Dynamic polygonization in 9%Cr heat resistant steel

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Abstract. Structural changes in 9%Cr martensitic steel during creep were examined. The grip section of the crept specimen was characterised by a lath martensite structure, which hardly changed during the test. In contrast, quite different microstructure developed in the necking portion of the specimen. The structural changes were characterized by the evolution of relatively large equiaxed subgrains with remarkably lowered density of interior dislocations at places of initial martensite laths. The development of the well-defined subgrains in the necking portion was accompanied with a coarsening of second phase precipitations. The structural mechanism responsible for the microstructure evolution during creep is considered as a dynamic polygonization.

1. Introduction

Nowadays, tempered martensitic 9-11%Cr steels are considered as promising creep resistant structural materials for fossil power plants [1-4]. The creep properties of these materials at elevated temperatures depend on the stability of martensite laths [5, 6]. Structural changes leading to significant changes of original microstructures during hot deformation are commonly related to the development of dynamic recrystallization [7]. Two general mechanisms for dynamic recrystallization were proposed [8]. The discontinuous dynamic recrystallization was shown to occur in alloys with relatively low stacking fault energy, when the new grains nucleate and grow due to bulging and migration of original grain boundaries over long distances. Another mechanism of dynamic recrystallization takes place in materials with high ability to dynamic recovery and involves continuous development of new grains as a result of formation of strain-induced subboundary network and gradual increase in misorientations between subgrains. However, the mechanisms of microstructure evolution operating in martensitic steels during the creep at elevated temperatures have not been detailed. Efficiency of different second-phase particles for microstructure stabilization against dynamic evolution is still unclear. High dislocation densities in martensite laths may promote a local bulging of grain boundaries, leading to discontinuous recrystallization. On the other hand, the rearrangement of lath boundaries and interior dislocations may result in the development of new recrystallized grains that is similar to continuous recrystallization by subgrain coalescence. The aim of the present work is to study the mechanism of microstructure evolution in a 9%Cr steel during the creep at a temperature of 650°C, which is just above the service temperature of 620°C for this material.

2. Experimental

A P92-type steel, 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 subjected to solution treatment at 1050°C followed by air cooling and then tempered at 730°C for 3 hours. The tensile specimens of ∅10 mm with the gauge length of 50 mm were crept up to rupture at 650°C under starting stresses of 118 MPa. The sample ruptured after 1271 hours was selected for detailed structural examination. Structural investigations were performed by using a Quanta 600 FEG 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. The subgrain sizes were measured on the TEM micrographs by the linear intercept method, including those at all the clear visible (sub)boundaries. The dislocation densities were evaluated by counting the individual dislocations in grain/subgrain interiors. Hardness measurements were carried out with the load of 5 N on the central section along the specimen axis.

3. Results and discussion

The starting martensite microstructure was obtained by air cooling of austenite with an average grain size of ~20 μm. A typical tempered fine structure is shown in figure 1a. The tempered martensite consists of lath blocks subdividing original austenite grains (figure 1). Parameters of microstructure is shown in table 1. The lath interiors are homogeneously filled with plate-shaped fine V(C,N) particles (figure 2a). The particles obey the Baker-Nutting type orientation relationship [9], *i.e.* $(100)_{V(C,N)} \parallel (100)_{\alpha}$, $[011]_{V(C,N)} \parallel [001]_{\alpha}$. The average longitudinal size and thickness of these fine carbonitrides are about 8 and 2 nm, respectively.

The hardness of initial tempered steel remarkably decreased after the creep test. However, the softening is not uniform along the crept specimen. The hardness gradually decreases from the portion close to the grip section to the specimen neck. The softening during creep correlates with the total creep strain.

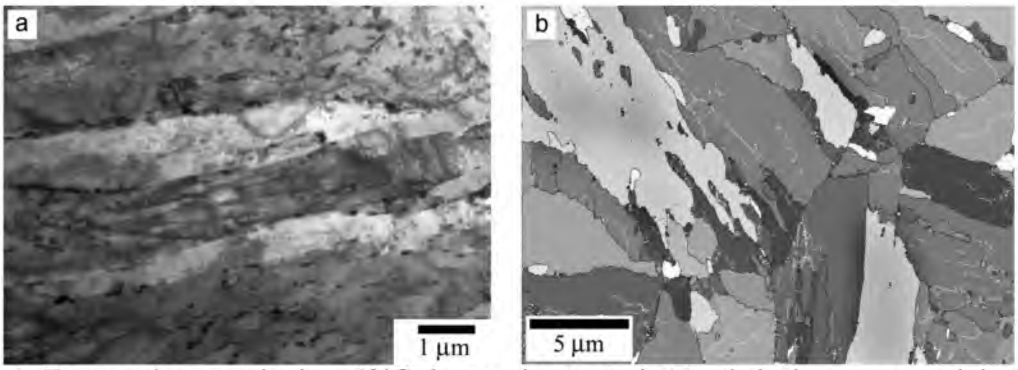

Figure 1. Tempered martensite in a 9%Cr heat resistant steel; (a) relatively coarse precipitations of carbides and Laves phases at boundaries of blocks and laths; (b) OIM map.

Table 1. Microstructure parameters of P92 steel.

	Transverse size of laths, nm	Densities of dislocation in the lath, m^{-2}	Average size of particles, nm	Portion of CSL boundaries, %	Hardness, MPa
Tempered microstructure	330	6.2×10^{14}	85	9	2680
Head of specimen	330	4.4×10^{14}	112	7	2340
Neck of specimen	740	10^{14}	211	3	1820

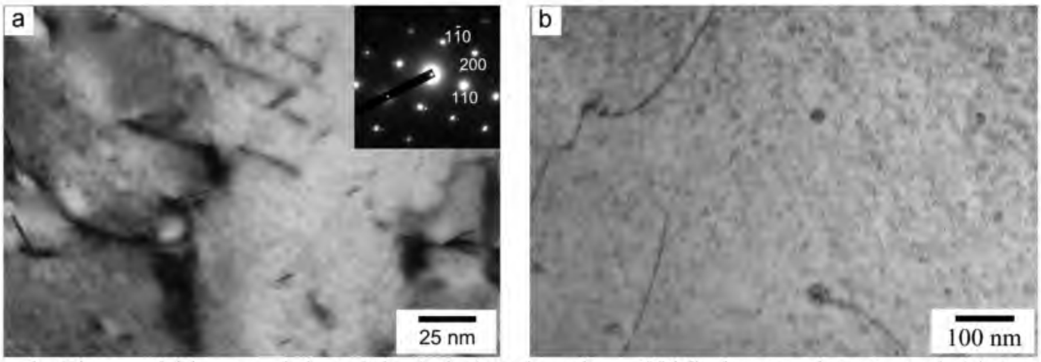

Figure 2. Fine carbides precipitated in lath interiors in a 9%Cr heat resistant steel (a) tempered martensite; (b) carbonitrides close to the neck portion of sample after the creep test.

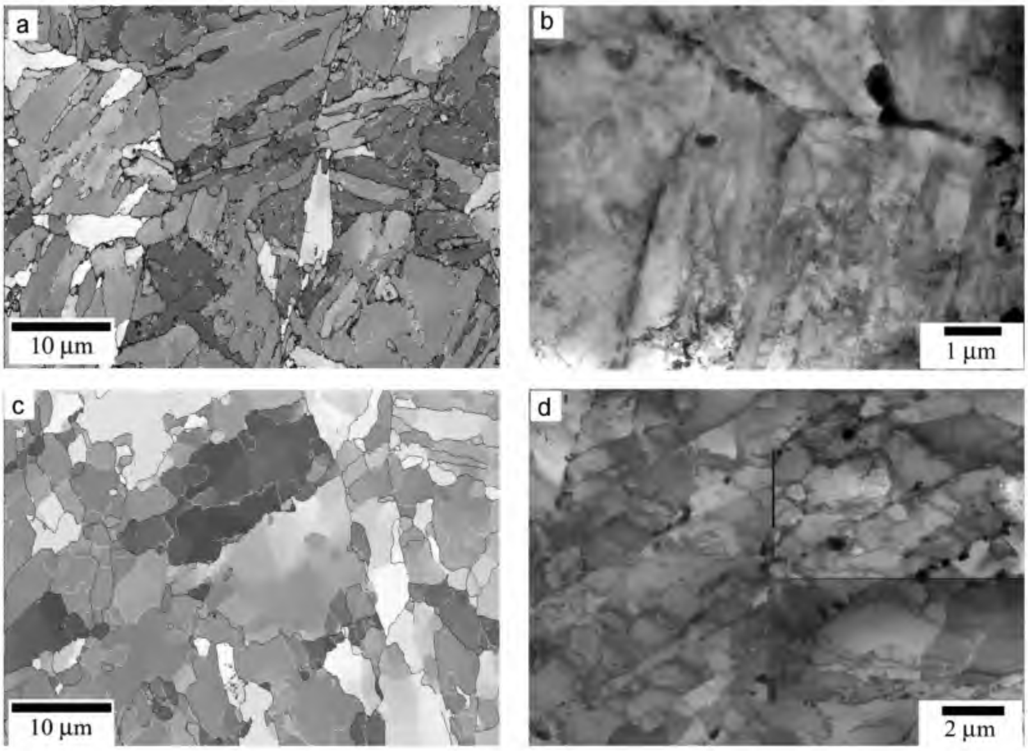

Figure 3. OIM maps and TEM photographs for (a, b) thread and (c, d) neck portions of a 9%Cr heat resistant steel specimen after the creep test.

The effect of hot plastic deformation on the structural changes in the tempered martensite of 9%Cr steel is clearly illustrated by OIM and TEM micrographs in figure 3. The grip section of the crept specimen did not experience a remarkable plastic deformation. Therefore, the structural changes in the grip section can be simply considered as a result of static recovery or recrystallization. The creep test did not change significantly the lath martensitic microstructure evolved in the grip section that looks like typical tempered martensitic microstructure (figures 3(a, b)). In contrast, the microstructure developed in the neck part of the crept specimen is quite different from the initial one. The microstructures in figure 3(c, d) look like typical dynamically recrystallized that is composed by almost equiaxed grains/subgrains with irregular boundaries. It should be noted that the fraction of special boundaries with high density of coincident sites decreased after the creep test (table 1). Coarsening of microstructure during the creep test is accompanied by a growth of fine V(C, N) precipitations. A few of round-shaped fine particles with an average size of about 30 nm can only be observed with in interiors of subgrain after creep test (figure 2b).

The development of relatively large subgrains in necking portion of crept specimen and the creep softening indicate that the plastic flow can significantly accelerate the coarsening of all second phase particles. The dislocation fluxes promote the diffusion processes leading to the particle coagulation during creep. The mobility of dislocation subboundaries increases due to decreasing the pinning forces. Also, the dislocation motion and interaction with the special boundaries result in the change of orientation relationship and, therefore, facilitate the boundary mobility. Then, the dislocation rearrangement within martensite substructure and the development of dynamic recovery result in the evolution of conventional hot worked substructure. The formation of new subgrains takes place continuously during the deformation due to rearrangements of interior dislocations; that is in-situ without nucleation and growth of new recrystallizing grains. Any local bulging of high-angle grain boundaries scarcely accompanies the microstructure evolution. The fraction of high-angle grain boundaries does not rise and the (sub)boundary misorientation distributions do not significantly change during the process. Therefore, such evolutional process is similar to a continuous dynamic recrystallization, which frequently develops during hot working of metallic materials with high stacking fault energy [10]. In the present study the structural changes took place on the substructural level; namely, the martensitic laths gradually transformed to hot deformation subgrains. Thus, the considered mechanism of microstructure evolution should be discussed as a dynamic polygonization.

4. Conclusions

The structural changes depended significantly on the amount of plastic strain. The initial microstructure of tempered martensite with the transverse lath size of 330 nm was changed to relatively coarse substructure with the average subgrain size of about 740 nm in the neck portion of the crept specimen, where the experienced strain (reduction in area) was about 60%. In contrast, the martensitic microstructure in the grip section of the specimen scarcely changed during the test.

The subgrain coarsening in the necking part of the specimen during the creep test was accompanied by corresponding growth of second phase particles that originally precipitated at boundaries of blocks and laths. The average particles size increased from 85 to 211 nm during the test.

The mechanism of microstructure evolution in the neck portion of the crept specimen is considered as a dynamic poligonization.

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