Creep behaviour of ultrafine-grained CP titanium processed by multi-pass rolling

J. Dvorak 1,2, A.G. Kadomtsev 3, V.I. Betekhtin 3, V. Sklenicka 1,2, P. Kral 1,2 and M. Kvapilova 1,2

1 CEITEC – IPM, Institute of Physics of Materials, Czech Academy of Sciences, Zizkova 22, 616 62 Brno, Czech Republic

2 Institute of Physics of Materials, Czech Academy of Sciences, Zizkova 22, CZ-616 62 Brno, Czech Republic

3 Ioffe Physical-Technical Institute, Russian Academy of Sciences, Politechnicheskaya 26, 194 021 St.Petersburg, Russia

E-mail: dvorak@ipm.cz

Abstract. Two commercially pure (CP) Ti heats containing the same total impurity content have been used under investigation. Both materials were subjected to the processing procedure method of severe plastic deformation (SPD). This procedure consists of two steps of hot rolling (at 673 K) and the final rolling step at ambient temperature. The difference in individual chemical contents of impurities resulted in two states, both with ultrafine-grained (UFG) microstructures. For comparison reason, coarse-grained (CG) state was prepared by annealing of UFG state at temperature 823 K/1h. Microstructure investigations were performed using scanning electron microscope equipped with EBSD unit (SEM/EBSD) and transmission electron microscopy (TEM). Constant load tensile creep tests were conducted at temperature 673 K and applied uniaxial stress of 200MPa. Creep tests were run up to the final fracture of the creep specimens. The main objective of this work was to evaluate how the synergistic effect of SPD and the resulting microstructure affects the creep behaviour and properties of the studied materials.

1 Introduction

It is well known that heavy deformation, e.g. by rolling, extrusion, forging or drawing, can result in significant refinement of the microstructure of metals and alloys [1]. In recent years, severe plastic deformation (SPD) processing has been developed as a new method of manufacturing bulk specimens having ultrafine/nano-crystalline grained structures. Using the idea of this grain refinement effect, recently various bulk nano-structured materials processed by several methods of SPD were prepared [1-4]. It is known that the metals produced by these processes have very small average grain sizes of less than 1μm, with grain boundaries of mostly high-angle misorientation. The high-angle grain boundaries (HAGBs) in particular allow additional deformation mechanisms
to operate including grain-boundary sliding during low-temperature straining. So far, the most detailed investigations have focused on fcc metals such as Al, Cu and their alloys [3-6]. However, modern technological progress still requires light materials with special mechanical properties e.g. for application at elevated and high temperatures. Nowadays, titanium is an element which is frequently used in design of modern high temperature components. Increase of the fraction of HAGBs contributes to enhance some mechanical properties of UFG Ti. Microstructure refinement and formation of UFG structures in pure titanium result in considerable improvement in mechanical properties, such as the tensile strength limit [7], the compressive strength limit [8], and fatigue strength [9]. A new interesting example of grain boundaries (GB) engineering is investigation of mechanical properties of UFG Ti produced by combined SPD processing in the shape of long-length rods. In this work, we suggest this way of producing a stable submicrocrystalline structure: SPD by longitudinal rolling in combination with helical rolling under different conditions [10,11]. The main objective of this work was to evaluate how the combined way of SPD influences the creep properties and to investigate the effect of microstructure on creep behaviour.

2 Experimental Materials and Methods
Two commercially pure Ti heats (marked as State 1 and State 2) with the same total impurity content of 0.3 wt % (0.004C, 0.003N, 0.12Fe, 0.143O, 0.0008H, 0.001Al, 0.002Si) have been used in this study. The main distinction between the two states, as established by energy-dispersion X-ray spectroscopy, was that the content of carbon in State 1 was twice that in State 2. Both titanium heats were received in the form of the rods of 30 mm in diameter. The rods were processed under the same conditions consisting helical rolling at 673 K; lengthwise rolling at 673 K; and again helical rolling to final 8 mm diameter at ambient temperature [10,11]. The difference in chemical content of impurities resulted in two states both with UFG microstructures. To relieve internal stresses and improve the stability of the structure, titanium samples were annealed at 623 K for 3 h. For comparison, these two states were compared with CP Ti of conventional grain size. Coarse-grained state was obtained by annealing of UFG state at temperature 823 K for 1 hour. Creep specimens were cut from SPD processed and coarse-grained (CG) billets. The orientation of the tensile axis corresponded to the rolling direction. Flat tensile specimens had the gauge lengths of 25 mm and the cross-section of 3 mm². Constant tensile load creep tests were conducted at temperature 673 K under different levels of the applied uniaxial stress and under protection atmosphere of argon. Creep tests were run up to the final fracture of the creep specimens. For each level of the applied stress UFG specimens and CG specimen were tested. Microstructure investigations were performed using microscope Tescan Lyra 3 XMU FEG/SEM-FIB equipped with EBSD unit and transmission electron microscopy (TEM).

3 Results and discussion
3.1 Creep behaviour
Representative creep curves are shown in Fig. 1. All of these plots were obtained at an absolute temperature of 673 K (~ 0.35 Tₘ, Tₘ=melting temperature of the material) and at applied tensile stress of 200 MPa. Fig. 1a show standard ε vs. t curves for coarse-grained state and for the UFG materials. To identify individual stages of creep these standard strain ε versus time t creep curves can be easily replotted in the form of the instantaneous strain rate dε/dt versus time t (as shown in Fig. 1b) and/or in the form of the instantaneous strain rate dε/dt versus strain ε (Fig. 1c). As demonstrated by figures, there are differences in creep behaviour between the two states of materials. First, State 1 exhibits markedly better creep resistance in comparison with State 2. The creep life of State 1 specimen is seen to be longer than that of State 2 by a factor of more than 3. Second, comparing of UFG and CG states, UFG states exhibit better creep life than CG one.
Further, the minimum creep rate for the CG material is about one or more order of magnitude higher than that of the UFG counterparts. Finally, fracture plasticity (elongation) is not improved by applying SPD methods. Lower values of elongation measured for CG State 1 are probably caused by scatter of results. The creep curves are clearly different in appearance between the two states as illustrated in Fig. 1. Supposing that the creep rate \( \frac{\text{d} \varepsilon}{\text{d} t} \) at given stress is a certain measure of the “softness” of the microstructure, than the \( \frac{\text{d} \varepsilon}{\text{d} t} – \varepsilon \) plots (Fig. 1c) reveal the strain evolution of this “softness”. In UFG condition, hardening was observed at the beginning of creep exposure followed by a rapid softening. The secondary stage of creep is there very short. For State 1, gradual course of tertiary creep is finished by rapid accelerating of the last tertiary part of creep exposure shortly before fracture, while State 2 has steady softness progress. By contrast, in CG materials the primary creep stage is markedly longer followed by a distinct tertiary stage with gradually increasing creep rate.

![Figure 1. Creep curves for UFG and for CG Ti samples: (a) standard creep curves, (b) creep rate vs. time, (c) creep rate vs. strain.](image)

### 3.2 Microstructure before creep testing

Two types of microstructure were achieved by application of three steps of SPD rolling operation. Due to the effect of SPD deformation the grain size is reduced to nanometer range for both states of material. The EBSD results showed inhomogeneous microstructure with a small submicron elongated grains along the rolling direction for the State 1 (Fig. 2a). The mean grain size was measured about 0.2 µm. State 2 is represented by a similar microstructure (Fig. 2b). The mean grain size was measured as \( \sim 0.25 \) µm. Further, EBSD measurements were taken to determine the grain boundary misorientations. The mean value of the relative fraction of high-angle (\( \theta > 15^\circ \)) grain boundaries (HAGB) was estimated to be \( \sim 64\% \). TEM figures revealed high dislocation density in the interior of grains created by large plastic deformation imposed into material predominantly by the last step of rolling operation at room temperature (Fig. 2d).

The microstructure of CP titanium in the coarse grain (CG) state is shown in Fig. 2c. This state was obtained from UFG states by annealing at temperature 823 K for one hour. It contains a relatively homogenous microstructure with a presence of large bands of equiaxed grains. This annealing lead to coarsening of grains and the mean grain size measured by EBSD analysis was about 1.2 µm with presence about 50% of HAGB. The annealing process led to complete recovery of dislocations formed during last rolling process at room temperature.
3.3 Microstructure after creep testing

After creep exposures at 673 K and 200 MPa the microstructures of State 1 and 2 contain clusters of grains with nearly the same orientation. In microstructure are still visible bands of elongated grains. Creep loading also leads to coarsening of grain size up to 1.75 µm and 1.45µm for State 1 and State 2 (Fig. 3a), respectively. The microstructure of CG state after subsequent creep exposure show a change in the mean grain size to 2 µm with occurrence mixed small and large predominantly equiaxed grains (Fig. 3b). In compare with as-annealed state, bands of elongated grains was not observe. Finally, no expressive change has been observed on fracture surface using SEM fractography. In all states typical mix mode of transgranular and intergranular fracture was observed (Fig. 4).
Discussion

Since both titanium States 1 and 2 were fabricated under the same SPD conditions creating almost the same structure, the proved difference between their the high temperature static and long-term characteristics is thought to be caused by their defect substructure, nanoporosity, and the presence of nonmetallic disperse carbide type inclusions. Higher resistance observed for State 1 can be attributed to increased content of carbon which create titanium carbide disperse particles [12,13] and thus can be responsible for more effective particle hardening. In connection with this effect, during static tests and tension under high temperature creep conditions, the expected negative effect of nanoporosity is likely to be effectively compensated just by the hardening induced by disperse particle (carbides). The presence of such inclusions could explain the high static strength and long-term strength during creep of titanium in State 1.

The results also show that creep life at 673 K is increased in microstructure with an occurrence of large elongation grains. Thus, the attained microstructure is created by large degree of straining during the last cold steps of SPD process. When the frequency of elongated grains in microstructure is increased, the total area of grain boundaries in a constant volume is decreased and
this can enhance the creep resistance due to reducing of grain-boundary mediated deformation processes activities. Static annealing of CG state at 823 K /1h does not lead to expressive differences in structure of UFG states. Generally, higher creep resistance of UFG states can be also explained by higher dislocation density. During heating and soaking on creep temperature (673 K), imposed dislocations probably are not fully recovered and thus dislocations could strengthen materials by deformation hardening. By contrast, high annealing temperature (823 K) of CG state can be associated with completely recovery of dislocations. Operating deformation mechanism is probably not qualitatively different for all three states of materials under investigation as evidenced by similar values of fracture elongation. The high values of elongation suggest that the fracture proceeds due to the loss of plastic stability matrix of the material (local necking of the specimen section) and the resulting final fracture is mixed intergranular and transgranular mode.

4 Conclusions

Commercial Ti were processed by method of SPD by means of two rolling hot steps and one step at room temperature. The microstructure changes after application of SPD, annealing and creep testing were investigated. It was observed that short-term annealing of UFG state led to the coarsening of grain size and fully recovery of dislocation situated in the interior of grains. It led to degradation of creep resistance. The higher content of carbon in State 1 and potential formation of highly dispersed carbide particles was found to enhance the thermal stability of SPD titanium.

Acknowledgements

The authors acknowledge financial support from the Czech Science Foundation (grant No. 20-14450J), the joint project running within Russian and Czech Academies of Sciences. This research was carried out under the project CEITEC 2020 (LQ1601) with financial support from the Ministry of Education, Youth and Sports of the Czech Republic under the National Sustainability Programme II.

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