Strength and ductility-related properties of ultrafine grained two-phase titanium alloy produced by warm multiaxial forging

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A B S T R A C T

The most important room temperature mechanical properties of two-phase Ti–6Al–4V alloy with ultrafine grained microstructure were studied in the present work. Bulk preforms of the alloy with ultrafine grained microstructure were produced by warm multiaxial forging. The final structure consisted of alpha and beta particles with size of 150–500 nm depending on deformation temperature. The mechanical properties of ultrafine grained material were carried out in comparison with conventionally heat-strengthened condition of the alloy. Room-temperature strength of the ultrafine grained material was found to be 16–33% higher than that of the heat-strengthened alloy. However, ductility-related properties including tensile elongation, impact toughness, fatigue crack growth resistance and fracture toughness noticeably decreased with decreasing grain size. The efforts to increase ductility the ultrafine grained alloy by annealing was restricted by rather intensive softening of the material. Considerable enhancement of ductility of the alloy with a bi-modal microstructure consisting of large primary alpha in UFG alpha/beta matrix was shown.

Keywords:
Titanium alloy
Ultrafine grained microstructure
Mechanical properties
Strength
Ductility
Crack growth resistance

1. Introduction

Two-phase Ti–6Al–4V alloy is one of the most widely used titanium alloys due to an excellent combination of mechanical properties, corrosion resistance and workability. Considerable enhancement in the mechanical properties of the alloy can be attained by microstructure refinement to the ultrafine grained (UFG, grain size less than 0.5–1 μm) regime [1–4] that, in turn, may broaden application of the alloy. Improvement in the mechanical properties through grain refinement has contributed to the rapidly expanding field of materials engineering, in which UFG materials are produced using severe plastic deformation (SPD). Well-established SPD techniques include high-pressure torsion [5], equal channel angular extrusion [6] twist extrusion [7] and multiaxial forging (MAF) [8]. An advantage of these methods is the almost unlimited ability to accumulate deformation while maintaining the initial shape and size of the specimen.

Generally, it is well established that strength, hardness, high-cycle fatigue resistance and superplastic properties are improved by grain size reduction [1,2]. However considerable microstructure refinement usually results in a decrease in ductility-related properties (tensile elongation, impact toughness, crack propagation resistance) [1,2] due to low ability of such materials to strain hardening. Therefore, additional investigations are needed in order to evaluate the feasible area of application of UFG two-phase titanium alloys and to improve ductility of such materials.

The aim of the present work was to study the most important room temperature mechanical properties of two-phase Ti–6Al–4V alloy with UFG microstructure produced by warm multiaxial forging, and to develop approaches to further improvement of ductility-related properties.

2. Material and experimental procedures

The material used in this study was an alpha/beta titanium alloy Ti–6Al–4V with a measured composition (in weight pct.) of 6.3 Al, 4.1 V, 0.18 Fe, 0.03 Si, 0.02 Zr, 0.01 C, 0.18 O, 0.01 N. It was received in the form of a hot-rolled 40-mm diameter bar with a beta-transus temperature (at which alpha + beta → beta) of 980 °C.

Ultrafine grained microstructure in the alloy was produced by warm MAF consisted of sequential compression of a sample along variable directions as it is schematically shown in Fig. 1a. The alloy initial alpha′ martensite was produced by water quenching from the beta region (1010 °C) [8]. The size of alpha and beta particles in the alloy depends on deformation temperature (if other conditions are equal) (Fig. 1b). To attain the smallest grain size, deformation temperature should be reduced as much as possible. However, the workability of a coarse grained alloy at low temperature is limited

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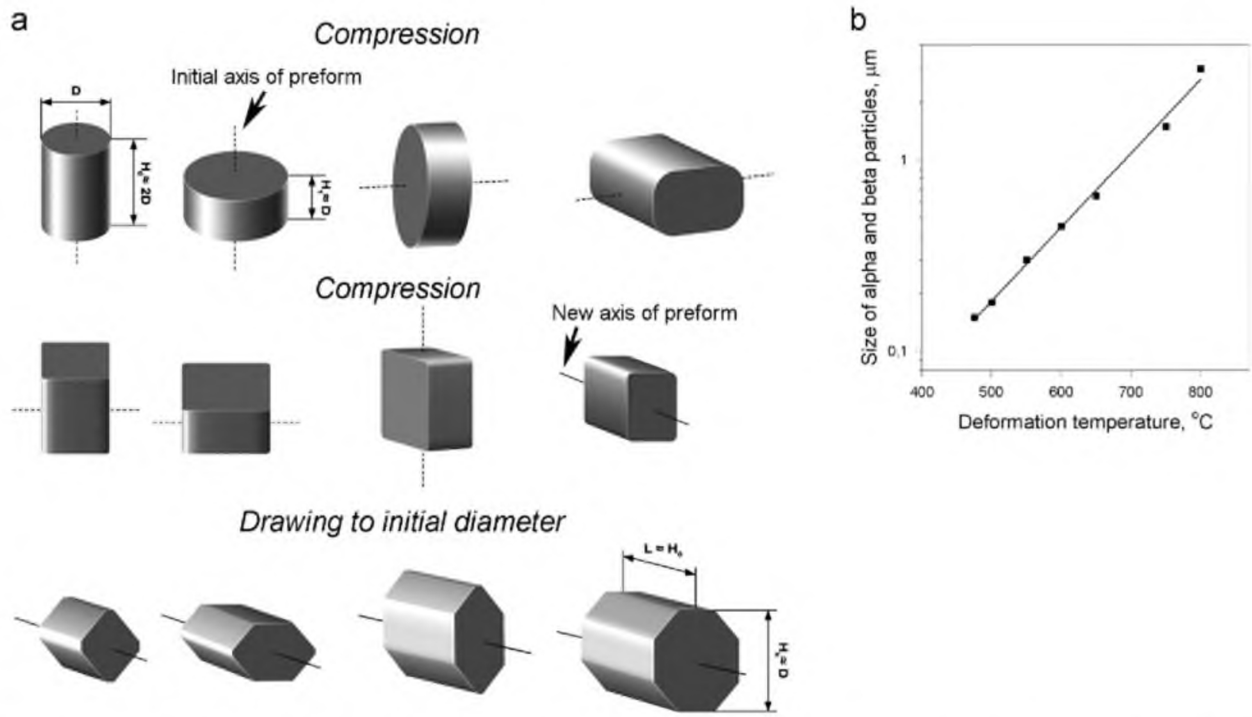

Fig. 1. Scheme of multiaxial forging (the loading direction is vertical) (a) and size of alpha and beta particles as a function of deformation temperature for two phase Ti–6Al–4V alloy for the strain rate ~10^{-3} s^{-1} (b).

whereas large deformation is required to produce samples with a homogeneous UFG microstructure. Therefore, the process usually was started from 700 °C to form a homogeneous fine grained microstructure. As workability of the sample was improved due to microstructure refinement, the deformation temperature in the next stage was decreased by 50–100 °C. The minimum attained temperature of MAF was 475 °C at ~10^{-3} s^{-1} (that gives the size of alpha and beta particles of about 150 nm), however specimens with larger grain size (up to 500 nm) was also produced to find the optimum deformation temperature–properties relationship. To attain a homogeneous UFG microstructure in a sample four–five stages (considering that in Fig. 1a one stage is shown) at each temperature are required. Deformation at each temperature was carried out under an isothermal condition at a strain rate of about 10^{-3} s^{-1}.

High strength UFG conditions were compared with a conventionally heat-strengthened microcrystalline (MC) condition. The heat strengthening consisted of water quenching from 945 °C and annealing 3 h at 500 °C [9].

Tensile tests were conducted at room temperature at the constant cross-head speed of 1 mm/min. Cylindrical specimens with the gauge dimensions of 3 mm in diameter and 18 mm in length, and flat specimens with the gauge dimensions of 1.5 mm × 3 mm and 16 mm in length were used for the tensile tests. The experimental errors did not exceed ±4.5% for the strength characteristics and ±7% for the ductile characteristics.

The impact toughness of the alloy was evaluated by the Charpy impact test using specimens measuring 10 mm × 10 mm × 55 mm with U-notch, V-notch and fatigue precracked V-notch.

The UFG alloy was subjected to a two stages annealing thus trying to improve ductility. The annealing included 1-h soaking within 550–650 °C followed by water quenching thereafter each specimen was additionally soaked at 480 °C for 2 h. The first stage was aimed at the formation of a stable substructure with decreased dislocation density. A somewhat nonequilibrium state was fixed by water quenching from the temperature of the first annealing. Therefore, some effect of aging due to the formation of segregations in grain boundaries could be expected as a result of the second soaking stage.

The microstructure was examined by using optical microscopy, a JEOL JEM-2100FX transmission electron microscope (TEM) and a FEI Quanta 600 FEG scanning electron microscope (SEM). Specimens for TEM were mechanically thinned to ~100 μm and then twin-jet electro-polished at 30 V and −40 °C using a solution consisting of perchloric acid, methanol and butanol at a ratio of 1/6/10. The dislocation density was measured by counting the individual dislocations in grain/subgrain interiors on at least six arbitrarily selected typical TEM images for each sample at the magnitude 150K×.

3. Results and discussion

3.1. Mechanical properties of the UFG Ti–6Al–4V alloy

The microstructures of the Ti–6Al–4V alloy in the UFG and MC conditions are shown in Fig. 2. The microstructure of the UFG alloy consisted of alpha and beta particles with a mean grain size of 150 nm (last deformation temperature was 475 °C) (Fig. 2a). The MC condition consisted of primary alpha globules in the beta transformed matrix (Fig. 2b). During cooling from the top of the alpha/beta field the beta phase decomposed into a mixture of very fine (less than 1 μm) alpha and beta lamellae. The size of alpha globules was about 6 μm while the average space between adjacent globular alpha particles was about 18 μm. The microstructure and properties of a heat strengthened Ti–6Al–4V alloy are well known and are summarized in a number of publications [e.g. 9,10].

The effect of grain size on strength and ductility of the alloy is shown in Table 1. Decrease in a grain size from 500 nm to 150 nm resulted in a considerable increase in strength and some decrease in total elongation. The alloy after MAF at 475 °C showed the maximum strength in 1400 MPa that corresponds to the highest level of strength of titanium alloys [9,10]. However the decrease in deformation temperature during MAF was associated with

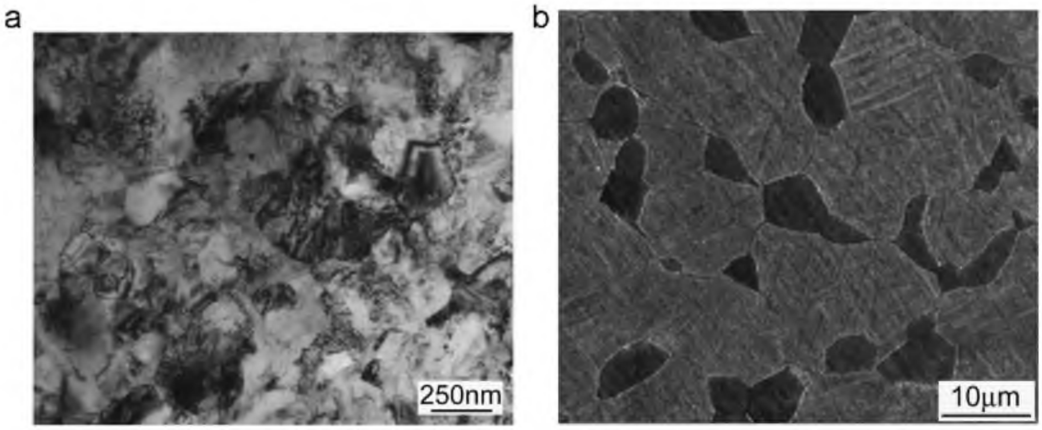

Fig. 2. Microstructure of Ti–6Al–4V alloy in the UFG (a) and MC (b) conditions: (a) TEM image; (b) SEM image.

Table 1
Tensile properties of Ti–6Al–4V alloy in UFG and MC conditions.

Condition of the alloy (grain size)/shape and size of the gauge	UTS, MPa	YS, MPa	TE, %	AR, %
MC (10 μm)/cylindrical Ø 3 mm	1050	960	9.0	32.0
UFG (150 nm)/flat 1.5 mm × 3 mm	1400	1280	6.8	–
UFG (300 nm)/cylindrical Ø 3 mm	1300	1210	7.1	60.0
UFG (500 nm)/cylindrical Ø 5 mm	1220	1190	9.5	56.5

UTS: ultimate tensile strength; YS: yield stress; TE: total elongation; AR: area reduction.

obvious difficulties caused by low workability and very high strength of the alloy. Deformation in a lesser critical temperature interval 550–600 °C also resulted in a high strength which is still noticeably higher than that of the MC condition.

Meanwhile, both the total elongation and the uniform deformation of the UFG condition were rather small. The engineering stress–strain curves of the UFG alloy with a grain size of 300 nm and the MC condition are shown in Fig. 3a. The UFG alloy showed smaller total elongation as well as deformation before necking even in comparison with the heat strengthened MC condition.

At the same time, after the strain localization (the uniform strain of the UFG alloy was about 0.5%), when the plastic deformation has become concentrated in the neck, the specimen has showed at additional ~6.5% elongation. Since the volume involved in the plastic deformation after the strain localization was considerably smaller than the nominal size of the gauge, the total elongation did not reflect properly ductility of the UFG condition. Meanwhile ductility of the UFG material in terms of the reduction in area was almost a twice as high as that for the MC alloy; 60% vs. 32%, respectively (Fig. 3b, Table 1). Hence, under some "soft" deformation schemes (rolling, compression) the UFG condition can manifest quite a high ductility although ductility under tensile stresses is low.

Such behaviour is a common feature of UFG materials irrespective of the method by which the structure is produced. Earlier necking and reduced tensile elongation, relative to their microcrystalline counterparts, has been observed in UFG materials produced by severe plastic deformation, by consolidation of nano-size particles and by electrodeposition [1–3,11]. The plastic instability in UFG materials manifests itself either as shear bands or by "early" necking is usually attributed to the lack of an effective hardening mechanism [1,2,11]. Indeed, the number of dislocations that become stored into the microstructure during the tensile test is no longer significant to make a difference over the already-high existing dislocation density. As a result, the flow stress does not increase remarkably during the tensile test.

Another manifestation of the stress localization in UFG materials is associated with the shrinkage of plastic zone in front of the crack tip [12,13]. The radius of the plastic zone can be found as [12]: $r_y = (1/2\pi)(K/\sigma_y)^2$, where K denotes the stress intensity factor in the crack tip and σ_y is the yield stress of the material. Small grain size and a very high density of dislocation result in increased yield strength of UFG materials whilst the fracture toughness trends to decrease with microstructure refinement [1].

Structurally, the size of the plastic zone in front of the crack tip is related to the grain size. When grains are equal to or smaller than the plastic zone, the spreading of the plastic deformation is associated with the passing strain through grain boundaries. Obviously, the presence of a number of boundaries restricts deformation volume in the vicinity of the crack tip thereby decreasing crack propagation energy [12]. In addition, the path of the crack propagation in the case of UFG materials should be much smoother in comparison with coarse grained counterparts [1].

These assumptions are corroborated by the results of impact toughness and crack growth resistance. The Charpy impact tests of specimens with different shapes of notches showed much more pronounced propensity of the UFG material to the notch acuteness. If in the case of the MC condition the ratio of the impact toughness between the rounded U-notch and the very sharp fatigue pre-cracked notch was approximately 2, for the UFG condition this ratio was about 4.5 (Table 2). Therefore, when the nucleation of a crack is needed, the impact toughness of both conditions does not differ drastically. However, when the crack is already formed and all the energy spends for the crack propagation, the difference becomes very large.

Table 2
Impact toughness of Ti–6Al–4V alloy in various conditions.

Condition	Impact toughness, MJ/m²		
	U-notch	V-notch	Fatigue precracked notch
UFG (500 nm)	0.37	0.18	0.08
UFG (500 nm) + annealing at 625/650 °C for 1 h	–	–	0.12/0.17
MC (10 μm) heat-strengthened	0.45	0.41	0.24
Bi-modal	0.43	0.24	0.13

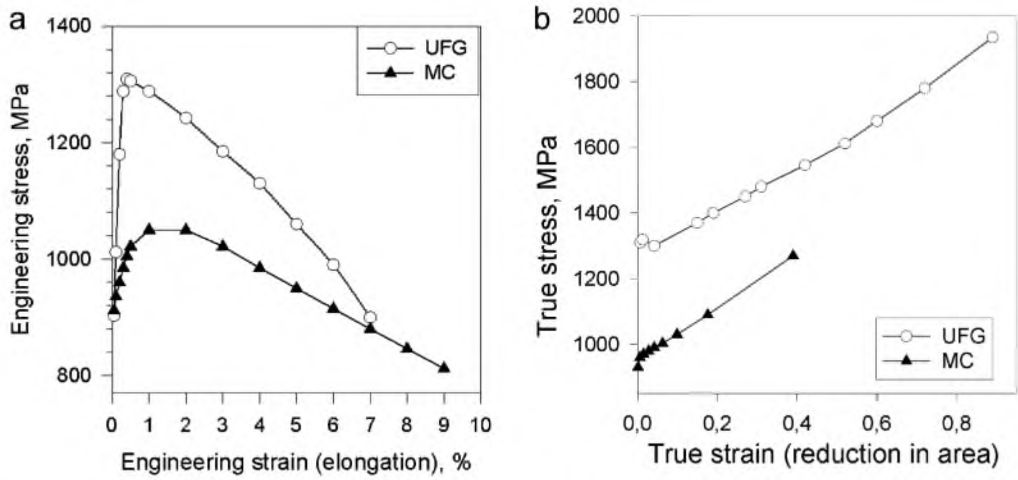

Fig. 3. Engineering (a) and true (b) stress–strain curves for the UFG Ti–6Al–4V alloy with a grain size of 300 nm in comparison with the MC condition.

Indeed, the fracture toughness of the UFG condition was found to be 30.5 MPa m$^{1/2}$ that is lower than the typical values for this alloy (which falls in the range 33–110 MPa m$^{1/2}$ [9]). However, the fracture toughness is quite reasonable considering relatively high strength and low ductility of the UFG material compared to coarse-grained counterparts.

At the same time, the rate of fatigue crack propagation in comparison with the published data [14] for Ti–6Al–4V alloy with a fine equiaxed (d = 2 μm), coarse equiaxed (d = 12 μm) and coarse lamellar microstructure (Fig. 4) does not show a drastic decrease. The rate of fatigue crack propagation of the UFG condition is close to or even better than that of the alloy with fine equiaxed microstructure.

3.2. Effect of annealing on strength and ductility of the UFG Ti–6Al–4V alloy

The most obvious way to increase the plastic zone in the vicinity of the crack tip and thereby to increase the energy of crack propagation is associated with the annealing of material. The annealing below the recrystallization temperature is likely to increase grain size insignificantly, but at the same time decrease dislocation density and thereby increase ability of the material to strain hardening.

Two-stage annealing had a positive effect on the total elongation, even if the ductility was accompanied by material softening (Fig. 5b). The total elongation increased after annealing above 600 °C that was associated with a pronounced decrease in the dislocation density (Fig. 5b). After this annealing, the ultimate tensile strength was maintained above 1350 MPa that is still quite high when compared to the MC condition. Further rise of the annealing temperature resulted in faster decrease in strength due to grain size coarsening and dislocation density reduction (Fig. 5a–d); so that after the annealing at 650 °C (and further soaking at 480 °C for 2 h), the strength of the material corresponded to the level of conventionally heat strengthened Ti–6Al–4V alloy. Obviously, going down significantly in strength is not an option since the main point of the microstructure refinement is to raise the yield strength. This fact reduces the actual temperature interval of heat treatment considerably, and therefore limits the possibility to improve ductility of the material without the strength loss.

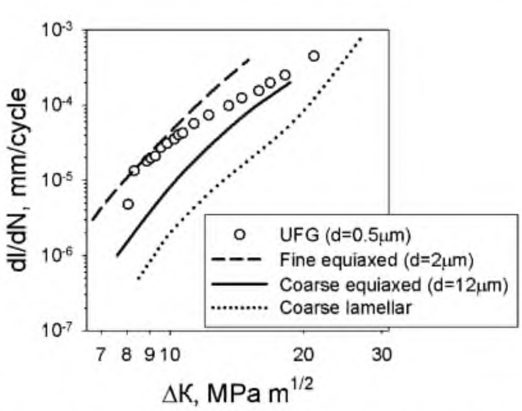

Fig. 4. Fatigue crack propagation rate in the UFG Ti–6Al–4V alloy with grain size of 500 nm in comparison with various condition of the alloy [14].

It should be noted that the present results do not fully agree with the data reported in [15] by R. Valiev with co-authors. They found that annealing of a UFG Ti–6Al–4V ELI alloy (the grain size was estimated to be 300 nm) at 500 °C for 2 h increased both the strength and the total elongation by 12%, and the uniform deformation increased by 1%. Such a pronounced effect of annealing in the case of [15] can be attributed to (i) higher purity of the alloy that may facilitate the rearrangement of dislocations into stable (sub)boundaries even at relatively low temperature, and (ii) higher dislocation density as well as sharper material texture since the alloy was subjected to multi-cycle extrusion at 300 °C with the total elongation λ = 4.2 following ECAP in a die set with channel intersection angle φ = 120° at 600 °C via B_c route. The last factor can result in the intensive formation of transversal (sub)boundaries in elongated grains during annealing thereby combining the processes of microstructure refinement and the decrease in dislocation density.

Use of a coarser-grained alloy with the initial grain size of 500 nm along with higher annealing temperature of 625 °C or 650 °C increased the value of impact toughness of fatigue pre-cracked notch specimens approximately by a factor of two (Table 2). However, the strength of such material was very close to that of the usual heat strengthened MC condition.

Therefore, annealing is able to increase ductility-related properties of UFG materials, but this improvement is quite limited due to the corresponding grain growth and the decrease in strength. Meanwhile, it is worth noting that quite a similar balance between strength and ductility can be obtained by higher temperature MAF solely, without any additional annealing (Table 1 and Fig. 5).

3.3. Enhancement of ductility of a high-strength Ti–6Al–4V alloy

Based on the results obtained, it can be concluded that ductility-related properties are a weak point of the UFG materials. This problem is closely related to the lack of work hardening and, as a result, the fast loss of plastic flow stability, early necking and

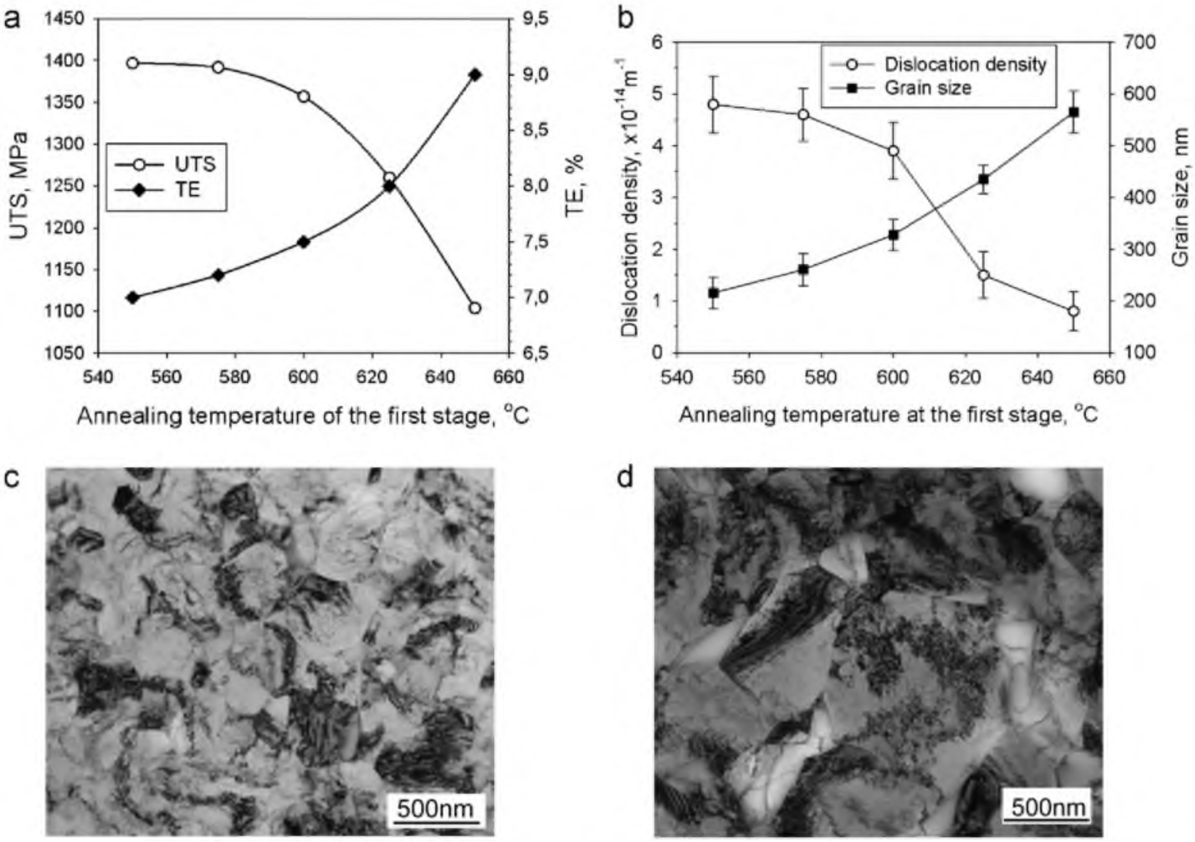

Fig. 5. Effect of two-stage annealing on grain size (a) and UTS and TE (b) of UFG alloy with the initial grain size of 150 nm (a). Annealing temperature of the first stage is shown in the plots; then each specimen was additionally soaked at 480 °C for 2 h. Duration of the first stage of annealing was 1 h. Typical microstructures of the alloy after annealing at 550 °C or at 650 °C for 1 h at the first stage and then at 480 °C for 2 h at the second stage in both cases are shown in (c) and (d), respectively.

low tensile elongation. Annealing has quite a limited ability to the mechanical properties improvement because any noticeable increase in ductility is associated with decrease in strength.

To increase the ability of UFG materials to work hardening a promising approach was proposed by Wang and Ma [16,17]. It consisted of formation of a bi-modal microstructure with relatively coarse recrystallized grains in nanostructured matrix.

In the two-phase Ti–6Al–4V alloy, the similar microstructure can be produced quite easily by means of MAF. However, the initial condition in this case would be obtained by air cooling from the top of the two-phase alpha/beta area. Such microstructure is typical for hot rolled or hot extruded industrial bars and consists of primary globular alpha and lamellar alpha/beta (Fig. 6a) [9,10]. During MAF, the lamellar constituent refined forming UFG grains while the interior of alpha globules after deformation consisted of mainly substructure (and almost no high-angle boundaries) (Fig. 6b and c). The mean grain size after the MAF at 550 °C was found to be ~300 nm. The volume fraction of the UFG grains in the microstructure was approximately 0.4.

The tensile test of specimens with such bi-modal microstructure showed considerable enhancement of both strength and ductility including total elongation and uniform elongation. In addition, the stage of work hardening was observed on the stress–strain curve which corresponded to the specimen with the bi-modal microstructure (Fig. 7a). The examination of surface of the tensile specimen with bi-modal microstructure in the vicinity of fracture showed two types of deformation relief: alpha grains with multiple sliding and areas with a very fine deformation relief typical of fine-grained microstructure (Fig. 7b).

The impact test also gave quite attractive results; the impact toughness of specimens with the bi-modal microstructure was found to be higher than that of the UFG condition in all cases (Table 2). For U-notch specimens the impact toughness of the bi-modal microstructure was very close to that of the MC condition.

The fracture toughness of the alloy with bi-modal microstructure increased in comparison with the uniform UFG microstructure to 39 MPa m$^{1/2}$. The absolute value is still not high enough in comparison with the typical values of fracture toughness; however, quite high increment by 30% is very promising results itself.

The last operation of MAF was drawing (Fig. 1a) that formed metallographic texture in the specimen. The axis of the tensile specimens coincided with the metal flow direction (horizontal direction at the Fig. 6b) while the direction of crack propagation was perpendicular to the metal flow direction (vertical direction in Fig. 6b). Obviously, the texture contributed into the increased ductility and the crack growth resistance. However, the evaluation of texture intensity as well as microstructure and mechanical properties homogeneity in UFG Ti–6Al–4V alloy after MAF [8,18] indicates that the effect of those factors should not be very high.

Therefore, formation of a bi-modal microstructure consisting of relatively large grains in UFG matrix can enhance ductility-related properties (particularly uniform deformation, total elongation and crack resistance), corroborating the above cited assumption of Wang and Ma [16,17]. Appearance of the stage of work hardening at the stress–strain curves indicates the enhanced ability of the material with bi-modal microstructure to the accumulation of extra dislocations during tensile test. Deformation relief (Fig. 7b) implies that primary alpha globules have a great contribution in this process. Due to the lack of developed substructure and high angle boundaries within alpha particles, they can act as places for dislocations storage. The presence of large grains in the microstructure can also either increase the length of crack path or blunt the

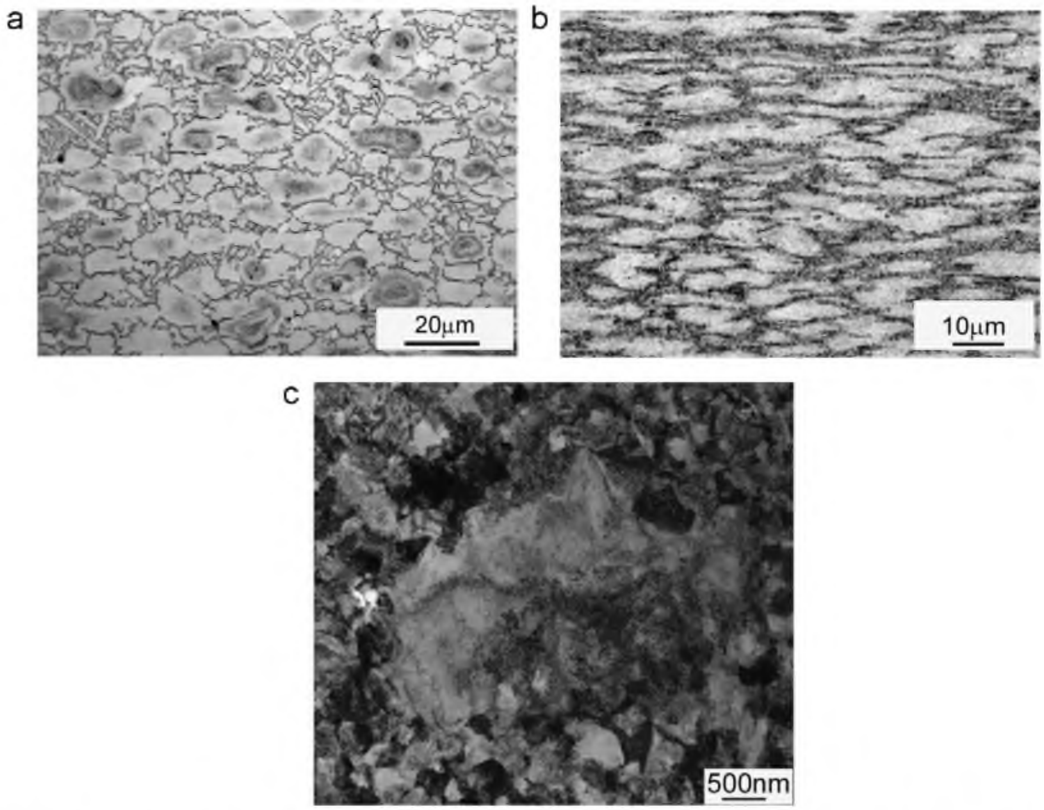

Fig. 6. Microstructure of Ti–6Al–4V alloy with a bi-modal microstructure before (a) and after MAF at 550 °C (b and c); (a and b) SEM images; (c) TEM image.

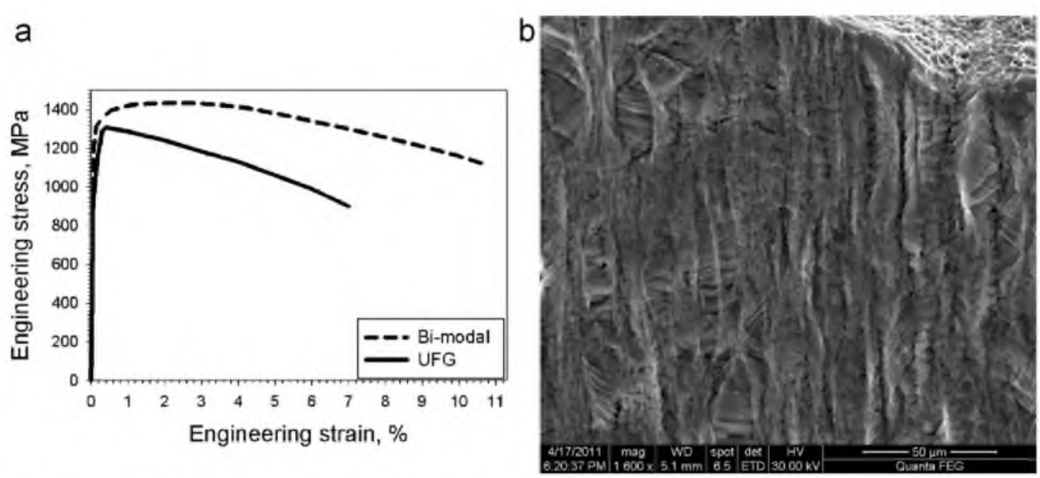

Fig. 7. Engineering stress–strain curves of the alloy with the bi-modal and the MC microstructures (a) and SEM image of deformation relief after tensile test in the vicinity of the fracture of the specimen with the bi-modal microstructure (b).

crack tip thereby increasing the energy of fracture in comparison with the uniform UFG microstructure (Table 2).

However, further investigations are needed to clarify the features of the mechanical behavior of two-phase titanium alloys with a bi-modal microstructure.

4. Summary

Mechanical properties of two-phase Ti–6Al–4V alloy in the ultrafine grained condition produced by multaxial forging and in the heat strengthened microcrystalline condition were studied. The strength of the UFG condition was found to be up to 33% higher than that of the MC condition (1400 and 1050 MPa, respectively). However, ductility-related properties including tensile elongation, impact toughness and fracture toughness decreased with decreasing the grain size. Any noticeable increase in ductility caused by annealing was accompanied by the softening of the material to the level of strength of the conventional heat strengthened MC condition. The formation of a bi-modal microstructure consisting of relatively large grains in UFG matrix enhanced ductility-related properties by 20–60% in comparison with the uniform UFG microstructure without the strength loss.

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