Microstructure changes occurring in heat-resistant steels during severe plastic deformation and subsequent creep

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Abstract. This work investigates creep and microstructure characteristics in P92 martensitic and 304L austenitic stainless steel after the imposing of large value of plastic strain. The as-received coarse-grained steels were strongly deformed at room temperature (RT) before tensile creep testing. The flat specimens were tested at 873 K and 923 K under different applied stresses. Microstructures were analysed by means of scanning electron microscope with EBSD (electron back-scatter diffraction) camera and transmission electron microscope. It was observed that application of severe plastic deformation (SPD) techniques at RT significantly reduces grain size and changes phase structure of 304L austenitic steel. The microstructure investigations revealed that the growth of new phases is enhanced in plastically deformed P92 martensitic and 304L austenitic stainless steels compared to their undeformed states. Creep results showed that the mean grain size of SPD-processed steels is still near ultrafine-grained region even after long-term creep testing and the coarsening of microstructure is predominantly influenced by creep time and strain.

1 Introduction

The methods of allowing to impose the large deformation into the microstructure provide a unique opportunity to study creep processes in ultrafine-grained or even nano materials [1]. The grain size of these materials is comparable or even finer than stationary subgrain size in their coarse-grained counterparts. For this reason creep mechanisms related to the high-angle grain boundaries (HAGBs) may significantly affect the mechanical properties of materials at high temperatures [2,3]. The grain
boundaries can strengthen the material because they act as strong obstacles to dislocation glide. But they can also reduce strength of materials through the processes associated with grain boundaries, such as grain-boundary sliding [4] or enhanced recovery of dislocations at grain boundaries [5].

The martensitic and austenitic chromium steels are very often used for the high-temperature components of steam power plants. Their creep resistance is significantly influenced by the density of free dislocations and low-energy boundaries, such as low-angle grain boundaries and martensitic lath boundaries whose movement is restricted by precipitates [6,7]. The microstructure of martensitic and austenitic chromium steels change and deteriorate their mechanical properties during long-term annealing and creep exposure at high temperatures. The deterioration of creep properties and degradation of microstructure in these steels is caused by martensite recovery, growth of carbidic and formation of new coarse phase or σ phases which reduce the content of W, Mo or Cr in solid solution [6].

It was found that the microstructure refinement through severe plastic deformation leads to the faster precipitation of Laves phase in ultrafine-grained martensitic P92 steel [8]. By contrast, the previous works showed that cold strain imposed into austenitic steels can enhance the precipitation of secondary phases [9-11] which lead to the degradation of properties in stainless steels, in particular an increase in brittleness and a decrease in corrosion resistance [12-14]. It was also shown that plastic strain of many austenitic steels at low temperature may cause the formation of a large amount of deformation-induced martensite [15].

The aim of the present investigation is to evaluate the influence of severe plastic deformation at room temperature (RT) on microstructure and creep in P92 martensitic and 304 austenitic steels.

2 Experimental Materials and Methods

The experimental materials used in the present investigation were coarse-grained P92 and 304 L austenitic steels. Their chemical composition (in wt%) and heat treatment can be found elsewhere [8,9].

The specimens with a thickness of 1.1 mm in the form of either disc with 30 mm in diameter or sheets 100 mm long and 10 mm wide were cut from coarse-grained P92 and 304L steels. The discs were deformed by 1 rotation of high-pressure torsion (HPT) at room temperature under the pressure of 6 GPa and rotation speed of 0.1 mm per second. The sheets were processed by high-pressure sliding (HPS) at room temperature with the sliding distance of 5 mm under the pressure of 4 GPa.

The amount of imposed equivalent strain during HPT depends on the radius of the disc and can be estimated by following equation [1]:

$$\varepsilon_{eq} = \frac{2\pi N}{\sqrt{3} t}$$  \hspace{1cm} (1)

where \( r \) is the distance from the disc centre, \( N \) is the number of turns and \( t \) is the thickness of disc. The tensile specimen located about 7.5 mm from the disc centre. From this reason the minimum of equivalent strain \( \varepsilon_{eq} \) is about 23 and the maximum is about 37 (if we consider the areas near grip parts). The amount of imposed equivalent strain during HPS process can be determined by this equation [16]:

$$\varepsilon_{eq} = \frac{x}{\sqrt{3} t}$$  \hspace{1cm} (2)

where \( x \) is the sliding distance of plunger with respect to anvil and \( t \) is the thickness of the sample. Thus equivalent strain imposed during HPS was about 7.8. The further details of sample preparation are given elsewhere [17,18]. Constant load tensile creep tests of P92 steel were conducted at 873 K and austenitic 304L steel was tested at 923 K. The experimental materials were tested under different loads using flat specimens of a gauge length of 8 mm, thickness of 1 mm and width 3 mm.
Microstructure investigated by scanning electron microscope (Tescan Lyra 3) equipped with a NordlysNano EBSD detector. The specimens of ultrafine-grained P92 martensitic steel processed by HPT was analysed on the section perpendicular to the rotation axis of the disc. The microstructure of HPT processed states was investigated on areas about 48 µm² with EBSD step size of 25 nm. The microstructure processed by HPS and after creep exposure was investigated on areas about 3000 µm² with EBSD step size of 50 nm. The boundaries with $\theta \geq 15^\circ$ are referred as high-angle grain boundaries (HAGBs) and boundaries with $\theta < 15^\circ$ are referred as low-angle grain boundaries (LAGBs). The mean intercept spacing of LAGBs and HAGBs was performed along test longitudinal and transverse lines.

![Microstructure of SPD-processed steels](image)

Figure 1. Microstructure of SPD-processed steels: a) P92 processed by HPT ($\varepsilon_{eq} \sim 23$), b) P92 deformed by HPS with $\varepsilon_{eq} \sim 2.6$, c) 304L processed by HPT ($\varepsilon_{eq} \sim 23$).

3 Results and discussion

3.1 Microstructure processed by HPT

Figure 1 shows the microstructure of P92 steel processed by HPS and also the microstructures of P92 and 304L steels after one HPT revolution at RT situated about 7 mm from the centre of the HPT disc. The average size of grain in P92 steel in reduced to the value about 0.15 µm after imposed strain $\varepsilon_{eq}$ higher than 20. The microstructure of HPT-processed 304L steel is formed by grains with the mean size about 0.37 µm.
In the previous studies [1] was shown that the grain refinement by is meaningly affected by imposed strain value and also by processing temperature. It was reported [19] that the largest changes of microstructure characteristics take place during $\varepsilon_{eq}$ up to 10 and $\varepsilon_{eq}$ higher than 20 usually leads to the negligible changes of the mean grain size and number of HAGBs. The saturation of microstructure characteristics after $\varepsilon_{eq}$ higher than 20 was found in several materials. It means that the change in the number of HAGBs and the reduction of average grain size in steels after $\varepsilon_{eq}$ higher than 20 is insignificant. This implies that the saturation process occurring in the microstructure may be significantly affected by dynamic recovery or recrystallization, where boundaries act as sinks and/or sources of dislocations. The previous works [20] revealed that cold SPD may cause not only to the refinement of the microstructure but also phase transformations. This was recently observed.

**Figure 2.** Microstructures of HPT-processed steels after short-term annealing for 5 h: a) UFG P92 annealed at 873 K, b) UFG 304L steel after annealing at 923 K. Comparison of misorientation distributions for HPT state and its annealed counterpart c) UFG P92 and d) UFG 304L steel.
also in the HPT-processed 304L austenitic steel [9]. It was found that during HPT deformation occurs the subsequent transformation of FCC into BCC phase. After $\varepsilon_{eq}$ higher than about 20 the microstructure of 304L steel contains only BCC phase [9,21]. This result is valid for the SPD conditions (applied pressure and rotation speed) used in this study because the applied pressure and rotation speed have the influence on the microstructure changes [see 21-23].

3.2 Microstructure at the beginning of the creep testing
This microstructure at the beginning of the creep testing was simulated by annealing at the creep testing temperature for 5 h. This leads to the grain size similar to that in the structure at the beginning of testing. One can see (Fig. 2a) that the fine grains of short-term annealed UFG P92 steel can contain dislocations in their interiors. It means that the dislocations, generated during HPT, are not recovered during annealing at 873 K for 5h. The comparison of misorientation distributions demonstrates that the short-term annealing has neglect effect on the misorientation of boundaries (Fig. 2c). The HPT formed dislocation and LAGBs structure is partially preserved up to higher temperatures and can strengthens the microstructure by deformation hardening. It can be suggested that the influence of possible deformation hardening coming from HPT at room temperature on creep strength can be high at short-term creep test and low creep temperatures.

However, significant changes of misorientation distributions occurred in UFG 304L steel after annealing at 923 K/5 h (Fig. 2d). The results demonstrate the occurrence of low energy $\Sigma 3$ $(111/60^\circ)$ in the microstructure after short-term annealing. The significant changes of misorientation distribution in UFG 304L are related to the reversible transformation of BCC phase into austenite. The annealed microstructure of UFG 304L steel exhibited grain coarsening and the means grain size at the beginning of the creep testing was about 450 nm. In the microstructure of annealed UFG 304L steel was observed $\sigma$-phase precipitates (Fig. 2b). The enhanced precipitation of secondary phases in SPD-processed materials was found also in other works [8]. It was concluded that SPD supports the precipitation in UFG microstructure because dislocations act as places for nucleation of precipitates [24].

3.3 Microstructure after creep exposure
Figure 3 shows that grain growth occurs during creep exposure. The grain size was measured after the final fracture of specimens. The comparison of the grain size in the grip (unstressed) part and gauge length of UFG P92 steel tested at 873 K demonstrates that the grain coarsening can be influenced by stress and/or creep strain. One can see that mean grain size of UFG P92 steel is near UFG level even after creep testing at 50 MPa with creep time about 10 500 h. It can be suggested that good thermal stability of microstructure is supported by the presence of carbides and Laves phase precipitates [8] along grain boundaries which restrict their growth. Even if the grain coarsen during
creep testing of UFG P92 and 304L steel, the mean grain size of both steels is finer than predicted quasi-stationary subgrain size \( w_0 \) [5] for their coarse-grained counterparts. This result demonstrates that grains do not contain subgrains. Thus creep processes associated with boundaries, such as grain boundary sliding or enhanced recovery of dislocations, can significantly influence creep in HPT-processed steels.

4 Conclusions
Heat-resistant steels P92 and 304L were subjected to severe plastic deformation at room temperature. The microstructure changes after application of SPD, annealing and creep testing were investigated. It was observed that the short-term annealing of UFG P92 caused only minor changes in misorientation distribution and the dislocation formed during HPT at RT are not fully recovered. However, short-term annealing of UFG 304L led to the formation of \( \Sigma 3 (111/60^\circ) \) twin boundaries. It was further observed that creep exposure caused the growth of grains but the average grain size was finer than subgrain size predicted for coarse-grained materials.

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