Materials Science Forum Vols. 783-786 (2014) pp 1021-1026
© (2014) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/MSF.783-786.1021

Static Grain Growth in an Austenitic Stainless Steel Subjected to Intense Plastic Straining

Marina Tikhonova[a], Andrey Belyakov[b] and Rustam Kaibyshev[c]
Belgorod State University, Pobeda 85, Belgorod 308015, Russia
[a]tikhonova@bsu.edu.ru, [b]belyakov@bsu.edu.ru, [c]rustam_kaibyshev@bsu.edu.ru

Keywords: austenitic stainless steel, ultrafine grains, post-dynamic recrystallization, grain growth.

Abstract. The post-dynamic recrystallization of an ultrafine grained 304-type austenitic stainless steel was studied during annealing at 800 and 1000°C for 7.5 to 480 minutes. The initial ultrafine grained microstructures have been developed by continuous dynamic recrystallization during isothermal multidirectional forging to a total strain of ~4 at temperatures ranging from 500 to 800°C. The post-dynamic recrystallization involves a rapid softening at early stage of annealing followed by a sluggish decrease of hardness upon further annealing. A transient recrystallization at early annealing stage results in somewhat heterogeneous microstructures in the samples subjected to previous deformation at relatively low temperatures of 500-600°C. This structural heterogeneity disappears with increasing the annealing time. Commonly, the post-dynamic recrystallization behavior can be considered as a kind of continuous recrystallization.

Introduction

Nowadays, large strain deformations are considered as one of the most effective processing methods for production of various engineering steels and alloys with ultrafine grained structures [1, 2]. The ultrafine grained steels have been reported to exhibit enhanced strength properties due to high Hall-Petch response [3-5]. However, plasticity of severely deformed materials leaves much to be desired that limits any engineering applications of ultrafine grained materials in the as-deformed state. Generally, ductility can be recovered by an appropriate heat treatment. The heat treatments of particular interest are those that allow keeping the developed microstructure against remarkable grain coarsening; and, therefore, provide improved strength-plasticity combinations.

In contrast to primary recrystallization taking place during annealing of conventional cold worked metals and alloys [6], the recrystallization behavior of ultrafine grained structures developed by large strain deformation has not been studied systematically. The annealing behavior of such materials has been reported to involve a rapid recovery followed by a gradual grain coarsening that is much similar to continuous static recrystallization [7-9]. However, the effects of conditions of previous large strain deformation and characteristics of ultrafine grained structures on the recrystallization behavior during subsequent annealing are still unclear. The aim of the present study is to clarify the effects of the temperature of multidirectional forging, which was utilized as severe plastic deformation to produce an ultrafine grained austenitic stainless steel, and the temperature of subsequent annealing on the post-dynamic recrystallization behavior and the finally developed microstructures.

Experimental Procedure

An S304H austenitic stainless steel, 0.10%C–18.2%Cr–7.85%Ni–2.24%Cu–0.50%Nb–0.008%B–0.12%N–0.95%Mn–0.10%Si and the balance Fe (all in weight%), with an average grain size of about 7 μm was used as the starting material. Rectangular samples were subjected to multidirectional forging (MF), which was carried out using isothermal multi-pass compression tests with a change in the compression direction in 90° in order of three orthogonal axes from pass to pass at temperatures of 500, 600, 700 and 800°C. A total strain of 4 was applied for each sample.

This MF resulted in almost full development of ultrafine grained microstructures. The mean dynamic grain sizes developed in the largely strained samples at temperatures of 500, 600, 700, 800°C were 0.22, 0.3, 0.36 and 0.69 μm, respectively. The detailed descriptions of these microstructures can be found elsewhere [10, 11]. Finally, these MF samples were annealed during 7.5 to 480 min at temperatures of 800 and 1000°C. The structural investigations of the annealed samples were carried out using an FEI Quanta 600F scanning electron microscope equipped with an electron back scatter diffraction (EBSD) analyzer incorporating an orientation imaging microscopy (OIM) system. The OIM images were subjected to clean-up procedures, setting a minimal confidence index of 0.1. The grain sizes and the grain boundary distributions were evaluated by OIM software (EDAX TSL, ver. 5). The annealing softening was studied by means of Vickers hardness tests with a load of 3 N.

Results and Discussion

Annealing Softening. The changes in the room temperature hardness during annealing at 800°C and 1000°C of the S304H austenitic stainless steel subjected to MF at 500-800°C are shown in Fig. 1. The change in the hardness during annealing at 800°C depends on the temperature of previous MF. The hardness of 2800 MPa does not change remarkably in the sample subjected to MF at 800°C irrespective of annealing time. In contrast, the hardness of the samples subjected to MF at 500-700°C significantly decreases to about 2750 MPa during annealing for about 2 hours and then remains almost constant upon subsequent annealing. On the other hand, annealing at 1000°C leads to a rapid decrease in the hardness of all samples at an early annealing stage. The hardness decreases to 2100-2300 MPa after 7.5 min annealing and then slightly decreases to approx. 2000 MPa with increasing the annealing time. Therefore, the hardness levels of about 2750 and 2000 MPa are obtained in all samples annealed for rather long time at 800 and 1000°C, respectively.

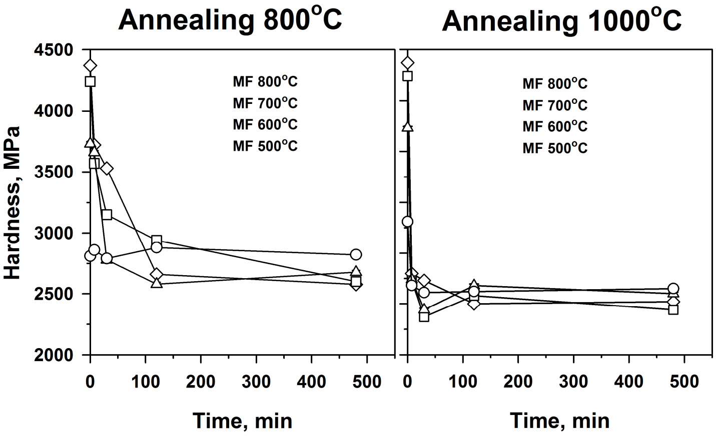

Fig. 1. Changes in the room temperature hardness during annealing at 800°C and 1000°C of the S304H austenitic stainless steel subjected to MF at 500-800°C.

Figure 2 presents the effect of the annealing duration and temperature on the fractional softening (X) of the S304H austenitic stainless steel subjected to MF at 500-800°C. The fractional softening was calculated as: $X = (HV_{MDF} - HV_{Annealing}) / (HV_{MDF} - HV_{Initial})$, where HV_{MDF} is the hardness of the steel just after MF, $HV_{Annealing}$ is the hardness of the MF samples after subsequent annealing at different temperatures for various time, $HV_{Initial}$ is the hardness of original steel samples, which have been fully annealed at 1100°C. The fractional softening of the samples subjected to MF at 500-700°C increases to its saturation level during annealing for about 2 hours; and the samples subjected to MF at lower temperature exhibit higher values of structural softening after subsequent annealing. In contrast, the sample subjected to MF at 800°C does not soften during the annealing at 800°C. On the other hand, all MF samples are characterized by the sharp softening during annealing for 7.5 min at 1000°C, when the fractional softening attains about 0.7-0.9, depending on the temperature of preceding MF. The fractional softening of the samples subjected to MF at 800°C saturates at ~0.7 during annealing at 1000°C, while that of the samples, which were previously processed at 500-600°C, approaches about 0.95. It is worth noting that the annealing does not result in complete softening under the studied conditions that is similar to a common annealing behavior of dynamically recrystallized metallic materials [12].

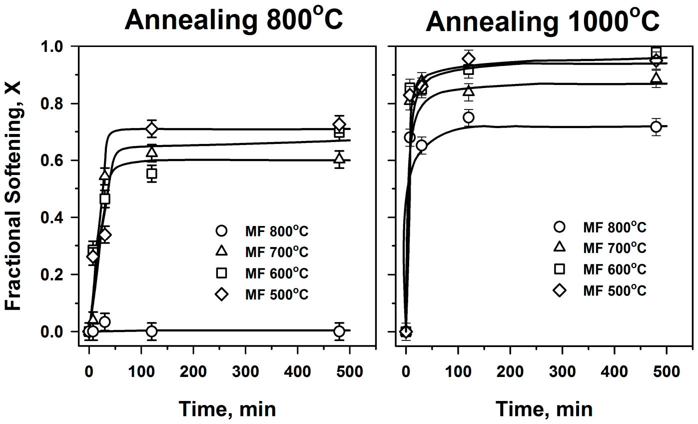

Fig. 2. Effect of annealing time and temperature on the fractional softening (X) of the S304H austenitic stainless steel subjected to MF at 500-800°C.

Annealed Microstructures. Typical microstructures evolved in the S304H austenitic stainless steel subjected to MDF at 800 and 600°C and then annealed at 800 and 1000°C during 30 and 120 min are shown in Fig. 3. The samples processed by MF at relatively high temperature of 800°C are quite stable against grain coarsening during the subsequent annealing at 800°C. The grain size of 0.69 μm that evolved by MF slightly increases to 0.78 μm after 30 min annealing and then to 1.06 μm with increase in the annealing time to 2 hours (Fig. 3a). In contrast, the samples processed by MF at relatively low temperatures are characterized by somewhat accelerated grain growth during the annealing at 800°C. For instance, the 30 min annealing results in twofold increase in the mean grain size from 0.3 to 0.68 μm in the sample subjected to MF at 600°C (Fig. 3a). Some microstructural inhomogeneity should also be noted for this sample. Namely, rather large annealed grains appear as islands surrounding by much fine grains. Increase in the annealing time to 2 hours leads to further grain growth; the mean grain size increases to 1.46 μm. The grain growth during annealing for rather long time removes the structural inhomogeneity inherent in early annealing stages.

The changes in the microstructures during annealing at 1000°C are qualitatively the same as those observed at 800°C. The ultrafine grains evolved by MF at 800°C are characterized by a sluggish growth to 2.2 and 4.4 μm during annealing at 1000°C for 30 min and 2 hours, respectively (Fig. 3b). The ultrafine grained microstructures developed by MF at relatively low temperature (600°C in Fig. 3b) demonstrate inhomogeneous coarsening at early annealing at 1000°C, when the mean grain size increases to 3.5 μm, similar to annealing at 800°C. The increase in annealing time to 2 hours results in the development of almost uniform microstructure with an average grain size of 5.08 μm.

The effects of the MF and annealing temperatures on the structural uniformity are clearly illustrated by the grain size distributions (Fig. 4). The grain size distributions developed after annealing in the samples, which were previously subjected to MF at 800°C, are single peak-type with narrow peaks against grain sizes being a function of annealing time/temperature. The sharp peaks developed after 30 min annealing shift towards larger grain sizes with increase in the annealing time. On the other hand, the annealing for 30 min at 800 and 1000°C results in wide bimodal grain size distributions in the samples, which were previously processed by MF at 600°C. However, increase in the annealing time equalizes the grain sizes in these samples. Thus, the uniform microstructures develop in the samples after sufficiently long annealing.

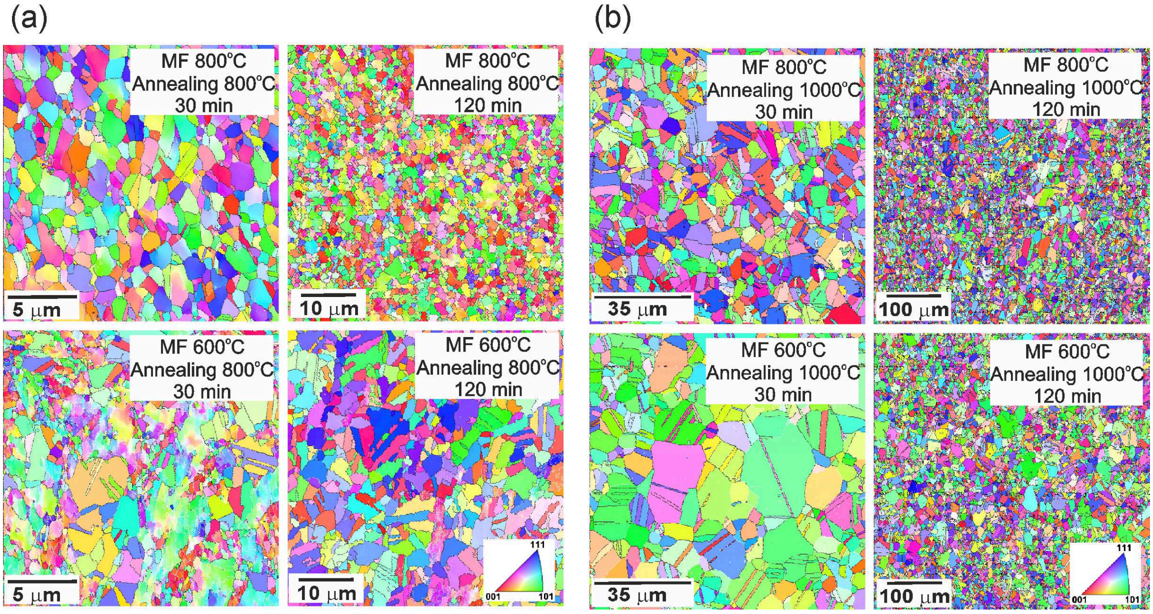

Fig. 3. Typical microstructures evolved in the S304H austenitic stainless steel subjected to MF at 800°C and 600°C and then annealed for 30 and 120 min at 800°C (a) and 1000°C (b). The white and black lines correspond to the subgrain/grain boundaries with misorientations of 2° ≤ θ < 15° and θ ≥ 15°, respectively.

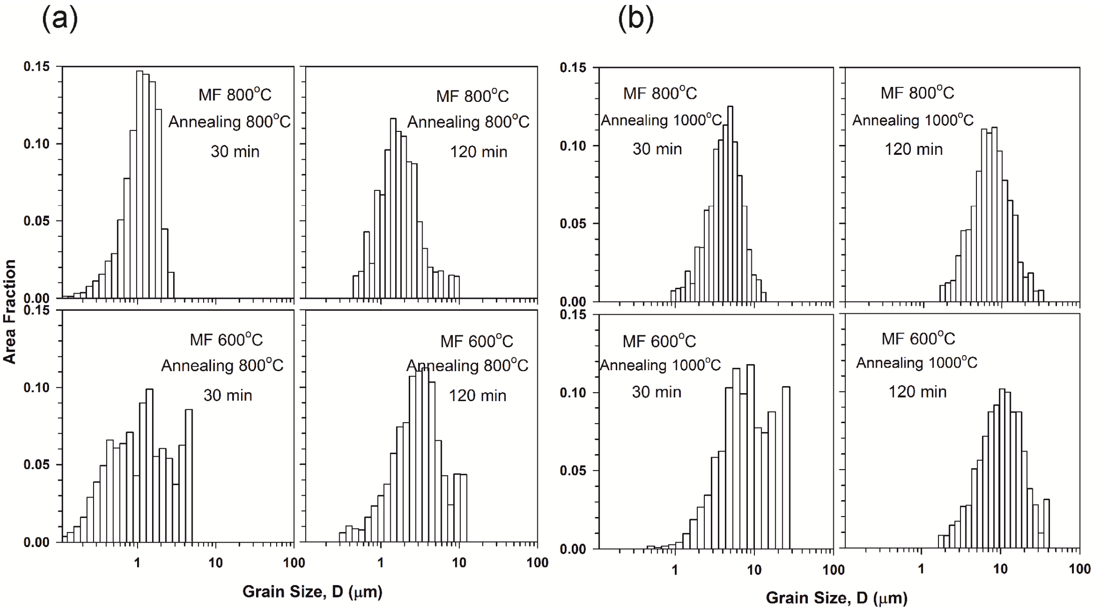

Fig. 4. Grain size distributions developed in the S304H austenitic stainless steel subjected to MF at 800°C and 600°C and then annealed for 30 and 120 min at 800°C (a) and 1000°C (b).

The evolution of ultrafine grained microstructures that developed by severe deformations during subsequent annealing is usually discussed in terms of static grain growth and/or post-dynamic recrystallization [7, 12]. The annealing behavior of an austenitic stainless steel after large strain forging has been considered as continuous recrystallization involving a rapid recovery at an early annealing followed by a transient recrystallization and subsequent gradual grain growth [7]. In the present study the development of transient recrystallization corresponds to the range of rapid softening in Figs. 1 and 2. The present results suggest that the microstructures developed during transient recrystallization depend on the temperature of previous deformation. Namely, higher deformation temperature leads to more uniform microstructure after subsequent transient recrystallization. Strictly speaking, the structural uniformity at a beginning of continuous recrystallization seems to be depended on the difference between the temperature of severe plastic deformation and the temperature of subsequent annealing; a larger temperature difference corresponds to a larger structural inhomogeneity.

The grain growth kinetics in the ultrafine grained steel depends on the temperature of previous large strain deformation. The annealed grain size is commonly expressed by a power law relationship of the annealing time [6], $D = k\,\tau^{1/n}$, where τ is the annealing time, k is a constant, n is a grain growth exponent. The magnitude of n should be related to the mechanisms controlling the grain growth. Theoretically predicted n for a normal grain growth in pure single-phase materials should be equal to 2, although much larger n up to 10 have been frequently obtained experimentally [6, 8]. In the present study, the grain growth exponents of approx. 2 are obtained for the samples subjected to MF at 600°C and then annealed at 800°C and for the samples subjected to MF at 800°C and then annealed at 1000°C, whereas larger n values (4.6 and 3.7) are obtained for the samples annealed at 800°C following the MF at the same temperature and for the samples annealed at 1000°C following the MF at 600°C (Fig. 5). The grain growth with n ~ 2 seems to be attributed to a relatively fast transient recrystallization at an early annealing stage. In contrast, the larger grain growth exponents correspond to the normal grain growth in the present ultrafine grained steels much similar to other studies on grain coarsening. It is interesting to note that the difference between the dynamically and statically recrystallized grain sizes depends significantly on the difference between the deformation and annealing temperatures (Fig. 5). The change in grain size after annealing increases with a decrease in the deformation temperature and an increase in the annealing temperature.

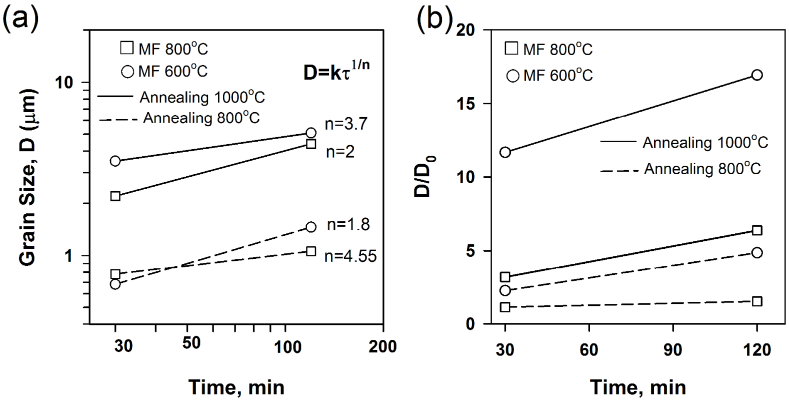

Fig. 5. Effect of annealing time on (a) the mean grain size (D) and (b) the change in grain size (D/D_o) in the S304H austenitic stainless steel subjected to MF at 800 and 600°C and then annealed at 800 and 1000°C. The D_0 corresponds to the mean grain size evolved by MF.

Summary

The annealing behavior of a 304-type austenitic stainless steel with ultrafine grained structures, which have been developed by large strain multidirectional forging at various temperatures in the range of 500-800°C, was studied at 800 and 1000°C. The annealing behaviour is associated with the development of continuous static recrystallization and its kinetics decelerates with increase in the temperature of previous deformation. Therefore, a large grain size is obtained after annealing in the samples subjected to previous deformation at lower temperature. The continuous static recrystallization involves a transient recrystallization resulting in a rapid softening at early stage of annealing and a gradual grain growth at later annealing stage. The transient recrystallization is characterised by a rapid grain growth and leads to somewhat heterogeneous grain size distributions in the samples subjected to previous deformation at relatively low temperatures of 500-600°C. This structural heterogeneity, however, disappears with increasing the annealing time. The grain growth can be expressed by a power law function of annealing time with a grain growth exponent of about 2-4 depending on the deformation and annealing temperatures.

Acknowledgements

This study was supported by the Russian Foundation for Basic Research, under grant №12-08-31115. The authors are grateful to the staff of the Joint Research Centre, Belgorod State University, for their assistance with instrumental analysis.

References

[1] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov, Bulk nanostructured materials from severe plastic deformation, Prog. Mater. Sci. 45 (2000) 103-189.

[2] Y. Estrin, A. Vinogradov, Extreme grain refinement by severe plastic deformation: a wealth of challenging science, Acta Mater. 61 (2013) 782-817.

[3] Z. Yanushkevich, A. Mogucheva, M. Tikhonova, A. Belyakov, R. Kaibyshev, Structural strengthening of an austenitic stainless steel subjected to warm-to-hot working, Mater. Character. 62 (2011) 432-437.

[4] I. Shakhova, V. Dudko, A. Belyakov, K. Tsuzaki, R. Kaibyshev, Effect of large strain cold rolling and subsequent annealing on microstructure and mechanical properties of an austenitic stainless steel, Mater. Sci. Eng. A 545 (2012) 176-186.

[5] S.V. Dobatkin, V.F. Terent'ev, W. Skrotzki, O.V. Rybalchenko, M.N. Pankova, D.V. Prosvirin, E.V. Zolotarev, Structure and fatigue properties of 08Kh18N10T steel after equal-channel angular pressing and heating, Russian Metallurgy (Metally) 2012 (2012) 954-962.

[6] F.J. Humphreys, M. Hatherly, Recrystallization and Related Annealing Phenomena, Elsevier, 1996.

[7] A. Belyakov, T. Sakai, H. Miura, R. Kaibyshev, K. Tsuzaki, Continuous recrystallization in austenitic stainless steel after large strain deformation, Acta Mater. 50 (2002) 1547-1557.

[8] A. Belyakov, K. Tsuzaki, Y. Kimura, Y. Mishima, Annealing behaviour of a ferritic stainless steel subjected to large strain cold working, J. Mater. Res. 22 (2007) 3042-3051.

[9] N. Dudova, A. Belyakov, R. Kaibyshev, Recrystallization behaviour of a Ni-20%Cr alloy subjected to severe plastic deformation, Mater. Sci. Eng. A 543 (2012) 164-172.

[10] M. Tikhonova, R. Kaibyshev, X. Fang, W. Wang, A. Belyakov. Grain boundary assembles developed in an austenitic stainless steel during large strain warm working. Mater. Character. 70 (2012) 14-20.

[11] M. Tikhonova, A. Belyakov, R. Kaibyshev. Strain-induced grain evolution in an austenitic stainless steel under warm multiple forging, Mater. Sci. Eng. A 564 (2013) 413-422.

[12] T. Sakai, Dynamic recrystallization microstructures under hot working conditions, J. Mater. Process. Technol. 53 (1995) 349-361.