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Coarse-grained and ultrafine-grained titanium high-temperature creep

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Abstract. The publication describes the study of durability in tensile creep of VT1-0 commercial titanium in its two states - coarse-grained and ultrafine-grained. It is shown that the best temperature for log-term testing is 350^0C. At this temperature, the ultrafine-grained titanium structure remains stable both during free annealing and durability testing. The obtained data enable retrieving the difference in fracture initiation energies for titanium in its coarse-grained and ultrafine-grained states.

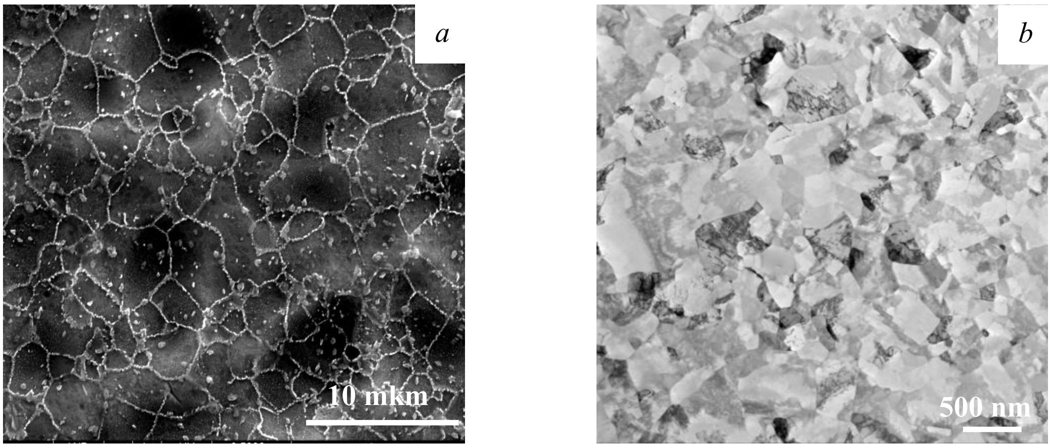
1. Introduction

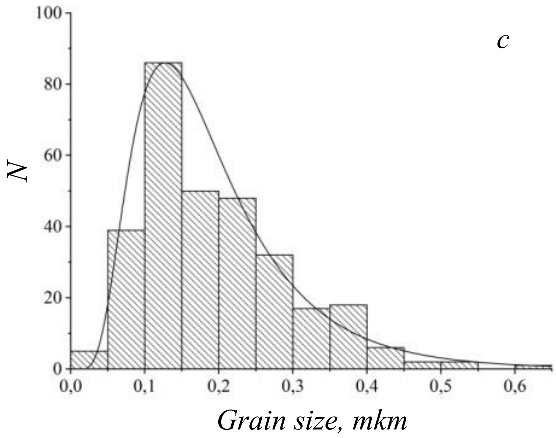
Severe plastic deformation (SPD) methods yield unparalleled mechanical properties of materials by generating an ultrafine-grained (UFG) structure which imparts high mechanical performance to such materials. Stabilizing such nanocrystal or microcrystal structure under long-term thermal and mechanical impact (creep, fatigue) is the critical theoretical and practical challenge for modern materials science. Creep resistance is strongly associated with the microstructure and thermal stability of UFG materials during creep. Thus, the study of creep behavior of UFG microstructure is important to define strain control mechanisms.

It is known that starting from certain temperatures UFG metal grains begin to coarsen causing changes in mechanical performance of the metal [1]. We ventured to run high-temperature creep tests of titanium in such temperature and strength range where the grain re-crystallization process may be ignored.

2. Materials and methods

Test samples were made of Ø 8 mm VT1-0 alloy bars produced by longitudinal and transverse rolling [2] and subjected to 3 hours of finish annealing at 673 K to relieve first-order internal stress. UFG grain size was equal to (193±11) nm, and that of re-crystallized coarse-grained (CG) material was 2.35 µm. Figure 1 shows the CG (figure 1, a) and UFG (figure 1,b) microstructure states of VT1-0 alloy, as well as grain-size distribution bar chart for UFG state of alloy (figure 1, c).





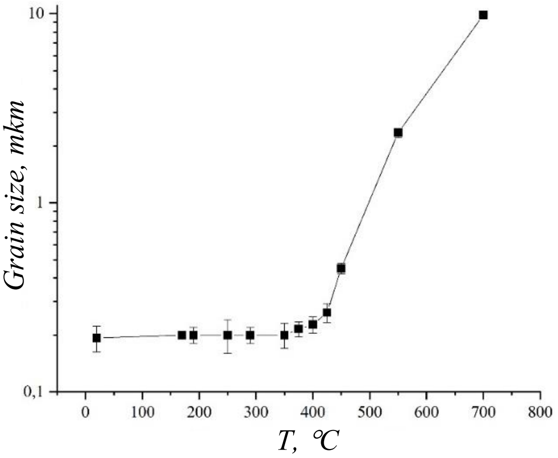


Figure 1. CG microstructure of VT1-0 alloy (a - scanning electron microscopy), UFG microstructure (b - transmission scanning electron microscopy) and histogram of grain size distribution for the UFG state (c).

Figure 2. Annealing temperature influence on the average grain size of VT1-0 UFG titanium

3. Results

To identify test temperature states for VT1-0 titanium, additional detailed study of the structure stability was made for material heated to $150...700^0$C°, its exposition ranging from 1 to 1000 hours. Figure 2 shows the dependence of average grain size on annealing temperature for VT1-0 (UFG). The phase composition of UFG state is represented by a single phase - α titanium alloy. UFG VT1-0 tends to decrease the level of microstress with higher annealing temperatures and longer exposure. The material manifests a slightly defined texture by preferred orientation of its grains in the (100) plain in its initial as-rolled and as-annealed state after 3 hour exposition at 350°C. A slight decrease in internal stress level observed during X-ray diffraction analysis is apparently associated with decreased local microstress at grain boundaries. The latter is detected by the change in grain boundary contrast after long-term annealing.

Tensile creep durability testing time varied from several seconds to several days. Figure 3 shows typical CG and UFG creep curves at 350^0C. They are similar in quality, strain-to-fracture for CG titanium is lower, and deformation increases slightly with the decreasing stress applied for both types of

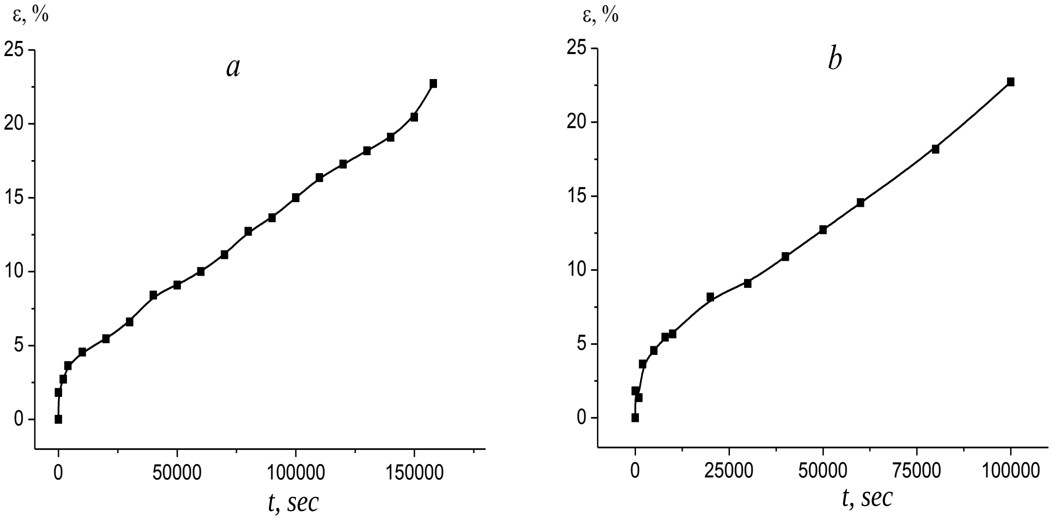


Figure 3. VT1-0 creep curves: (a) – CG state, 350^{0}C, σ=209 MPa;
(b) – UFG state, 350^{0}C, σ=279 MPa.

titanium. The obtained data analysis for both its strength dependence (figure 4) and creep rate (figure 3) let to assess both fracture and creep initiation energies. Within the accuracy margin these values are close to each other yielding ≈80 kcal/mol for CG state and ≈60 kcal/mol for UFG state, respectively. It is essential that titanium durability in UFG state is higher than that in CG state across the full test stress range. Note that this difference depends on the test stress value - the difference decreases with the stress decrease. This is likely to happen due to a lower fracture initiation energy of UFG titanium.

Surely, the cause for such difference in fracture initiation energy of CG and UFG titanium calls for a more detailed study. This publication is probably the first to reveal the difference in titanium fracture initiation energies in the temperature range where virtually recrystallization doesn't take place. However, there are multiple data [3,4] that indicate that the parameters of diffusion initiation in UFG materials are lower than those in CG materials. This is quite reasonable to expect, since UFG materials feature grain boundaries extending an order of magnitude length while the state of these boundaries is highly unbalanced itself. Thus, the obtained results prove that SPD treatment at selected temperature when there is virtually no re-crystallization results in low fracture initiation energy of UFG titanium,

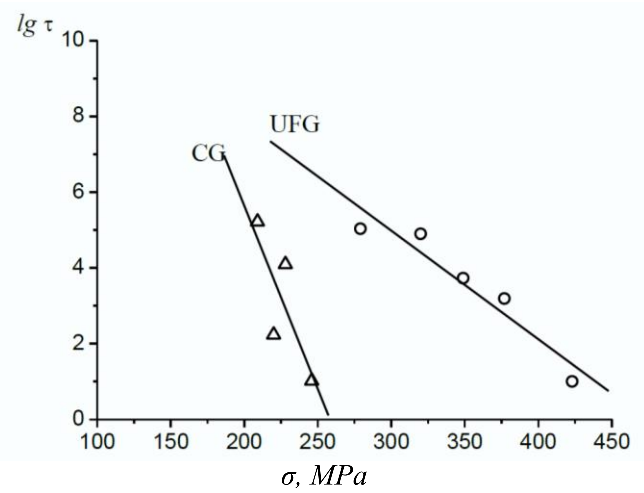


Figure 4. lg τ vs. stress dependence for tensile creep test of CG and UFG titanium at 350^{0}C

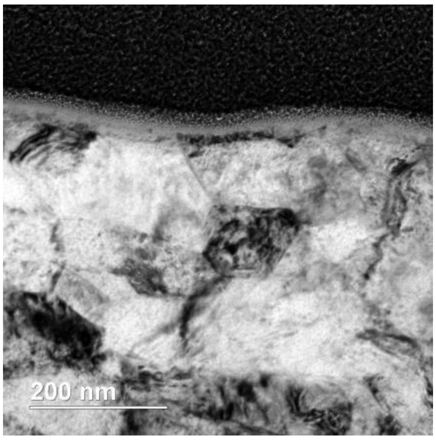


Figure 5. Ti UFG microstructure after creep test (350^{0}C, 30 hours).

while its mechanical performance is higher than that of CG titanium. Figure 5 shows VT1-0 sample microstructure in UFG state after its creep test (350^{0}C, 30 hours) to prove that the grain size remains virtually unchanged after the test.

It was found that after the creep test of initially recrystallized VT1-0 CG titanium at 350°C (44 hours), grain structure is refined to sub-microcrystalline range (grain size diminishing to 100-200 nm from its initial size of 2.35 µm) near the fracture surface. The grain shape in those ranges is close to globular. At distances exceeding 3 µm from the fracture surface, the grain shape becomes elongated.

In ultrafine-grained state, a similar, but less pronounced change takes place. At 2 µm from the fracture surface, grain morphology is changed from globular to platelet as in recrystallized coarse-grained state. Bend extinction contours indicate that internal stresses take place in the material crystal lattice.

Fracture behavior analysis of CG and UFG samples has shown that they are similar and correspond to a viscous pitted profile on the fracture surface (figure 6). Pitting and elongation zone (areas at adjacent pit boundaries) distribution pattern for UFG is more homogeneous as compared to the coarse-grained sample. The difference is that there are lower size structural elements and the number of bridges between pits is increased, which indicates the higher ductile properties of the sample in creep test states. Nanostructurizing near the fracture surface, shares of micro- and nanopores and fractures in their total amount in the fracture area (1%) are common for CG and UFG titanium. This amount is obviously the main parameter preceding the transfer to macrofracture.

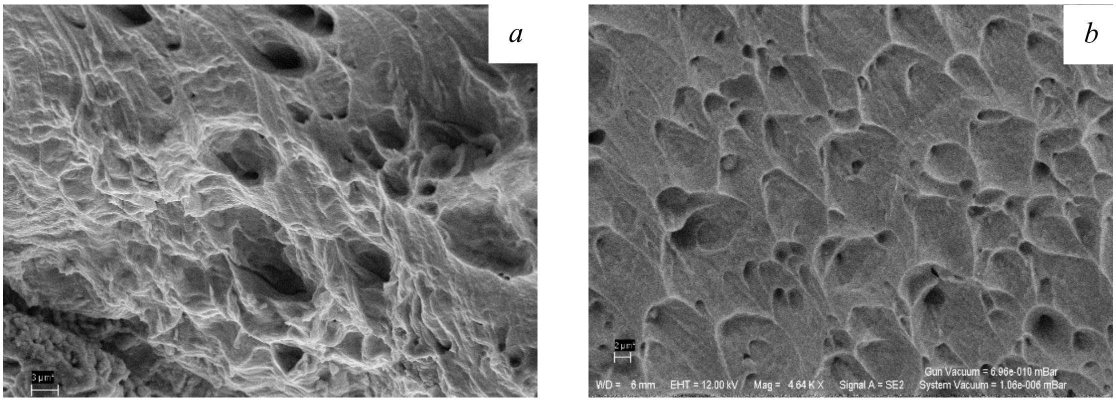


Figure 6. VT1-0 sample rupture fractography in CG (a) and UFG (b) states after creep test at 350^{0}C (30 hours). Electron beam normal to fracture surface. Scanning electron microscopy.

4. Conclusion

The analysis of findings obtained during the study also shows that the severe plastic deformation method [2] used by the authors results in the formation of UFG structure which ensures high strength and durability as compared to recrystallized CG state. Figure 3 shows that, after the creep test at 350^{0}C, CG titanium durability is comparable with that of UFG titanium tested at significantly higher stress (load).

In view of this, it should be noted that, as shown by the authors earlier, the durability of UFG titanium obtained using other severe plastic deformation method (equal channel angular pressing at 400^{0}C) and creep tested at 400^0 C is decreased as compared to CG state [5]. This result was attributed to several factors. In particular, to sharp increase in grain size due to re-crystallization during creep and to increase in nanopore size within new grain boundaries [6]. It is essential that for correct assessment of influence of various severe plastic deformation methods on long-term creep durability of titanium, UFG titanium should be tested under similar conditions (temperature, stress).

Acknowledgments

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