

# STATIC RECRYSTALLIZATION OF A WARM WORKED AUSTENITIC STAINLESS STEEL

BONDAREVA E., TIKHONOVA M., BELYAKOV, A.  
Belgorod State National Research University, ul. Pobedy 85, Russia

## Abstract

The effect of worm worked microstructures on the development of static recrystallization in a 304-type austenitic stainless steel was studied during annealing at 800 and 1000°C for 7.5 to 120 minutes. The initial microstructures have been developed by single-pass compression to a strain of ~1.2 and multidirectional forging to a total strain of ~4 at temperatures ranging from 500 to 800°C. The samples subjected to single-pass compression are characterized by the development of static recovery during annealing at 800°C irrespective of the compression temperature. The static recrystallization readily develops in these samples upon annealing at 1000°C. On the other hand, the static recrystallization takes place in the samples processed by multidirectional forging during subsequent annealing at the both temperatures; and the recrystallization kinetics increases with decrease in the temperature of preceding multidirectional forging.

## Keywords

austenitic stainless steel, ultrafine grains, recrystallization, grain growth.

## Introduction

Nowadays, the recrystallization of ultrafine grained metallic materials is of particular interest. The ultrafine grained metals and alloys are considered as promising structural materials, because the mechanical properties of structural metallic materials can be enhanced by grain refinement (Valiev, 2002; Kimura, 2008; Zharebtsov, 2012; Dobatkin, 2012). Large strain deformations have been proposed as one of the most effective processing methods for production of various engineering steels and alloys with ultrafine grained structures (Humphreys, 1999; Valiev, 2006; Tsuji, 2010; Estrin, 2013). The steels after large strain deformation have been reported to exhibit enhanced strength properties, but low plasticity properties. Generally, the 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. Generally, the primary recrystallization takes place during annealing of cold-to-warm worked metals and alloys. However, the recrystallization behavior of ultrafine grained metallic materials processed by large strain warm working has not been studied in sufficient detail. The effects of conditions of previous large strain deformation and the characteristics of deformation microstructures on the recrystallization behavior during subsequent annealing are still unclear.

The aim of the present study is to clarify the effects of the deformation temperature and total strains, and the temperature and duration of subsequent annealing on the recrystallization behavior and the finally developed microstructures in an austenitic stainless steel.

## 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 single-pass forging (SPC) to a strain of 1.2 and multidirectional forging (MF) to a total strain of 4 at temperatures of 500, 600, 700 and 800°C. The multidirectional forging 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 (Zherebtsov, 2012; Belyakov, 2001; Tikhonova, 2013). After forging these samples were annealed during 7.5 to 120 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. 6). The annealing softening was studied by means of Vickers hardness tests with a load of 3 N.

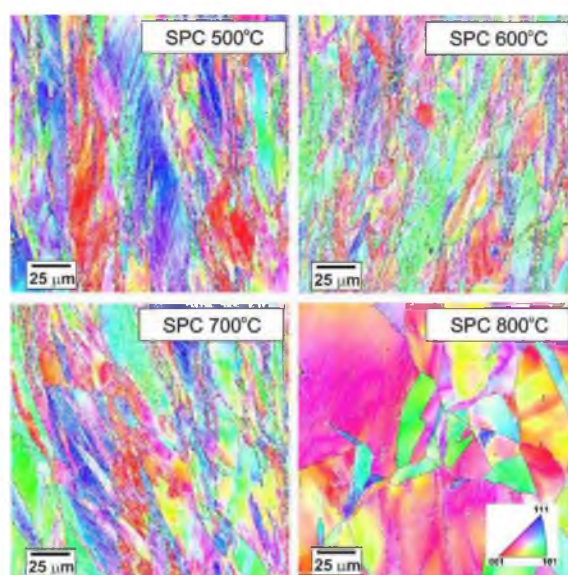


Fig. 1: Typical microstructures evolved in the S304H austenitic stainless steel subjected to SPC at 500, 600, 700 and 800°C.

## Results and Discussion

### 1. Deformation Microstructures

Typical microstructures that developed in the steel samples subjected to SPC are shown in Fig. 1. The deformation microstructures with grains flattened crosswise to compression axis were obtained after SPC irrespective of deformation temperature. Any evidence of dynamic recrystallization were not observed in the SPC samples. Representative micrographs of microstructures evolved by MF to total strains of approximately 4 at different temperatures are shown in Fig. 2. The ten forging passes resulted in the formation of ultrafine-grained microstructures. The mean dynamic grain size in the highly strained steel samples increased from 0.22 to 0.69  $\mu\text{m}$  with increasing the MF temperature from 500 to 800°C (Tikhonova, 2013).

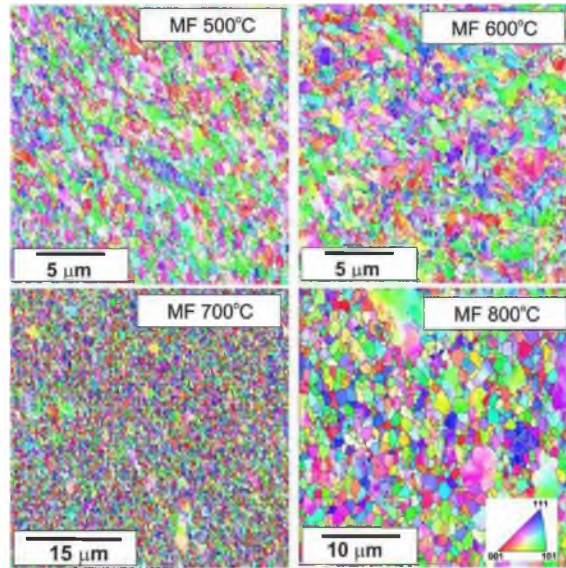


Fig. 2: Typical microstructures evolved in the S304H austenitic stainless steel subjected to MF at 500, 600, 700 and 800°C

## 2. Annealed Microstructures

The microstructures evolved in the S304H austenitic stainless steel subjected to SPC at 800 and 600°C and then annealed at 800°C for 120 min are shown in Fig. 3. The samples processed by SPC at these temperatures are quite stable against any static recrystallization and grain coarsening during the subsequent annealing at 800°C. In the steel samples subjected to SPC at 500-800°C and then annealed at temperature of 800°C only static recovery could be only a mechanism of static restoration during annealing. The deformation grains kept their shape pancaked in the direction of metal flow.

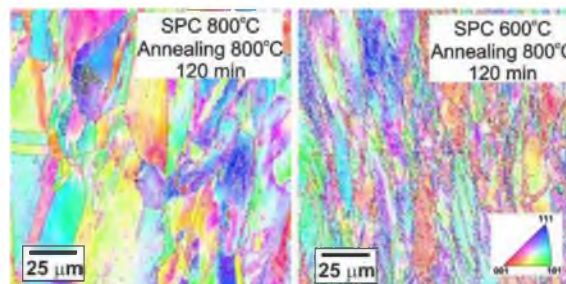


Fig. 3: Typical microstructures evolved in the S304H austenitic stainless steel subjected to SPC at 800°C and 600°C and then annealed for 30 min at 800°C

Figure 4 shows the microstructures that developed in the steel samples subjected to SPC at 800 and 600°C and then annealed at 1000°C during 30 and 120 min. In contrast to the samples annealed at 800°C, the static recrystallization was readily developed during annealing at 1000°C. The initially warm worked microstructures were completely recrystallized even after short annealing for 7.5 minutes. Annealing for a longer time was accompanied by a gradual grain coarsening. Further annealing did not have a significant influence on the annealed microstructures because all recrystallization processes are completed and the developed structure consisted of an equiaxed grains (Fig. 4).

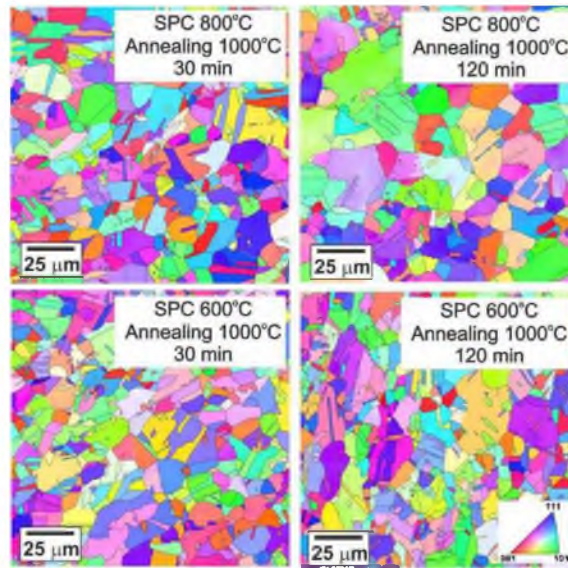


Fig. 4: Typical microstructures evolved in the S304H austenitic stainless steel subjected to SPC at 800°C and 600°C and then annealed for 30 and 120 min at 1000°C

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. 5, 6. The steel samples subjected to MF at relatively high temperature of 800°C are quite stable against grain coarsening during annealing at 800°C. To the contrary, the samples processed by MF at relatively low temperatures (600°C) were characterized by somewhat accelerated grain growth during the annealing at 800°C. Namely, rather large annealed grains appeared as islands surrounding by much fine grains. Increase in the annealing time to 120 min led to further grain growth.

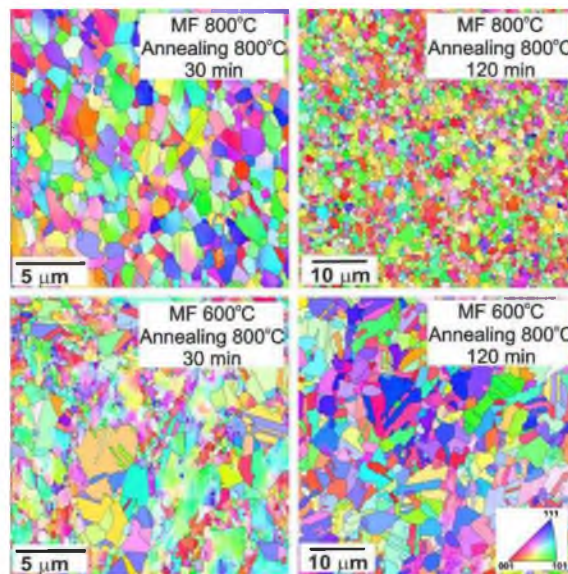


Fig. 5: Typical microstructures evolved in the S304H austenitic stainless steel subjected to MF at 800°C and 600°C and then annealed for 30 and 120 min at 800°C.

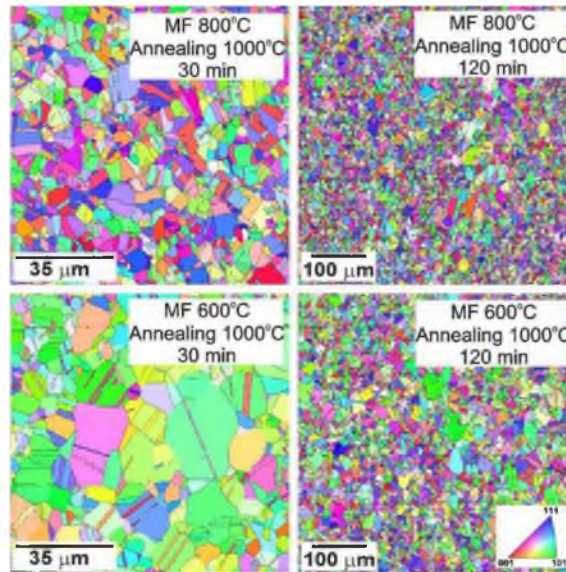


Fig. 6: Typical microstructures evolved in the S304H austenitic stainless steel subjected to MF at 800°C and 600°C and then annealed for 30 and 120 min at 1000°C

The changes in the microstructures during annealing at 1000°C are qualitatively the same as those observed at 800°C. The ultrafine grained microstructures developed by MF at relatively low temperature of 600°C (Fig. 6) demonstrate inhomogeneous coarsening at early annealing at 1000°C, when the mean grain size increased, similar to annealing at 800°C. The increase in annealing time to 120 min results in the development of almost uniform microstructure.

### 3. Fractional Softening

Figures 7 and 8 present the effect of the annealing duration and temperature on the fractional softening ( $X$ ) of the S304H austenitic stainless steel subjected to warm working by SPC and 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 warm working,  $HV_{Annealing}$  is the hardness of the 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.

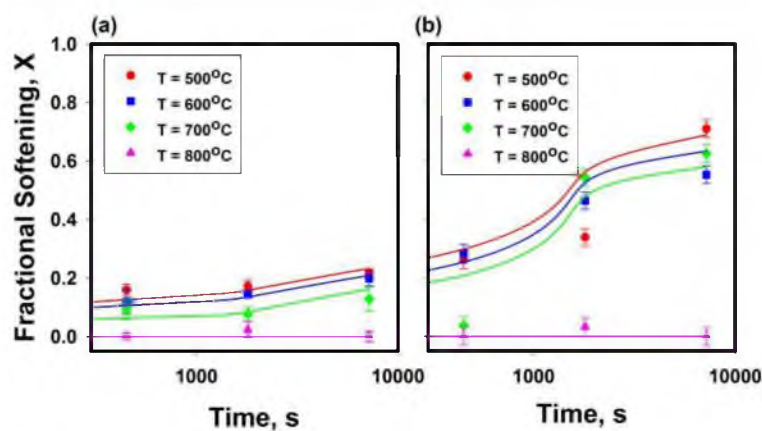


Fig. 7: Effect of annealing time at 800°C on the fractional softening ( $X$ ) of the 304H austenitic stainless steel subjected to SPC (a) and MF (b) at 500-800°C

The fractional softening of the samples subjected to SPC and MF and then annealed at 800°C is shown in Figs. 7a and 7b, respectively. The sample subjected to SPC at 800°C did not soften during the annealing at 800°C. A slight softening was observed for the samples subjected to SPC at lower temperatures. The fractional softening gradually increased to about 0.2 with increase in the annealing time to 120 min; and the samples warm worked at lower temperatures are characterized by somewhat faster softening during annealing. In the samples subjected to MF at 500, 600, 700 and 800°C and then annealed at 800°C a process of continuous static recrystallization was developed (Fig. 7b). The behavior of softening curves for MF significantly differed from the softening curves for SPC, although the softening curves for SPC and MF during annealing at 800°C are similar. The fractional softening of the samples subjected to MF at 500-700°C increases to about 0.6 during annealing for about 120 min; and the samples subjected to MF at lower temperature exhibit higher values of structural softening after subsequent annealing.

Figure 8 shows the fractional softening of the samples subjected to SPC and MF and then annealed at 1000°C. In the samples after SPC at 500-800°C and then annealed at 1000°C the primary recrystallization developed (Fig. 8a). The fractional softening rapidly increased above 0.8 after short annealing time. The annealing time of 7.5 min was enough for completion of the primary recrystallization. An increase of the annealing time did not lead to significant changes in the fractional softening, which tends to saturate at about 0.9 irrespective of the temperature of preceding SPC. The MF samples are also characterized by the sharp softening during annealing for 7.5 min at 1000°C (Fig. 8b). However, the fractional softening of the MF samples saturated at different levels ranging from 0.7 to approx. 1.0 depending on the temperature of preceding MF. This behavior is considered as a result of continuous static recrystallization (Sakai, 1995).

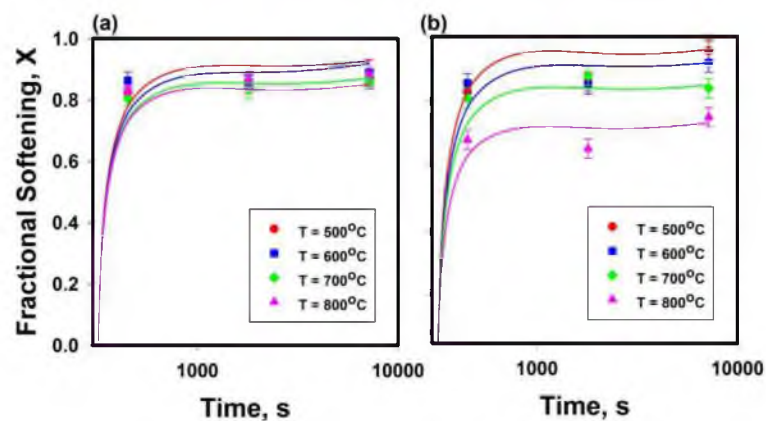


Fig. 8: Effect of annealing time at 1000°C on the fractional softening (X) of the 304H austenitic stainless steel subjected to SPC (a) and MF (b) at 500-800°C.

## Summary

The recovery/recrystallization behavior of an S304H stainless steel subjected to single-pass compression and multidirectional forging at temperatures of 500-800°C was studied during annealing at 800 and 1000°C. The samples subjected to single-pass compression were characterized by the development of static recovery during annealing at 800°C irrespective of the compression temperature, whereas increase in the annealing temperature to 1000°C resulted in rapid development of primary static recrystallization. On the other hand, continuous static recrystallization took place in the samples processed by multidirectional

forging during subsequent annealing at the both temperatures. The kinetics of continuous static recrystallization are faster in the samples subjected to multidirectional forging at lower temperatures.

## References

- [1] Belyakov A, Sakai T, Miura H, Tsuzaki K. Grain refinement in copper under large strain deformation. *Philosophical Magazine A* 2001;81:2629-43.
- [2] Dobatkin SV, Terent'ev VF, Skrotzki W, Rybalchenko OV, Pankova MN, Prosvirin DV, Zolotarev EV. Structure and fatigue properties of 08Kh18N10T steel after equal-channel angular pressing and heating. *Russian Metallurgy (Metally)* 2012;2012:954-62.
- [3] Estrin Y, Vinogradov A. Extreme grain refinement by severe plastic deformation: a wealth of challenging science. *Acta Mater* 2013;61:782-817.
- [4] Humphreys FJ, Prangnell PB, Bowen JR, Gholinia A, Harris C. Developing stable fine-grained microstructures by large strain deformation. *Phil Trans R Soc Lond A* 1999;357:1663-81.
- [5] Kimura Y, Inoue T, Yin F, Tsuzaki K. Inverse temperature dependence of toughness in an ultrafine grain-structure steel. *Science* 2008;320:1057-60.
- [6] Sakai T., Dynamic recrystallization microstructures under hot working conditions, *J. Mater. Process. Technol.* 53 1995;349-361.
- [7] Tikhonova M, Belyakov A, Kaibyshev R. Strain-induced grain evolution in an austenitic stainless steel under warm multiple forging. *Mater Sci Eng A* 2013; 564; 413-22.
- [8] Tsuji N. New routes for fabricating ultrafine-grained microstructures in bulky steels without very-high strains. *Adv Eng Mater* 2010;12:701-7.
- [9] Valiev RZ. Materials science: Nanomaterial advantage. *Nature* 2002;419:887-9.
- [10] Valiev RZ, Estrin Y, Horita Z, Langdon TG, Zehetbauer MJ, Zhu YT. Producing bulk ultrafine-grained materials by severe plastic deformation. *JOM* 2006;58:33-9.
- [11] Zharebtsov S, Kudryavtsev E, Kostjuchenko S, Malysheva S, Salishchev G. Strength and ductility-related properties of ultrafine grained two-phase titanium alloy produced by warm multiaxial forging. *Mater Sci Eng A* 2012;536:190-6.