Russian Physics Journal, Vol. 43, No. 9, 2000

SOLID-STATE PHYSICS

EFFECTS OF ULTRASONIC SURFACE TREATMENT ON THE STRUCTURE AND PROPERTIES OF POLYCRYSTALLINE AND NANOSTRUCTURED TITANIUM

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UDC 539.43

The effects of ultrasonic surface treatment on the structural and phase-state of subsurface layers of polycrystalline and ultrafine-grained titanium are investigated. The changes in microhardness, mechanical properties, and regularities of microplastic deformation buildup are studied under static and cyclic loading. It is found that the characteristics of polycrystalline titanium suffer most from the ultrasonic treatment, while its influence on ultrafine-grained titanium is considerably weaker.

INTRODUCTION

Many properties of metals and alloys are to a great extent dictated by the structural and phase state of the surface. Special methods of mechanical and thermal treatment are, therefore, used for predetermined modification of their physical, chemical, and mechanical properties, which gives rise to changes in structural and phase state of the surface layers alone [1–3]. All other factors being equal, the fashion in which this or that treatment affects the surface layer structure, and thereby the properties, depends on the initial metal state (dislocation density and distribution, grain size, *etc*.).

Ultrafine-grained (nano and submicrocrystalline) metals (UFG) produced under severe plastic deformation (SPD), in particular by the method of equichannel angular pressing (ECAP), are advantageous over recrystallized coarse-grained (CG) metals as concerns high deposition of elastic energy, which is largely associated with the nonequilibrium grain boundaries [4–7]. Hence, given one and the same treatment, the structural and phase state in the surface layers of nanostructured (NS) metals may differs from that in CG metals, i.e. their physical, chemical, and mechanical properties may be different as well.

In the general case it seems, therefore, impossible to use the data on changes of properties of coarse-grained metals under surface modification to interpret the NS-metal properties. To date, there have been no studies aimed at identifying common mechanisms or special features of the effects of surface treatment on the metals in question.

With regard to the above-mentioned, the purpose of this paper is to elucidate the effects of plastic deformation on the surface on the structure and mechanical behavior of statically and cyclically loaded CG and NS-titanium produced by SPD. In so doing, we proceeded from the fact that plastic deformation of the surface (shot blasting) improves mechanical performance of cyclically loaded CG titanium, while chemical-thermal treatment (nitriding and oxidation) degrades it [8].

MATERIALS AND EXPERIMENTAL TECHNIQUE

The studies were carried out on commercial-grade titanium alloy specimens, IT, composition in wt.% (0.12O, 0.18 Fe, 0.07 C, 0.04 N, and 0.01 H) in the initial recrystallized and ultrafine-grained states. The structure was formed by SPD using the ECAP method [9]. Two types of UFG-titanium states were studied, the first – following ECAP (hereinafter referred to as NS-0) and the second – after ECAP with a subsequent large volume plastic deformation (referred to as NS-1).

Plastic deformation of the surface was performed by the ultrasonic method. Ultrasonic surface modification of materials aimed at improving their performance is well known [10–15], and is widely used in production practices. The

Institute of Strength Physics and Materials Science; Siberian Physicotechnical Institute at Tomsk State University. Translated from Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika, No. 9, pp. 45–50, September, 2000. Original article submitted June 27, 2000.

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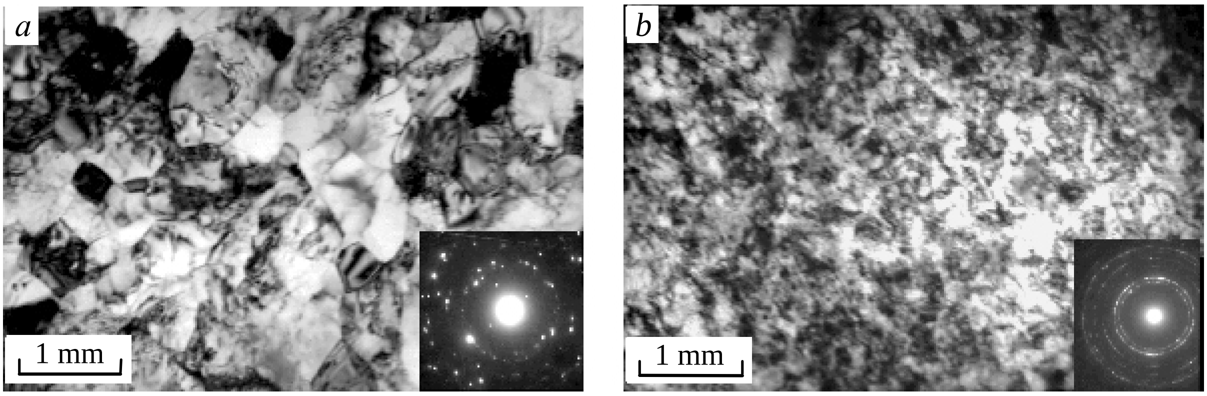

Fig. 1. Microstructure of subsurface layers of nanostructured titanium before (*a*) and after (*b*) ultrasonic modification.

ultrasonic treatment of the specimen surface was done in an ultrasonic Moskit setup [16]. The working tool in this machine is a hard-alloy rod measuring 5 mm in diameter and having a spherical tip. An ultrasonic generator excites ultrasonic-frequency oscillations in the rod, which results in deformation of the surface. Due to the amplitude and stepwise output power control provided for in the machine design, the surface treatment can be varied within a wide range of modes.

In this work we used flat specimens to investigate the mechanical and fatigue properties. To this end, an *ad hoc* method was developed for ultrasonic treatment of flat specimens 0.2–0.4 mm in thickness in the Moskit machine. The specimen was placed onto a specially manufactured platform, its longer side parallel to the direction of reciprocating motion of the tool. The ultrasonic treatment resulted in "cross-hatching" of both sides of the specimens in the longitudinal direction by the deformation stripes with a step of 0.2 mm.

The surface layer structure was examined by transmission electron microscopy in an EM-126K device. Microhardness measurements were made using a PMT-3M microhardness tester with an indentor load of 0.1 kg. Tensile testing was carried out at room temperature at a strain rate of $10^{-3}·s^{-1}$. To measure the microplastic deformation and to determine the macroscopic elasticity limit σ'' under static loading, use was made of the loading–unloading technique at uniform bending of a flat specimen measuring $46 \times 6 \times (0.2–0.3)$ mm along the mandrel of a preset radius [6, 7]. Fatigue tests were performed in the same machine with constant-sign bending from zero at a frequency of 1.7 Hz.

EXPERIMENTAL RESULTS

A detailed investigation of microstructure of titanium specimens in the initial CG (IT) and UFG (NS-0 and NS-1) states was made earlier in [5–7]. The average grain size in the initial polycrystalline titanium d = 10 mm. The dislocation density typical of annealed recrystallized metals was ~10^{13} m^{-2}. The NS-0 state exhibits a bimodal average grain size distribution (appearing in the electron diffraction pattern as regions of the same contrast), including grains from 0.3–0.35 to 1 mm in diameter. The fraction of coarse grains can be as large as 40%. The dislocation density in coarse grains is comparable with that in the CG state, while in fine grains it is by far higher – up to 10^{14} m^{-2}. SPD (NS-1 state) gives rise to the formation of grains elongated along the direction of deformation, their transverse and longitudinal size being 0.26±0.7 mm and 0.6–1.5 mm, respectively. The dislocation density in most grains was 10^{15} m^{-2}, with occasional grains of low dislocation density.

The ultrasonic treatment of CG and UFG-titanium plates using the above-mentioned procedure resulted in severe buckling of the specimens. Their middle part was invariably warped towards the treated surface, which testifies to the formation of residual compressive stresses in the subsurface layer.

The ultrasonic modification produces the most pronounced changes in the sub-surface layers of CG titanium (IT). The dislocation density grows abruptly and the material exhibits regions with a characteristic grain size of 0.12 mm. The electron microscopy images from the boundaries between these regions are indistinct and smeared (Fig. 1). An analysis of the electron diffraction patterns showed that the misorientation of the adjacent regions ranges from 2 to 40°. These diffraction patterns demonstrate smearing of the reflections appearing as nearly regular rings. This structure is similar to the structure of metals subjected to SPD followed by the formation of a nanostructured state. These changes are observed in the subsurface layer of the material up to a depth of about 100 mm.

TABLE 1. Properties of Coarse-Grained and Nanostructured Titanium before and after Ultrasonic Treatment

Material	State	H_μ, MPa	σ'', MPa	$\sigma_{0.2}$, MPa	σ_{UTS}, MPa	δ, %
IT BT1-0	initial	1800	110	370	470	21.9
d = 10 mm	UST-modified	2750	110	480	530	13.4
NS-0	initial	2300	210	650	710	18.9
	UST-modified	3170	150	650	710	13.2
NS-1	initial	3090	153	1040	1090	5.0
	UST-modified	3515	110	1020	1060	5.8

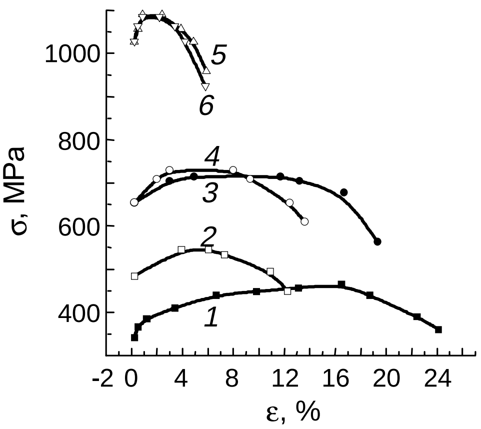

Fig. 2. Tensile curves of IT (*1, 2*), NS-O (*3, 4*), and NS-1 titanium (*5, 6*) before (*1, 3, 5*) and after (*2, 4, 6*) ultrasonic modification.

Under supersonic treatment, the microstructure of the initial UFG titanium (NS-0) is still further refined, with the characteristic grain size being as small as 0.12 mm. It is clear from the microphotographs of the longitudinal section of the NS-1 titanium specimens that upon ultrasonic treatment the grains retain their elongated shape, however, breaking into smaller fragments.

Table 1 shows the results on microhardness, Hm, macroscopic elasticity limit, σ'', ultimate tensile strength, UTS, yield strength, $\sigma_{0.2}$, and relative elongation to fracture, δ, of the NS-0, and NS-1 titanium specimens prior to and following the ultrasonic modification.

It was found that following ultrasonic treatment all specimens studied showed increased microhardness. The maximum increase (by a factor of 1.5) was observed for the initial IT titanium, while the NS-1 state was least affected. The changes caused by UST in the CG-titanium differ from those in the NS titanium (Fig. 2). Following UST, the ultimate tensile strength and yield strength in CG titanium increase, while the total elongation to fracture sharply decreases. No pronounced changes in the yield strength or tensile strength are observed in the NS-0 and NS-1 states, while the relative elongation to fracture decreases in the NS-0 specimens and remains virtually the same in the NS-1 titanium. Note that upon UST, the behavior of the flow stress curves from all specimens studied showed no changes.

The macroscopic elasticity limit, σ'', characterizing the law of plastic deformation buildup in the microdeformation zone, remained the same in the IT specimens treated by ultrasound, while the same treatment of the NS-0 and NS-1 titanium resulted in a change of its σ''.

It was reported earlier [5, 7] that the highest values of fatigue limit were observed in the NS-1 titanium. Fatigue studies were, therefore, performed on the NS-1 specimens only. The limited fatigue strength of the NS-1 state specimens following 10^6 cycles of loading according to the scheme proposed in this work was found to be 520 MPa. To reduce the time of testing, the maximum stress per cycle was varied from 520 to 625 MPa. The results of these tests are summarized in Table 2. It is clear that durability of the material suffered virtually no changes upon UST. These data suggest that the limited fatigue strength did not undergo any marked changes either.

TABLE 2

State	Initial maximum stress per cycle, σ_{max}	Number of cycles to fracture, *N*
NS-1	520	$>10^6$
	550	$9{\cdot}10^4$
	625	$6{\cdot}10^4$
NS-1 after UST	560	$12{\cdot}10^4$
	580	$6.5{\cdot}10^4$
	590	$3{\cdot}10^4$

DISCUSSION OF RESULTS

The experimental results cited in the previous section show that UST most severely affects the properties of the initial CG IT titanium. The diffraction analysis and the results summarized in Table 1 allow us to compare the efficiency of affecting the material properties demonstrated by surface modification by UST and that of SPD via ECAP. Both methods result in a differently changed microstructure. The ECAP method forms an UFG state with prevailing high-angle grain boundaries. Though UST does cause refining of the grain size, and misorientations of the adjacent regions tend to approach large values (up to 40º), the boundaries between these regions are, nevertheless, so smeared that it is hard to identify individual grains, which are rather cells of a sort. We refer to this as the cellular-granular structure. Evidence of the differences between the structures formed by UST and ECAP also comes from the data on the macroscopic elasticity limit σ''. This is a structure-sensitive characteristic. ECAP gives rise to a nearly twofold increase in σ'', while UST has no effect on its value.

The change in microhardness is higher in the initial IT titanium modified by UST than by ECAP. The major effect is likely to belong to the material structure components (grains or cells). One cannot, however, rule out the differing distribution and the value of average residual stresses.

When analyzing the mechanical properties, it is necessary to take into account that in the case of UST it is only the subsurface layers that are modified, the bulk of the material remaining unchanged. Considering that the depth of the modified layer is about 100 mm given the total thickness of the specimen of 400 mm, then even for the equal efficiency of ECAP and UST the latter technique will cause half as small changes in ultimate tensile strength and yield strength. The data presented in Table 1 support this in general. At the same time, the relative elongation to fracture tends to be higher after UST than ECAP.

Thus, we may conclude that even though UST of the surface of CG-titanium specimens (IT) gives rise to the formation in the subsurface layers of a microstructure differing from that produced by ECAP, its efficiency of influencing the mechanical characteristics under study compares well with the formation of a nanostructured state in the material.

When, however, we use UST to modify titanium specimens with the submicrocrystalline structure already formed by ECAP, then the properties vary in a manner somewhat different from those described above. Since the NS-1 state is in fact the NS-0 state that underwent severe plastic deformation, we can compare the effects of UST and volume deformation on the properties of the NS-0 titanium (Table 1). Both methods resulted in approximately the same changes in microhardness and macroscopic elastic limit. Whereas UST virtually does not influence the elastic limit and tensile strength, the volume deformation increased those characteristics considerably. Its influence on the relative elongation to fracture was also more pronounced. That is to say that UST of UFG NS-0 titanium exerts the most influence on the material properties controlled by the state of the surface. The same refers to ultrasonic modification of the NS-1 titanium specimens.

From the data reported in [5–8] one can draw the conclusion that fatigue properties of both CG and NS states somehow correlate with strength characteristics, i.e., the higher the yield strength the higher the fatigue limit. Proceeding from this, one would expect that durability of NS-titanium under cyclic loading and its fatigue limit do not change essentially as a result of UST, because the flow curve characteristics suffered virtually no changes either. This assumption was supported experimentally (Table 2).

SUMMARY

The ultrasonic modification of both coarse-grained and NS-titanium gives rise to a change in their structure and increases the dislocation density. A peculiar granular-cellular structure with a characteristic size of structural units of about

0.12 mm is formed in the subsurface layer. This structure differs from that produced by ECAP in which the majority of boundaries are high-angled. The change in the subsurface layer microstructure caused by UST influences the physico-chemical properties of titanium. In all states, there is an increase in microhardness, with the most pronounced effect observed in the coarse-grained titanium. The macroscopic elastic limit, σ'', behaves under UST in the same fashion as in cold rolling or extrusion. The tensile strength of CG titanium (IT) is increased while the elongation to fracture decreases. No marked variation in strength characteristics of the UFG titanium is observed.

The above-mentioned facts permit us to make the following conclusion. Plastic deformation of IT has nearly exhausted its potential resources of improving mechanical properties through the formation of nanostructured state. That is why additional modification of its subsurface layers by UST exerts but a weak influence on the properties in question. There is, however, reason to believe that a subsequent thermal treatment would lead to improved mechanical properties.

The work was performed with financial support from the Russian Foundation for Basic Research, grant No. 00–02–17911, the Ministry of Education-project No 202.03.01.19, and Los Alamos National Laboratories.

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