

Characteristics of creep of fine-grained zirconium

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Creep studies in the temperature range 300 – 700 K have shown that the deviation from depending the Hall-Petch relationship for a nano-structured zirconium is associated with the decrease of the contribution of the mechanisms of intragranular slip in the plastic deformation and increasing of the contribution of the mechanisms more characteristic of high-temperature creep.

All studied materials have instability of the structural state after creep deformation in the all temperature range. That is a result of the change in the geometry of the applied stress and low strain rate and significantly influences on the creep characteristics.

Keywords: creep, intense plastic deformation, structural instability.

Проведені в області температур 300 - 700 К дослідження повзучості показали, що відхилення від залежності Холла-Петча для наноструктурованого цирконію пов'язано зі зменшенням вкладу в пластичну деформацію механізмів внутрізеренного ковзання і зростанням вкладу механізмів повернення, більш характерних для високотемпературної повзучості.

Всі досліджені структури цирконію у всій дослідженій області температур виявляються нестійкими при зміні параметрів деформації, що є результатом зміни геометрії прикладених напружень і температурно-швидкісних умов деформації і суттєво впливає на характеристики повзучості.

Ключові слова: повзучість, інтенсивні пластичні деформації, структурна нестійкість

Проведенные в области температур 300 - 700 К исследования ползучести показали, что отклонение от зависимости Холла-Петча для наноструктурированного циркония связано с уменьшением вклада в пластическую деформацию механизмов внутрзеренного скольжения и ростом вклада механизмов возврата, более характерных для высокотемпературной ползучести.

Показано, что все изученные структурные состояния циркония во всей исследованной области температур оказываются неустойчивыми в условиях ползучести, что является результатом изменения геометрии приложенных напряжений и температурно-скоростных условий деформирования, и существенно влияет на характеристики материала.

Ключевые слова: ползучесть, интенсивные пластические деформации, структурная неустойчивость

Introduction

The interest in materials with ultra fine grain is conditioned by their unique mechanical, physical and chemical properties differ significantly from the corresponding polycrystalline samples with small and large grains.

At present, various methods intense plastic deformation (IPD) are developed and used: the equal channels angular pressing, rolling, screw under high pressure, pressing, and the methods of combining the IPD with the doping hydrogen [1-5]. One of the most perspective methods of obtainment of nanocrystalline metallic materials is based on the use of IPD. However, large deformed materials are characterized by high internal stresses and low plasticity at cold deformation, which don't improves their processability. Effectiveness of heat treatment in terms of stress relaxation increases with increasing temperature. Simultaneously with increasing of annealing temperature the probability of

substantial grain growing increases, what can lead to loss of the nanocrystalline advantage, such as high strength. Thus, the effective use of large plastic deformations, i.e. selection of method and the optimum modes of processing for this material is not possible without a systematic analysis of the regularities of formation of nano- and microcrystalline structures after large plastic deformations, as well as studies of the mechanisms of evolution of the structure of highly deformed materials by heating and subsequent deformation.

Because the systematic study of polycrystalline ultra fine grain began relatively recently (the 80 years of the last century), so far is no generally accepted terminology in this field. One classification of polycrystals grain size is shown in Table 1 [6] and we will stick to it:

It should be noted, that specificity of nanomaterials according to theory of defects, is that the main processes controlling their behavior and properties, are not realized in

the crystal lattice (in grains), as in traditional materials, and in the grain boundaries [1-9]. And the main type of defects, which determine the nature of these processes are not the dislocations and vacancies (both in traditional materials), but the internal boundaries. The peculiarities of grain-

Table 1.

The classification of crystal grain size

The scale level	The type of polycrystals	The grain size
Mezo	coarse grained ordinary fine-grained	0,1-10 mm 10-100 μm ; 1-10 μm
Micro	ultrafinegrain submicrograin nanocrystals	0,2–1 μm ; 100-200 nm 3–100 nm

boundary flow processes are caused by the interaction of grain boundaries with the dislocations and point defects, which adjudged to them from the lattice. Moreover, qualitative and quantitative difference of the properties of these materials is determined not only by the size of elements of grain structure but also by the nonequilibrium state of grain boundaries formed in the process of IPD. Therefore, can change the strength and creep rate of nanocrystalline materials by changing the structure of the boundaries.

Besides the perspective of industrial using and scientific interest be associated with the idea that in the case of the small grain size the main mechanisms of plastic deformation of metal materials in creep at low and middle temperatures ($T < 0,4T_{pl}$) can be the high-temperatures deformation mechanisms. This determines the importance of studying of the creep characteristics of nanostructured metallic materials obtained by the methods of IPD. Moreover an important issue is the stability of obtained structural states in a wide temperature range. It is known that the nature of the external action determines the resistant type of the structural state in relation to it. Changing the geometry of plastic deformation is accompanied by structural restructuring so as to cause the maximum intensity of the internal stress relaxation in line with the desire to maintain continuity of the crystal [2]. The newly formed structure as a result of alteration also can be fragmented, but steady in relation to the new scheme of the elastic-stress condition and the temperature-rate mode of deformation.

The purpose of this paper is to analyze the characteristics of creep and structural evolution, and the influence of state of the grain boundaries on the development of plastic deformation of zirconium with different grain size, obtained by the combined processing by rolling and subsequent annealing.

Material and experimental procedure

The investigated material was the polycrystalline Zr with grain size 80 nm – 5 μm ., obtained by electron beam melting and deformed by rolling at 100 K with next annealing.

For the study of defect structure of materials we used the method of measuring of electrical resistance after each treatment and in the process of creep. Electrical resistance was measured at $T=300$ K for 4th point scheme by a compensative method with the use of potentiometer of P- 363. The measuring error did not exceed +0,05 %, and variation of values of specific electrical resistance did not exceed $\pm 0,5$ %. Monitoring of the structure evolution was carried out by electron microscopy.

Creep tests were carried out in the step loading regime at 300-700 K, the measurement accuracy was $5 \cdot 10^{-5}$ cm. The activation parameters were determined using the differential methods described in [10].

Results and discussion

Based on the analysis of experimental data and theoretical estimates is established, that in all investigated temperature range there is a big creep rate for all samples and the dependence of the deformation value from test time is described by a power law ($\epsilon \sim t^{1/3}$), which may be the result of the simultaneous action of the hardening and recovery [11-13], i.e. plastic flow is caused by the combined action of several mechanisms.

The dependence of the creep rate of zirconium with grain size 80 nm – 5 μm ; from the applied stress are shown in Figure 1.

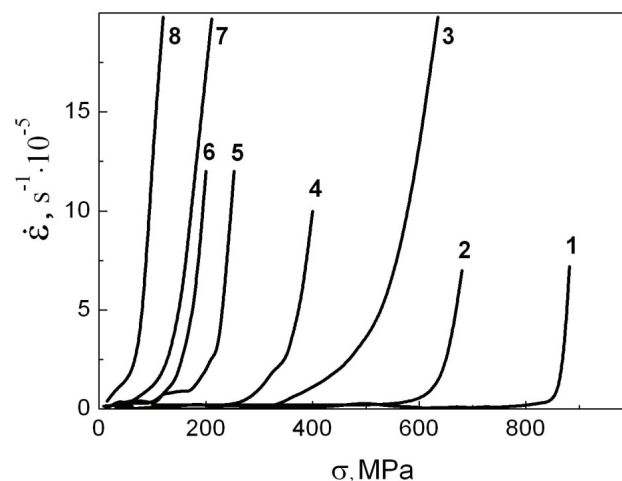


Fig.1. Dependence of creep rate of zirconium with different grain size at 300 K(1,2,3,4) and 700 K(5,6,7,8) from the applied stress: 4,8 – 5 μm ; 3,7 – 1 μm ; 2,6 – 300 nm, 1,5 – 80 nm.

Strength characteristics of zirconium (yield strength $\sigma_{0,2}$ and tensile strength σ_b) increases with decreasing grain size up to $d = 300$ nm, that is the evidence of fulfillment the Hall-Petch law. Further the grain refinement to $d = 80$ nm

leads to the sharp increase in strength characteristics, particularly at 300 K, and it is connected with the dependence of Hall-Petch coefficient from the grain size [6-10]. At the same time sharply reduced the plasticity of nanostructured zirconium, which is due to the high level of long-range stresses on the nonequilibrium boundaries grain.

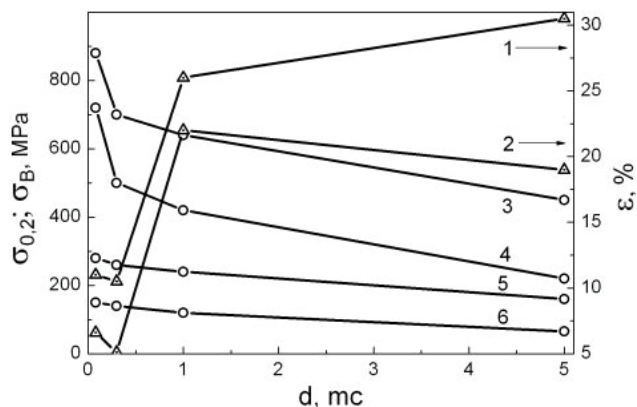


Fig. 2. Dependence of creep characteristic of zirconium from grain size at 300 K (1,3,5) and 700 K (2,4,6): 1,2 – plasticity (ϵ); 3,4 - tensile strength (σ_B); 5,6 - the yield strength ($\sigma_{0.2}$).

Now, let us analyze the data obtained. The basic mechanisms of plastic deformation of the fine crystalline and ultrafine zirconium (1-5 microns) during the creep in the temperature range 300 – 700 K [11-14] are: the cross-slip, climb and annihilation of dislocations at the grain boundaries, diffusion creep and grain-boundary slip and the basic mechanism of accommodation of grain boundary slip - intragranular dislocation slip, controlled by dislocations crossed of the point defects and dislocations of the forest. All these processes lead to micro localization of deformation and stress relaxation. The contribution of

each of mechanisms in the material deformation depends on the test temperature, applied stress and the state of the grain boundaries. The contribution of the intragranular slip, as accommodative mechanism, to the development of the creep of zirconium with a grain size of 300 nm is very small, and for nanostructured zirconium - is practically absent, especially at $T = 300$ K. Thus, at $T = 300$ K, there is a sharp increase in strength characteristics.

Microstructure of ultrafine zirconium having an average grain size of 1 μm ; (density of dislocations in the grains $\sim 10^8 \text{ cm}^{-2}$) shown in Fig.3a. During creep at 300 K in this samples is formed the cellular structure with the size $\sim 0.1 - 0.3 \text{ mm}$, extending along the direction of the tensile load, the cell walls are friable. It should be noted that the essential role as an accommodative mechanism carried out by the intragranular slip. This leads to the formation of the cellular structure, and as consequently, to some increase of the electrical resistivity. During creep at 700 K (fig.3b) is formed highly defective state characterized by a large number of boundaries and high dislocation density, that are the result of translational and rotary deformation modes. Micro volumes are unfolding completely, forming fragments, adjusting to the direction of strain. There are the large numbers of long high angle boundaries, while the disorientations between the fragments are small.

Structural studies of the submicrograined and nanostructured zirconium have shown that the formed structure is sufficiently homogeneous by volume. The dislocation density in the body of grains is $\sim 3.4 \cdot 10^{10} \text{ cm}^{-2}$. The main part of dislocations is concentrated at the grain boundaries and triple junctions (fig.3c).

The plastic deformation of the submicrograin and nanostructured zirconium is due to the reorganization of the defect structure and is accompanied by stress relaxation. This process includes the destruction of the initial

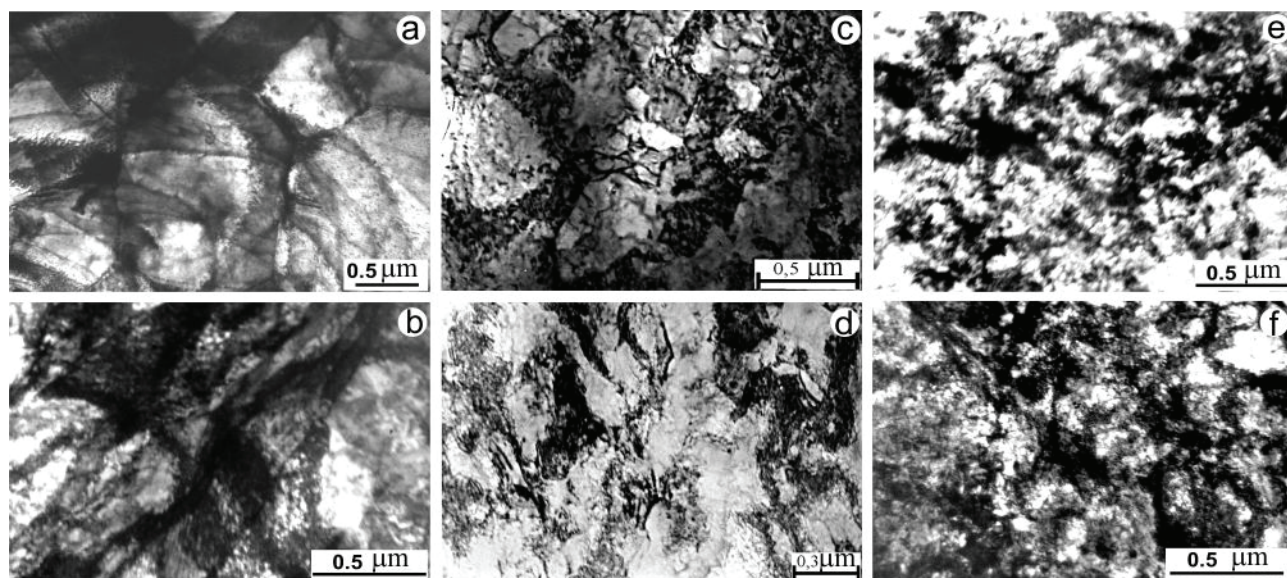


Fig. 3. TEM images of zirconium with different grain size: a) $d=1 \mu\text{m}$; b) $d=1 \mu\text{m}$ + creep at 700 K; c) $d=300 \text{ nm}$; d) $d=300 \text{ nm}$ + creep at 300 K; e) $d=100 \text{ nm}$; f) $d=100 \text{ nm}$ + creep at 700 K.

structural configuration, created as a result of deformation by rolling, and the formation of a new structure, which also is fragmented, but less tense and steadier to subsequent deformation.

At the first, being in the other mode of stress- and rate deformation, being unable to change during plastic deformation, the initial boundaries are destroyed, trying to adjust the orientation of micro volumes to the direction of tension. After that, developing a uniformly oriented crystal, plastic deformation creates new boundaries of disorientation, but already steady in relation to new geometry and rate of deformation influence. And to the end of uniform elongation the fragmented structure with the grain size $d \sim 0.5 \mu\text{m}$ is formed; and the high level of internal stresses is observed. The high density of dislocations in clusters is observed. On the boundaries of fragments are the powerful high angle boundaries, which can later open a crack (fig.3d).

During creep at 700 K the initial structure experiences a number of transformations (fig.3f). At the beginning the recrystallization is occurring. In the future the new grains are destructed and formed the equilibrium distribution of dislocations with a homogeneous density of $N_d \sim 5 \cdot 10^{10} \text{ cm}^{-2}$, and in their place a cellular structure with disorientations of $\sim 3\text{-}7$ degrees is formed. This results in a sufficiently equilibrium structure with uniform distribution micro stress. Recovery processes are due to the presence in the material the high level of internal stresses, so that the accumulate energy is sufficient to accelerate the kinetics of dynamic recrystallization [11-14].

Studies of zirconium submicrocrystalline and nanostructure (80 – 300 nm) obtained by IPD have shown that plastic deformation and relaxation of stress during creep can occur due to the destruction of the original structural configuration by the intense deformation by rolling, and forming a new structure, a less stressful and more stable with respect to a given temperature and high-speed mode and the geometry of loading. In the temperature range 300 - 700 K deformation of zirconium with a grain size of 80 – 300 nm is due to the combined action of several mechanisms is more characteristic of high-temperature creep: climb of dislocations, grain boundary diffusion, grain boundary sliding, and the intragranular slipping virtually nonexistent. The contribution of each of them depends on the test temperature, applied voltage and the boundary structure.

As noted above, metallic materials with submicrocrystalline and nano - structures obtained by severe plastic deformation, have non-equilibrium grain boundaries. Fields stress non-equilibrium boundaries are inhomogeneous and depend strongly on the orientation of the facet, ie changed within a single grain. This makes the non-equilibrium boundaries of the source of strong overlapping long-range stress fields, which made difficult

the accommodative slip inside grains and leads to strain localization at the borders. As a result are increasing the deforming stresses required for plastic deformation of the material. At the same time the contribution of the recovery process, which determined by the dislocations climbing and by absorption of dislocations at the boundaries, as well as by development the grain-boundaries slip, rises with increasing of stresses, which explains the big creep rate. A small value of the activation volume $\sim 10^{-22} \text{ cm}^3$ also indicates an increase in strain localization.

Conclusions

1. Studies of creep in the temperature range 300 – 700 K have shown that the deviation from Hall-Petch law for the nanostructured zirconium is associated with the decreasing of the contribution of the intracrystal slip mechanisms in the plastic deformation and with increasing of the contribution of the mechanisms characteristic more for high-temperature creep.

2. So, it should be noted, that all studied materials have instability of the structural state after creep deformation. That is a result of the change in the geometry of the applied stress and low strain rate and significantly influences on the creep characteristics.

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