

# Recrystallization Mechanisms in Severely Deformed Dual-Phase Stainless Steel

Andrey Belyakov<sup>1, a</sup>, Rustam Kaibyshev<sup>1, b</sup>, Yuuji Kimura<sup>2, c</sup>  
and Kaneaki Tsuzaki<sup>2, d</sup>

<sup>1</sup>Belgorod State University, Pobeda 85, Belgorod 308015, Russia

<sup>2</sup>Structural Metals Center, National Institute for Materials Science,  
Sengen 1-2-1, Tsukuba, Ibaraki 305-0047, Japan

<sup>a</sup>belyakov@bsu.edu.ru, <sup>b</sup>rustam\_kaibyshev@bsu.edu.ru, <sup>c</sup>kimura.yuuji@nims.go.jp,  
<sup>d</sup>tsuzaki.kaneaki@nims.go.jp

**Keywords:** Austenite/ferrite stainless steel; severe deformation; grain refinement; internal stresses; recovery; recrystallization.

**Abstract.** The structural recrystallization mechanisms operating in an Fe – 27%Cr – 9% Ni dual-phase (ferrite-austenite) stainless steel after large strain processing to total strain of 4.4 were investigated in the temperature range of 400-700°C. The severe deformation resulted in the development of an ultrafine grained microstructure consisting of highly elongated grains/subgrains with transverse dimensions of 160 nm and 130 nm in ferrite and austenite, respectively. The annealing mechanism operating in ferrite phase was considered as continuous recrystallization, which involved recovery leading to the development of essentially polygonized microstructure. On the other hand, the mechanism of discontinuous nucleation took place at an early recrystallization stage in austenite phase.

## Introduction

Ultrafine grained metals and alloys with grain sizes ranging from tens to hundreds nanometers are a subject of much current interest because of their enhanced mechanical properties [1-5]. Recently, severe plastic deformations have been successfully utilized as novel processing methods for production of nano- and submicrocrystalline metallic materials [6, 7]. Structural metals and alloys subjected to large strain cold-working are characterized by high residual stresses, which make difficult any practical applications. The residual stresses can be partially or fully released by an appropriate heat treatment. The final microstructures and associated mechanical properties of products depend on the evolutionary mechanisms operating during heat treatment.

Two recrystallization mechanisms, continuous and discontinuous, have been recognized in conventional processing technologies [8-10]. The discontinuous or primary recrystallization takes place in almost all cold-worked metallic materials during annealing at temperature above a critical recrystallization temperature [10]. Following a recovery, the new grains nucleate and grow out consuming the work-hardened structures, leading to replacement of deformation structure by annealed one. The continuous or in-situ recrystallization was very seldom observed in some alloys containing second phase particles [9]; the new grains evolve continuously as a result of subgrain coalescence that is controlled by particle growth. In contrast, the recrystallization mechanisms operating in severely deformed materials are a currently debated topic among material scientists. A rapid recovery in largely strained materials was shown resulting in homogeneous nucleation of recrystallizing grains upon heating [11, 12]. Hence, almost all strain-induced subgrains quickly transformed into the new grains, leading to continuous-like recrystallization behavior. On the other hand, the heterogeneous nucleation of recrystallized grains that suggests the operation of discontinuous mechanism was also evident in studies on severe deformation of materials with a relatively low stacking fault energy [3, 13]. The aim of the present study is to clarify the effect of stacking fault energy on the operating recrystallization mechanisms after large strain deformation. A

dual-phase stainless steel consisting of ferrite and austenite, which are typical representatives of materials with high and low stacking fault energies and bcc and fcc lattice, respectively, is selected to simplify this comparative investigation.

## Experimental

A ferrite/austenite dual phase stainless steel (Fe – 0.017%C – 0.01%Mn – 0.01%P – 0.001%S – 26.8%Cr – 9.03%Ni – 0.002%N) was used as the starting material. The ferrite/austenite ratio was about 3/2 in the initial state. The severe deformation was carried out at ambient temperature by caliber rolling from  $21.3 \times 21.3$  to  $7.8 \times 7.8$  mm<sup>2</sup> square cross-section of bars followed by swaging from  $\varnothing 7.0$  to  $\varnothing 2.1$  mm, providing a total strain of 4.4. The processing details and the deformation microstructures have been described elsewhere [14]. Note here, the severe deformation resulted in the development of a ribbon-like microstructure consisting of highly elongated grains/subgrains. The transverse (sub)grain size and the fraction of high-angle grain boundaries in the ferrite phase were about 160 nm and 0.64, respectively. The transverse austenite (sub)grain size was about 130 nm; and the fraction of high-angle grain boundaries comprised 0.7. These two phases were quite different in the density of intergranular dislocations. The dislocation density in the (sub)grain interiors within ferrite phase was about  $9 \times 10^{14}$  m<sup>-2</sup>, while that evolved within the austenite (sub)grains amounted up to  $8 \times 10^{15}$  m<sup>-2</sup>.

The severely deformed samples were cut into 10-mm pieces and annealed in an argon atmosphere at temperatures from 400 to 700°C. Structural investigations were performed on sections parallel to the swaging axis, using a Jeol JEM 2100 transmission electron microscope (TEM). The grain/subgrain sizes were measured on TEM images by a linear intercept method. Misorientations across the (sub)grain boundaries were analyzed by the conventional Kikuchi-line method. The annealing softening was evaluated by Vickers hardness tests with a load of 3 N.

## Results and Discussion

**Annealed Microstructures and Softening.** Typical annealed microstructures are shown in Fig. 1. Annealing for 30 min at temperatures below 500°C hardly results in significant changes of the deformation microstructure, which is composed of elongated grains/subgrains with high density of lattice dislocations in their interiors. The numerous bend contours on the TEM images suggest that high internal stresses still exist in these annealed samples. The process of static recrystallization develops upon heating to temperatures above 500°C. Annealing at 600°C leads to partially recrystallized microstructure consisting of equiaxed recrystallized grains and elongated work-hardened grains/subgrains (Fig. 1c). Almost fully recrystallized microstructure evolves during 30 min annealing at 700°C (Fig. 1d). It should be noted that the structural changes at temperatures of  $T \geq 600^\circ\text{C}$  are accompanied with the formation of  $\sigma$ -phase in within the ferrite phase. Therefore, the mixture of ultrafine grained austenite and  $\sigma$ -phase develops at 700°C.

In contrast to cold-worked single-phase metals and alloys [10, 12], the heat treatment of the dual-phase steel does not lead to remarkable softening (Fig. 2). The softening rate slightly increases with temperature in the range of 400-600°C; however, the hardness of the annealed samples is just below the value recorded for the as-processed state. Annealing at 700°C results in rapid hardening, which is associated with the  $\sigma$ -phase formation. The transverse size of grain/subgrain evolved in the both ferrite and austenite phases scarcely changes during 30 min annealing at temperatures below 500°C (Fig. 2). An increase in the annealing temperature results in gradual coarsening of the microstructure. However, the scale of structural elements remains on submicron level even after annealing at rather high temperature of 700°C. Let us detail the mechanisms of microstructure evolution that operate in severely deformed ferrite and austenite during annealing.

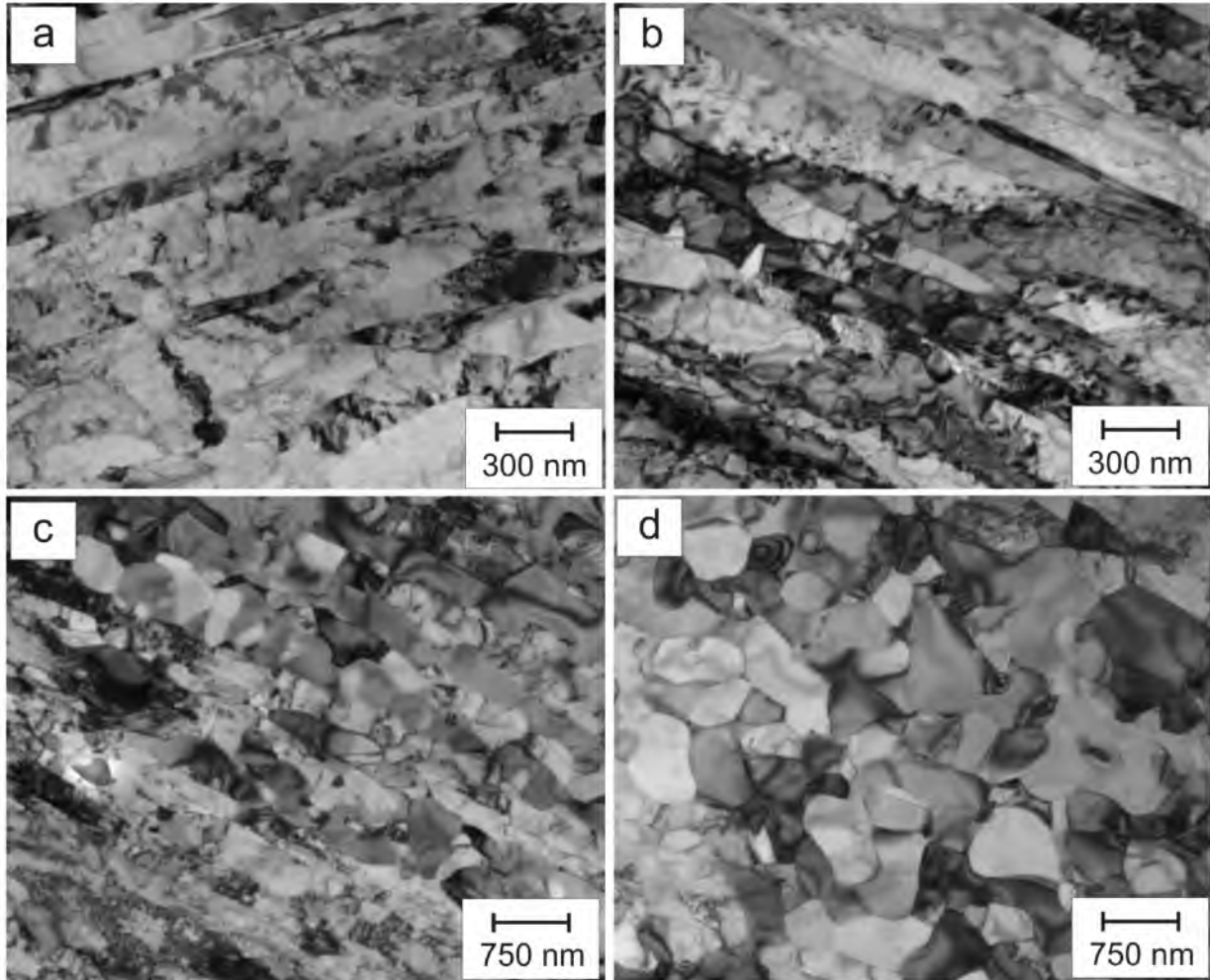


Fig. 1. Typical microstructures developed in severely deformed dual-phase steel after annealing for 30 min; (a)  $T = 400^{\circ}\text{C}$ ; (b)  $T = 500^{\circ}\text{C}$ ; (c)  $T = 600^{\circ}\text{C}$ ; (d)  $T = 700^{\circ}\text{C}$ .

**Static recrystallization in severely deformed ferrite.** The large strain unidirectional deformation brought about the formation of highly elongated (sub)grains with low- to high-angle boundaries within the ferrite phases. The most of high-angle boundaries were aligned parallel to the processing axis, while the angular misorientations along individual deformation (sub)grains did not exceed several degrees [14]. Such lattice curvatures within elongated (sub)grains are created by interior dislocations as well as high internal stresses originated from strain-induced (sub)boundaries. An early annealing is accompanied with a reduction of dislocation density and leads to a release of internal stresses, while the (sub)grain size remains the same with that in as-processed state. The density of interior dislocations in the severely strained ferrite decreases down to  $2.5 \times 10^{14} \text{ m}^{-2}$  after annealing for 30 min at  $500^{\circ}\text{C}$ . It is clearly seen in Fig. 3a that static recovery develops homogeneously in the elongated ferrite (sub)grains and does lead to appearance of small perfect subgrains, which could serve as nuclei for primary discontinuous recrystallization.

The structural changes in the ferrite phase during annealing for a relatively long time are characterized by a remarkable decrease of dislocation density in (sub)grain interiors that takes place along with an increase of (sub)grain size (Fig. 3b). It should be noted that the grain coarsening does not lead to any significant changes in the shape of elongated grains. The recovery-controlled rearrangement of interior dislocations results in the formation of dislocation walls, which subdivide the elongated grains by low-angle subboundaries. This is in consistence with other studies dealt with annealing behavior of single-phase ferritic steels after large strain processing [12, 15]. The structural

changes during annealing involve essentially two phenomena; namely, a gradual decrease of lattice dislocation density and a grow of deformation induced (sub)grains. The first process is a recovery and the last one is much similar to a normal grain growth. Therefore, this mechanism of microstructure evolution can be interpreted in terms of continuous recrystallization.

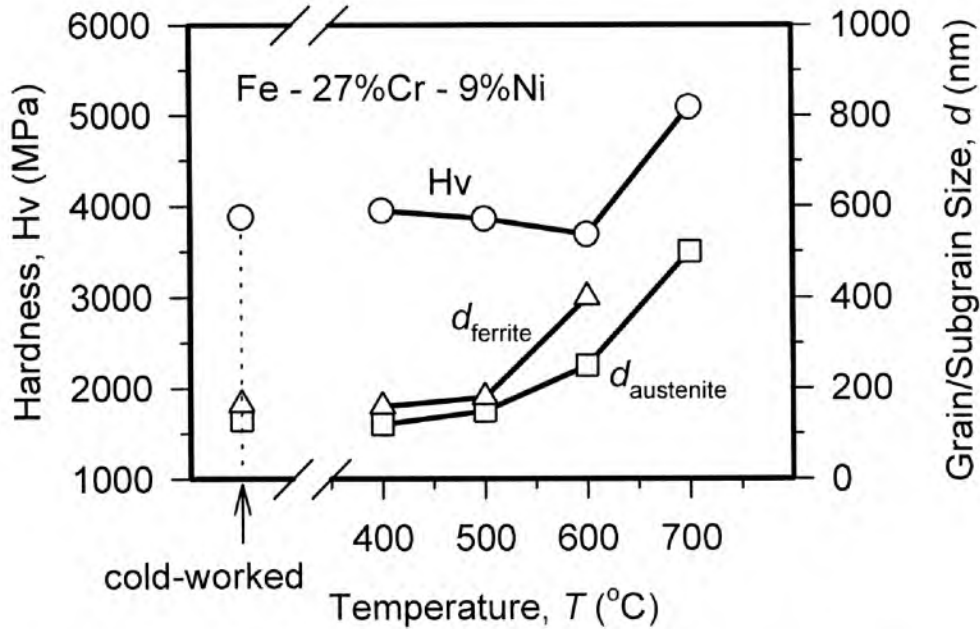


Fig. 2. Temperature effect on hardness and transverse grain/subgrain size of dual-phase steel annealed for 30 min.

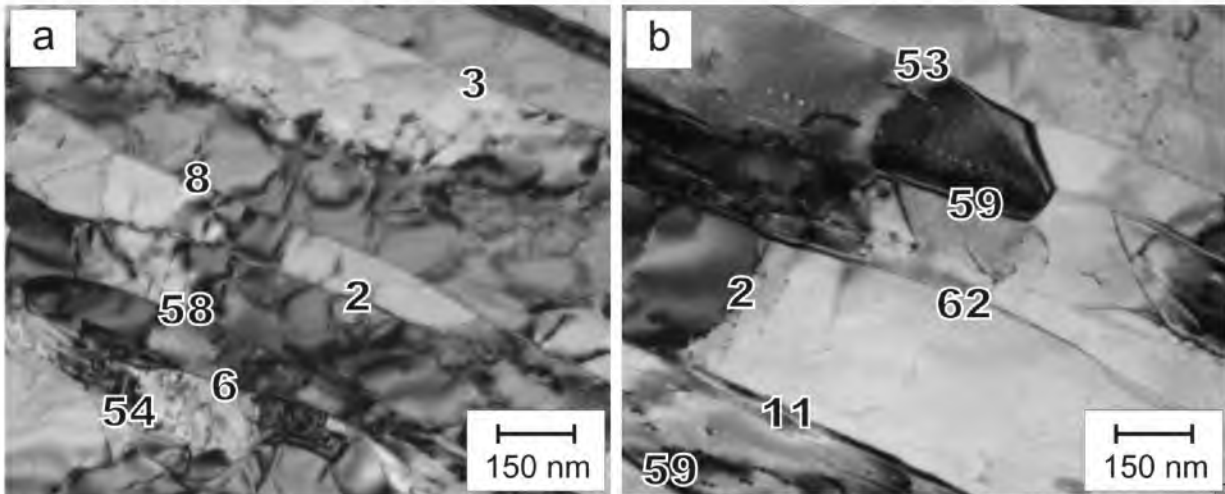


Fig. 3. Continuous recrystallization in ferrite phase of Fe – 27%Cr – 9%Ni steel annealed at 500°C for (a) 30 min and (b) 2 hours. The numbers indicate the (sub)boundary misorientations in degrees.

**Static recrystallization in severely deformed austenite.** After severe deformation, the elongated austenite phases are composed of largely misoriented (sub)grains [14]. The feature of the austenite is a very high dislocation density in (sub)grain interiors (an order of magnitude higher than that in ferrite) and large misorientations between strain-induced crystallites along the elongated phases. The large lattice distortions associated with high internal stresses provide a condition for rapid nucleation of a great number of recrystallizing grains. Typical example of the recrystallizing

austenite nuclei is shown in Fig. 4a. The several equiaxed submicrocrystallites are outlined by sharp high-angle boundaries and they are almost free of any interior dislocations. Therefore, these ultrafine (sub)grains can be considered as potential recrystallized grains. It should be noted that the recrystallizing nuclei are closely located together (Fig. 4a). Contrary to the recrystallization nucleation in conventionally cold-worked metals and alloys, the severely deformed austenite is characterized by a high density of nucleation sites, *i.e.* the nuclei are spaced on submicron scale.

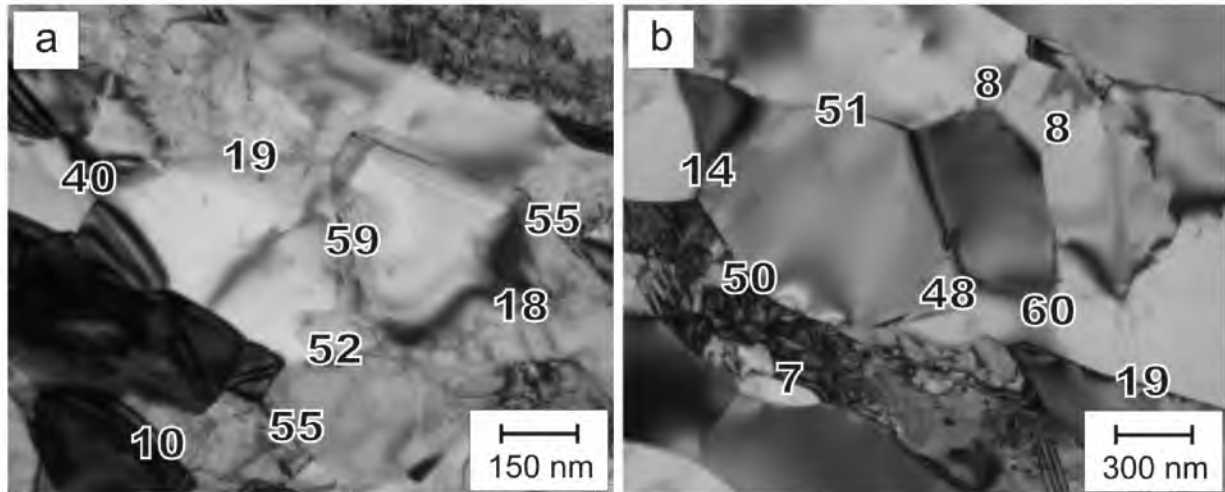


Fig. 4. Recrystallization in austenite phase of Fe – 27%Cr – 9%Ni steel annealed for 30 min at (a) 600°C and (b) 700°C. The numbers indicate the (sub)boundary misorientations in degrees.

The development of recovery processes in severely strained austenite results in the simultaneous formation of many recrystallization nuclei at place of strain-induced submicrocrystallites at the initial stage of annealing. Such transformation of deformation grains/subgrains into recrystallizing nuclei was discussed as a transient recrystallization [11]. Ultrafine grains/subgrains are evolved by the transient recrystallization. Next, these (sub)grains grow out upon further annealing through continuous recrystallization mechanism. Careful inspection of the transient recrystallization mechanism in the austenite phase reveals that the nucleation of recrystallized grains differs from the early stage of continuous recrystallization in ferrite. It is clearly seen in Fig. 4b that the recrystallizing nuclei grow out towards the work-hardened portions. Two structural components, *i.e.* the fine recrystallizing grains and the deformation substructures, are concurrently presented during the recrystallization development. Therefore, the early stage of static recrystallization in the severely deformed austenite is essentially the discontinuous process.

### Summary

The annealing behavior of an Fe – 27%Cr – 9%Ni ferrite/austenite stainless steel subjected to large strain cold deformation by bar rolling/swaging to total strain of 4.4 was studied at temperatures of 400 – 700°C. The present results suggest that the recrystallization behaviors of ferrite and austenite are different in the operating structural mechanisms. The annealing behavior of severely deformed ferrite is characterized by the development of continuous recrystallization including dislocation rearrangement and subgrain coalescence followed by a normal grain growth. In contrast, the nucleation of recrystallized grains in austenite occurs through the discontinuous mechanism. The large angular misorientations evolved in austenite substructures after severe plastic working are considered as prerequisites for discontinuous recrystallization.

## Acknowledgements

The financial support received from the Japan Society for the Promotion of Science and Russian Foundation for Basic Research is gratefully acknowledged. The authors are grateful to Mr. E. Kudryavtsev, Center of Common Facilities, Belgorod State University, for his assistance in specimens preparation.

## References

- [1] C. Suryanarayana: *Int. Mater. Rev.* Vol. 40 (1995), p. 41.
- [2] C.C. Koch: *NanoStructured Materials* Vol. 9 (1997), p. 13.
- [3] Y. Wang, M. Chen, F. Zhou and E. Ma: *Nature* Vol. 419 (2002), p. 912.
- [4] S. Ohsaki, K. Hono, H. Hidaka and S. Takaki: *Scripta Mater.* Vol. 52 (2005), p. 271.
- [5] Y. Kimura, T. Inoue and K. Tsuzaki: *Science* Vol. 320 (2008), p. 1057.
- [6] F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia and C. Harris: *Phil. Trans. R. Soc. Lond.* Vol. 357 (1999), p. 1663.
- [7] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov: *Prog. Mater. Sci.* Vol. 45 (2000), p. 103.
- [8] R.W. Cahn, in: *Grain Growth and Textures*, ASM, Ohio (1966).
- [9] E. Hornbogen and U. Koster, in: *Recrystallization of Metallic Materials*, edited by F. Haessner, Verlag, Germany (1978).
- [10] F.J. Humphreys and M. Hatherly: *Recrystallization and related annealing phenomena* (Pergamon Press, UK 1996).
- [11] A. Belyakov, T. Sakai, H. Miura, R. Kaibyshev and K. Tsuzaki: *Acta Mater.* Vol. 50 (2002), p. 1547.
- [12] A. Belyakov, K. Tsuzaki, Y. Kimura, Y. Mishima: *J. Mater. Res.* Vol. 22 (2007) p. 3042.
- [13] X. Molodova, S. Bhaumik, M. Winning and G. Gottstein: *Mater. Sci. Forum* Vol. 503-504 (2006), p. 469.
- [14] A. Belyakov, Y. Kimura and K. Tsuzaki: *Acta Mater.* Vol. 54 (2006), p. 2521.
- [15] A. Belyakov, Y. Kimura and K. Tsuzaki: *Mater. Sci. Eng. A* Vol. A403 (2005), p. 249.