UDC 669.296: 539.377

ULTRASOUND INFLUENCE ON THE CREEP NANOSTRUCTURED Zr

V.I. Sokolenko, V.M. Gorbatenko, E.V. Karaseva, A.V. Mats, E.S. Savchuk, V.A. Frolov
National Science Center “Kharkov Institute of Physics and Technology”,
Kharkov, Ukraine
E-mail: vsokol@kipt.kharkov.ua

The peculiarities creep and electro-physical characteristics of nanostructured zirconium subjected to severe treatment was studied in the temperature range 300…700 K. It is shown that plastic flow of nanostructured zirconium is passed by rearrangement of the defect structure and is accompanied by stress relaxation. Ultrasonic treatment reduces the stress level, but do not lead to a noticeable change in the size of the nanostructure in contrast to the annealing.

INTRODUCTION

One of the most perspective methods of obtainment nanocrystalline metallic materials is based on the use of intense plastic deformation (IPD) [1–5]. However, large deformed materials are characterized by high internal stresses and low plasticity at cold deformation, which improves thear processability. Effectiveness of heat treatment in terms of stress relaxation increases with increasing temperature. Simultaneously with increasing annealing temperature increases the probability of substantial grain growing, what can lead to loss of nanocrystalline state advantage, such as high strength.

It is also known, that as a result of low-intensity ultrasonic treatment (UST) on nanostructures deformation origin, observed the level reduction and alignment of the spectrum of internal stresses in the bulk material while maintaining the morphology nanostructure and increasing structure homogeneity [6, 7].

However, if the mechanisms of microstructure evolution during hot deformation are well studied, the laws of the formation and evolution of nanostructures at low-intensity ultrasonic treatment are not clearly established. 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 < 0.4 Tpl) can be the high-temperatures deformation mechanisms. This determines the importance of studying the creep characteristics of nanostructures metallic materials obtained by the methods of IPD. An important issue is the stability of obtained structural states in a wide temperature range, because strength and plastic characteristics of the material may observably change in the case of an irregular structure and structural instability [1–7].

The purpose of this paper is to study the laws of creep and evolution nanostructure of zirconium obtained with the use of rolling and subsequent annealing and ultrasonic treatment.

MATERIAL AND EXPERIMENTAL PROCEDURE

The investigated material was the polycrystalline Zr, obtained by electron beam melting and deformed by rolling at 100 K. With the purpose of maximally effects on structure and properties were used next modes of influence:

1) MT-1 – rolling at 100 K, the residual deformation (ε) was 3.9;

2) MT-2 – rolling at 100 K + UST at 300 K;

3) MTT-1 – rolling at 100 K + annealing at 750 K during of 1 h.

For the study of defect structure of materials 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. A measuring error did not exceed + 0.05%, and variation of values of specific electrical resistance did not exceed ± 0.5%. Monitoring 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 [7].

Part of rolled samples was subjected to UST (f = 20 kHz) at T = 300 K using the method described in [6]. The amplitude of the ultrasonic treatment was 70 MPa, duration of exposition – 10 min. The preliminary ultrasound treatment, as previously shown, produces softening effect on the deformed material [6, 8].

RESULTS AND DISCUSSION

After rolling and different treatments the values of the residual electrical resistivity (ρ) of zirconium were determined and was showed, that both treatments of rolled samples (MT-2 and MTT-1) reduces the level of stress. The results are given in Table.

Zirconium residual electrical resistivity

Measurement characteristics	Modes of treatment		
	MT-1	MT-2	MTT-1
$\rho_{300\,K}/\rho_{77\,K}$	3.63	4.0	4.55

On Fig.1 the creep rate of the samples after all MTT in all investigational intervals of stresses and corresponding change of specific electrical resistance at 300 K are present.

As can be seen (see Fig. 1,a) the samples of rolling (ε ≈ 3.9) zirconium tested at 300 K have high strength at low plasticity and creep rate. At the same time the specific electrical resistance, measured at 300 K (see Fig.1,b), decreased during creep after all types of treatments, indicating that the stresses decreased. Studying the creep process we have calculated, by the formularies of the thermofluctuational plastic deformation theory, the effective activation volume V_{ef}. The calculated value of V_{ef} is ~ 10^{-22}cm^3, which indicating that plastic flow is caused by moving of point defects and has the high degree localization.

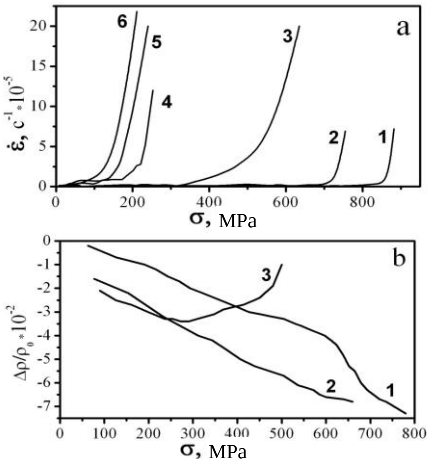

Fig. 1. Dependence of creep rate nanostructured zirconium at 300 and 700 K (a) and specific electrical resistance at 300 K from the applied stress (b): 1 – MT-1; 2 – MT-2; 3 – MTT-1

Numerous steps on the curve ε(σ) at 700 K after MT-1 indicate the restructuring during the creep and only at stresses close to the ultimate strength the deformation can occur by means of dislocation slip on the boundaries of the fragments.

In [10–12], we have shown that the plastic flow in this case is the result of simultaneous action of the hardening and recovery, i. e. plastic flow is caused by the combined action of several mechanisms: cross-slip, climb and annihilation of dislocations at the grain boundaries, diffusion creep and grain-boundary slip. All these processes lead to microlocalization 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.

Structural studies have shown that after rolling to 3.9 at 100 K the nanostructure is formed with the grain size ~ 80 nm, sufficiently homogeneous by volume. The dislocation density in the body of grains is ~ $3.4\cdot10^{10}$ cm^{-2}. The main part of dislocations is concentrated at the grain boundaries and triple junctions.

During creep at 300 K the initial nanostructure is destroyed and to the end of uniform elongation is formed the fragmented structure with the grain size d ~ 0.5 mc and the high level of internal stresses. It is noted the high density of dislocations in clusters. On the boundaries of fragments are the powerful high angle boundaries, which can later open a crack.

During creep at 700 K the initial structure experiences a number of transformations (Fig. 2). 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 $N_d \sim 5\cdot10^{10}$ cm^{-2}, and in their place a cellular structure with disorientations of ~ 3…7 degrees is formed. This is results in a sufficiently equilibrium structure with uniform distribution micro stresses. 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 [13–15].

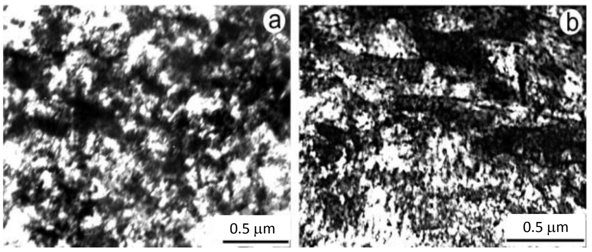

Fig. 2. TEM images of zirconium after follows influences: a – MT-1; b – MT-1 + creep at 700 K

In order to reduce the internal stress after rolling (MT-1) the samples were annealed at the temperature corresponding to the first recrystallization stage (MTT-1). The tensile strength of annealed samples decreased by 30% at 300 and at 700 K compared to rolling samples. At same time increased plasticity at 15% and the creep rate.

Fig. 3,a shows the structure of zirconium after rolling and annealing at 750 K 1 h which corresponds to the first stage recrystallization. The grain size increased to 0.6…1.5 mc (average grain size ~ 1 mc), which indicates the transition of zirconium from nanostructures to fine-grained condition. The grains have an equiaxed shape, at the same time the intragrain structure and the structure of the grain boundaries are changed. The dislocation density less than 10^8 cm^{-2}.

During creep at 300 K of annealing samples is formed the cellular structure with the size ~ 0.1…0.3 mm, extending along the direction of the tensile load, the cell walls are friable. Plastic deformation caused by the action of several mechanisms [10–12], moreover 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 (see Fig. 3,b) 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. Microvolumes are unfolding completely, forming fragments, adjusting to the direction of strain. There is the large number of long high angle boundaries, while the disorientations between the fragments are small.

Thus, due to annealing, the coarsening of the grains is occurring, resulting in reduction of strength of zirconium. In this connection, it is appropriate to carry out such processing, for example, low-intensity ultrasonic treatment, when the changes of the internal stress do not associate with the variation of structural parameters.

It should be noted, that specificity nanomaterials according to theory of defects, is that the main processes controlling their behavior and properties, do not realized in the crystal lattice (in grains), as in traditional materials, and in the grain boundaries [9–15].

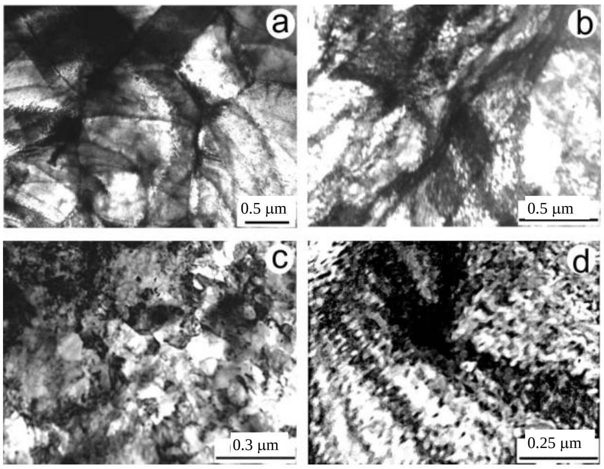

Fig. 3. TEM images of nanostructured zirconium after follows influences: a – MTT-1; b – MTT-1 + creep at 700 K; c – MT-2; d – MT-2 + creep at 700 K

And the main type of defects, which determined the nature of these processes are not the dislocations and vacancies (both in traditional materials), but the internal boundaries. The peculiarities flow of grain-boundary 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 grain structure but also by the nonequilibrium state of grain boundaries formed in the process of IPD. Therefore, can to change the strength and creep rate of nanocrystalline materials by changing the structure of the boundaries.

It is known that as a result of effects low-intensity ultrasound (UST), occur the relaxation of internal stresses in the material volume, which is the consequence of the number of factors. As a result of high-frequency alternating loading is generated the large number of vacancies, which stimulate non-conservative slip [6, 15]. Moreover, the dissipation of the vibration energy occurs mainly at the interfaces, which may lead to local heating, local stress decrease and activation of dislocation sources. It is known [16], that the treatment of ultrasonic field is started the process of multiplication of dislocations through two channels. One of the channels is the activation of Frank-Read source, but due to the ultrasound treatment the activation of each of the source occurs at a lower value of the shear stress (the effect of ultrasound is the similarity to the action of the trigger). The second channel is the increase in the number of sources of Frank Reid. It stimulates the active movement, interaction and annihilation of dislocations and reconstruction of the structure boundaries.

The result of our ultrasonic treatment is slightly decreased of the strength characteristics of the material on ~ 15% at 300 and at 700 K compared with the rolling samples, with the simultaneous increase of plasticity and creep rate. Herewith changed the nature of the creep specimens, decreased specific electrical resistivity during deformation, which indicates the decrease in the internal stresses and the change in the deformation mechanism.

According to the analysis of the curve of creep rate by the applied stresses for the samples of zirconium after MT-2 (see Fig. 1), the numerous restructuring during deformation is absent. From the value of the activation volume ($V_{ef} \sim 10^{-22}$ cm^3) plastically flow caused by moving of point defects and is controlled by several mechanisms, which more character for the high-temperatures deformation: the climb of dislocations, grainboundary diffusion and slipping along the grain boundaries,

Investigations of the structure have shown that the ultrasonic treatment of nanostructured Zr does not lead to the noticeable change in the size of the initial nanostructure, however, there are the signs indicating the beginning of the recovery process. In electron micrographs observed the clear fragments boundaries and low dislocation density in fragments (see Fig. 3,c). It should be noted that, due to creep at T = 700 K and stresses close to the ultimate strength (see Fig 3,d), the original nanostructure is destroyed. In its place the dense elongated dislocation formations, slightly disoriented (~ 4 degrees) between them, are nucleated.

According to some investigations [16], the self-organization of the dislocation subsystem in an ultrasonic field leads to the fact that each of boundaries is formed by the dislocations of the same sign, but any two adjacent are formed by the dislocations of opposite sign. In addition, the dislocations in the boundaries do not settled on the one plane, and have a fine boundaries structure, i.e. contain dislocation dipoles and multiples. This distinguishes them from the dislocation walls formed during annealing in the absence of ultrasonic field. Possibly, the higher thermostability of structure during creep of Zr after UST can be associated with this interpretation, at least in the area of stresses $\sigma < 0.9\sigma_B$.

CONCLUSIONS

The studies have shown that nanostructured Zr has a high strength but low plasticity in the temperature range 300…700 K.

The ultrasonic effect on imperfect grain boundaries leads to the redistribution of dislocations, modification in the structure and formation of the more perfect and ordered grain boundaries, i. e., there is the self-organization of the dislocation subsystem.

The ultrasonic treatment allows keeping the higher strength of material, at the same time lead to the plasticity increases, homogeneity of material and greater structural stability in the process of plastic flow.

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Article received 25.11.2015

ВЛИЯНИЕ УЛЬТРАЗВУКОВОГО ВОЗДЕЙСТВИЯ НА ПОЛЗУЧЕСТЬ НАНОСТРУКТУРИРОВАННОГО Zr

В.И. Соколенко, В.М. Горбатенко, Е.В. Карасева, А.В. Мац, Е.С. Савчук, В.А. Фролов

В области температур 300…700 К изучены особенности ползучести и электрофизические характеристики наноструктурированного циркония после различных воздействий. Показано, что пластическое течение наноструктурированного циркония осуществляется вследствие перестройки дефектной структуры и сопровождается релаксацией напряжений. Ультразвуковая обработка снижает уровень внутренних напряжений, но не приводит к заметному изменению масштабности наноструктуры.

ВПЛИВ УЛЬТРАЗВУКОВОЇ ДІЇ НА ПОВЗУЧІСТЬ НАНОСТРУКТУРОВАНОГО Zr

В.І. Соколенко, В.М. Горбатенко, Є.В. Карасьова, О.В. Мац, Є.С. Савчук, В.О. Фролов

В області температур 300…700 К вивчені особливості повзучості й электрофізичні характеристики наноструктурованого цирконію після різних впливів. Показано, що пластична течія наноструктурованого цирконію здійснюється за рахунок перебудови дефектної структури й супроводжується релаксацією напруг. Ультразвукова обробка знижує рівень внутрішніх напружень, але не призводить до помітної зміни масштабу наноструктури.