studied in more detail by isothermal annealing test. Fig. 4 presents the effect of isothermal annealing on the room temperature hardness (Hv), the average grain size (D), and the internal stress (τ/G). Fig. 4 shows that the annealing process can virtually be subdivided into the three sequential stages as indicated by broken lines in the figure. At early annealing, i.e. during the first stage, the hardness of the annealed samples gradually decreases. Upon further annealing, the hardness steps down in the second annealing stage, which is relatively short. Then, at the third annealing stage, the softening kinetics slows down and is roughly the same as that for the first stage.

The changes in the average grain size with annealing (Fig. 4(b)) correspond roughly to those in the hardness (Fig. 4(a)). At early annealing there is no remarkable grain coarsening in spite of some

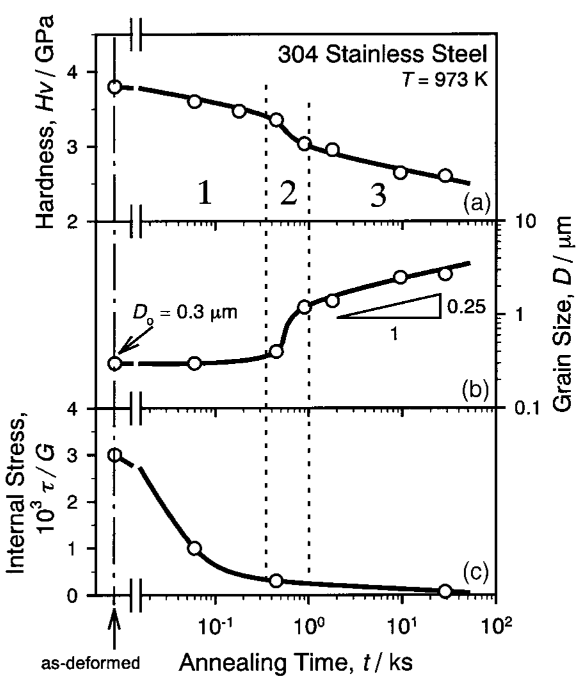

Fig. 4. Changes in the room temperature hardness (Hv), the grain size (D), and the internal stress (τ/G) during isothermal annealing of the fine-grained 304 stainless steel at 973 K.

softening taking place. This suggests that recovery can be the structural restoration mechanism operating in stage 1. During stage 2, the average grain size increases rapidly and is accompanied by a decrease in the hardness. Therefore, we can conclude that a kind of recrystallization process may develop in annealing stage 2. This recrystallization will be discussed in more detail in the following sections. The third annealing stage is characterized by a relatively slow grain coarsening, which is similar to the kinetics of normal grain growth [15]. The average grain size follows a power law function of annealing time at this stage. The grain growth exponent of about 0.25 in Fig. 4(b) is in good agreement with that of 0.1 ~ 0.5, which was reported in other studies on the normal grain growth of single-phase materials [15,16].

Fig. 4(c) shows that high internal elastic distortions developed in the fine-grained steel are rapidly released and approach almost zero at stage 1. These high internal stresses can result from the non-equilibrium grain boundaries [12]. Fig. 4(c) suggests that the recovery develops quite easily at the grain boundaries, resulting in a rapid annihilation of any strain-induced defects such as grain boundary dislocations and disclinations [6,11,17,18]. Fig. 5 presents TEM microstructures and corresponding lattice images at the grain boundary vicinities before and after a short annealing at 973 K. Bent crystallographic planes and many dislocations are evolved near the grain boundary in the as-processed state, as indicated by arrowheads in Fig. 5(c). Moreover, this boundary has a spread and diffuse image, so it cannot be positioned precisely on the TEM micrograph. The small circles in Fig. 5(c) roughly indicate the edge of the boundary image. On the other hand, in the sample annealed for 0.45 ks, the grain boundaries are distinctively sharp and narrow, and the straight crystallographic planes near the grain boundary can be clearly defined as indicated in Fig. 5(d). Therefore, a rapid progress of recovery takes place especially at the grain boundaries, leading to a remarkable relaxation of such locally diffused boundaries.

We can conclude from the results described above that three sequential time intervals can be distinguished during the annealing of the strain-induced fine-grained microstructure. These annealing stages can be differentiated by the softening kinetics and/or the grain coarsening kinetics, that resulted from some differences in structural change mechanisms that operated upon reheating. The fastest operating process is a conventional recovery, because it does not require an incubation period and develops just after reheating. Following the recovery, a kind of recrystallization process can result in rapid grain growth accompanied by a sharp softening. Since the second annealing stage is relatively short and the grains coarsen to only a few times that of the as-processed grain size, this recrystallization should be considered as a "transient" recrystallization. Finally, a conventional normal grain growth following the transient recrystallization takes place in the final annealing stage.

3.3. Transient recrystallization

A conventional recovery creates the appropriate conditions for the development of the transient recrystallization at the end of stage 1. A certain amount of fine grains starts to grow and consumes neighbouring grains with high-density dislocations. Returning to Figs. 2(b) and 5(b), which show some growing grains, we can see that these grains are still not perfectly free from dislocations in their interiors. At least a portion of such grains should be recovered to provide the growth. The other parts of the growing grains may be occupied frequently by high-density dislocations. We can conclude that the strain-induced fine grains annealed at the end of stage 1 may potentially grow and become the nuclei for recrystallization. The ability of certain grains to grow rapidly can be motivated by any structural inhomogeneity that is inherent in the strain-induced state and, consequently, the variety of the recovery kinetics in each grain.

Typical microstructures that evolved in annealing stages 2 and 3 of the strain-induced ultra fine-grained steel are presented in Fig. 6. The microstructure at the beginning of the transient recrystallization shows some variety in each grain and is quite inhomogeneous (Fig. 6(a)). A certain portion of these grains accommodates some high-density dislocations, whereas other grains evolve

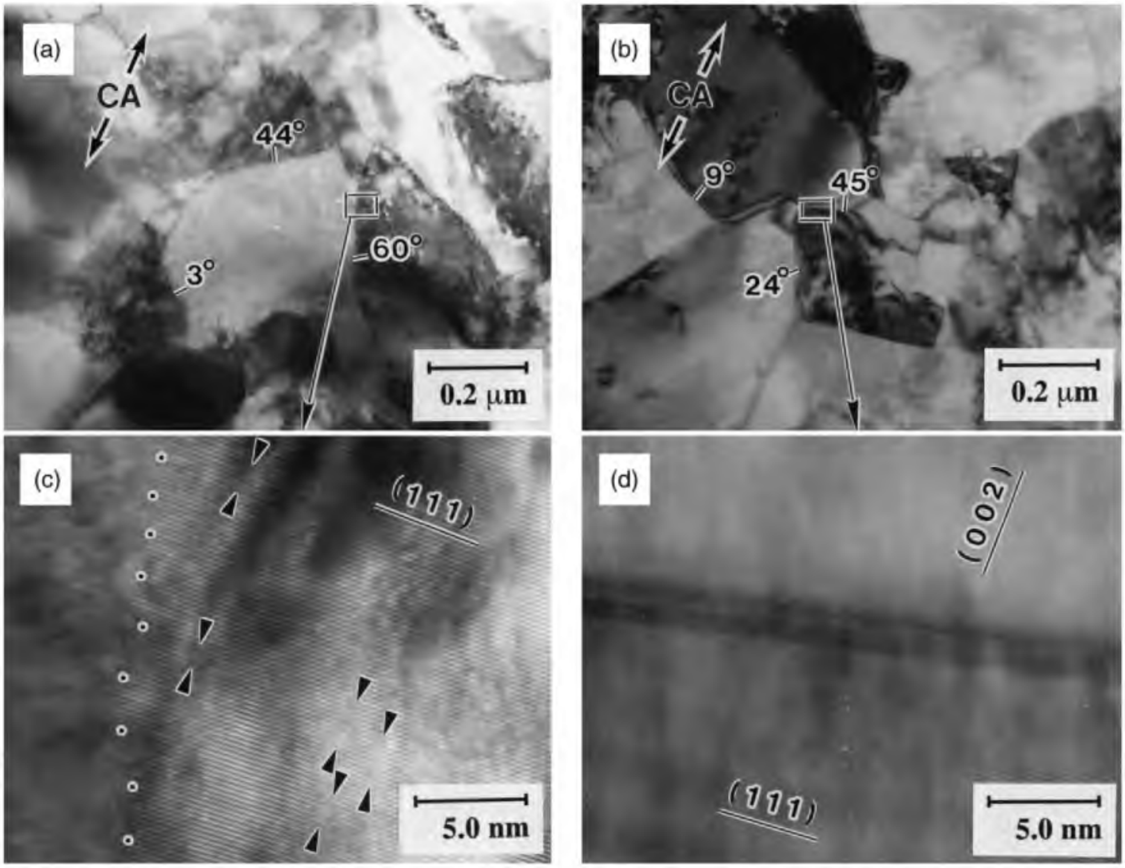

Fig. 5. TEM microstructures (a,b) and corresponding lattice images (c,d) of the fine-grained 304 stainless steel; (a,c) as-deformed state, and (b,d) annealed for 0.45 ks at 973 K. The small circles roughly indicate the grain boundary, and the arrowheads encompass some dislocations in the as-processed state.

in relatively low dislocation densities in their interiors. The former ones do not demonstrate any evidence of the growth, while the latter ones are considered to coarsen and become the recrystallized grains. With further annealing to the end of the transient recrystallization, the average grain size increases and the grain boundaries become sharper and clearly observable (Fig. 6(b)). Some of those recrystallized grains still contain substructures like subboundaries and dislocations. In the final annealing stage (stage 3), such dislocation substructures in grain interiors disappear almost completely, and fully recrystallized coarser grains develop including several twins (Fig. 6(c)). This is considered as a normal grain growth.

It is interesting to note in Fig. 6(a) that the resulting grain boundary misorientations that developed in the recrystallizing grain clusters differ from those which evolved in the strain-induced state. The former consist mainly of high-angle boundaries including some twins, but the latter involve a relatively large fraction of low-angle boundaries. Such a difference in the misorientations between the recrystallized and strain-induced grain boundaries should result in a gradual change of the grain boundary misorientation distribution. Fig. 7 represents the quantitative analysis of the changes in the grain boundary misorientations by annealing at 973 K. The misorientation distribution of the strain-induced fine-grained boundaries in the as-deformed state (Fig. 7(a)) is characterized by a roughly flat one with no clear

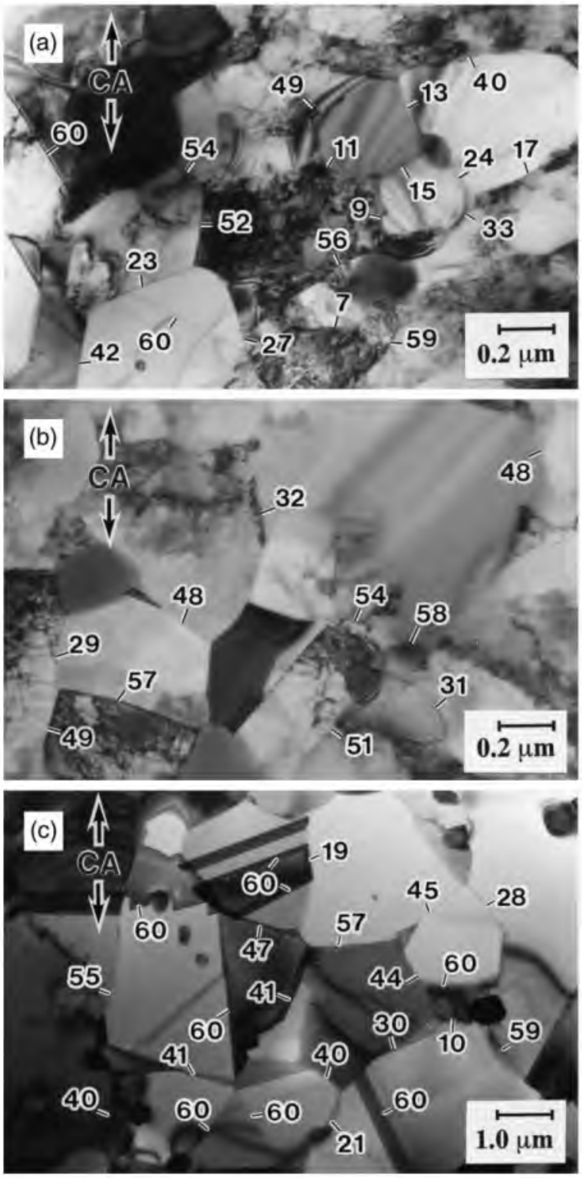

Fig. 6. Typical TEM microstructures evolved in the fine-grained 304 stainless steel after annealing at 973 K for (a) 0.45 ks, (b) 1.8 ks, and (c) 28.8 ks. The numbers indicate the misorientation in degrees.

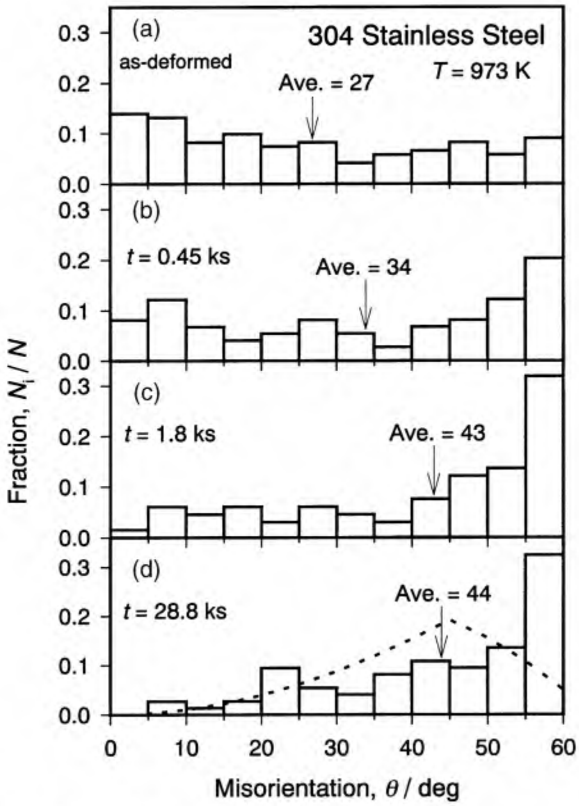

Fig. 7. Effect of annealing at 973 K on (sub)grain boundary misorientation distributions in the fine-grained 304 stainless steel. The broken line indicates the random misorientation distribution by Mackenzie [19].

peaks ranging from 0° to 60°. The fraction of high-angle misorientations above 40° increases considerably during the transient recrystallization, i.e. in stage 2, while the low-angle misorientations below 15° gradually disappear, as shown in Figs. 7(b) and (c). There is a clear increase of the fraction of misorientations around 60° indicating that annealing twins frequently evolve during grain coarsening in the transient recrystallization. With further annealing in stage 3 (Figs. 7(c) and (d)), the misorientation distribution and the average misorientation value do not change remarkably. They are roughly similar to the random misorientation distribution with its average value of about 41° predicted by Mackenzie [19], as shown by the dotted line in Fig. 7(d). Therefore, we can conclude that the grain structure which developed after annealing at stages 2 and 3 can be almost the same as that after a conventional recrystallization including a normal grain growth.

4. Discussion

The experimental results described above suggest that the structure evolution during the heating of strain-induced fine-grained austenitic steel follows a general tendency for the annealing behavior of conventional deformed materials. In other words, recovery leads to recrystallization followed by a normal grain growth. However, some distinctive characteristics inherent in the annealing behavior of the strain-induced ultra fine-grained materials are found in the present study. Note that the strain-induced fine-grained structures are formed by a grain fragmentation process, that is the initial grains are split by the formation of new internal high-angle dislocation boundaries [4–9,20]. The development of fine grains can be described as a consequence of continuous reactions on a substructural scale caused by severe plastic working, i.e. continuous dynamic recrystallization. Such strain-induced ultra fine grains are distributed homogeneously in the space of the order of their grain size; however, they are characterized locally by a heterogeneous distribution of dislocation densities and high internal stresses, which developed in the grain interiors [7–12].

High internal stress developed in the strain-induced ultra fine-grained boundaries can be reduced by a recovery process operating during the early annealing, as shown in Fig. 4. It was proposed [18,21] that the rate of recovery operating at such grain boundaries may be higher than that associated with the dislocation substructures in the grain interiors. The recovery developing at the first annealing stage should be considered as a process associated mainly with the relaxation of the strain-induced grain boundaries, leading to the formation of rather narrow and straight grain boundaries. Thus, many strain-induced grains transform into a great number of potential nuclei for static recrystallization at the end of stage 1. With further annealing, the grain coarsening can take place homogeneously in the whole area of the material. The recrystallizing nuclei transformed from the ultra fine strain-induced grains can grow and consume neighbouring grains containing higher density dislocations. However, there is a close limit for the dense nuclei to grow because the growing grains impinge on each other. Under such conditions it is difficult to find any clear transition borders between the recrystallized and the un-recrystallized areas in stage 2. Therefore, the present transient recrystallization should be stated as a continuous phenomenon similar to the continuous static and dynamic recrystallizations, which take place under specific conditions [9,15,22–28].

The structural softening processes that operated during the annealing of the strain-induced ultra-fine grained steel, i.e. the recovery in stage 1, the transient recrystallization in stage 2 and the normal grain growth in stage 3, are all based on the continuous mechanisms of the structure evolution. By recalling that the ultra fine-grained microstructure itself was also developed as a result of such continuous processes, i.e. continuous dynamic recrystallization [8], it may be interesting to consider the ultra fine grain evolution under a large strain deformation followed by annealing as a unified sequence of structural changes. The changes in the average (sub)grain boundary misorientations and the average (sub)grain sizes with deformation and subsequent annealing are summarized in the unified sequence in Fig. 8. Stage 0 in Fig. 8 represents the evolution stage of fine grains under severe plastic working [8]. The annealing stages 1 to 3 following stage 0 are defined above.

The mechanism of fine grain evolution under

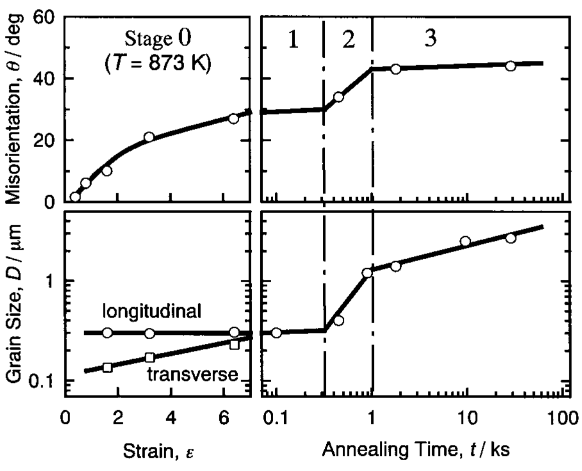

Fig. 8. Changes in the average (sub)grain boundary misorientation, and the average (sub)grain size with large multi-axial deformation [8] followed by annealing at 973 K.

large strain deformation appears in the grain fragmentation by increasing the (sub)boundary misorientations up to values of conventional high-angle grain boundaries. During deformation, in stage 0, the average (sub)boundary misorientation gradually increases from zero and approaches 25 ~ 30° at the end of this stage, while the average (sub)grain size does not change significantly. Under multiple and multi-axial deformation, the fine grains evolved at large strains are more equiaxed compared to the preceding subgrains [8]. The annealing behavior of such strain-induced fine grains will be described next.

At early annealing, the recovery operating in stage 1 does not evolve any changes in either the grain size or the grain boundary misorientation, while the internal stresses due to the non-equilibrium grain boundaries are almost released. The subsequent stage 2 is characterized by a rapid increase in both parameters. The rapid grain coarsening under transient recrystallization can be motivated by two driving forces, which result from the substructural inhomogeneities and the grain boundary energy. The former should be considered as a distinctive feature of transient recrystallization when compared to the conventional normal grain growth. Dislocation substructures resulting in high stored energy that are frequently associated with low-angle subboundaries are consumed mainly by the growing grains in stage 2. Further annealing in stage 3 leads to the occurrence of conventional normal grain growth. The average grain size follows the power law function of annealing time, and the average grain boundary misorientation of about 45° is almost constant. A high fraction of annealing twins in a fully recrystallized microstructure (see Figs. 6 and 7) can be responsible for the difference between the average misorientation obtained in the present work and that of about 41° calculated for randomly disoriented grains [19].

Finally, let us compare the continuous recrystallization operating in the strain-induced fine grains with the conventional discontinuous recrystallization. The restoration characteristics appearing in each process stage are summarized in Table 1. First, the difference between these two types of recrystallization should be mentioned for the deformed states (stage 0). The discontinuous

Table 1
Comparison of restoration sequences in discontinuous and continuous processes

Stages	Discontinuous reactions	Continuous reactions
0	*Deformation to low/medium strains* Strain/orientation gradients accompanied by dislocation substructures evolved in deformed matrices	*Deformation to large strains* Network of high-angle (sub)boundaries due to grain fragmentation evolved in grain interiors
1	*Recovery* Rearrangement and annihilation of dislocations, polygonization followed by heterogenous nucleation (low density of nuclei)	*Recovery* Non-equilibrium strain-induced grain boundaries change to conventional ones, leading to homogenous nucleation (high density of nuclei)
2	*Recrystallization* Large-scale boundary migration RCV = $10^6 \sim 10^9$[a]	*(Transient) recrystallization* Small-scale boundary migration RCV < 10[a]
3	*Normal grain growth* Driven by grain boundary energy	

[a] RVC is the relative volume change of typical recrystallized grains against nuclei.

recrystallization (frequently called primary recrystallization) takes place in the materials deformed to low-to-moderate strains, where the deformed substructures are characterized by high-density dislocations arranged in rich dislocation layers, cells, subboundaries, etc., as well as local orientation gradients that frequently evolved in the deformed matrices. In contrast, continuous recrystallization can occur in materials with fine-grained microstructures produced by large strain deformation. A principal feature of such materials is the large fraction of high-angle grain boundaries closely spaced at the (sub)grain size scale, and this has been considered to be the main condition preventing conventional discontinuous recrystallization [5,14]. Secondly, the rearrangement and annihilation of some dislocations leading to polygonization are an essence of recovery operating in stage 1 for discontinuous recrystallization, whereas recovery occurring at the non-equilibrium fine grain boundaries is the main process operating at the beginning of the continuous restoration. Thirdly, the mechanisms for new grain formation are quite different. The discontinuously recrystallized grains result from nucleation (mainly by the grain boundary bulging [15,28–30]) taking place at special sites with strain/orientation gradients followed by large-scale boundary migration, i.e. a two-step reaction. In contrast, the nuclei of the continuously recrystallized grains are the strain-induced ultra fine grains in themselves that can grow in small-scale only, i.e. essentially a one step reaction [28]. There are no isolated regions for the latter ones that coarsen over the surrounding matrix. This makes the strain-induced ultra fine-grained structures essentially stable against a rapid coarsening, even though it might be expected from the high stored energy of severe deformation. On the other hand, the normal grain growth in stage 3 driven by the grain boundary energy seems to be almost the same in both cases.

5. Conclusions

The annealing behavior of an ultra fine-grained 304 stainless steel, with a grain size of about 0.3 μm that was produced by large strain multi-axial deformation at 873 K, was studied at temperatures from 873 K to 1173 K. The main results can be summarized as follows:

1. Two temperature regions separated at around 1000 K can be distinguished by their effect on the microstructural changes during isochronal annealing for 450 s. The microstructure is relatively stable against coarsening at temperatures below 1000 K, while heating to higher temperatures results in a rapid grain growth.
2. The annealing behavior can be characterized by the operation of the following major sequential processes: recovery, transient recrystallization, and normal grain growth.
3. At early annealing, recovery develops readily at the grain boundaries, leading to a fast release of high internal stresses associated with the non-equilibrium state of the strain-induced grain boundaries. Then some fine grains rapidly grow to a close limit that results in the transient recrystallization. The normal grain growth takes place at late annealing stages, leading to a slow grain coarsening with a grain growth exponent of about 0.25.
4. The transient recrystallization following recovery results from the homogeneous nucleation mechanism operating in the uniformly distributed strain-induced fine grains and, therefore, should be considered as a continuous phenomenon.
5. The transient recrystallization is accompanied by a change in the grain boundary misorientation distribution. In other words, the misorientation distribution gradually changes from that containing a relatively large number of low-angle boundaries to a nearly random distribution corresponding to a fully recrystallized state. The latter includes a large fraction of annealing twins.

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