

SUBSTRUCTURES AND INTERNAL STRESSES DEVELOPED UNDER WARM SEVERE DEFORMATION OF AUSTENITIC STAINLESS STEEL

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Introduction

Ultrafine-grained materials having grain sizes of tens and hundreds of nanometers, that offer much improved mechanical and physical properties, recently have aroused great interest among researches in the materials science [1–3]. Since the evolution of fine grains in metallic materials can be achieved by plastic deformation, the studies on grain refinement under thermomechanical processing are of specific practical importance. Formation of new grains during deformation is usually associated with the operation of dynamic recrystallization (DRX). Considerable refinement of initial microstructure due to DRX can be obtained by decrease in deformation temperature or increase in strain rate [4]. Studies on the microstructural changes taking place at high strains, however, are often complicated by some limited workability of most metallic materials at low-to-moderate temperatures. Recently, several techniques have been proposed to provide very high plastic deformation, such as torsion under high pressure, equal-channel angular pressing, multiple forging, etc. [3,5–13].

It has been shown that the new grain formation at around or below $0.5 T_m$ cannot result from classical DRX, but from a strain-induced continuous reaction during deformation, i.e., the development of substructure composed of high-density dislocation subboundaries with moderate-to-high angle misorientations, followed by the transformation from these subgrains into fine grains [2,6–9,11,12]. These very fine grains evolved through severe plastic deformations sometimes contain relatively lowered dislocation densities in their interiors [11,12]. This may indicate the operation of any recovery process during deformation. On the other hand, the materials prepared by high cold-to-warm deformation offer very high strength and can be characterized by high internal stresses irrespective of dislocation-free grains [2,12]. The interrelationship between such microstructures and the properties, therefore, have not been fairly clarified.

The aim of this work is to study submicron-scale substructure evolution in a 304 type stainless steel caused by severe warm deformation at $0.5 T_m$. The effects of strain-induced grain boundaries on the internal stresses and the related lattice distortions evolved in these grain interiors are discussed in detail.

Experimental

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 and the balance Fe) with an initial grain size of about 25 μm was used as the samples for compression tests. For multiple compression, the samples were machined in a rectangular shape with the starting dimension of 9.8:8.0:6.5 mm. Such dimensional ratio of about 1.5:1.22:1 did not change during subsequent compression to a strain of 0.4 in each pass. Multiple compression tests were carried out with consequent changing of the loading direction in 90° in each pass (i.e. x to y to z to x . . .). The samples were compressed in vacuum with a powder of boron nitride as a lubricant at 873 K (0.5 T_m) under a strain rate of about 10^{-3} s^{-1} . The deformed samples were quenched in water, machine ground to right-angle shape, and then reheated to 873 K within 0.6~0.8 ks in each deformation pass. The substructures evolved were examined on the sections parallel to the compression axis with JEM-2000FX transmission electron microscope operating at 200 kV. Misorientations on strain-induced dislocation subboundaries were studied using a conventional Kikuchi-line technique. The small lattice curvatures inside fine grains were studied by analysis of component for the rotation vector lying in the plane of electron diffraction pattern with accuracy within 0.1 degree.

Results and Discussions

Early multiple warm deformation brought about the evolution of high density dislocations roughly homogeneously arranged in parallel rich dislocation layers which were crossed by dense dislocation walls with low-to-middle angle misorientations. Further multiple deformation to moderate strains of above 1.0 led to the formation of recognizable subgrains. Typical example of such substructures developed at a strain of 1.6 is presented in Fig. 1a. Note here that second phase precipitations (mainly of σ -phase (FeCr)) placed along initial grain boundaries makes clear the position of the latter. It is clearly seen in Fig. 1a that many subgrains containing high density dislocations in their interiors are developed, especially, in the vicinity of initial grain boundaries.

Upon further multiple deformation to high cumulative strains the volume fraction of the highly misoriented substructures increased substantially, leading to a full development of well defined subgrains and/or fine grains throughout. The subboundaries became more narrow accompanied with increase in their misorientations and the dislocation densities in grain interiors decreased in average. It can be stressed here that some fine grains/subgrains contain quite few dislocations in their interiors. A typical substructure evolved by severe warm multiple deformation to a total strain of 6.4 is presented in Fig. 1b. It should be noted in Fig. 1b that the secondary phase precipitations do not exist at the initial grain boundaries and are randomly distributed throughout as compared to the preceding deformations. The initial grain boundaries, therefore, cannot be recognized in Fig. 1b. Such a microstructure can be generally described as fine grains with medium-to-high angle misorientations (see Fig. 2), an average grain size of which is about 0.3 μm .

The new fine grain evolution described above is assisted by dynamic and static recovery processes taking place during multiple deformation at 0.5 T_m . The transformation of strain-induced dislocation subboundaries into conventional grain boundaries, in general, should be accompanied by the increase in their misorientations as well as the dislocation rearrangements inside subboundaries. The latter may be similar to the usual spreading of dislocations taking place in grain boundaries, leading to the disappearance of lattice dislocations trapped in the boundaries. The dynamic and static recovery processes, therefore, can be important mechanisms operating in the development of strain-induced fine grains through the promotion of movement and dissociation of lattice dislocations at grain boundaries.

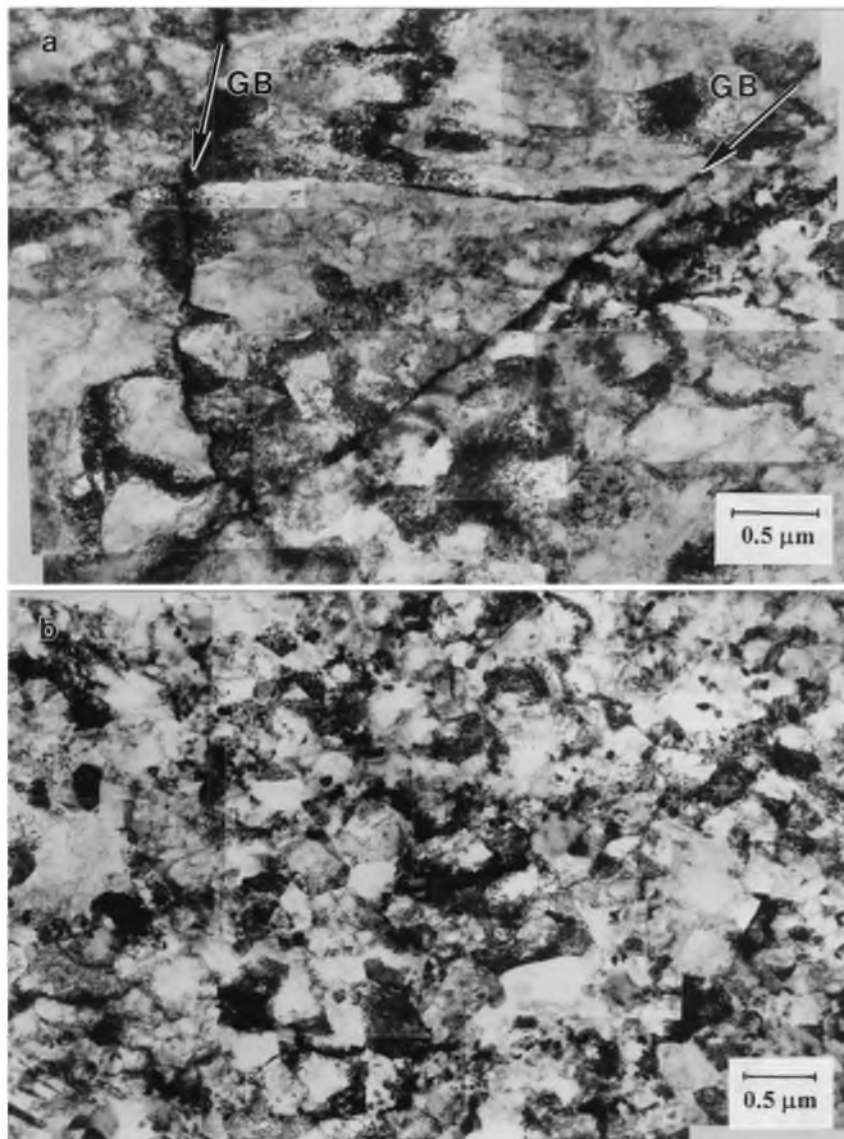


Figure 1. Typical microstructures evolved in 304 stainless steel at warm multiple deformation at $0.5T_m$. (a) Highly dislocated substructures evolved at a moderate cumulative strain of 1.6. GBs indicate the grain boundaries. (b) Fine-grained microstructure developed at a high cumulative strain of 6.4. Note here, the initial grain boundaries cannot be distinguished from those of new fine grains.

It should be noted that local migration of strain-induced subboundaries can also be assisted by recovery processes and may lead to the evolution of equiaxed fine grains.

It has been shown [2,7,12] that the fine grain evolution under severe plastic deformation was frequently accompanied by the development of high internal stresses. It is generally believed that the strain-induced grain boundaries can produce long-range stress fields in fine grain interiors because of their non-equilibrium character in addition to high density dislocations evolved by relatively low temperature deformation. To study the elastic lattice curvatures associated with such non-equilibrium

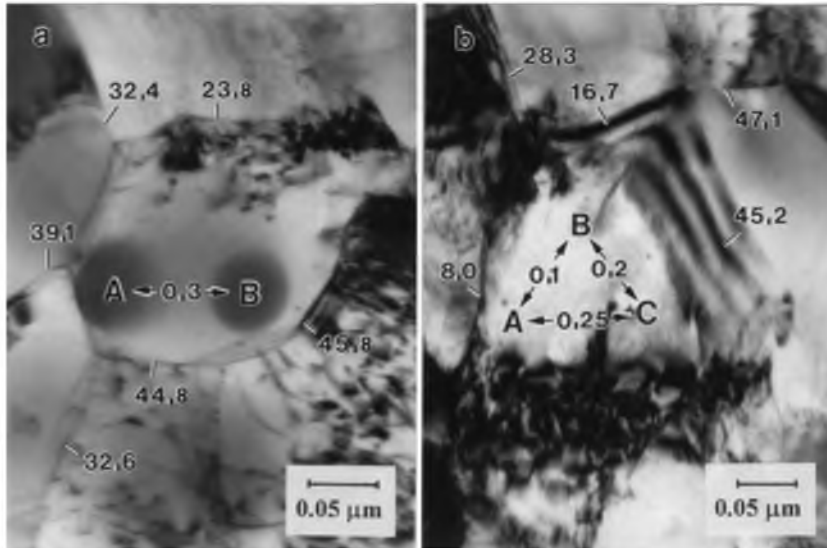


Figure 2. Typical fine grains with low dislocation densities evolved in 304 stainless steel under warm multiple deformation to cumulative strains of 3.2 (a) and 6.4 (b). The numbers indicate the misorientations in degrees. The misorientations between lettering points in grains suggest some high internal stresses evolved.

grain boundaries, some fine grains with quite low dislocation densities in their interiors were chosen. Two typical examples of such grains with medium-to-high angle boundaries are represented in Fig. 2. The misorientations evaluated in the diffraction plane between lettered points range from 0.1° (i.e. between “A” and “B” in Fig. 2b) to 0.3° (between “A” and “B” in Fig. 2a). It should be noted here the true misorientations between the selected points in Fig. 2 may be higher than those presented in Fig. 2, because only one component of rotation vector was analyzed. Since these grains are almost free of dislocations in their interiors, it is evident that the grain boundaries of fine grains produced by severe plastic deformation may have some high elastic distortions as suggested from Fig. 2. Let us consider origin of such high internal stresses evolved by warm deformation.

The material having a heterogeneous distribution of dislocation densities on subgrain scale can be considered as a composite composed of the dislocation subboundaries and the subgrain interiors as hard and soft phases, respectively [14,15]. The dislocation densities in subgrain interiors, therefore, are determined by actual stresses acting in the soft regions, which are less than applied stress by a value of internal back stress. Since the flow behaviors under warm multiple deformation are characterized by steady-state-like flows at high strains [11,12], a dynamic equilibrium between strain hardening and softening may be attained. It is reasonable to consider, however, that full annihilation cannot be reached in these strain-induced geometrically necessary subboundaries because of any difference in slip systems operating in neighboring subgrains. This leads to the accumulation of so-called grain boundary dislocations, which can be responsible to both the increase in misorientations between subgrains and the sliding along subboundaries. The excess dislocations are, generally, inhomogeneously distributed at the boundaries surrounding a grain and cannot be fully relaxed by some grain rotation. As a result, elastic lattice distortions remain in a grain. By the way, grain boundary sliding also leads to the evolution of long range elastic stresses due to accumulation of misfit dislocations at boundary junctions and legs.

The internal stresses produced by the inhomogeneously distributed grain boundary dislocations as well as the misfit dislocations accumulated at grain boundary junctions can be considered in terms of a junction distortion network [16,17]. Let us consider the interaction between a moving edge dislocation

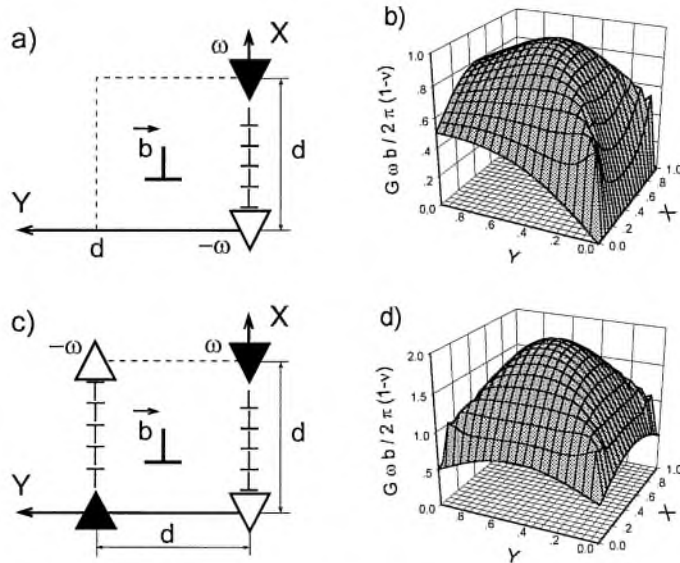


Figure 3. Interaction between a dislocation and subboundaries with junction distortions. (a, c) Schematic drawings for mutual arrangement of an edge dislocation moving towards the subboundary and the wedge disclinations at the corners. (b, d) Corresponding distributions of back forces acting on a dislocation per unit length.

and a segment of subboundary with excess grain boundary dislocations, as a simple case. This subboundary segment can be represented by that containing two distortion sources, as schematically shown in Fig. 3a. The internal stresses created by such distortion sources are analogous to those produced by two wedge disclinations with rotation vectors of ω placed at a distance of d (Fig. 3a). The back force (F) acting on a dislocation per unit length with a Burgers vector of b moving towards the grain boundary can be evaluated by the following relation [17]:

$$F = \frac{bG\omega}{2\pi(1-\nu)} \left(\frac{xy}{x^2 + y^2} + \frac{y(d-x)}{(d-x)^2 + y^2} \right) \quad (1)$$

where G and ν are the shear modulus and Poisson's ratio, respectively. This interaction force depends on the relative position of the dislocation. The force distribution developed inside a square area is shown in Fig. 3b in units of $G\omega b/2\pi(1-\nu)$. On the other hand, Fig. 3c shows a modified case for the mutual arrangement of junction disclinations and a moving dislocation. This model takes into account the accumulation of grain boundary dislocations of opposite signs at opposite boundaries of a deformed grain. Let us assume, for simplicity, the same nature for disclinations and dislocation as above. In view of a symmetry of the stress fields induced by the opposite boundaries in Fig. 3c, the back force acting on a dislocation can be evaluated through Eq.1 by simple superposition of the forces applied by opposite boundaries. The distribution of the resulting force is represented in Fig. 3d.

Of course, the exact solution for the interaction between dislocations and strain-induced grain boundaries requires more precise investigation of the latter's characters. The present simple models roughly allow to analyze the interaction between dislocation and non-equilibrium boundaries in very simplified cases. Assuming the square areas in Fig. 3a and Fig. 3c to be a cross section of subgrain, one can readily see that a maximum back force appears at a central part of the subgrain. $G = 58500$ MPa and $\nu = 0.27$ for 304 stainless steel at 873 K [18]. The maximal back shear stress in a model presented in Fig. 3c is evaluated to be $\tau \approx 130-260$ MPa for $\omega = 0.005-0.01$, i.e. $0.3^\circ-0.6^\circ$ close to the values

in Fig. 2. It is concluded that such high internal stresses are comparable with the flow stresses of $\sigma \approx 650$ MPa at $0.5 T_m$ [12] and so may be responsible to the decrease in dislocation densities evolved in subgrain interiors.

Conclusions

Strain-induced fine grain evolution taking place in a 304 type austenitic stainless steel under severe warm deformation was studied in multipass compression at a temperature of 873 K ($0.5 T_m$) with changing of the loading direction in 90° in every pass. The microstructural changes are characterized by the evolution of subgrains with their dense dislocation boundaries at low to moderate strains. These subgrains become more equiaxed and the misorientations between them gradually increase with increase in cumulative strain, finally leading to the fine grain formation with an average size of about $0.3 \mu\text{m}$. Such new fine grain evolution can be assisted by dynamic recovery, which promotes any dislocation rearrangement at their boundaries. Further, some new fine grains evolved at high strains contain quite few dislocations in their interiors, resulting in decreasing average dislocation densities with the evolution of fine-grains having highly misoriented boundaries. On the other hand, these fine grains can be characterized by high lattice curvatures irrespective of lowered dislocation densities. Such high internal stresses may be explained by consideration of the junction distortions developed at non-equilibrium boundaries under severe plastic deformation, resulting in the decrease in dislocation densities evolved in fine grain interiors.

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References

1. H. Gleiter, *Prog. Mater. Sci.*, 33, 1 (1990).
2. R. Z. Valiev, *Ann. Chim. Fr.* 21, 369 (1996).
3. F. J. Humphreys, P. B. Prangnell, and R. Priestner, in *Recrystallization and Related Phenomena (ReX'99)*, ed. T. Sakai and H. G. Suzuki, p. 69, The Japan Institute of Metals (1999).
4. T. Sakai and J. J. Jonas, *Acta Metall.* 32, 189 (1984).
5. V. M. Segal, V. I. Reznikov, A. E. Drobyshevskiy, and V. I. Kopylov, *Russian Metallurgy*, 1, 99 (1981).
6. N. A. Smirnova, V. I. Levit, V. I. Pilyugin, R. I. Kuznetsov, L. S. Davydova, and V. A. Sazonova, *Phys. Met. Metallogr.* 61, 127 (1986).
7. A. Belyakov, R. Kaibyshev, and T. Sakai, in *Interface Science and Materials Interconnection (iib'96, JIMIS-8)*, ed. Y. Ishida et al., p. 495, Japan Institute of Metals (1996).
8. M. Furukawa, Z. Horita, M. Nemoto, R. Z. Valiev, and T. G. Langdon, *Acta Mater.* 44, 4619 (1996).
9. G. A. Salishchev, R. G. Zaripova, A. A. Zakirova, and H. J. McQueen, in *Hot Workability of Steels and Light Alloys-Composites*, ed. H. J. McQueen et al. p. 217, TMS-CIM, Montreal (1996).
10. O. V. Mishin and G. Gottstein, *Phil. Mag. A*, 78, 373 (1998).
11. A. Belyakov, W. Gao, H. Miura, and T. Sakai, *Metall. Trans. A*, 29A, 2957 (1998).
12. A. Belyakov, T. Sakai, H. Miura, and R. Kaibyshev, *ISIJ Int.* 39, 593 (1999).
13. M. Richert, Q. Liu, and N. Hansen, *Mater. Sci. Eng. A*, A260, 275 (1999).

14. W. D. Nix and B. Ilshner, in *International Conference on Strength of Metals and Alloys (ICSMA 5)*, ed. by P. Haasen et al., p. 1503, Pergamon Press, Oxford (1980).
15. H. Mughrabi, *Acta Metall.* 31, 1367 (1983).
16. A. A. Nazarov, A. E. Romanov, and R. Z. Valiev, *Scripta Mater.* 34, 729 (1996).
17. A. E. Romanov and V. I. Vladimirov, in *Dislocations in Solids*, ed. F. R. N. Nabarro, vol. 9, p. 191, Elsevier Science, New York (1992).
18. H. J. Frost and M. F. Ashby, *Deformation-Mechanism Maps*, p. 62, Pergamon Press, Oxford (1982).