Ultrafine Grain Formation in Ferritic Stainless Steel during Severe Plastic Deformation

T. SAKAI, A. BELYAKOV, and H. MIURA

The development of submicrocrystalline structures in Fe-20 pct Cr ferritic stainless steel was studied in multidirectional forging to large total strains. The structural changes are characterized by the development of microshear bands in high density dislocation substructures. The multidirectional deformation promotes the multiple shearing, which results in the formation of a spatial net of mutually crossed microshear bands subdividing the original grains. The new grains with high-angle boundaries appear primarily at the microshear band intersections and subsequently along the bands. The fraction of ultrafine grains gradually increases with increasing the density of microshear bands as a result of continuous increase in misorientations among deformation subgrains during processing. An increase in the processing temperature can accelerate remarkably the kinetics of ultrafine grain evolution at large strains. The mechanism of strain-induced grain formation is discussed in detail.

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I. INTRODUCTION

THE growing interest in studies on ultrafine-grained metallic materials is motivated by engineering requirements for production of fine-scale parts mainly for electromechanical and medical devices as well as by attractive mechanical properties, such as high strength with sufficient ductility,[1] enhanced impact toughness,[2] low-temperature or high-strain-rate superplasticity,[3] and many others occasionally reported for submicrocrystalline metals and alloys.[4,5] One of the most promising methods for production of ultrafine-grained materials is based on severe deformation, *i.e.*, processing by large strain plastic working at relatively low temperatures.[5,6] Several special techniques were developed to achieve very large strains. Among those are mechanical milling,[7,8] torsion under high pressure,[9] equal channel angular extrusion,[10,11] accumulative roll bonding,[12] *etc*., which are applicable to a wide variety of structural metals and alloys. These processing methods, however, require costly equipment and have to use some additional consolidation working to make a bulky product. On the other hand, some conventional processing methods such as multiple forging, which is well known from ancient times,[13] can also provide a very large strain in forged sample. Recently, the multidirectional or three-dimensional (3-D) cross forging was successfully used as a method of severe deformation.[14,15] The advantages of this method are not only standard facilities and processing of sizeable samples, but also provision of flow stress behaviors during severe deformation.[16]

It is generally agreed that the structural changes leading to submicrocrystalline structures during severe deformation follow a common sequence of microstructure evolution irrespective of differences in the processing methods.[14–21] The new fine grains result from a kind of strain-induced continuous reaction, which is the formation of dislocation sub-boundaries, especially geometrically necessary sub-boundaries, subdividing microvolumes with different operating slip systems, and gradual rise in the sub-boundary misorientations up to typical values of ordinary high-angle boundaries (HABs) in large strain. Any deformation heterogeneities, therefore, are expected to play an important role in the grain refinement by severe deformation. Large shear strains were shown to result in a rapid increase of misorientations among strain-induced sub-boundaries.[16,21–25] However, regulations of such a mechanism of microstructure evolution are not perfectly understood. The final grain size that evolved in strain-induced submicrocrystalline structures can be reduced by decreasing the deformation temperature.[18–20,23,26,27] On the other hand, it is not clear how the processing conditions affect the kinetics of submicrocrystalline structure development. The role of dynamic recovery has not been studied systematically in sufficient detail. Decreasing the deformation temperature in some studies on copper is shown to retard the formation of new ultrafine-grained microstructure, while strain-induced HABs start to develop at a critical strain irrespective of the temperature.[14,16,27] As the processing temperature decreases, the submicrocrystalline structures with certain fraction of HABs are developed at larger strains. In contrast, an increase in the processing temperature leads to decreasing the volume fraction of new fine grains in

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an aluminum alloy.[28] This can be attributed to relaxation of strain compatibility between grains due to frequent operation of dynamic recovery at higher temperature.

The aim of the present work was to study the operating structural mechanism responsible for ultrafine grain evolution in a ferritic stainless steel during multiple forging to large strains at temperatures below 0.5 of the melting point (T_m). Specifically, the intents of this study are to clarify the role of deformation bands in continuous dynamic recrystallization and to make clear the effect of dynamic recovery on the kinetics of strain-induced submicrocrystalline structure development during warm working. The multidirectional forging at 773 K was scheduled to elucidate the sequence of structural changes, while the forging at 573 K was intended to study the temperature effect on the microstructure evolution.

II. EXPERIMENTAL PROCEDURE

A ferritic stainless steel, Fe-0.002C-0.04Si-0.004Al-20Cr (all in mass pct), was selected as a typical material with high ability to recovery. The starting material with grain size of 240 μm was obtained after homogenization annealing at 1003 K for 1.8 ks. The multidirectional forging was carried out by multipass compressions, while the loading direction was rotated 90 deg from pass to pass (*i.e.*, x to y to z to x …).[14–16] The tests were conducted with a strain rate of 10^{-3} s^{-1} at temperatures of 573 K (0.32 T_m) and 773 K (0.43 T_m). The specimens were machined in a rectangular shape with dimensions of 9.0 mm:7.3 mm:6.0 mm. The strain applied in each pass was 0.4 to keep the initial dimensional ratio of 1.5:1.22:1.0 constant through the all compressions. The deformed samples were quenched in water, slightly ground to right-angle shape, and then reheated to the testing temperatures. The specimens were subjected to 20 subsequent compression passes; therefore, the total strain of 8 was attained. Microstructural observations were carried out by using an optical microscope and a JEM-2000FX (JEOL*) transmission electron microscope operating at 200 kV on sample sections parallel to the last pass compression axis. Misorientations between deformation grains/subgrains were analyzed by the conventional Kikuchi-line technique. The total number of the (sub)boundaries was about 100 at each studied strain level.

*JEOL is a trademark of Japan Electron Optics Ltd., Tokyo.

III. RESULTS

A. *Stress-Strain Curves during Multidirectional Forging*

Two sets of interrupted stress-strain curves obtained during multidirectional forgings at 573 and 773 K are shown in Figure 1. Commonly, the differences between the yield stresses at reloading and the flow stresses just before unloading are quite small for all compression passes at both temperatures. Any static recrystallizations, therefore, hardly take place during the studied multidirectional forging. In this case, the deformation behavior including structural changes can be considered as that resulting from strain accumulation, much similar to a deformation behavior upon monotonous processing that is accompanied by only recovery processes.

The general shape of the flow curve envelope at both temperatures suggests a remarkable strain hardening at early deformation to total strain about 1 followed by a decrease of hardening rate during subsequent processing. The flow stresses at both temperatures are almost the same in the strain range of $0 < \varepsilon < 1.5$, whereas larger strains are characterized by different deformation behavior with higher flow stresses at lower temperature. During the multidirectional forging at 773 K, the rate of strain hardening smoothly decreases to almost zero, leading to steady-state-like flow behavior at total strains beyond 1.5. In contrast, the deformation at 573 K is accompanied by continuous strengthening of the samples, when the rate of strain hardening gradually decreases during the processing.

Both types of deformation behaviors are typical of plastic deformations, which are controlled by dynamic recovery. A steady-state deformation behavior is frequently observed during warm and hot working of materials with high dislocation mobility,[29] while an attenuating strain hardening is commonly reported for cold working of various metallic materials.[30] Deformation microstructures in these cases change in accordance to a total strain and approach some final state, which corresponds to steady-state flow behavior, when the main structural parameters remain invariant during

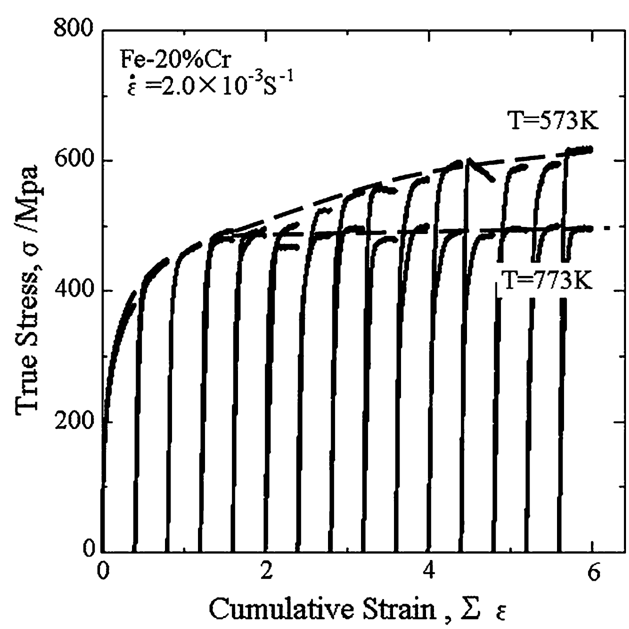

Fig. 1—Stress–total strain curves plotted over 15 multidirectional forging passes at temperatures of 573 and 773 K.

further processing. It is worthwhile, therefore, to consider the microstructural evolution taking place during the studied multidirectional forgings as a strain-induced phenomenon.

B. *Deformation Microstructures*

Typical deformation microstructures that developed at different strain levels at 773 K are presented in Figure 2. Two sets of deformation microbands intersecting at almost 90 deg angles are clearly revealed in the sample after two forging passes (Figure 2(a)). The number of microband families running in various directions increases with following multiple deformations. Many sets of the mutually crossed deformation microbands appear at a strain of 1.6 (Figure 2(b)). An increasing density of the deformation microbands is a feature of further processing to large total strains. Individual deformation microbands become hard to be resolved by optical microscopy when their density increases significantly (Figures 2(c) and (d)). After sufficiently large total strains, the deformation microbands homogeneously fill in the volume of the forged sample. The deformation microstructure in Figure 2(d) looks like a uniform ultrafine-grained one. This suggests that submicrocrystalline structure is completely evolved in the sample that has been subjected to multidirectional forging to a total strain of 6 at 773 K.

Figure 3 represents the enlarged micrographs corresponding to the selected portions of the deformation structures in Figures 2(a) and (b). The details of the microband interaction can be seen more clearly. After the second forging pass (Figure 3(a)), one of two families of deformation bands appears as wavy lines that are sheared and slipped off by another family of deformation bands. Therefore, the volumes of these deformation microbands correspond to localized plastic shearing, and the microbands should be considered as *microshear bands*. Evidently, the slipped bands are primary microshear bands and the straight ones are secondary microshear bands, which probably evolved during the second forging pass. The number of various shear bands increases with increasing the number of forging passes (Figure 3(b)). In other words, the multiple forging with changing the loading direction results in the development of multiple shearing. An operation of multiple slips is an important condition for subgrain formation and dynamic recrystallization during hot working.[29,31–33] In a similar manner, the operation of multiple shearing may be important for strain-induced

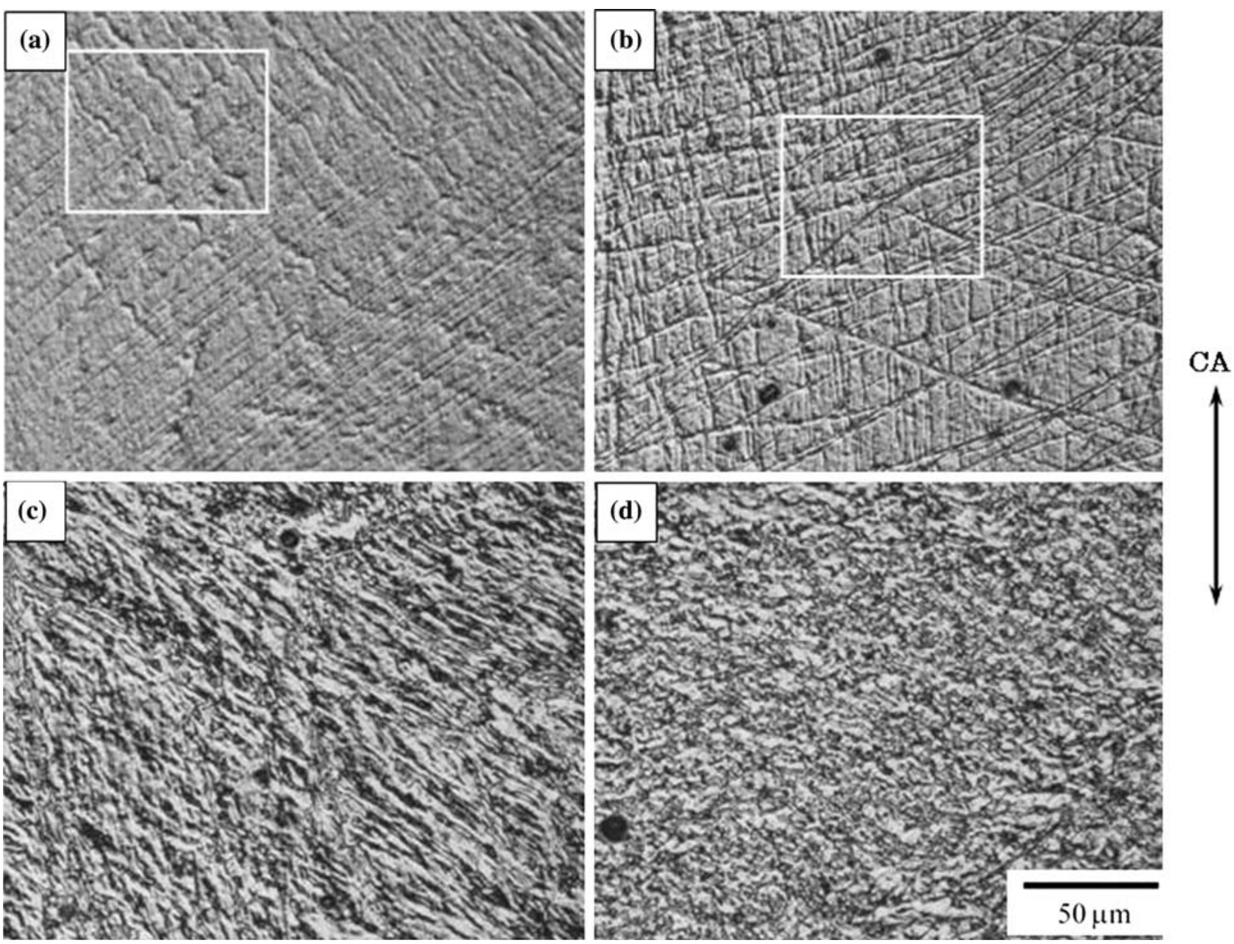

Fig. 2—Typical microstructures evolved in Fe-20 pct Cr steel after multidirectional forging at 773 K to total strains of (*a*) 0.8, (*b*) 1.6, (*c*) 3.6, and (*d*) 6.0. The CA indicates the direction of the forging axis at the last pass.

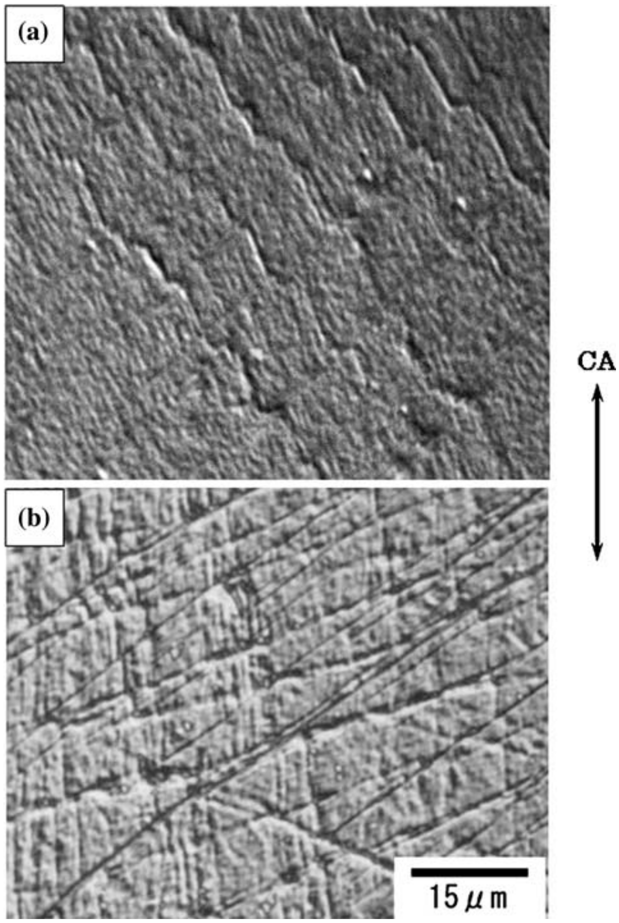

Fig. 3—Detailed micrographs showing the development of microshear bands in Fe-20 pct Cr steel after multidirectional forging at 773 K to total strains of (*a*) 0.8 and (*b*) 1.6. The pictures represent the selected portions in Figs. 2(a) and (b).

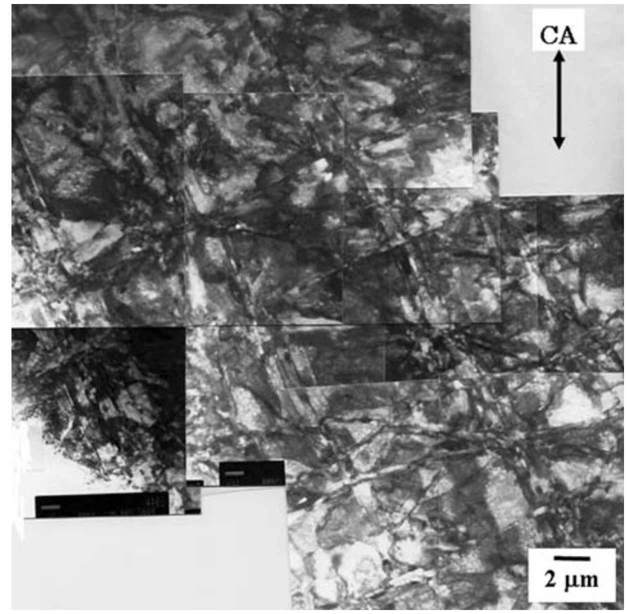

Fig. 4—TEM microstructure of microshear bands that cross the deformation substructures in Fe-20 pct Cr steel after multidirectional forging at 773 K to a total strain of 1.6.

submicrocrystalline structures upon severe cold working. Let us consider the deformation microstructures in more detail.

The development of microshear bands due to multiple shearing subdivides the cellular substructure that was homogeneously evolved at early plastic deformation. The deformation substructure with a number of mutually crossed microshear bands evolved at a total strain of 1.6 is shown in Figure 4. The multiple shearing results in a spatial net of microshear bands that separate the microvolumes containing high density dislocations, which are arranged in rather uniform dislocation cells. Cell blocks separated by strain-induced geometrically necessary sub-boundaries are commonly observed in cold-worked metallic materials.[17,34] The present microshear bands are considered as a kind of geometrically necessary sub-boundaries crossing over initial grains. Figure 5 represents a portion of the deformation substructure corresponding to a microshear band intersection. The intersection of microshear bands is characterized by the evolution of almost equiaxed ultrafine subgrains with relatively sharp sub-boundaries, many of which have high-angle misorientations. On the other hand, the dislocation sub-boundaries that located far from the microshear intersection are diffuse in the image and bear low-angle misorientations. This confirms that the microshear band intersections can be considered as preferential sites for the development of highly misoriented submicrocrystalline structure.[27]

Because the number of the microshear band intersections increases upon further multidirectional forging, the ultrafine-grained regions expand and take larger fractions among deformation substructures (Figure 6). The deformation substructure after processing to a total strain of 3.6 is characterized by large angular misorientations between (sub)grains, as suggested by a ringlike diffraction pattern taken from a selected area, although dislocation cells with low-angle diffuse sub-boundaries are also clearly seen (Figure 6(a)). Thus, a mixed deformation structure consisting of the equiaxed ultrafine grains and the cellular substructure between microshear bands is evolved at an intermediate strain level of about 2 to 4. Severe deformation to total strain of 8 results in almost complete development of the ultrafine grained structure, which propagates throughout the forged sample (Figure 6(b)). The average grain size is about 500 nm. This strain-induced submicrocrystalline structure involves highly misoriented equiaxed ultrafine grains with a relatively low dislocation density in their interiors as compared with high dislocation densities in cell substructures that evolve at smaller strains (Figure 5). The ultrafine grains are outlined by sharp grain boundaries with distinct extinction fringes, which are typical of ordinary grain boundaries in recrystallized microstructures.

Generally, the sequence of structural changes during the multidirectional forging at lower temperature (573 K) is essentially the same as described previously. The multiple deformation to large total strains results in

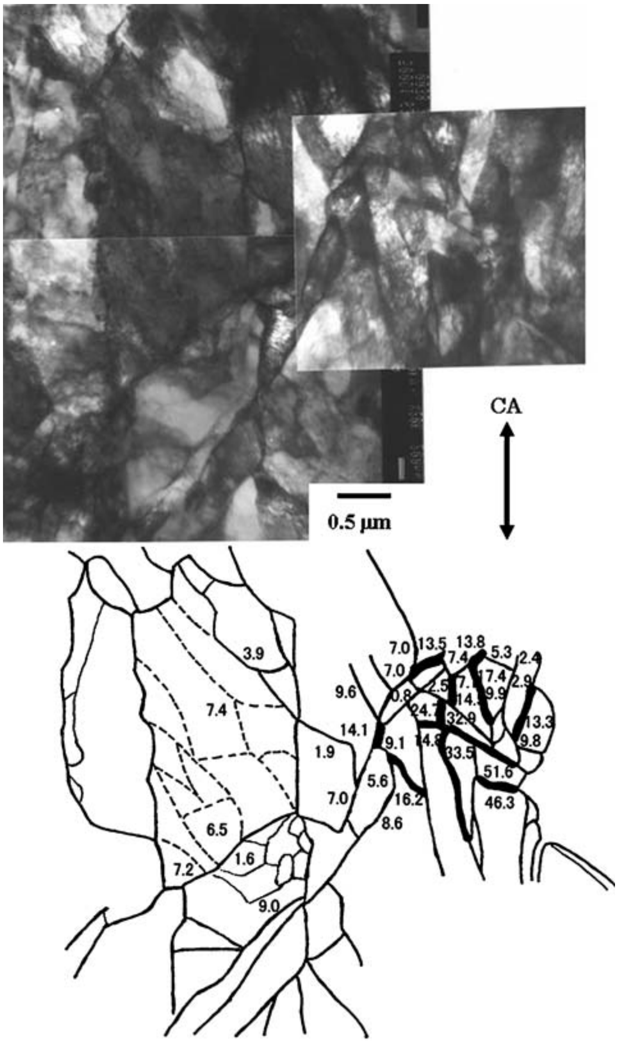

Fig. 5—Ultrafine grains developed at a microshear band intersection at a total strain of 1.6 at 773 K. The numbers indicate the misorientation angles in degrees.

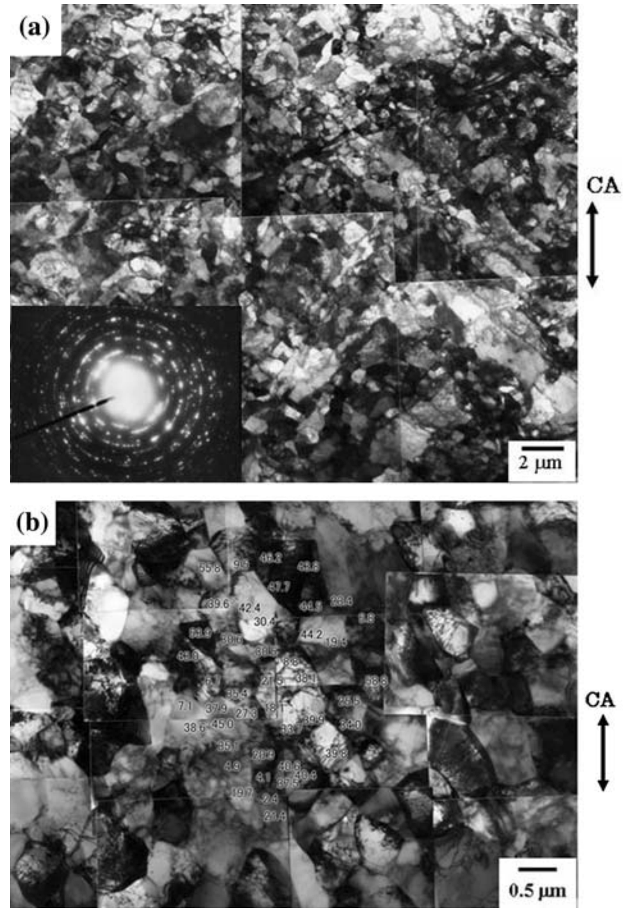

Fig. 6—Development of submicrocrystalline structures in Fe-20 pct Cr steel upon multidirectional forging at 773 K to total strains of (*a*) 3.6 and (*b*) 8.0.

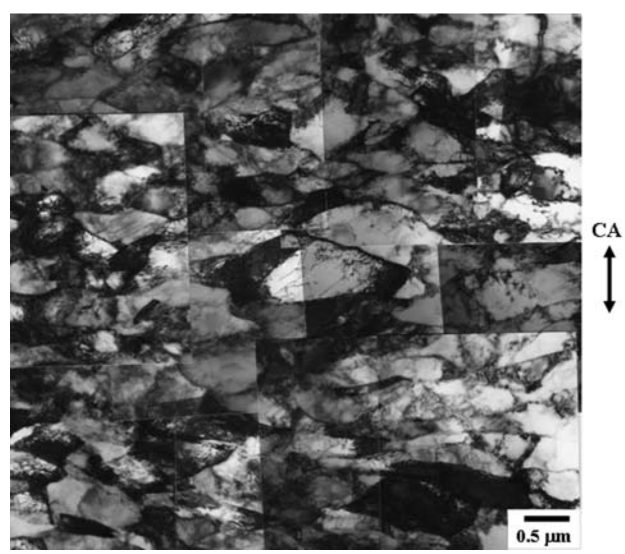

Fig. 7—Ultrafine grains evolved in Fe-20 pct Cr steel after multidirectional forging to a total strain of 6.0 at 573 K.

development of ultrafine-grained microstructure, an example of which is presented in Figure 7. Compared to the ultrafine-grained microstructure after processing at the higher temperature, the microstructure developed at a strain of 6 in Figure 7 shows a mixed one containing high- and low-angle dislocation boundaries and incorporates the following distinctive features: higher dislocation densities in fine grain interiors, more ragged grain boundaries, which do not display extinction fringes in their images, and somewhat flattened grains with respect to the deformation axis in the last forging pass.

The evolution of misorientations in deformation structures is shown in Figure 8. The distribution of (sub)boundary misorientations after processing to a relatively small total strain of 1.6 is characterized by a large fraction of low-angle dislocation sub-boundaries, which are associated with cellular substructures. The high-angle boundaries making up a small fraction at this strain level result from rare ultrafine-grained regions that developed at microshear band intersections (Figure 5). Upon further multiple deformations, the fraction of high-angle grain boundaries gradually increases. Finally, the misorientation distribution with almost equal fractions of various grain/subgrain

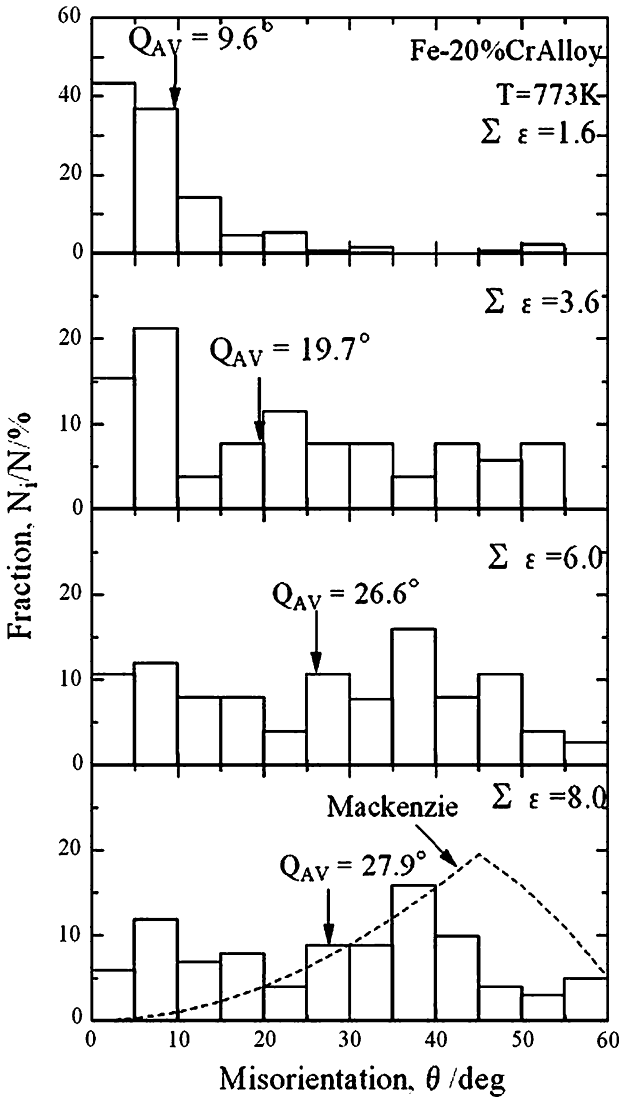

Fig. 8—Misorientation distributions for grain/subgrain boundaries that evolved in Fe-20 pct Cr steel during multidirectional forging at 773 K to various total strains.

boundaries evolves after severe deformation to a total strain of 8. The random misorientation distribution of cubic crystals is shown by the dotted line in Figure 8 for a reference.

The continuous increase in misorientations between deformation subgrains during plastic working is an essential feature of continuous dynamic recrystallization. Kinetics of this process can be illustrated by the relationship between total strain and the average (sub)boundary misorientation (θ_{av}) or the fraction of HABs (F_{HAB}) in Figures 9(a) and (b), respectively. The process of the misorientation increase can be virtually subdivided into three stages. The first stage is characterized by a rapid rise in average misorientation of deformation (sub)boundaries followed by a small plateau at a level of about 5 deg. Then, after some critical strain of around 1.5, the misorientation of strain-induced (sub)boundaries starts to increase again rapidly. This can be considered as the second stage of continuous development of submicrocrystalline structure. The third

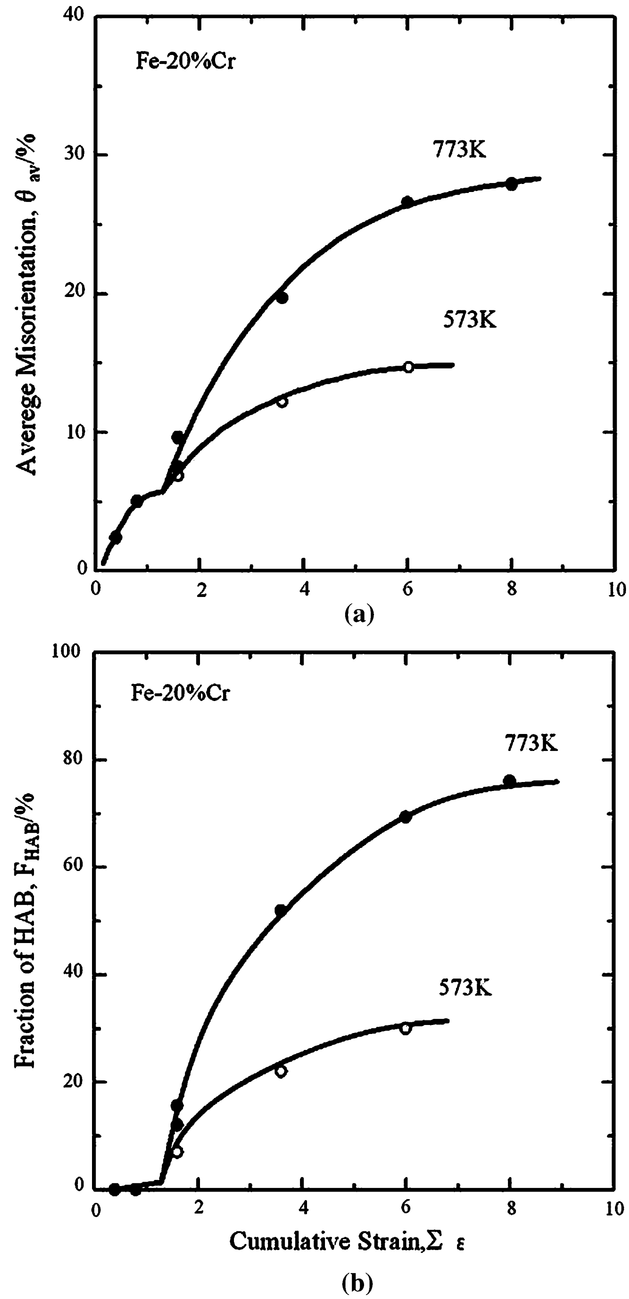

Fig. 9—Relationship between (*a*) the average misorientation of strain-induced (sub)boundaries (θ_{av}) or (*b*) the fraction of strain-induced HABs (F_{HAB}) and the total strain during repeated MDF at 573 and 773 K.

stage corresponds to large strains, when the average misorientation between strain-induced ultrafine grains approaches its saturation level. Changes in the fraction of HABs (F_{HAB}) with deformation are also similar to those in the θ_{av} (Figure 9(b)). Namely, the strain-induced HABs are scarcely developed at $\varepsilon < 1.5$, and then the F_{HAB} starts to increase and approaches a saturation value of about 75 pct at 773 K or that of 30 pct at 573 K.

It is clearly seen in Figure 9 that the temperature effect on the kinetics of continuous development of new ultrafine-grained structure for the studied material and processing method is negligibly small within the first stage of the evolutional process. The average value of misorientation for deformation (sub)boundaries rapidly increases with straining at the beginning of the second stage irrespective of the processing temperature. However, the decrease of deformation temperature slows the rate of misorientation rise significantly during the third stage of the process. As a result, the saturation level of average grain boundary misorientation at large strains is remarkably lower for deformation at lower temperature.

IV. DISCUSSION

A. *A Process of Strain-Induced Grain Formation*

The present results testify that the development of submicrocrystalline structures in ferritic stainless steel subjected to multidirectional forging to large total strains is due to a general process of grain refinement that is observed in various metallic materials during severe plastic deformations.[15–23] The fraction of ultrafine grains and the average misorientation of strain-induced (sub)boundaries gradually increase to their saturation levels upon processing. Therefore, the process of the microstructure evolution can be discussed as *in situ* or continuous dynamic recrystallization.

The structural changes leading to the submicrocrystalline structure after large strains by multidirectional forging can be illustrated by a schematic drawing in Figure 10. Early deformation brings about high density dislocations that are homogeneously arranged in the cellular substructure. In the present study, the strain range of $0 < \varepsilon < 1.5$ can be considered as an incubation period of the process of continuous dynamic recrystallization, when the plastic deformation actuates multiple slip leading to fragmentation of initial coarse grains into small domains through the development of microshear bands. The average angular misorientation in such a dislocation cell block structure is about 5 deg (Figure 9). Similar values of sub-boundary misorientations are frequently reported for rather homogeneous dislocation substructures evolved by cold working.[30,34] The development of narrow microshear bands followed by the fragmentation of original grains is not enough for remarkable rotations of individual domains within an initial grain. The crystal orientation of small adjacent domains separated by a sharp microshear band, *e.g.*, the domains labeled with A and B in Figure 10(a), seems to be the same.

The subsequent multiple deformation to strains above 1.5 is characterized by a rapid increase in the density of microshear bands and the number of high-angle (sub)boundaries. However, the new fine grains are not evolved homogeneously throughout the deformation substructures, but mainly inside the microshear bands and, especially, at the intersections of the microshear bands (Figure 10(b)). The latter ones are the microvolumes with local accumulation of geometrically necessary dislocations, which are caused by the operation of different slip systems. The local variation of active slip systems is considered as a necessary condition for lattice

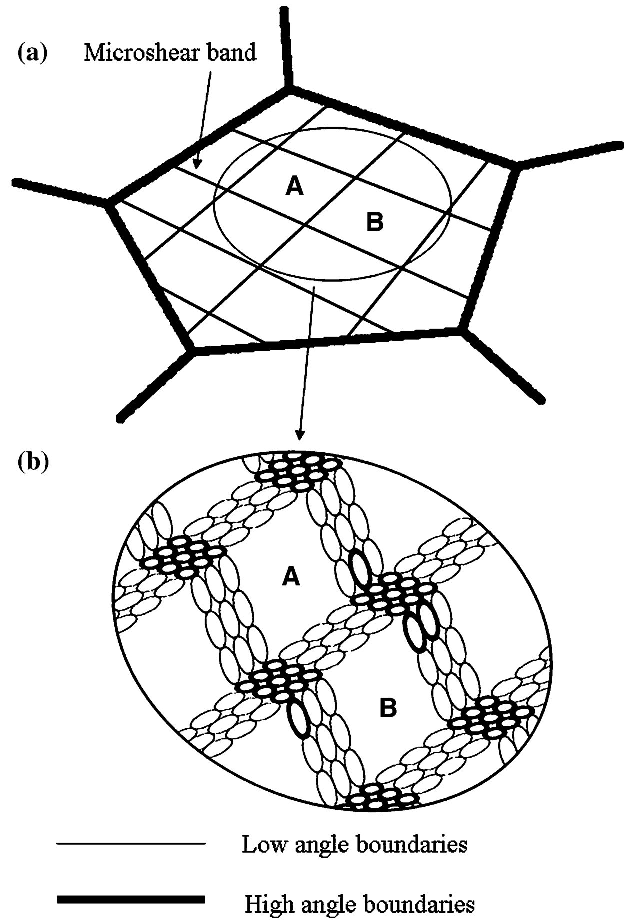

Fig. 10—Schematic drawing of the development of (*a*) microshear bands at low strains and (*b*) subsequent formation of new grains at the intersections and along the microshear bands at sufficiently large strains.

rotations leading to the formation of new grains along the microbands.[35] Dynamic recovery seems to be another important condition for the development of new grains by assisting the dislocation rearrangements in microshear bands and improving the shape and internal structure of strain-induced grain boundaries. The fraction of the ultrafine-grained structure gradually increases with increasing the number of microshear intersections, which is promoted by the multidirectional deformation mode. This suggests that the strain-induced submicrocrystalline structure develops in an apparently heterogeneous manner on a mesoscale level, *i.e.*, size of initial coarse grains. The deformation cell substructures and the new ultrafine grains are simultaneously present as two structural components at strains above 1.5, and the relative fractions of these two components continuously change with increasing the total strain. This is a feature of continuous dynamic recrystallization, when the plastic flow of metallic material is localized in crossing microshear bands.

B. *Temperature Effect*

The kinetics of continuous dynamic recrystallization slows remarkably with a decrease in deformation temperature, as can be seen in Figure 9. The average misorientation of the strain-induced boundaries developed after multidirectional forging to large strains above 5 at a temperature of 573 K comprises about half that obtained at 773 K. However, the difference in the misorientations among deformation (sub)boundaries evolving at different processing temperatures can be observed after large strains beyond 2. In the range of relatively small strains, $0 < \varepsilon < 2$, the rise of the average misorientation between subgrains is associated with the development of microshear bands. It is clear from Figure 9 that the average misorientation between subgrains is almost temperature independent in this strain region. Similar results were also reported for the early stage of continuous dynamic recrystallization developing upon large strain deformation of copper and aluminum alloy.[27,28] Therefore, the initial grain subdivision by the microshear bands can be considered as an athermal process that is a mechanically induced event.

On the other hand, the temperature effect on the evolution of submicrocrystalline structures becomes significant with subsequent processing to large total strains. An increase in the processing temperature results in a rapid rise of misorientations among deformation subgrains in this stage, which is considered as a thermal process. This may be related to the improvement of dynamic recovery, which leads to acceleration of dislocation rearrangements inside microshear bands and their intersections at elevated temperatures. The rapid dynamic recovery results in the steady-state-like deformation behavior during multiple forging at 773 K (Figure 1). The submicrocrystalline structures evolved at higher temperature are more sophisticated; *i.e.*, they are composed of more equiaxed grains with sharp grain boundaries and lower dislocation densities (Figures 6 and 7). The slow rise of the sub-boundary misorientations during deformation at lowered temperature also suggests that the strain localization in shear bands on macroscale level (within an entire sample) is more pronounced on decreasing the processing temperature. Regardless of the microshear bands leading to deformation heterogeneities on the mesoscale level (individual original grain), processing at higher temperatures is characterized by more uniform deformation microstructures on the macroscale level. That is to say, an increase in deformation temperature promotes the formation of a rich spatial net of microshear bands, which in turn lead to rather rapid evolution of strain-induced submicrocrystalline structures during multidirectional forging. In contrast to the warm-to-hot working of an aluminum alloy,[28] when the fraction of dynamically recrystallized grains decreased with increase of deformation temperature, the ferritic steel demonstrates an opposite tendency during warm deformation.

V. CONCLUSIONS

The development of submicrocrystalline structures in Fe-20 pct Cr ferritic steel during multidirectional forging at temperatures of 573 and 773 K was studied. The main results are summarized as follows.

1. The general shape of integrated stress-strain curves, plotted over a number of forging passes to a total strain of 6, is similar to a flow curve reflecting the recovery-controlled dynamic processes. Namely, after rapid increase of the flow stresses at early deformation, the rate of strain hardening gradually decreases upon subsequent multiple deformation. The processing at 573 K is accompanied by the continuous increase in the flow stresses within the studied strain range. On the other hand, the rate of strain hardening decreases to zero during the deformation at 773 K, leading to steady-state-like behavior at strains above 1.5.
2. The structural changes are associated with the development of microshear bands. The multidirectional deformation promoted multiple shearing, which results in the formation of a spatial net of mutually crossed microshear bands subdividing the original grains. This is a mechanically induced event and so an athermal process.
3. The submicrocrystalline structures with a grain size of about 500 nm are developed at 773 K in the forged samples as a result of a continuous increase in misorientations among deformation subgrains during processing. The new ultrafine grains with high-angle boundaries appear primarily at the microshear band intersections. The fraction of ultrafine grains gradually increases with increasing the density of microshear bands running in various directions due to multidirectional deformation.
4. The fast operation of recovery processes at elevated temperature accelerates the kinetics of ultrafine grain evolution during large strains, which is considered as a thermal process. The average value of misorientations across the strain-induced subboundaries, evolved during multidirectional forging to total strains above 2 at 773 K, is remarkably larger than that developed at 573 K.

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REFERENCES

1. Y. Wang, M. Chen, F. Zhou, and E. Ma: *Nature*, 2002, vol. 419, pp. 912–15.
2. V.V. Stolyarov, R.Z. Valiev, and Y.T. Zhu: *Appl. Phys. Lett.*, 2006, vol. 88, Art. no. 041905.
3. F. Musin, R. Kaibyshev, Y. Motohashi, and G. Itoh: *Scripta Mater.*, 2004, vol. 50, pp. 511–16.
4. H. Gleiter: *Progr. Mater. Sci.*, 1989, vol. 33, pp. 223–315.
5. R.Z. Valiev, R.K. Islamgaliev, and I.V. Alexandrov: *Progr. Mater. Sci.*, 2000, vol. 45, pp. 103–89.
6. R.Z. Valiev, Y. Estrin, Z. Horita, T.G. Langdon, M.J. Zehetbauer, and Y.T. Zhu: *JOM*, 2006, vol. 58, pp. 33–39.

7. C.C. Koh: *Nanostr. Mater.*, 1997, vol. 9, pp. 13–22.
8. S. Takaki, T. Tsuchiyama, K. Nakashima, H. Hidaka, K. Kawasaki, and Y. Futamura: *Met. Mater. Int.*, 2004, vol. 10, pp. 533–39.
9. I. Saunders and J. Nutting: *Met. Sci.*, 1984, vol. 18, pp. 571–75.
10. V.M. Segal, V.I. Reznikov, A.E. Drobyshevskiy, and V.I. Kopylov: *Russ. Metall.*, 1981, vol. 1, pp. 115–23.
11. Y. Iwahashi, Z. Horita, M. Nemoto, and T.G. Langdon: *Acta Mater.*, 1997, vol. 45, pp. 4733–41.
12. N. Tsuji: in *Severe Plastic Deformation*, A. Burhanettin, ed., Nova Science, New York, NY, 2005, pp. 543–64.
13. J. Wadsworth and O.D. Sherby: *Progr. Mater. Sci.*, 1980, vol. 25, pp. 35–68.
14. A. Belyakov, W. Gao, H. Miura, and T. Sakai: *Metall. Mater. Trans. A*, 1998, vol. 29A, pp. 2957–65.
15. A. Belyakov, K. Tsuzaki, H. Miura, and T. Sakai: *Acta Mater.*, 2003, vol. 51, pp. 847–61.
16. T. Sakai, H. Miura, and X. Yang: *Mater. Sci. Eng. A*, in press.
17. B. Bay, N. Hansen, D.A. Hughes, and D. Kuhlmann-Wilsdorf: *Acta Metall. Mater.*, 1992, vol. 40, pp. 205–19.
18. F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia, and C. Harris: *Phil. Trans. R. Soc. London*, 1999, vol. 357, pp. 1663–81.
19. P.B. Prangnell, J.R. Bowen, and A. Gholinia: in *Science of Metastable and Nanocrystalline Alloys*, A.R. Dinesen, M. Eldrup, D. Juul Jensen, S. Linderoth, T.B. Pedersen, N.H. Pryds, S.A. Pedersen, and J.A. Wert, eds., Risø National Laboratory, Roskilde, Denmark, 2001, pp. 105–26.
20. A. Belyakov, T. Sakai, H. Miura, and K. Tsuzaki: *Phil. Mag. A*, 2001, vol. 81, pp. 2629–43.
21. O. Sitdikov, T. Sakai, A. Goloborodko, H. Miura, and R. Kaibyshev: *Phil. Mag.*, 2005, vol. 85, pp. 1159–75.
22. M. Umemoto: *Mater. Trans.*, 2003, vol. 44, pp. 1900–11.
23. A. Belyakov, Y. Kimura, and K. Tsuzaki: *Acta Mater.*, 2006, vol. 54, pp. 2521–32.
24. A. Belyakov, M. Murayama, Y. Sakai, K. Tsuzaki, M. Okubo, M. Eto, and T. Kimura: *J. Electron. Mater.*, 2006, vol. 35, pp. 2000–08.
25. T. Inoue, F. Yin, and Y. Kimura: *Mater. Sci. Eng. A*, 2007, vol. A466, pp. 114–22.
26. S.V.S. Narayana Murty, S. Torizuka, and K. Nagai: *Mater. Trans.*, 2005, vol. 46, pp. 2454–60.
27. C. Kobayashi, T. Sakai, A. Belyakov, and H. Miura: *Phil. Mag. Lett.*, 2007, vol. 87, pp. 751–66.
28. I. Mazurina, T. Sakai, H. Miura, O. Sitdikov, and R. Kaibyshev: *Mater. Sci. Eng. A*, 2008, vol. 486, pp. 662–71.
29. H.J. McQueen and J.J. Jonas: in *Treatise on Materials Science and Technology*, R.J. Arsenault, ed., Academic Press, New York, NY, 1975, pp. 393–493.
30. J. Gill Sevillano, P. Van Houtte, and E. Aernoudt: *Progr. Mater. Sci.*, 1981, vol. 25, pp. 69–412.
31. T. Sakai and J.J. Jonas: *Acta Metall.*, 1984, vol. 32, pp. 189–209.
32. T. Sakai and J.J. Jonas: in *Encyclopedia of Materials: Science and Technology*, K.H. Buschow, R.W. Cahn, M.C. Flemings, B. Ilschner, E.J. Kramer, and S. Mahajan, eds., Elsevier, Oxford, United Kingdom, 2001, vol. 7, pp. 7079–84.
33. A. Dehghan-Manshadi, M.R. Barnett, and P.D. Hodgson: *Mater. Sci. Eng. A*, 2008, vol. 485, pp. 664–72.
34. G. Langford and M. Cohen: *Metall. Trans. A*, 1975, vol. 6A, pp. 901–10.
35. D. Dorner, Y. Adachi, and K. Tsuzaki: *Scripta Mater.*, 2007, vol. 57, pp. 775–78.