DOI 10.1007/s11182-015-0588-4

*Russian Physics Journal, Vol. 58, No. 7, November, 2015 (Russian Original No. 7, July, 2015)*

# ULTRAFINE-GRAINED STRUCTURE AND ITS THERMAL STABILITY IN LOW-CARBON STEEL

**A. V. Korznikov,[1] I. M. Safarov,[1] R. M. Galeyev,[1] S. N. Sergeev,[1] and A. I. Potekaev[2]**

UDC 669.14: 53.096

*The influence of annealing temperature on ultrafine-grained structure (UFG) of low-carbon steel 05G2MFB, produced by multiple isothermal forging (MIF) and warm rolling is investigated. Following 1-hour annealing (from 20 to 550°C), the fibrous UFG-structure formed as a result of rolling remains virtually the same. The equiaxial UFG-structure is found to be stable as the annealing temperature is increased up to 600°C. An examination of the resulting UFG-states by the method of electron backscatter diffraction (EBSD) provided a way to identify the differences in softening processes for a number of structure types. When the temperature of annealing treatment of as-rolled steel specimens is increased to 600°C, the fraction of low-angle boundaries (LAB) is found to remain at about 56%, while the average grain/subgrain size in the rod cross-section increases from 0.4 to 0.9 μm. In the MIF-processed specimens, an increase in the annealing temperature up to 625°C gives rise to a gradual decrease in the fraction of LAGBs from 53 to 30%, with the average grain/subgrain size increasing from 0.4 to 0.6 μm.*

**Keywords:** low-carbon steel, ultrafine-grained structure, heat stability.

## INTRODUCTION

Steels are most commonly used metal materials that exhibit a variety of physical-chemical properties depending on their composition and structural-phase states. Varying the pattern of severe plastic deformation (SPD) treatment, one can produce materials with different UFG-structure types, which would possess improved strength, processability [1], and other functional properties [2]. This is especially critical for materials that cannot be effectively heat hardened using conventional processes, in particular, for widely used low-carbon steels. It was shown earlier [3, 4] that SPD-treatment provides a variety of UFG-structures in low-carbon steels, whose strength and impact strength are exceptionally high. A UFG-structure of a predetermined type offers a possibility of forming a set of the desired physical-mechanical properties. On the other hand, it is well known that UFG-states are risky in terms of structure non-equilibrium, high internal stresses, and higher defect density; hence the issue of thermal stability of UFG-structure is ever more demanding.

This study aims at investigating the influence of temperature on ultrafine-grained structure types formed in low-carbon steel 05G2MFB, produced by the techniques of multiple isothermal forging and rolling, and their thermal stability.

[1]Institute for Metals Superplasticity Problems, Ufa, Russia, e-mail: korznikov@imsp.ru; ilfat@anrb.ru; galeyev@imsp.ru; sem17@mail.ru; [2]V. D. Kuznetsov Siberian Physical-Technical Institute at Tomsk State University, Tomsk, Russia, e-mail: kanc@spti.tsu.ru. Translated from Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika, No. 7, pp. 24–28, July, 2015. Original article submitted April 20, 2015.


1064-8887/15/5807-0898 ©2015 Springer Science+Business Media New York

TABLE 1. Chemical Composition of Steel 05G2MFB, wt.%

| Fe | C | Mn | Si | Nb | V | Mo | P | S |
|---|---|---|---|---|---|---|---|---|
| Base | 0.07 | 1.65 | 0.29 | 0.05 | 0.08 | 0.0022 | 0.008 | 0.007 |

## MATERIAL AND EXPERIMENTAL PROCEDURES

In this study, we used the method of electron backscatter diffraction (EBSD) that allows the structure, microstructure and distribution of grain-boundary misorientations in a material to be analyzed to a high statistical confidentiality [5].

The chemical composition of the low-carbon steel 05G2MFB under study is presented in Table 1.

The structure of as-received specimens consisted of ferrite grains and platy cementite colonies (7 vol.%). The average ferrite grain size was 6 μm.

The UFG-structure was produced by the MIF process [6, 7] and warm rolling [8]. Multiple isothermal forging of steel specimens was performed by a stepwise decrease of temperature from 700 to 500°C, with the accumulated strain being $\varphi = 6.2$. Warm rolling was carried out at the temperature gradually reduced from 750 to 550°C, with the accumulated strain being $\varphi = 2.7$. The specimens thus deformed were then annealed at the temperatures within the range 500–700°C for 1 hour, followed by quenching into water.

The structure of MIF-specimens was investigated in two planes: in the central plane, parallel to the direction (ND) of strain during upsetting in the final MIF-stage, and in the plane normal to ND, which corresponded to the direction of plastic flow of the material. In as-rolled specimens, TD is the transverse rolling direction and ND is the normal direction. Microstructure was investigated using SEM examination in a mode of orientation-phase contrast in a TESCAN MIRA 3LMH electron microscope and by analyzing the diffraction patterns of backscatter electrons (EBSD-analysis). The latter analysis was performed with the CHANNEL-5 software code. The scanning step was varied from 50 to 300 nm. A criterion that provided the distinction between low- and high-angle grain boundaries (LAGBs and HAGBs, respectively) was the value of misorientation equal to 15°, with the boundaries misoriented by less than 2° neglected due to insufficient accuracy of their determination [5].

No analysis of the carbide particle composition by EBSD was performed due to a small size of these particles, which was less than the scanning step.

## RESULTS AND DISCUSSION

The structural studies of steel 05G2MFB demonstrated that following warm rolling different types of UFG-structure are formed: from equiaxial to fibrous, depending of the strain pattern. Under conditions of multiple isothermal forging, UFG-structure is formed, which consists of grains and subgrains measuring on average 0.4 μm (Fig. 1*b*). This structure is characterized by the grains slightly elongated in the direction of plastic flow of the material during upsetting in the final MIF-stage. No sharp differences in the grain structure were observed, and on the whole it was homogeneous throughout the bulk of the specimen. Warm rolling generally results in the structure with the grains and subgrains severely elongated in the direction of rolling. In the direction transverse to rolling, the cross-sections of these grains are equiaxed and are found to measure 0.4 μm on average, while in the longitudinal direction the fibers are as long as 15 μm (Fig. 1*a*).

An EBSD-analysis of different structural states demonstrated that a MIF-treatment gave rise to the formation of grain-subgrain structure. The resulting UFG-structure contains both low- and high-angle boundaries showing in the EBSD orientation maps as white and black lines, respectively (Fig. 2). The fraction of LAGBs in the forged specimens was found to be 53% (Table 2). In the as-rolled sample, the fraction of HAGBs in the longitudinal direction was found to be 52%, which is 7% smaller than that in the transverse cross-section.

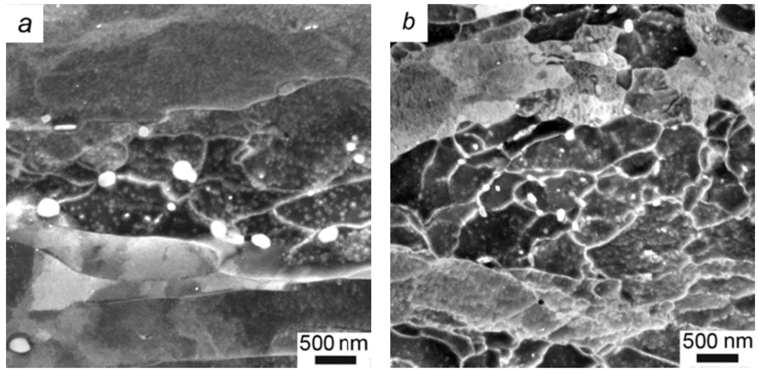


Fig. 1. SEM image of microstructure of steel 05G2MFB: after rolling in the longitudinal section (*a*) and following MIF (*b*).

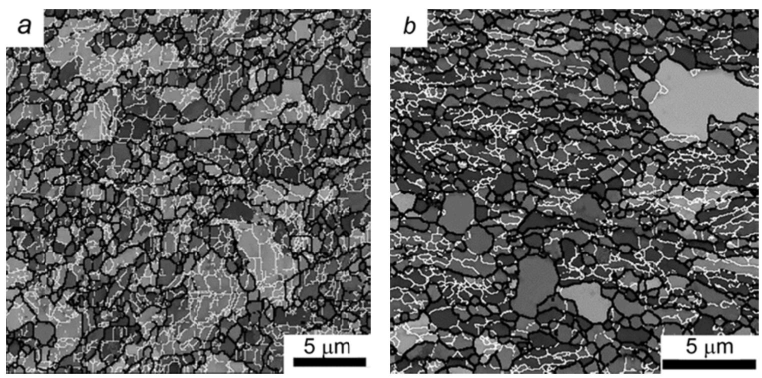


Fig. 2. An EBSD-map of microstructure scanned with a step of 50 nm: after warm rolling followed by annealing at 600°C (*a*) and after MIF-treatment followed by annealing at 625°C (*b*).

Construction of direct pole figures (DPFs) after MIF-treatment revealed the presence of multicomponent deformation microtexture, specifically: {111}<112>, {110}<111>, {100}<010> (Fig. 3*a*). An analysis of the texture component after rolling revealed the presence of such microstructure as {110}<010> (Fig. 3*b*). The fraction of LAGBs in the cross-sections differently oriented with respect to the rolling direction is presented in Table 2. After annealing at 600°C in as-forged specimens, the fraction of LAGBs was observed to decrease from 43 to 37%. An investigation of thermal stability of the structure types, formed by different strain-hardening techniques, allowed us to identify considerable differences in the evolution of the fibrous and equiaxed structure types under the influence of annealing within the temperature interval from 500 to 700°C.

The grain/subgrain boundaries in the specimen produced by MIF and annealing at 550°C become thinner and smoother; the grain and fragment size is still around 0.4 μm, while the fraction of LAGBs is decreased from 53 to 43%. As the annealing temperature is increased up to 600°C, the microstructure formed remains the same.

In as-rolled specimens annealed at 550°C, a slight increase in the grain/subgrain size is observed, without any noticeable changes in the content of LAGBs and microtexture. After annealing at 600°C, the percentage of LAGBs in as-forged specimens is found to decrease from 43 to 37% (Table 2) and there appears a slight fraction of grains with large-angle grain boundary misorientations and lower dislocation density (Fig. 2*a*). On the other hand, the structure is still dominated by areas with deformation microtexture and the average grain/subgrain size ~0.4 μm. In subgrains and recrystallized grains there are negligible amounts of secondary carbide precipitates, measuring less than 20 nm, which are observed both in the bulk of the grains/subgrains and at their boundaries. Annealing of the rolled specimens at

TABLE 2. Average grain/subgrain dimensions, *d*, fraction of LAGBs after MIF-treatment, warm rolling and subsequent annealing treatments.

| Treatment conditions | *d*, μm | Fraction of LAGBs, % |
|---|---|---|
| After MIF | 0.4 ± 0.2 | 53 |
| After MIF + annealing at 550°C | 0.4 ± 0.2 | 43 |
| After MIF + annealing at 600°C | 0.4 ± 0.2 | 37 |
| After MIF + annealing at 625°C | 0.6 ± 0.3 | 30 |
| After MIF + annealing at 650°C | 3.1 ± 0.8 | 15 |
| After MIF + annealing at 700°C | 3.1 ± 0.8 | 8 |
| After warm rolling (longitudinal) / (transverse cross-section) | –/0.4 ± 0.2 | 59/52 |
| After warm rolling + annealing at 550°C (longitudinal) /(transverse cross-section) | –/0.5 ± 0.3 | 60/54 |
| After warm rolling + annealing at 600°C | 0.9 ± 0.4 | 56 |
| After warm rolling + annealing at 650°C | 3.4 ± 0.8 | 9 |
| After warm rolling + annealing at 700°C | 4.8 ± 0.8 | 7 |

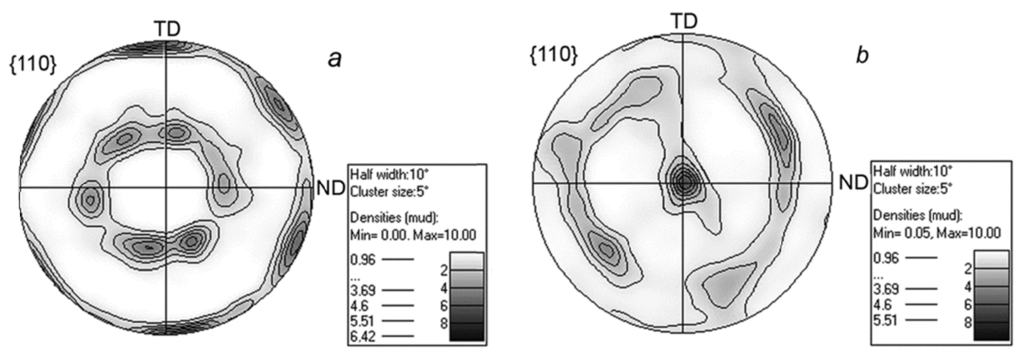


Fig. 3. Direct pole figures constructed for the steel specimens: after MIF-processing (*a*) and after warm rolling (*b*).

600°C gives rise to a further increase in the average transverse grain size and a slight decrease of the percentage of LAGBs. There are no changes in the texture components.

After annealing at 625°C, recrystallized regions appear in as-forged specimens (Fig. 2*b*), where the size of certain grains has anomalously increased to as large as 1 μm. The fraction of recrystallized structure is ~10%. In the regions that avoided recrystallization, the fragment size has increased but insignificantly, to as large 0.6 μm. At the same time, the percentage of LAGBs has decreased down to 30%. The secondary phase carbide precipitates measure ~30 nm. The microstructure is observed to partially smear, and a new microtexture component is seen to form: {110}<112>, which is associated with the onset of recrystallization.

After annealing of as-forged specimens at 650°C, the percentage of recrystallized regions in them becomes as high as 87%, with their structure consisting of grains as small as (3.1 ± 0.8) μm on average and, at the same time, quite large grains measuring more than 10 μm. The fraction of LAGBs is found to be 15%. The content of the secondary carbides is also significantly increased, their size being more than 40 nm. The fraction of the annealing-induced microtexture is observed to increase.

Annealing of as-rolled specimens at 650°C results in the formation of homogeneous recrystallized structure with the average grain size ~3.4 μm and the fraction of LAGBs is 9%. An analysis of the resulting microtexture demonstrated that a {100}<120> annealing component was added to the {110}<010> component. An increase in the annealing temperature up to 700°C in the forged state gives rise to a virtual completion of the process of UFG-structure recrystallization. The content of recrystallized region is as high as 99%, the percentage of LAGBs is reduced to 8%, and the average grain size is found to be (3.1 ± 0.8) μm. The texture is severely smeared. In as-rolled samples after annealing at 700°C we also observe the formation of completely recrystallized structure with the average grain size ~4.8 μm, which is accompanied by a decrease in the percentage of LAGBs. The formation of a completely recrystallized structure is also indicated by the development of an absolutely different microtexture: {110}<211>, {110}<100>.

In as-forged specimens, the average grain/subgrain size in UFG-structure remains unchanged as the annealing temperature is increased up to 600°C, while the fraction of LAGBs is somewhat reduced, which is due to the processes of non-equilibrium structure recovery and relaxation. Stability of the resulting UFG-structure up to the above-mentioned temperature is accounted for by the fact that deformation of the specimen is accompanied by a partial solution of cementite resulting in oversaturation of ferrite with carbon and easy formation of finely dispersed carbides. Segregations of carbon atoms and formation of finely dispersed carbides on dislocations and grain boundaries, in their turn, block their motion. In the forged state, an annealing run at the temperature as low as 550°C gives rise to an increase in the transverse average grain/subgrain size, though the fraction of LAGBs is virtually unchanged as the annealing temperature increases to 650°C.

In accordance with the Zenner criterion

$$\frac{2r}{D} \leq \frac{3\nu}{2} \tag{1}$$

and the Hellman–Hillart criterion

$$\frac{2r}{D} \leq \left(\frac{\nu}{6}\right)^{\frac{1}{3}}, \tag{2}$$

where $r$ is the particle radius, $D$ is the average grain size, $\nu$ is the volume fraction of particles. In order to retard grain/subgrain boundary migration, a large number of fine particles is needed on the side of a grain, and in the case of a large size of particles – their pinning in the apexes [9]. It is evident from (1) and (2) that the smaller the size of the secondary particles, the lower is the boundary mobility, hence, the structure is more stable. According to the structure examination, the volume fractions of carbides in as-rolled and as-forged specimens are virtually the same, while the size of the particles in as-rolled state is larger (see Fig. 1). This suggests that the structure after rolling would be less stable than that produced by the MIF processing. This is supported by the data on a faster grain growth in as-rolled specimens compared to as-forged sample after annealing at 550°C. Therefore, grain-boundary migration during annealing of as-rolled specimens is less impeded.

During annealing at the temperatures higher than 600°C, precipitation of finely dispersed special carbides [10] results in the pinning of grain- and subgrain-boundaries, which retards their growth. In the latter case, the secondary-phase particle distribution within the structure is invariably inhomogeneous. Due to higher grain-boundary mobility with increasing temperature [9], the grains and subgrains, not stabilized by the particles, begin intensively to grow, which results in the formation of a consertal structure. This structure is observed in as-forged state after annealing at 625°C (Fig. 2*b*).

When the annealing temperature is increased to 650°C in as-rolled specimens, a recrystallized structure is formed with a larger average grain size (Table 2) than in as-forged samples after the same type of annealing. This also supports the assumption on weaker deceleration of grain-boundary migration due to carbides in as-rolled samples.

**SUMMARY**

It has been shown that processing of low-carbon steel 05G2MFB by multiple isothermal forging results in the formation of equiaxial UFG-structure with the average grain and sibgrain size 0.4 μm and the deformation microtexture of the following type: {111} <112>, {110} <111>, {100} <010>.

After warm rolling of low-carbon steel, fibrous UFG-structure is formed with the average transverse grain and subgrain size being 0.4 μm and the longitudinal grain size being 15–20 μm; this structure has a typical rolling microtexture {110} <010>.

The changes in the average grain size and in the percentage of low-angle grain boundaries observed in the fibrous UFG-structure begin at the annealing temperature that is 50°C lower than that in the equiaxial structure. This is due to predomination of the static recovery over the static recrystallization when the annealing temperature in as-forged specimens is increased up to 600°C and vice versa in as-rolled specimens. The difference in thermal stability of the resulting UFG-states is controlled both by the grain structure type and by the differences in the disperse phase morphology.

## REFERENCES


1. V. A. Valitov, R. R. Mulyukov, A. A. Nazarov, *et al*., Zh. Probl. Mashinostr. Avtomatiz., No. 3, 51–56 (2013).
2. I. I. Musabirov, I. M. Safarov, R. R. Mulyukov, *et al*., Lett. Mater., **4**, Issue 4, 265–268 (2014).
3. I. M. Safarov, A. V. Korznikov, S. N. Sergeev, *et al*., J. Phys. Met. Metallogr., **113**, No. 10, 1–6 (2012).
4. I. M. Safarov, A. V. Korznikov, R. M. Galeyev, *et al*., J. Phys. Met. Metallogr., **115**, No. 3, 315–323 (2014).
5. V. N. Danilenko, S. Yu. Mironov, A. N. Belyakov, and A. P. Zhilyaev, Zh. Zavod. Lab. Diagnost. Mater., **78**, No. 2, 28–46 (2012).
6. S. N. Sergeev, I. M. Safarov, A. V. Korznikov, *et al*., Pis'ma Mater., **2**, No. 3, 117–120 (2012).
7. R. M. Imayev, A. A. Nazarov, R. R. Mulyukov, *et al*., Lett. Mater., 4, Issue 4, 295–301 (2014).
8. S. N. Sergeev, I. M. Safarov, A. V. Korznikov, *et al*., Pis'ma Mater., **2**, No. 2, 74–77 (2012).
9. M. A. Stremel, Strength of Alloys. Part I. Lattice Defects [in Russian], MISIS Publ. (1999).
10. E. G. Astafurova, G. G. Zakharova, E. V. Naidenkin, *et al*., J. Phys. Met. Metallogr., **111**, No. 1, 64–73 (2011).