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EFFECTS OF TEMPERATURE ON THE LAWS OF PLASTIC DEFORMATION AND MECHANICAL CHARACTERISTICS FOILS Al COATED WITH TITANIUM NITRIDE

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The experimental results define the role of the coating of titanium nitride on a two-dimensional pattern of plastic deformation of polycrystalline aluminum at room and elevated temperatures. It is shown that the hardening of the titanium nitride coating 3 μm thick unable increase the strength of aluminum specimens, but can block the emergence and development rotational modes of plastic deformation and relaxation processes at elevated temperatures, leading to the occurrence of oscillation of the deformation stress on the deformation curves It is experimentally shown that the nature of the destruction of the coating, which leads to the destruction of the sample depends on the deformation temperature.

INTRODUCTION

It is well known that by applying an external load to the sample in it there are various relaxation processes - crystallographic slip, rotational effects of various kinds, and finally destruction [1, 2]. This typical sequence of occurrence and development of relaxation processes may be changed, for example, by forming a texture structure [3] in the samples after thermomechanical processing under certain conditions, or by applying hardening coating of various types [4, 5]. It may lead the change not only the mechanical characteristics of the sample, but the sequence and nature of the development of various relaxation processes.

In the "cold" strain (0.2…0. 25 $T_{melting.}$) plastic deformation develops mainly due to the translation mode (single and multiple dislocation slip). With the development of plastic deformation turns on the rotational mode mechanism (fragmentation, individual and collective reorientation bands, twinning, etc.). Under conditions of "warm" (≤ 0.5T melting) and "hot" (> 0.5 T melting) deformation along with the mechanisms mentioned above, occurs slipping along the grain boundaries (GBS) and thermally activated processes: dynamic recrystallization, thermal and dynamic recovery, etc. [6–8].

Thus, the change of deformation temperature should lead to a change in the role of the basic mechanisms of the the state of stress relaxation and the emergence of the new mechanisms of relaxation. In general, it can lead to a change patterns of strain hardening and, ultimately, to change the mechanical characteristics of the samples.

At deformation two-dimensional of polycrystalline aluminum with titanium nitride coating is found another nature of relaxation processes. It is experimentally shown [9] that the titanium nitride coating a few microns thick blocks the emergence and development of the rotational mode at all stages of plastic deformation. This primarily leads to an increase in strain hardening at the initial stage of deformation, and, ultimately, to reduce the magnitude of plastic deformation of samples prior to failure and increases conventional yield strength. In these samples, at an early stage of plastic deformation occurs only dislocation glide, the development of which continues until the destruction of the sample.

The distinguishing feature process of sliding in aluminum samples with titanium nitride coating is the possibility of matching plastic deformation by sliding at various levels (coarse and fine slip) in different grains at their boundary.

Thus, the experimental results suggest of essentially different nature of the structural and orientational changes accompanying the plastic deformation of the two-dimensional aluminum polycrystals with hardening of titanium nitride coating and without it.

The coating of titanium nitride on the two-dimensional polycrystalline aluminum unable to change the tensile strength of samples, presumably because of its low thickness, but such a coating thickness sufficient to suppress the rotational mode of plastic deformation. The rotational mode of plastic deformation in the uncoated samples significantly contributes to the plastic deformation of the sample as a whole.

The aim of the work is experimental study of the effect of the all-round coating of titanium nitride on the mechanical characteristics and the pattern of occurrence and development of the accommodative-relaxation processes in plastic deformation in the two-dimensional aluminum polycrystals in the temperature range from 25 to 300 °C.

MATERIALS AND INVESTIGATIONS METHODS

As the object of study were selected single-crystal on thickness aluminum samples (two-dimensional polycrystalline), the working part of the sample was 100×20×0.15 mm. The choice of the such samples are not random for the study. It is experimentally shown, due to the lack of constraint in the direction, normal to the sample surface, in two-dimensional polycrystalline (foils, samples containing only through the grain boundaries) rotational effects are most pronounced. To prepare the samples, aluminum foil (99.96%), 0.15 mm thick was used. Research by means of x-ray fluorescence analysis was shown that the main impurity in a foil are: Fe – 0,03%, Ti – 0,01%. For samples with a large grain size cut out strips of aluminum foil, subjected to thermomechanical treatment, which included a primary annealing at T = 400 °C for one hour, uniaxial tension deformation is 2…2.5% and subsequent annealing at T = 300 °C for one hour (primary recrystallisation) and T = 630 °C for one hour (collective recrystallization). The average grain size of the samples after the recrystallization was ≈ 10 mm.

Grain structure was revealed by chemical etching using an etchant Keller. After identifying the grain boundaries of one of the working surfaces of the sample polished carefully in order to detect the strain relief on it in the form of slip lines and determination its characteristics in situ during the deformation of the sample by laser technique [10].

Other surface remained intact with quasiperiodic relief, arising therein by chemical etching. It served for studying of regularities of emergence and development of substructural and orientation changes using received color orientation maps from this surface [11] and their visualization [12]. This made it possible to trace for substructural and orientation changes of the sample in the process of deformation.

On the thus prepared samples are after preliminary ionic cleaning was applied thorough coating of titanium nitride thickness ~ 3 μm on the “Bulat-3T” in the following mode: precleaning (I = 75 A, U = 600 V) along with heating for one minute, followed by the precipitation of titanium nitride (I = 75 A, U = 100 V), the nitrogen pressure in the chamber was $2\ 10^{-1}$ Pa.

All the samples were deformed in conditions of uniaxial tension with constant speed of deformation $\acute{\varepsilon} = 5\cdot10^{-5}\ s^{-1}$. Record deformation diagrams carried out using a digital multimeter (Nnit-T UT70D), connected to a PC. Color orientation maps were registered by means of the Webcam connected to the computer. To identify a possible grain boundary sliding (GBS) on the surface of the individual samples was applied using a PMT-3 microhardness series of parallel scratches. The distance between them made 100 μm.

The required temperature regime was provided by means of the heating furnace with a temperature regulator or by passing an alternating electric current through the sample. The sample temperature controlled by several thermocouples “chromel-alumel” in different parts of the sample. The temperature was maintained with an accuracy ± 5 °C. The method of heating the sample as showed control measurements, did not exert noticeable impact on character of deformation curves.

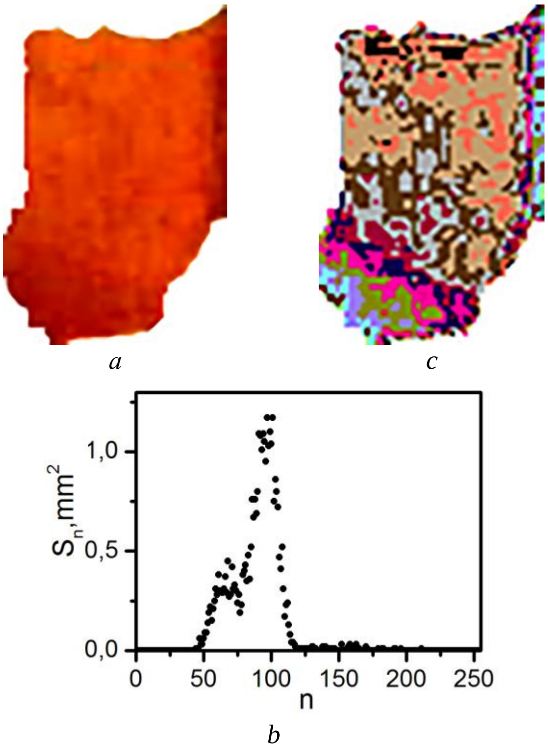
For studying the nature of emergence and development of rotational structure and definition of characteristics of a deformation relief in situ during the deformation of the samples two techniques were used at the same time. The first used the diffraction effect of white light into a quasiperiodic structure of the surface of the sample that occurs after chemical etching, in the second – the effect of laser diffraction by the deformation relief.

It is known [11] that at chemical etching on the surface of crystalline sample the quasiperiodic relief is arise, the characteristics of whose depend on the crystallographic orientation of the surface. The experimentally detected effect of diffraction of white light on this relief [11], which become visible for the color staining image of the sample surface. When the surface of a sample has orientation inhomogeneity, each region with the identical crystallographic orientation there corresponds the own quasiperiodic relief and therefore its hue on the color image of the surface. As an example, Fig. 1,a shows the color image of the surface of one of the grains of a polycrystalline – color orientation map where the color of the image specific area uniquely defines its crystallographic orientation.

Because the color orientation card is received in the RGB color space containing 16.7 million shades on it is impossible to determine the sizes of areas with visually indiscernible shades of color. The method of visualization of these shades which essence consists in consecutive consideration of color orientation cards in shades of red (R), green (G) and blue flowers (B) which number makes 256 was developed.

As an example, Fig. 1,b shows the distribution of areas of the color image of the surface of grain (S) in shades of red (n), where n - conditional crystallographic orientation; S - area of the grain surface region with the same crystallographic orientation (subgrains). In other words, the curve shown in Fig. 1,b is a distribution subgrains by their size S and conditional crystallographic orientation n.

The second stage of the visualization method allows obtaining the map of accommodation subgrains on the grain surface (see Fig. 1,в).



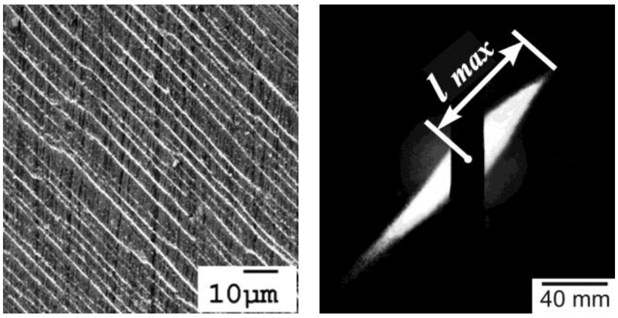
a

c

b

Fig. 1. The color orientation map of the surface of a single grain (a), the distribution subgrains by size and crystallographic orientation (b) and map of placement a subgrains on the surface of grain (c)

Fig. 2 shows the quasiperiodic deformational relief which occurs on the grain surface of the sample and corresponding scattering patterns of laser radiation (He-Ne, $\lambda = 630$ μm), are given on this relief. Value l_{max} characterized the minimum distance between the slip lines [10], thus, defines intensity of development of plastic deformation by sliding in this grain.



a *b*

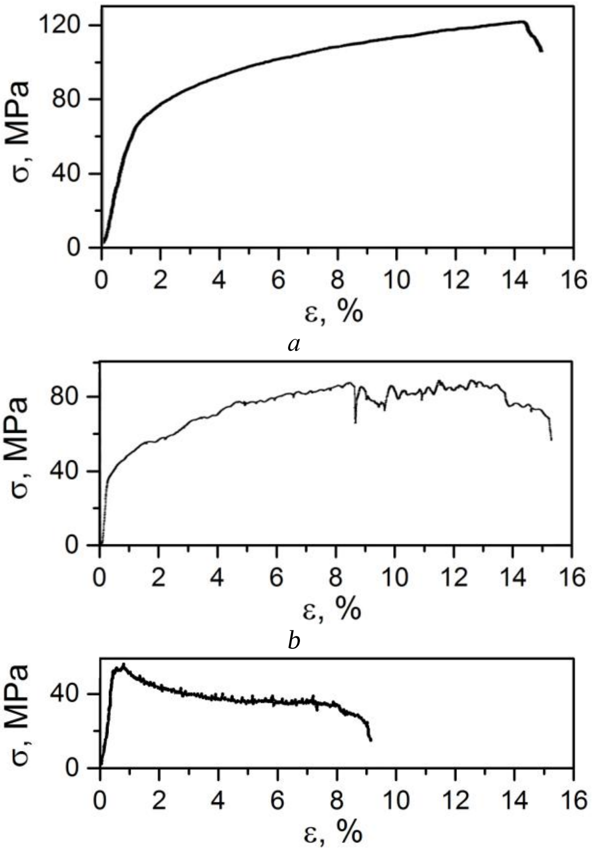
Fig. 2. The deformation relief on the surface of one of the polycrystal grains Al (a) and pattern the laser diffraction from this relief (b)

It should be noted especially that the covering from titanium nitride 3 μm thick does not prevent to receive color orientation maps and the diffraction maps from a surface of a polycrystalline aluminum specimen. It was shown experimentally that coating completely repeats the morphology of the surface sample of the aluminum. This result allows to use above given techniques for research of emergence and development of rotational and translation modes of plastic deformation for aluminum specimens with a covering. To register of the deformation relief and determining its characteristics used interferometer MII 4, optical (MIM-7) and scanning (Jeol JSM-840) microscopy.

EXPERIMENTAL RESULTS

For definition of a role of the strengthening covering of titanium nitride on regularity of development of a plastic strain of aluminum polycrystals were conducted researches in the same conditions on samples with a covering and without it Fig. 3 shows the deformation curves of polycrystalline aluminum samples without covering received at various tests temperatures: 25, 240, and 340 °C. These curves substantially are different both in form and the nature of the changes of strain hardening.

The deformation curve obtained at room temperature ("cold" deformation), has parabolic character, typical for polycrystalline samples (see Fig. 3,a). The deformation curve that obtained in condition of a "hot" deformation (T = 240 °C) has the same parabolic character, as in the "cold" deformation, however it detects leaps deforming stresses which become visible when the strain $\varepsilon \approx 2\%$ (see Fig. 3,b). Ultimate stress of the samples under such deformation conditions are decreases. "Hot" deformation (T = 340 °C) leads to a softening of the sample (see Fig. 3,c). Despite the softening of the sample, the deformation curve that given on Fig. 3,c, also finds out oscillations of deforming tension.



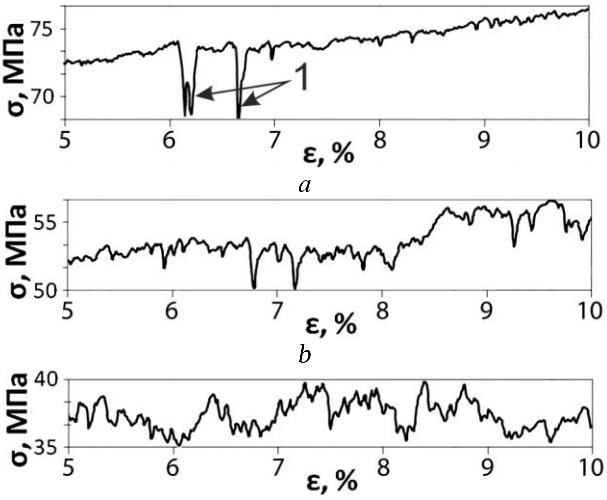
a

b

c

Fig. 3. The deformation curves of Al samples without covering that are tested at different temperatures: a – T_1 = 25 °C; b – T_2 = 240 °C; c – T_3 = 340 °C

Fig. 4 shows fragments deformation curves for aluminum samples without covering that are deformed at temperatures in the range from 200 to 300 °C. All the deformation curves are characterized by oscillation of the deforming stress. From the figure shows that the temperature increases deformation grows the amplitude of oscillations of the deforming stress.



a

b

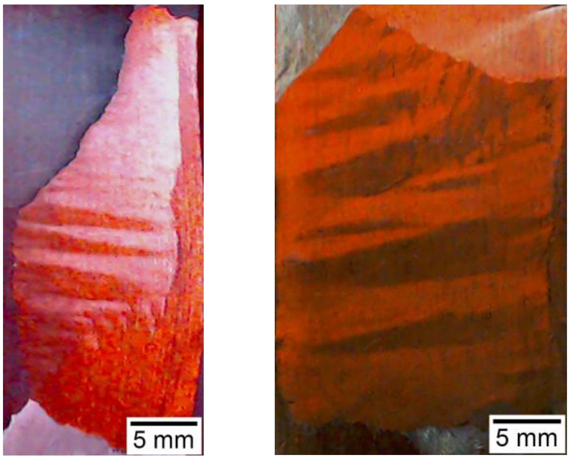
c

Fig. 4. Fragments of the deformation curves of aluminum samples without covering tested at temperatures ranging from 200°C to 300°C (a – 200 °C; b – 250 °C; c = 300 °C)

On Fig. 4 are given fragments of deformation curves in two-dimensional polycrystalline aluminum in a temperature range from 200 to 300°C. These curves show that the deformation is characterized by an saltatory change in the flow stress, indicating about the heterogeneity of the plastic flow of samples, which increases with increasing temperature. On separate parts of the deformation curves, for example, in conditions of deformation at T = 200°C, are found out single leaps (1) with an amplitude considerably exceeding the average amplitude of the oscillation.

The large number of the experimental and the theoretical works are devoted to research of the phenomenon of saltatory deformation in the metal samples, for example [13, 14]. In these works it is shown that the oscillations of deforming stress in the conditions of deformation at elevated temperature, can arise at thermally activated release of dislocations from stoppers (impurity atoms). This process is the basis of the well-known effect Portevin Le Chatelier. As a result of the combined action of applied stress and thermal fluctuations occurs quasiperiodic detachment of dislocations from impurity atmospheres. At a detachment of dislocations from the impurity atmospheres the density of movable dislocations saltationally increases and provides deformation leap. In such cases for continuation of deforming of the sample requires the raising of stress capable to provide a further separation of dislocations from fixing points, and then the cycle continues.

A possible reason for the appearance on the deformation curves leaps type 1 in Fig. 4,a can be the appearance during deformation of the samples of the bands of reorientation. The use of a research technique of rotational structure *in situ* during the deformation of the samples at a temperature 200°C allowed on the surface of the individual grains of the sample to detect the occurrence of macroscopic reorientation bands crossing the surface of the whole grain (Fig. 5). Experimental studies have shown that with temperature increase of deformation in the aluminum samples without coating process of any rotational reconfiguration becomes more active.



a *b*

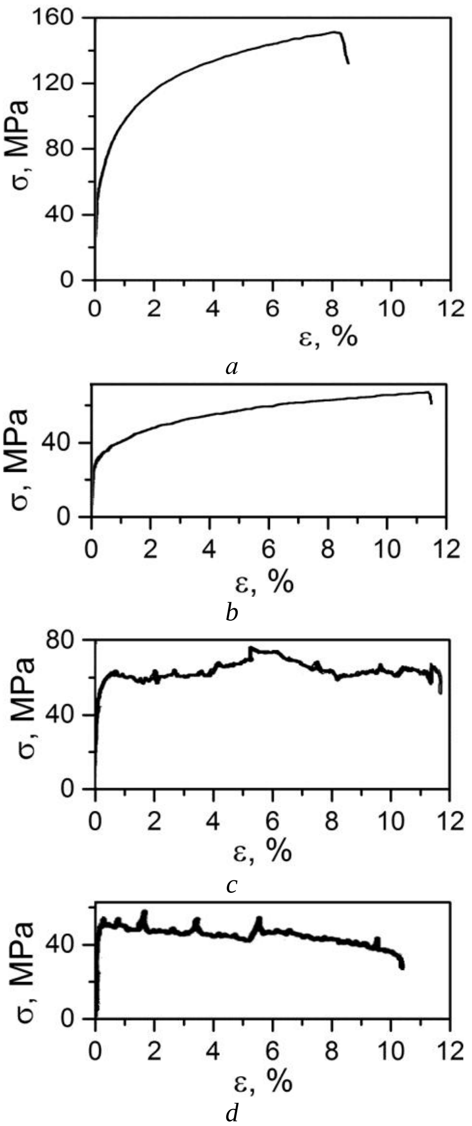
Fig. 5. Macroscopic reorientation band detected in a number of grains of the aluminum sample without coating during its deformation at a temperature 200°C

The experimental results shown in Figs. 3 and 4 for samples (aluminum foil) without coating and results of research rotational structure shows different nature of relaxation processes in a “cold”, “warm” and “hot” strain.

Fig. 6 shows the deformation curves of the samples of aluminum with a covering from titanium nitride for room temperature (a) and temperature increases: 100 °C (b), 240 °C (c), 340 °C (d).

Deformation curves obtained at room temperature and at 100 °C do not show the oscillation of the flow stress and have a typical parabolic form. In the conditions of deformation at room temperature hardening a covering from titanium nitride leads to slight increase in the ultimate stress and decrease plasticity of the sample. Increase of the conditional yield strength at ~ 25% and coefficient of straining hardening at the initial state of plastic deformation is characteristic of aluminum samples with a covering from titanium nitride in comparison with exemplars without covering in the conditions of deformation at room temperature. The oscillation of deforming stress in the samples with a covering is found only at temperatures ≥ 200 °C. The amplitude of the oscillations stress value is significantly less than the deformation curves of aluminum uncoated samples. It should be noted that none of the deformation curves obtained in the temperature range 100…300 °C, no separate leaps deforming stress were found with large amplitudes, typical for aluminum samples deformed at temperature increases. In order to explain the results of detailed studies of pattern occurrence and development of the rotational structure were carried out, which characterizes the contribution to deformation sample of the rotational mode, and patterns of development of translational mode of a plastic strain (dislocation sliding) were conducted. Use of the visualization technique of the orientation changes occurring on the surface of a polycrystalline aluminum sample with covering showed us that in the course of its deformation at room temperature (at strain 4…5%) on the surface of almost all the grains occurs slightly misoriented subgrain structure. The samples of uncoated aluminum at achievement of the same amount of deformation is characterized by a pronounced substructure (Fig. 7,a, b).

Fig. 8 is given a typical regularity of sliding development in the individual grains of polycrystalline aluminum specimen with a covering and without it. The figure shows that the nature of the slip in the process of deformation of aluminum samples with a covering and without it varies considerably. In samples with a covering from titanium nitride the initial state of plastic deformation ($\varepsilon \approx 2\%$) is characterized by intensive development of translational mode of plastic deformation (a sharp decrease d_{min} – the minimum distance between the slip lines). It finally leads to increase the coefficient of straining hardening. It is in full accordance with the effect of blocking of rotational mode of a plastic deformation found at deformation the samples with a covering. In aluminum exemplars without covering in the course of their deformation both modes of plastic deformation – translational and rotational are realized, and, the last not only makes a self-contained contribution a plastic strain of the samples, but also development of sliding is promoted for the account by the accommodative processes.



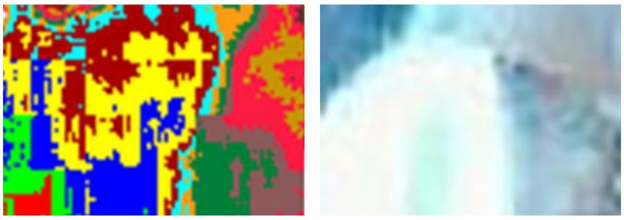
a

b

c

d

Fig. 6. Deformation curves for Al samples with titanium nitride coating, tested at different temperatures: a – T_1 = 25 °C; b – T_2 =100 °C; c – T_3 = 200 °C; d – T_3 = 300 °C



a *б*

Fig. 7. The substructure of the surface of individual aluminum grains samples without covering (a) and with a covering (b), arising after deformation of the sample 4…5% at room temperature.

The received results can unambiguously explain a role of the strengthening coverings from titanium nitride 3 μm thick applied on the coarse-grained aluminum samples at plastic deformation. On the one hand, a coating of this thickness may not significantly increase the tensile strength of the samples and on the other side a coating of titanium nitride, even such a small thickness can substantially alter the emergence and development of the relaxation processes and their role in the plastic deformation of the whole sample. Character of influence depends on the deformation temperature. The covering from titanium nitride significantly blocks development of rotational mode of a plastic deformation and changes regularity of emergence and development of sliding.

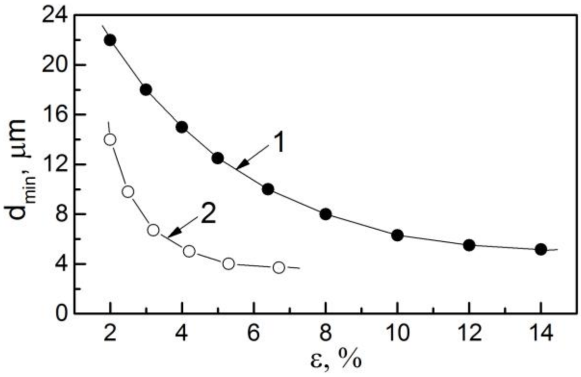
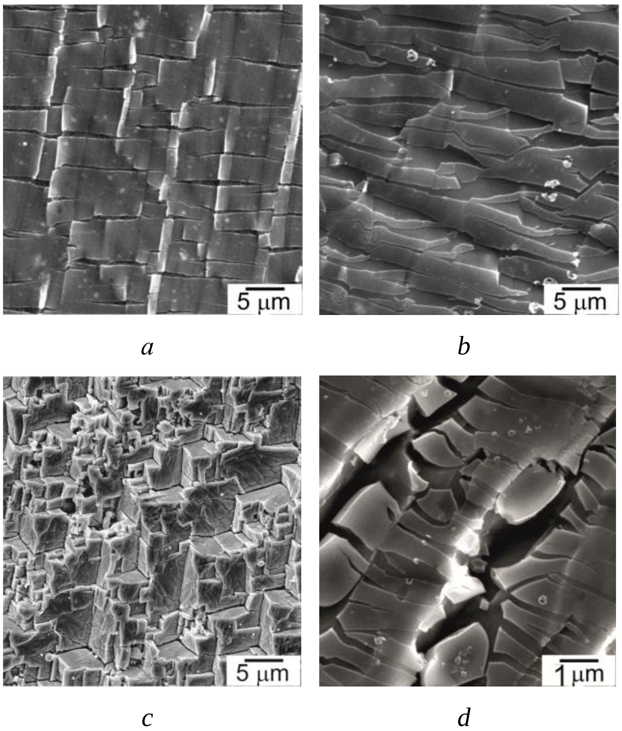


Fig. 8. Intensity of sliding during deformation aluminum samples at room temperature without cover (1) and with titanium nitride coating (2)



a *b*

c *d*

Fig. 9. Micrographs of the surface of the aluminum foil with a titanium nitride coating, illustrating the coating fracture when tested at different temperatures: a – T_{room}; b – T = 100 °C; c – T=200 °C; d – T =300 °C

The coating reduces the amplitude of oscillation deforming stress arising from the deformation of samples at elevated temperatures. For definition of the reason of decrease of aluminum plasticity samples with a covering from titanium nitride in the conditions of deformation at room and temperature increases character of destruction of aluminum samples with a covering was studied. It is experimentally shown that crack initiation in coverings is a cause of destruction of all the samples. Fig. 9 shows photomicrographs of the surface of samples with the titanium nitride coating obtained by the scanning microscope Jeol JSM-840.

The figure shows that the structure of emerging cracks is highly dependent on the deformation temperature. Thus for the samples deformed at room temperature, narrow cracks (up to 1 μm) are equally oriented, the length of which does not exceed 10 μm (a). With increasing temperature, their width and length increases (b), there is a mesh of cracks and finally at a temperature in a deformation 300°C the crack width comparable to the length, they becomes shapeless, the new cracks are forming that develop normal to the first.

CONCLUSIONS

- Experimental studies of regularities of development of a plastic deformation the coarse crystalline aluminum samples containing through grain boundaries with a covering from titanium nitride 3 μm thickness and without it in the range of temperatures from 25 to 300 °C are conducted.

- It is shown that the multifold covering from titanium nitride 3 μm thickness practically does not increase the strength of the studied aluminum samples, but can block emergence and development of relaxation processes.

At temperature increases (“warm” deformation) on deformation curves the oscillations of deforming stress and single leaps stress which amplitude considerably exceeds amplitude of oscillations is found.

- It is experimentally established that individual leaps of deforming stress are obliged to emergence in separate grains of polycrystalline samples without covering of macroscopic reorientation bands.

- It has been shown that the small thickness coating of titanium nitride at room temperature can almost completely block the emergence and development of the rotational structure and partly sliding, and at temperature increases - relaxation processes that lead to oscillations of the deforming stress. It is experimentally shown that decrease of plasticity of polycrystalline aluminum samples with a covering in the range from the room temperatures to 300 °C is caused by specifics of destruction of a covering.

- It is experimentally revealed that the grain boundary sliding as one of possible mechanisms of a plastic strain at alevated temperature, in the studied samples is not realized, apparently, because the samples are macrocrystalline and contain only the through grain boundaries.

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ВЛИЯНИЕ ТЕМПЕРАТУРЫ НА ЗАКОНОМЕРНОСТИ ПЛАСТИЧЕСКОЙ ДЕФОРМАЦИИ И МЕХАНИЧЕСКИЕ ХАРАКТЕРИСТИКИ ФОЛЬГ Al С ПОКРЫТИЕМ ИЗ НИТРИДА ТИТАНА

Е.Е. Бадиян, А.Г. Тонкопряд, О.В. Шеховцов, Р.В. Шуринов

Приведены экспериментальные результаты определения роли покрытия из нитрида титана на закономерность пластического деформирования двумерных поликристаллов алюминия при комнатной и повышенных температурах. Показано, что упрочняющее покрытие из нитрида титана толщиной 3 мкм не может увеличить прочность алюминиевых образцов, но может блокировать возникновение и развитие ротационной моды пластической деформации и релаксационные процессы при повышенных температурах, приводящие к возникновению на деформационных кривых осцилляции деформирующего напряжения. Экспериментально показано, что характер разрушения покрытия, приводящего к разрушению образца, зависит от температуры деформирования.

ВПЛИВ ТЕМПЕРАТУРИ НА ЗАКОНОМІРНОСТІ ПЛАСТИЧНОЇ ДЕФОРМАЦІЇ ТА МЕХАНІЧНІ ХАРАКТЕРИСТИКИ ФОЛЬГ Al ІЗ ПОКРИТТЯМ ІЗ НІТРИДУ ТИТАНУ

Є.Ю. Бадіян, А.Г. Тонкопряд, О.В. Шеховцов, Р.В. Шурінов

Наведено експериментальні результати визначення ролі покриття з нітриду титану на закономірність пластичного деформування двовимірних полікристалів алюмінію при кімнатній і підвищених температурах. Показано, що зміцнювальне покриття з нітриду титану товщиною 3 мкм не може збільшити міцність алюмінієвих зразків, але може блокувати виникнення і розвиток ротаційної моди пластичної деформації і релаксаційні процеси при підвищених температурах, що призводять до виникнення на деформаційних кривих осциляцій деформуючого напруження. Експериментально показано, що характер руйнування покриття, що призводить до руйнування зразка, залежить від температури деформування.