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CHARACTERISTICS OF THE CREEP AND EVOLUTION OF THE STRUCTURE IN NIOBIUM IN THE TEMPERATURE RANGE 77…300 К

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The changes in the structure, resistivity and plastic deformation characteristics of the polycrystalline niobium, annealed and also preliminary deformed by drawing to 80%, were studied at 77 and 300 K. It is shown that during the low-temperature creep of highly defect systems, the dislocation exhaustion mechanism is working even at stresses above the yield stress as the processes of linear defect generation under these conditions are strongly hindered. The development of low-temperature creep processes in the highly distorted fragmented structures cannot be described by the classical representations. A transition from the logarithmic creep to the power law creep, characteristic for much higher temperatures is observed, at the same time the total level of internal stresses being decreased. The variation of the law of plastic flow in the given stage of niobium creep at low temperatures does not depend on the method of highly distorted structure formation. The formation of new structures, which are steadier to tensile stresses, plays a determining role.

INTRODUCTION

The structure formation in metals with the use of large plastic deformations is one of the main steps in the many methods of mechanics-thermal treatments applied for a long time. However, the use of large plastic deformations in this-type treatments has had, in general, an empiric character, because of complexity and ambiguity of the processes being developed under these conditions. The notions on collective effects in the dynamics of dislocations were intensively developed during the recent years that advanced the understanding of the main processes and mechanisms of structure formation.

It is well-known, that under the critical external action the structure evolution and formation of new structure elements take place, which was the result of structural instability in the crystal [1]. The structural instability can cause a number of effects, e. g. a non-monotonic change in the mechanical properties, rotational plasticity, changes in the law of creep [2] etc. Of particular interest is the study of plastic deformation processes involved by the structural instability in the region of highly imperfect metals, where they are especially pronounced. For achievement a very high defect density in metals the most effective is the use of large plastic deformations at low temperatures [3].

The purpose of the present study is to investigate the characteristics of the low-temperature creep and corresponding changes in the structural state of annealed niobium and after large plastic deformation. The results obtained permit to make up more valid conclusions about the relation between the activated plastic flow processes and the structure elements of the material.

MATERIAL AND EXPERIMENTAL PROCEDURE

The material investigated was polycrystalline 99.9% niobium annealed at 1700 K during 1 hour and prestrained by drawing to 80% at 77 K.

Creep tests were carried out in the step loading regime at 77 and 300 K, the measurement accuracy was $\sim 5 \cdot 10^{-5}$ cm. Measurements of the electrical resistance were made by the compensation scheme. The error of resistivity determination did not exceed ±0.5%. The activation parameters were determined using the differential methods described in [4]. Investigations of the electron-microscopic niobium structure were made on the microscope EMV-100BR.

RESULTS AND DISCUSSION

Analysis of experimental data has shown that, practically throughout the entire investigated range of stresses and temperatures, the creep of annealed polycrystalline niobium is described by the logarithmic law and only at the stresses near the ultimate strength the transition to the power law is observed. The resistivity during the deformation process in the part of the logarithmic creep increases with deformation rise. This evidences on the increase of the total concentration of crystalline lattice defects that is characteristic for hardening mechanisms in the plastic flow process. At the stresses near the ultimate strength, where the change of the creep law is observed, the resistivity of metal decreased during deformation. This can be due to the redistribution of defects in the crystalline lattice, enhancement of the stress relaxation process, and, as a consequence, change in the mechanism of plastic deformation

Studying the creep process we have determined and calculated, by the formulae of the thermofluctuational plastic deformation theory, the effective activation volume V_{eff}, the effective activation energy U_{eff}, and the total activation energy U_{o}, which characterizes the value of barriers controlling the plastic flow of niobium during the creep. The activation parameters as a function of the temperature and applied stress were investigated.

The obtained values of the activation volume ($V_{\text{eff}} \sim 9b^3$) and the total activation energy ($U_{\text{o}} \sim 0.3$ eV) at the test temperature $T = 77$ K, and also the fact that both these values decrease with the stress increasing allow us to conclude that the dislocation motion is controlled by the low-energy barriers, the concentration of which increases with stress rise. Considering the activation volume value, these obstacles might be Peierls-Nabarro barriers, as well as, points defects and interstitial atoms, because the energy of dislocation interaction with them is 0.2…0.5 eV [5].

Let us evaluate the impurity contribution into the process of retardation of dislocations at this temperature. The average distance between the points of dislocation line pinning by the interstitial impurities, obtained by the formula $l_n \approx bc^{-2/3}$ (where c is the interstitial impurities concentration, b is the Burgers vector) [5], is $l_n \sim 5 \cdot 10^{-6}$ cm. Using the relation $V = bdl \sim b^2 d$ (where V is the activation volume, d is the barrier width) we can determine the length of dislocation segment 1, taking the part in the elementary slipping act. The calculation results show that $l \sim 3 \cdot 10^{-7}$ cm.

The above estimates permit to conclude that the activated motion of dislocations at $T = 77$ K is controlled by the Peierls-Nabarro barriers and points defects.

At 300 K the role of barriers with a higher energy ($U_o \sim 1$ eV, $V_{eff} \sim 30b^3$) increase. This may be dislocations, the contribution of which is determined by their density and distribution, depending on the stress applied. The observed decrease of V_{eff} and U_o with stress increasing, confirms this assumption. The average distance between dislocations in the grain is estimated by the formula $l_r \sim N_d^{-1/2}$, (where $N_d \sim 2 \cdot 10^{10}$ cm^{-2} is the dislocation density), the value obtained is $l_r \sim 7 \cdot 10^{-6}$ cm. The length of the dislocation segment taking the part in the elementary slipping act at this temperature is $l \sim 1.4 \cdot 10^{-6}$ cm.

However, as it was shown above, the distance between the points of dislocation pinning by the interstitial impurities is $l_n \sim 5 \cdot 10^{-6}$ cm. Consequently, the plastic flow of niobium during the creep at 300 K is controlled by the impurities and interaction between dislocations.

The electron-microscopic study of the niobium structure at different stages of the creep deformation has shown that at the initial stages of deformation there is a rather uniform dislocation distribution inside the grains and an increased concentration of the last in the near-boundary accommodation zones ($N_d \sim 2 \cdot 10^{10}$ cm^{-2}) (Fig. 1,a).

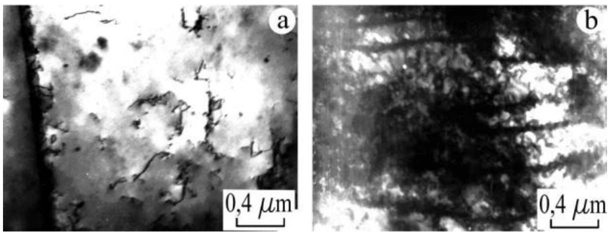

Fig. 1. TEM images of niobium after creep deformation (ε) at T = 77 K: a – ε ~ 0.5%; b – ε ~ 3%

Where deformation increasing up to the specimen destruction, a sharp dependence of the defect structure evolution on the crystalline orientation of grains relatively to the axis of tensile is observed. In this case the structure being formed differs by both the dislocation density and the pattern of dislocation distribution.

So, even a little deformation increase initiates, in some grains, the build up of elongated dislocation formations and power ($N_d \sim 9 \cdot 10^{10}$ cm^{-2}) dislocation clusters near the grain boundaries and their joints (see Fig. 1,b). At stresses close to the ultimate strength the formed dislocation boundaries and their closed configurations was observed.

Hence, during the step loading the structure imperfection increases and at stresses close to the ultimate strength of the material the structure comprises a sufficient number of elements being characteristic for highly distorted systems. The processes of their formation under dislocation interaction enhancement are strongly influenced by the dislocation mobility increased due to the transverse slipping. The latter can be, in this case, a main mechanism of stress relaxation.

A sufficient degree of transverse slipping, promoted by the low deformation rate under creep, leads to the activation of the processes of annihilation of unlike dislocations and their polarization, i.e. to the collective interaction and mobility of dislocation ensembles. It is assumed that at stresses close to the ultimate strength the collective effects become the predominant mode of plastic deformation that at the macro level is revealed by the variation of the character of the material plastic flow.

In this connection, it is interesting to study the peculiarities of the processes of highly distorted structure self-organization during low-temperature creep deformation. For this purpose the creep of niobium, preliminary deformed by drawing by 80% at 77 K, has been studied.

Investigations of the niobium structure after drawing have shown that a part of the material volume is occupied, besides the dislocation of a density ~ (5…7)·10^{10} cm^{-2}, by the boundaries, of a dislocation-disclination origin, stretched along the drawing direction (Fig. 2,a). Inside of fragments one can see many transverse boundaries and bend contours (close clusters of like-sign dislocations). Disorientation of fragments makes ~ 8…9.5^0, and the distance between them is ~ 0.12…0.3 μm.

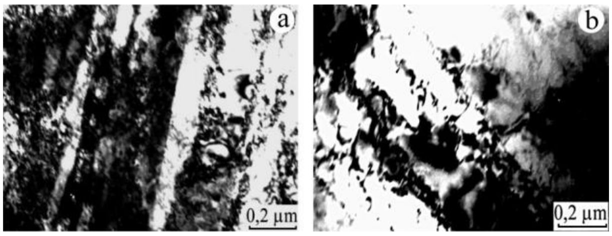

Fig. 2. TEM images of niobium after drawing to 80% at 77 K (a); after creep deformation (ε ~ 1%) at 77 K (b)

For the material with a preliminary formed higher distorted structure, the creep obeys a logarithmic law only at stresses near the yield stress, and during the further stress rise one observes the creep rate increasing and transition to the power law. And the electrical resistance of the sample is decreasing at all the deformation stages.

According to the analysis of obtained activation parameters and their dependencies on the stress at 77 K we can conclude that, under conditions of a highly distorted crystalline lattice, the value of barriers controlling the plastic flow decreases ($V_{\text{eff}} \sim 2b^3$, $U_o = 0.1$ eV). This result gives us a reason to assume that under these conditions the Peierls-Nabarro barriers become main obstacles controlling the activated motion of dislocations.

In concern with the values of activation parameters of niobium after drawing, obtained at the test temperature of 300 K ($V_{\text{eff}} \sim 9b^3$, $U_o = 1$ eV), the main contribution to the deformation made by the dislocation interaction localized in little volumes.

Structural investigation of specimens after creep deformation to ~ 1% (see Fig. 2,b) has shown, that the defect structure of niobium predeformed by drawing becomes instable, when the deformation conditions are changing. Thus, the density of randomly distributed dislocations sharply decreases, the dense elongated like-sign dislocation formations are nucleated, creating a rather large gradient of local internal stresses. The old boundaries are broken and new boundaries with smaller disorientation angles are formed. In the remained boundaries the processes of dislocation redistribution occur, being accompanied by some ordering, i.e. the effects of deformation microlocalization are observed.

Now, let us analyze the data obtained. Realization of the logarithmic creep law in the initial deformation stages means that the deformation occurs due to the individual dislocation mobility in the fragment-free micro zones. However, this plasticity mode is exhausting, that is evidenced by the decrease of the material resistivity. In essence, the mechanism of dislocation exhaustion is realized.

An increase in the creep rate with the stress and, as a consequence, a transition from a logarithmic dependence of the creep deformation to a power law, indicates on the decreased level of the elastic-stressed state in the lattice of drawing niobium, under the action tensile stresses caused by the integral effect of interaction between dislocations and their complexes.

At the microscopic level this process is followed by the fracture of the initial fragmented structure and by the formation of a new one, being less strained and more resistant to the extension [6].

We can mention possible mechanisms of such a structure transformation in the superposition of external and internal stress fields.

One of causes of structure instability can be the activation of new slip systems accompanied by the multiplication of dislocations with different Burgers vectors. The plane dislocation clusters formed lead to curving and destroying of boundaries, oriented definite way. For the most part, those boundaries are destroyed, in which the angle of location plane is close to 90^0 relatively to the axis of load application and the active slip planes are adjacent to these boundaries at a small angle [7].

Another cause of the boundary instability under tensile deformation might be the variation of the character of interaction between a part of ordered boundaries and nonequilibrium long-range stresses (clusters of like-sign dislocations, uncompensated boundaries etc.) [8]. If the arising stresses exceed the stresses of dislocation interaction at boundaries the latter can be ruptured. In both cases the effects of deformation microlocalization slow down the hardening rate.

And, at last, the new structure being formed with realization of translation and rotation modes under conditions of intensive interdislocations interaction and destructs the initial structure [9].

So, it should be noted, that the low-temperature creep deformation of a metal in the structural-hardened state of the lattice under the action of very low stresses leads to the defect structure transformation resulting in the distortion of the initial structure configuration and formation of a more tension-resistant structure. It should be noted that, on the whole, the defect structure develops with a decrease in the total background of internal stresses that is evidenced by the material resistivity decreasing during deformation.

CONCLUSIONS

The changes in the structure, resistivity and plastic deformation characteristics of the polycrystalline niobium, annealed and also subjected to large plastic deformation by low-temperature drawing, were studied at 77 and 300 K. It is shown that during the low-temperature creep of highly defect systems, the dislocation exhaustion mechanism is working even at stresses above the yield stress as the processes of linear defect generation under these conditions are strongly hindered.

The development of low-temperature creep processes in the highly distorted fragmented structures cannot be described by the classical representations. A transition from the logarithmic creep to the power law creep, characteristic for much higher temperatures is observed, at same time the total level of internal stresses being decreased. The variation of the law of plastic flow in the given stage of niobium creep at low temperatures does not depend on the method of highly distorted structure formation. The formation of new structures, which are steadier to tensile stresses, plays a determining role.

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ХАРАКТЕРИСТИКИ ПОЛЗУЧЕСТИ И ЭВОЛЮЦИЯ СТРУКТУРЫ НИОБИЯ В ИНТЕРВАЛЕ ТЕМПЕРАТУР 77…300 К

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Изучены изменения структуры, электросопротивления и характеристик ползучести при 77 и 300 К отожженного и предварительно деформированного волочением на 80% ниобия. Показано, что в процессе низкотемпературной ползучести высокодефектных систем действует механизм истощения дислокаций даже при напряжениях, превышающих предел текучести. Развитие процессов низкотемпературной ползучести в сильноискаженных фрагментированных структурах не описывается с помощью классических представлений. Наблюдается переход от логарифмической ползучести к степенной, характерной для гораздо более высоких температур, при этом общий уровень внутренних напряжений снижается. Изменение закона пластического течения на определенном этапе ползучести ниобия при низких температурах не зависит от способа получения сильноискаженной структуры. Определяющую роль играет формирование новых структур, менее напряженных и более устойчивых к изменениям геометрии действующих напряжений и скорости деформации.

ХАРАКТЕРИСТИКИ ПОВЗУЧОСТІ І ЕВОЛЮЦІЯ СТРУКТУРИ НІОБІЮ В ІНТЕРВАЛІ ТЕМПЕРАТУР 77…300 К

Є.В. Карасьова, О.В. Мац, В.І. Соколенко

Вивчено зміни структури, електроопору та характеристики повзучості при 77 і 300 К відпаленого та попередньо деформованого волочінням на 80% ніобію. Показано, що під час низькотемпературної повзучості високодефектних систем діє механізм виснаження дислокацій навіть при напругах, що перевищують межу текучості. Розвиток процесів низькотемпературної повзучості в дуже спотворених фрагментованих структурах не описується за допомогою класичних уявлень. Спостерігається перехід від логарифмічної повзучості до степеневої, характерної для більш високих температур, при цьому загальний рівень внутрішніх напруг знижується. Зміна закону пластичної течії на певній стадії повзучості ніобію при низьких температурах не залежить від способу отримання дуже спотвореної структури. Головним чинником є формування нових структур, менш напружених та більш стійких до змін геометрії діючих напружень та швидкості деформування.