Microstructure Evolution in a 9%Cr Heat Resistant Steel during Creep Tests

V. Skorobogatykh[1,a], I. Schenkova[1,b], V. Dudko[2,c], A. Belyakov[2,d] and R. Kaibyshev[2,e]

[1]Central Research Institute for Machine-Building Technology, Sharikopodshipnikovskaya 4, Moscow 115088, Russia

[2]Belgorod State University, Pobeda 85, Belgorod 306015, Russia

[a]polyform_k@mail.ru, [b]in_material@mail.ru, [c]dudko@bsu.edu.ru, [d]belyakov@bsu.edu.ru, [e]rustam_kaibyshev@bsu.edu.ru

Keywords: Heat resistant steel; tempered martensite; creep test; carbide coarsening; microstructure evolution.

Abstract. Dynamic structural changes during creep tests for about 10^3 hours at 600 and 650°C were investigated in a P92-type 9%Cr martensitic heat resistant steel. The structural changes are characterised by the development of relatively large equiaxed subgrains with relatively low dislocation densities in place of initial martensite laths. The coarsening of substructure was accompanied by a growth of second phase precipitates. The final grain/subgrain sizes and dislocation densities evolved after the creep tests were in rough correlation with applied stresses, *i.e.* larger (sub)grains developed under lower stresses. The structural mechanism responsible for microstructure evolution was considered as a kind of continuous dynamic recrystallization.

Introduction

Martensitic steels containing 9-12%Cr are considered as promising creep resistant structural materials [1-3]. Commonly, the strengthening of these steels results from tempered martensitic structures including the well developed lath martensite substructure with high dislocation density in the subgrain interiors and the various homogeneously distributed fine second-phase precipitations. The creep resistance, therefore, depends on the stability of original tempered martensitic structures at elevated temperatures. The coarsening of initial structural elements, i.e. martensite laths and second-phase particles at elevated temperatures can remarkably deteriorate the mechanical properties [4, 5]. In spite of much effort to discovering the crept microstructures, the evolutional mechanisms responsible for microstructure development in martensitic steels during high temperature creep are still unclear. Thus, the studies on the structural changes in the heat resistant steels during creep at elevated temperatures are of great practical importance.

The fine uniformly distributed particles in tempered martensite provide the dispersion strengthening of the steel and the stabilization of martensite lath substructure. Generally, the size of substructural elements (subgrains, laths, etc.) in single-phase metals and alloys gradually increases with increase in heating temperature and/or duration of annealing [6]. In the case of plastic deformation at high temperatures, the dynamic size of developing subgrains depends inversely on the level of flow stress (or temperature-compensated strain/creep rate) [7]. The presence of fine particles in every alloy retards the subgrain growth in static and dynamic conditions because of so-called pinning effect [8]. Therefore, much finer subgrain size can be predicted in the tempered heat resistant steel subjected to long term creep/annealing. The relationship between the evolved grain/subgrain size in multiphase matrix-type metallic materials and the size and volume fraction of second-phase particles has been fairly clarified for static annealing condition. However, effect of hot plastic deformation on the developing substructures in dispersion strengthened steels has not been investigated in sufficient detail. In the present work, dynamic structural changes in a 9%Cr martensitic heat resistant steel are studied in creep tests. The aim of this study is to evaluate the parameters of martensitic substructure and their relation to the second phase precipitates in samples subjected to creep tests at elevated temperatures.

Experimental

A P92-type steel with chemical composition as follows, Fe-0.1C-0.17Si-0.54Mn-8.75Cr-0.21Ni-0.51Mo-1.60W-0.23V-0.07Nb (all in mass%), was fabricated by Chelyabinsk Metallurgical Plant, Russia. The steel was solution treated at 1050°C followed by air cooling and then tempered at 730°C for 3 hours. The prior austenite grains size was about 20 μm. The tensile specimens of ∅10 mm with the gauge length of 50 mm were subjected to the creep test up to rupture at 600 and 650°C under starting stresses of 196 and 118 MPa, respectively. The structural changes were analysed on the longitudinal section close to the necking portion of crept specimens (about 1.5 mm away from the fracture surface) with a reference to the original tempered microstructure.

Structural investigations were performed by using a Quanta 600 scanning electron microscope equipped with an electron back scattering diffraction pattern (EBSP) analyser incorporating an orientation imaging microscopy (OIM) system, and a JEM-2100 transmission electron microscope (TEM). The lath/subgrain sizes were measured on the TEM micrographs by the linear intercept method, including all clear visible (sub)boundaries. The same method was applied to evaluate the block/grain sizes on the OIM images by counting the boundaries with misorientations above 15 degrees. Therefore, the grain size represents the inter-boundary spacing. The sizes of second phase particles were estimated by averaging the TEM data of about 100 measurements of individual particles in each sample. The dislocation densities were evaluated by counting the individual dislocations in grain/subgrain interiors on at least six arbitrary selected typical TEM images for each sample. Hardness measurements were carried out with the load of 5 N.

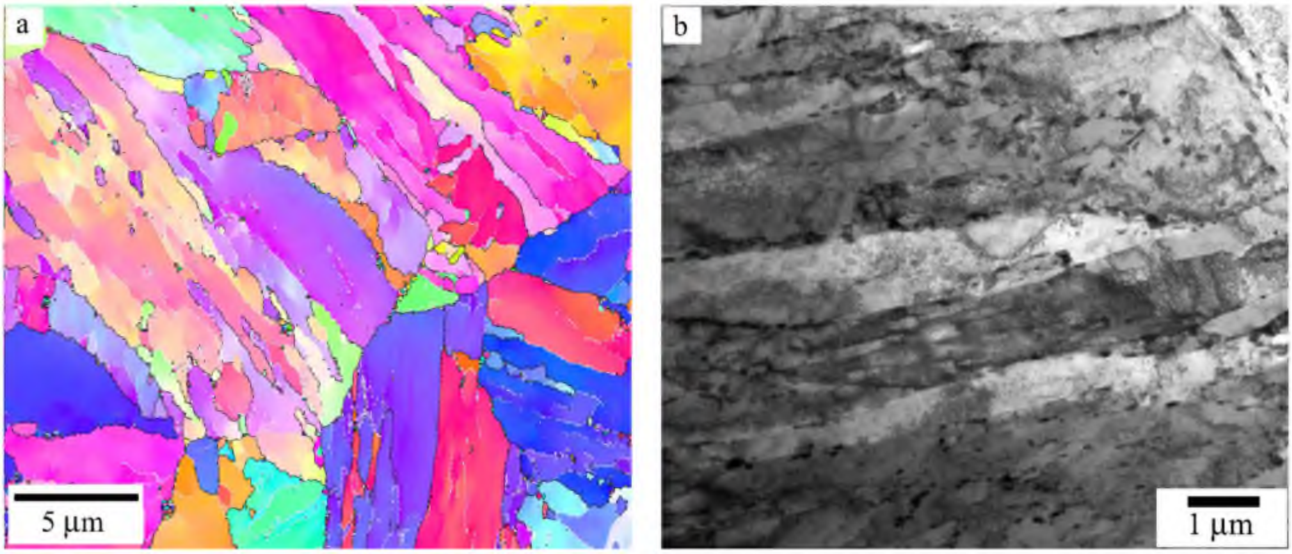

Fig. 1. Tempered martensite in a 9%Cr heat resistant steel; (a) typical OIM and (b) TEM images. The white and black lines in (a) indicate the low- and high-angle boundaries.

Results and Discussion

Tempered Microstructure. The tempered martensite structure is shown in Fig. 1. The tempered microstructure consists of lath blocks subdividing prior austenite grains. The block/grain size is about 2 μm. The fraction of special Σ3 boundaries among all boundaries with misorientations above

2^o comprises about 0.1. The transverse lath size is about 330 nm. The lath interiors are characterised by rather high dislocation density of 6.2×10^{14} m^{-2} in average. The tempering treatment resulted in the precipitation of second-phase particles. Relatively large $Me_{23}C_6$ carbide particles with an average size of about 85 nm and the volume fraction of about 0.08 are mainly located along the lath and block boundaries. The lath interiors are homogeneously filled with plate-shaped fine V(C,N) particles with average longitudinal and transverse sizes of about 8 and 2 nm, respectively. The volume fraction of fine carbonitrides is about 0.002, which was estimated by counting the number of particles per unit volume, $N_V = 2 \times 10^{22}$.

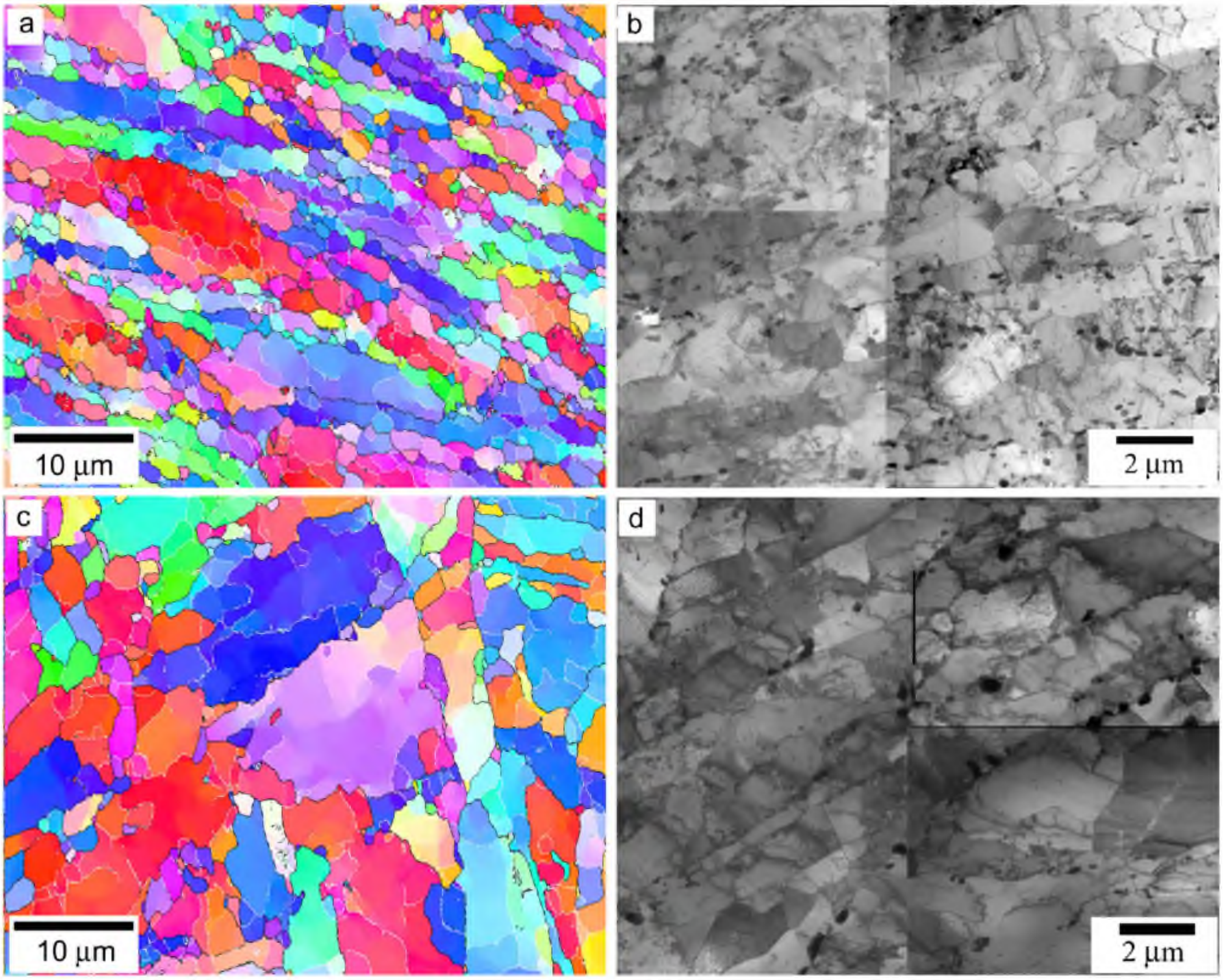

Fig. 2. Typical microstructures evolved in a 9%Cr heat resistant steel during creep tests at (a, b) 600°C and (c, d) 650°C. The white and black lines in (a, c) indicate the low- and high-angle boundaries.

Deformation microstructures. Typical microstructures evolved after the creep tests are presented in Fig. 2. The times to rupture of the samples tested at 600 and 650°C were 920 and 1271 hour, respectively. The microstructures developed in the crept specimens are quite different from that in the original tempered sample. The initial martensite structure was completely replaced by new ones consisting of almost equiaxed grains/subgrains. Generally, the crept microstructures look like partially dynamically recrystallized microstructures that develop during ordinary hot-working [7].

The plastic deformation at elevated temperatures results in increasing the size of structural elements, i.e. grains and subgrains, irrespective of testing conditions (*cf.* Fig. 1 and 2). However, the sample tested at lower temperature is characterized by a remarkably finer microstructure. The grains with sizes of 2.2 and 3.1 μm are evolved at 600 and 650°C, respectively. Similarly, the sizes of subgrains in transverse directions are about 600 and 740 nm in these samples. The coarsening of grains/subgrains during the creep tests of tempered martensite is accompanied by a decrease in the interior dislocation densities. The latter ones decrease to 2.8×10^{14} and 1.0×10^{14} m^{-2} at 600 and 650°C, respectively. The dislocation densities (ρ) in the crept samples can be related to the flow stresses (σ) by means of a conventional work-hardening mechanism, $\sigma = \alpha G b \sqrt{\rho}$ [9], where G is the shear modulus, b is the Burgers vector and α is a constant of about 0.7 in the present study. On the other hand, the sizes of grains and subgrains demonstrate much weaker stress dependence than it is reported for hot deformation of single-phase metallic materials [7].

The growth of grains/subgrains during the creep occurs concurrently with the coarsening of second-phase particles. It should be noted that plastic flow leads to coarsening of all-type precipitates. Figure 3 shows a row of rather large $Cr_{23}C_6$-type carbides, which were precipitated along the block and lath boundaries during the tempering treatment. Much similar to the tempered microstructure, the chains of these carbides are mainly located at grain/subgrain boundaries in the deformation structure after the creep tests (Fig. 2). The size of fine V(C, N)-type carbides that homogeneously precipitated throughout the tempered sample is also increased by creep tests. Relatively large carbonitrides sized up to about 100 nm are observed in the deformation structures as intragranular particles (Fig. 3).

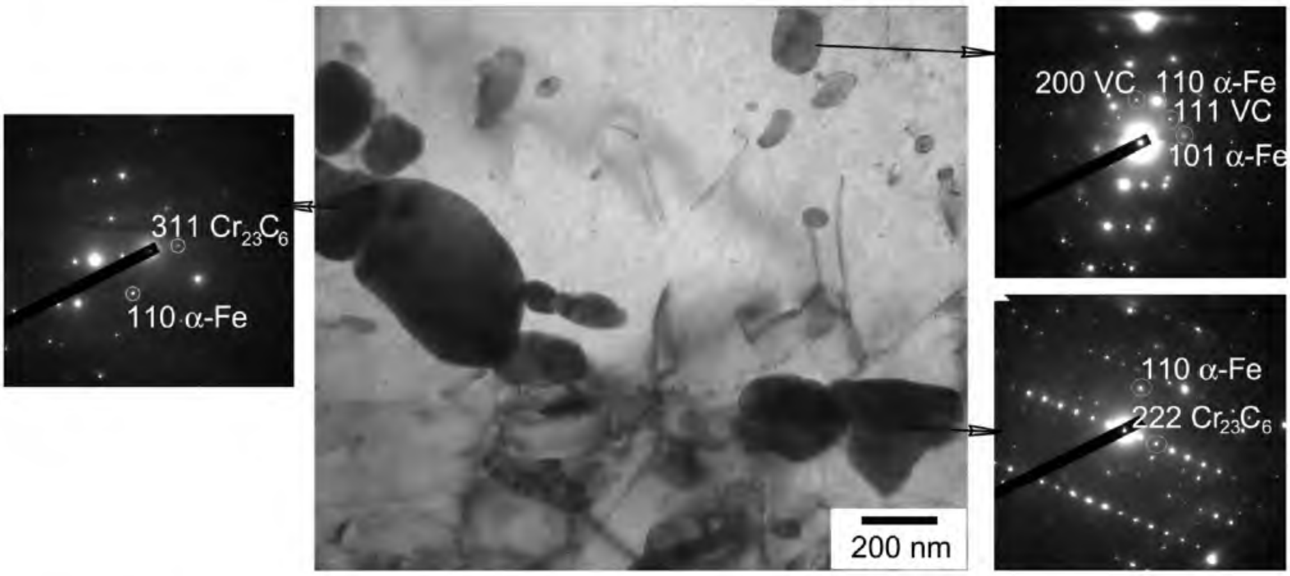

Fig. 3. Coarsening of second-phase precipitates in a 9%Cr heat resistant steel during creep test at 600°C.

The size distribution for second-phase particles after tempering and following creep tests is quantitatively represented in Fig. 4. The particle size distribution for initial tempered state is characterised by a narrow peak of sizes ranging mainly from 20 nm to 130 nm. Note here that the very fine V(C, N) particles were omitted from the measurements due to some methodical reasons. Commonly, the sharp peak against particle size of about 70 nm in the tempered sample decreases and spreads out towards larger sizes in the crept samples. The average particle size increases to about 110 and 210 nm during the tests at 600 and 650°C, respectively.

The effect of creep tests on the structural parameters evolved in the studied samples is summarized in Table 1. The plastic deformation at elevated temperature significantly alters the initial microstructures of tempered martensite. The development of almost equiaxed (sub)grains with low- to high-angle boundaries that accompanied by a remarkable decrease in the fraction of Σ3 boundaries suggests that the microstructure evolution results from a kind of continuous dynamic recrystallization. Generally, the dynamic sizes of grains and subgrains that evolve in single-phase alloys during hot deformation should follow the power functions of flow stresses, *i.e.* $\sigma \sim d^{-N}$, where N is about 0.7 and 1.0 for grains and subgrains, respectively [7]. Some deviation of the experimental stress dependence for grain/subgrain sizes from the equation above is probably caused by the presence of second-phase particles, although the measured sizes do not obey the Zener pinning relationship [8] as well. The present results suggest that the various particles in the P92-type steel confine more effectively the growth of relatively large grains/subgrains, while the size of small (sub)grains is less affected by second-phase particles because of higher driving force for their growth.

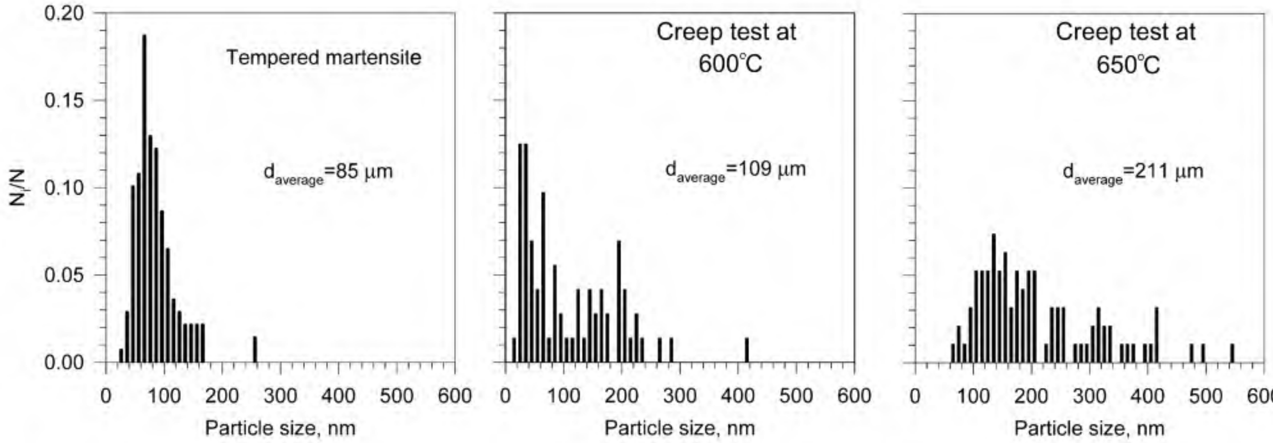

Fig. 4. Size distributions of relatively coarse second-phase precipitates in a 9%Cr heat resistant steel after tempering and creep tests.

Table 1. Some parameters of microstructure evolved in a 9%Cr heat resistant steel after tempering and creep tests.

Sample	Creep stress [MPa]	Total elongation [%]	Block/grain size [μm]	Transverse lath/subgrain size [nm]	Dislocation density [10^{14} m^{-2}]	Particle size [nm]	Fraction of Σ3 boundaries
Tempered	-	-	2.0	330	6.2	85	0.078
Crept at 600°C	196	28.2	2.2	600	2.8	109	0.018
Crept at 650°C	118	22.2	3.1	740	1.0	211	0.033

Summary

The structural changes during creep of a P92-type martensitic steel were studied at temperatures of 600 and 650°C. The plastic deformation of tempered martensite at elevated temperatures was accompanied by the evolution of almost equiaxed grain/subgrain structure that developed concurrently with the coarsening of second-phase particles. The size of structural elements increased with an increase of temperature and/or a decrease of flow stress similar to conventional hot working. The mechanism of microstructure evolution was considered as a kind of continuous dynamic recrystallization.

Acknowledgements

The study was supported by Federal Agency for Science and Innovations, grant No. 02.523.12.3019. Authors are grateful to Mr. E. Kudryavtsev, Center of Common Facilities, Belgorod State University, for his assistance in specimens preparation and instrumental analysis.

References

[1] K. Maruyama, K. Sawada and J. Koike: Iron Steel Inst. Jpn. Int. Vol. 41 (2001), p. 641.

[2] P.J. Ennis and A. Czyrska-Filemonowicz: Sadhana Vol. 28 (2003), p. 709.

[3] M. Taneike, F. Abe and K. Sawada: Nature Vol. 424 (2007), p. 294.

[4] K. Sawada, M. Takeda, K. Maruyama, R. Ishii, M. Yamada, Y. Nagae and R. Komine: Mater. Sci. Eng. A Vol. A267 (1999), p. 19.

[5] F. Abe, T. Horiuchi, M. Taneike and K. Sawada: Mater. Sci. Eng. A Vol. A378 (2004), p. 299.

[6] P.A. Beck, J.C. Kremer, L.J. Demer and M.L. Holzworth: Trans. AIME Vol. 13 (1948), p. 372.

[7] H.J. McQueen and J.J. Jonas, in: *Treatise on Materials Science and Technology*, edited by R.J. Arsenoult, Academic Press, NY (1975).

[8] P.A. Manohar, M. Ferry and T. Chandra: Iron Steel Ins. Jpn. Int. Vol. 38 (1998), p. 913.

[9] D. Hull and D.J. Bacon: *Introduction to Dislocations* (Butterworth Heinemann, UK 1984).