Microstructure and Tensile Properties of Cost-Efficient Thermally Hardenable $\alpha + \beta$ Alloys of Ti–Al–Mo–Fe and Ti–Al–Mo–Cr Systems

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Received: 10 August 2020 / Revised: 12 November 2020 / Accepted: 20 November 2020 / Published online: 2 December 2020
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Abstract

Four new compositions of titanium alloys of Ti–Al–Mo–Fe and Ti–Al–Mo–Cr systems with content of β -phase stabilizing elements in a range of 3.8–9.8 wt.% (in terms of “molybdenum equivalent” C_{Mo}) were studied. The alloys were melted by single-melt electron-beam cold hearth technique, thermomechanically processed, and subjected to conventional (in furnace) or continuous rapid (resistant heating by electric current) heat treatments with different regimes. The microstructure, phase composition, and mechanical properties both in as-deformed state and after heat treatments were studied in detail. It is found that the proposed thermomechanical processing allows to obtain a transformed microstructure characterized by nearly equiaxed α -phase particles with relatively small aspect ratio and weak crystallographic texture of a basal type. Subsequent annealing in the $\alpha + \beta$ field led to completion of the $\alpha + \beta$ microstructure transformation; a microstructure of globular type was obtained in the low-alloyed ($C_{Mo} = 3.8\%$) material. The phase transformations at various stages of strengthening heat treatments and their influence on the final microstructure and properties were studied, depending on the content of β -stabilizing elements and processing route. Particular attention was paid to the influence of the content of β -stabilizing element on the mechanical properties, as well as to the difference between the alloying systems with iron or chromium. It is shown that the alloys of proposed compositions provide high strength and reliability; they are competitive with conventional commercial alloys in terms of the balance of mechanical properties in hot deformed, annealed, and thermally strengthened states. Strengthening treatment based on rapid heating allows to achieve strength above 1500 MPa with sufficient ductility.

Keywords Cost-efficient titanium alloys · Microstructure · Phase composition · Heat treatment · Mechanical properties · High strength

Abbreviations

EBCH	Electron-beam cold hearth technique of melting
T_β	Temperature of $\alpha + \beta_0 \rightarrow \beta$ phase transformation completion (beta transus)

C_{Mo}	“Molybdenum equivalent”—expresses total content of β -phase stabilizing alloying elements in Ti-based alloy via equivalent amount of Mo
STA	Conventional strengthening heat treatment based on solid solution treatment (furnace annealing), water quenching, and final aging
RHT	Rapid heat treatment—special method of thermal strengthening based on rapid (under non-equilibrium conditions) heating to high temperatures, followed by water quenching or another mode of controlled cooling
WQ, FC	Water quenching and furnace cooling—mode of cooling during heat treatments
SEM	Scanning electron microscopy
SE	Secondary electrons (image)
BSE	Backscattered electrons (image)
EDX	Energy-dispersive X-ray spectroscopy (analysis)

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XRD	X-ray diffraction
CSR	Coherent scattering region (X-ray diffraction)
RD	Rolling direction (in X-ray pole figures)
TD	Transverse direction (in X-ray pole figures)
α	Primary α -phase in initial (in as-rolled or annealed) state of alloys
α_I	Remnants of primary α -phase particles after heat treatments in higher-alloyed alloys
α_{II}	Secondary α -phase formed upon strengthening heat treatments of higher-alloyed alloys
$\alpha_{II} + \beta$	Mixture of secondary α -phase particles and β -matrix (after strengthening heat treatments)
α_{gb}	Grain boundary α -phase (α -phase decorating the boundaries of β -grains)
α'	Hexagonal martensite precipitated in low-alloyed titanium alloys upon fast cooling (quenching)
α''	Orthorhombic martensite precipitated in high-alloyed titanium alloys upon fast cooling (quenching)
β_m	Metastable β -phase fixed by fast cooling (quenching)
YS	Yield strength (under tension)
UTS	Ultimate tensile strength (under tension)
$El_{unif.}, El_{tot.}$	Relative uniform and total elongations (under tension), respectively
RA	Reduction in area (under tension)

Introduction

Titanium alloys are very attractive structural materials in various fields of modern industry due to their excellent balance of strength and ductility, combined with low density, high fracture toughness, fatigue properties, corrosion resistance, and non-magnetic properties. These alloys are most widely used in the aerospace, chemical, medical, automotive [1–4], and military [5, 6] industries. The wider use of the titanium alloys is hindered by relatively high price of raw materials and production costs. This problem can be solved by two parallel approaches: reducing the cost of raw materials, and simplifying the methods of melting and subsequent processing of ingots. On the one hand, Montgomery and Wells [6] showed that single-melt electron-beam cold hearth (EBCH) melting followed by direct rolling after annealing is the best approach from the viewpoint of cost efficiency. EBCH method is more preferable compared to the conventional vacuum-arc melting with a consumable electrode (VAR), because the latter even after double melt does not always provide a uniform distribution of alloying elements throughout the ingot, especially for complex alloyed alloys [7, 8]. Also, VAR does not guarantee

the complete removal of dangerous inclusions (so-called low density inclusions and high density inclusions), which drastically reduce the ductility of titanium alloys [7, 9]. The use of EBCH approach allows guaranteed not only to melt refractory elements, but also to refine metal from all inclusions and impurities through the use of an intermediate Cold Hearth, which ensures good results in smelting both commercially pure titanium and complex alloyed alloys [8, 9]. Also, the use of even single-melt EBCH process allows to reduce essentially the cost of the resulting ingots due to the possibility of using up to 100% scrap in the initial charge, as well as eliminating the laborious operation of pressing the consumable electrode [9, 10]. On the other hand, a number of low-cost alloys of mainly β -metastable type (TIMETAL-LCB [8]), as well as a wide range of alloys on the basis of Ti–Al–Fe–Cr system [11–13] have been developed up to date. It should be considered that the advantage of titanium alloys in specific strength over other structural materials is most pronounced exactly in the thermally hardened state. However, the conventional methods of hardening heat treatment (based on heating in furnace under equilibrium conditions) do not allow to use the full potential of heat hardening embedded in alloys by alloying [2, 5]. At the same time, it was earlier shown that the application of special rapid heat treatment (RHT) [14–16] allows to improve strength characteristics of titanium alloys at least by 50% comparing to the conventional strengthening heat treatments [16–19]. In this case, the maximum effect of enhanced strength corresponds not to the alloys of β -metastable type (with general content of β -phase stabilizing elements in terms of equivalent content of molybdenum, so-called molybdenum equivalent C_{Mo} , above 11 wt.% [4]), but to high-alloyed $\alpha + \beta$ alloys of a martensitic type with $C_{Mo} = 8–10$ (wt.%) [16].

Thus, considering all the above mentioned and cost efficiency, the goal of the present study was to evaluate the potential and possibility of the following:

1. Application of single-melt EBCH melting approach followed by relatively simple hot deformation processing;
2. Using cheaper β -alloying elements and/or master alloys of Ti–Al–Mo–Fe(Cr) system with different contents of β -stabilizing elements (C_{Mo} from 3 to 4% up to 8–10%) in order to reduce the cost of alloys;
3. Achieving better balance of high strength and sufficient ductility of the alloys after the conventional (STA) and non-equilibrium rapid (RHT) heat treatments.

Materials and Experimental Procedure

Alloys of Ti–Al–Mo–Fe and Ti–Al–Mo–Cr systems with chemical compositions listed in Table 1 were melted by electron-beam cold hearth method using UE-208 M multipurpose electron-beam unit equipped with VTR-300

Table 1 Chemical composition and some characteristics of studied alloys

Alloys ##	Chemical composition, wt. %						$C_{Mo,(a)}$ wt. %	T_{β} , °C
	Al	Mo	Fe	Cr	Sum of Impurities	Ti		
Ti–Al–Mo–Fe system								
Low-alloyed								
1	3.9	0.9	0.9	...	<0.38	Balance	3.8	945
2	4.3	1.9	1.7	...	<0.41	÷	6.5	920
High-alloyed								
3	5	5	1.66	...	<0.39	÷	9.8	850
Ti–Al–Mo–Cr system								
4	5	5	...	2.7	<0.40	÷	8.6	865

(a) C_{Mo} values were calculated with formulae: $C_{Mo} = 1.0$ (wt%) Mo + 2.9 Fe + 1.6 Cr. Unlike other authors [4, 7], we do not take into account the presence of aluminum (with a minus sign), since in such a case the total amount of β -stabilizers should drop below a critical level (for example, in alloys like VT22 or Ti-5-5-5-3 essentially lower than 11.5% Mo), which would lead to non-fixation of the metastable β -phase upon quenching from temperatures of single-phase β field that does not actually happen

gas-discharge electron guns (Fig. 1) [20, 21]. Commercial pure titanium (Grade 2), A0 grade aluminum, 99% purity molybdenum, technical purity chromium, and ARMCO iron were used as initial components. The compositions of alloys were chosen in order to cover the content of β -stabilizing elements (in terms of molybdenum equivalent) in a range of 4–10 wt.% Mo. Melting was performed in a chamber with residual vacuum not lower than 6×10^{-2} Pa. The cast ingots had dimensions of 100 mm in diameter and about 300 mm in length (Fig. 2a). Melted ingots were machined, their top and bottom ends were cut off (Fig. 2b).

The regimes of subsequent thermomechanical processing and heat treatments were chosen basing on the technological approaches developed in [16, 22–24]. Machined semi-products were subjected to 3D pressing at initial temperature of 1100 °C (Fig. 2c) with total reduction not less than 75%. Pressed semi-products were rolled at 820–840 °C from 40 to 15 mm in 6 passes with rolling direction alternated by 90° (Fig. 2d).

The 15-mm-thick plates (Fig. 2d) were cut into $15 \times 15 \times 160$ mm pieces for subsequent heat treatments summarized in Table 2.

The choice of heat treatments was based on the T_β (beta transus) temperatures and the following aims:

1. To obtain as stable as possible initial state, annealing at temperatures approximately 30–45 °C below the T_β was applied;
2. To find out the potential of conventional (furnace heating) STA heat treatment in strengthening the new alloys;
3. To evaluate potential in obtaining the highest strength by means of special RHT hardening approach.

Microstructure of the alloys at different stages of treatment was studied with Scanning (TESCAN VEGA 3 equipped with EDS micro-analyzer Bruker QUANTAX) and

Fig. 1 UE-208 M multipurpose electron-beam unit

Transmission (JEOL 2000FX) Electron Microscopy. Phase composition and crystallographic texture were examined by X-ray Diffraction analysis at RIGAKU ULTIMA IV diffractometer. Tensile properties were determined according to ASTM E8 standard using cylindrical specimens with gage diameter 4 mm and length 25 mm at INSTRON 3376 unit.

Results and Discussion

Microstructure, Phase Composition, and Crystallographic Texture

In as-rolled condition, the microstructure of all of the alloys consisted of not recrystallized β -phase matrix with α -phase lamellas inside with heavily waved shape, which partially transformed during hot deformation into equiaxed particles

Fig. 2 Typical example of (a) as-melted and (b) turned ingot, (c) hot-pressed preform, and (d) rolled plate of Ti–5Al–5Mo–2Cr alloy

Table 2 Employed heat treatments

Alloy ##	Heat treatments/regimes		
	Annealing	Conventional STA strengthening	Rapid heat treatment
1	900 °C, 2 h, FC	900 °C, 1 h, WQ, 550 °C, 5 h, FC	20 °C/s to 1000° C, WQ, 550 °C, 5 h, FC
2	880 °C, 2 h, FC	880 °C, 1 h, WQ, 550 °C, 5 h, FC	20 °C/s to 950 °C, WQ, 550 °C, 5 h, FC
3	820 °C, 2 h, FC	840 °C, 1 h, WQ, 550 °C, 5 h, FC	20 °C/s to 900 °C, WQ, 550 °C, 5 h, FC
4	820 °C, 2 h, FC	840 °C, 1 h, WQ, 550 °C, 5 h, FC	20 °C/s to 900 °C, WQ, 550 °C, 5 h, FC
Ti–1.5Al–1Fe–7.2Cr [23, 24]			
5	800 °C, 2 h, FC	850 °C, 1 h, WQ, 550 °C, 5 h, FC	...

(Fig. 3a). Subsequent annealing at temperatures of upper section of the $\alpha + \beta$ field (approximately 35 °C below T_β) caused almost complete transformation into microstructure of globular type (Fig. 3b) in the low-alloyed alloy #1. In the alloys with higher content of alloying elements this

transformation into globular microstructure did not complete, and some remnants of previous α -lamellas mainly oriented along rolling direction remained after annealing (Fig. 3b–d), possibly due to relatively lower T_β and hence lower annealing temperatures. The latter fact, together with a

Fig. 3 SEM images (SE) of microstructure of the alloys: (a, b) #1, (c) #2, (d) #3, (e) #4 in (a) as-rolled, and (b–e) annealed conditions

Fig. 4 XRD patterns for alloys #2, #3, #4 in as-annealed state

high content of alloying elements, slows down the diffusion that determine the depth of recrystallization processes. After annealing the average size of primary α -particles varied within a range of 5–10 μm depending on chemical composition. In the alloy with a lowest content of β -stabilizers (#1), only the primary globular α -phase ($\varnothing \sim 10 \mu\text{m}$) was observed (Fig. 3b). In the alloys with higher content of β -stabilizers needles of secondary α -phase (designated as α_{II} -phase, Fig. 3c, e) inside the β -matrix were also observed, which precipitated obviously due to lower annealing temperature and thus slower diffusional redistribution of alloying elements upon furnace cooling.

All alloys after rolling and annealing had two-phase composition (Fig. 4). Alloys #2 and #3 of Ti–Al–Mo–Fe system had very similar XRD patterns (compare relevant curves in Fig. 4) for all iron contents. The main difference between them consisted in the ratio between α - and β -phases: with increase in C_{Mo} value (higher content of iron) the amount of β -phase increased. At the same time, XRD pattern of alloy #4 of Ti–Al–Mo–Cr system noticeably differed in positions of XRD peaks of both α - and β -phases (compare corresponding curves in Fig. 4), which indicates a significant difference in the features of structure formation during deformation and subsequent annealing (and partial recrystallization that occurred at least in α -phase). The phase compositions, as well as contributions of crystallite sizes (coherent scattering regions, CSR) and lattice strain in the peak broadening of the alloys were calculated with standard ULTIMA IV diffractometer program using Rietveld [25] and Williamson–Hall [26] methods. The diffraction patterns of all alloys contain all reflections of the α - and β -phases with slight variations in their intensity (Fig. 4). These patterns

have varying magnitudes of shift and broadening of reflections of phases, which characterize each alloy. According to calculations, the ratios of residual stresses and CSR sizes in α - and β -phases in relation to the alloy #3 are 0.05, 0.3, 1, and 0.8, 0.95, 1) for alloys #2, #4, #3, respectively. The splitting of (002) α lines in all these alloys (Fig. 4) suggests that they contain two types of α -phase with different chemical compositions [3, 27–29]. In our case, these are primary α_{I} - and secondary α_{II} -phases (see Fig. 3c, e). Local chemical analysis (EDX SEM)¹ showed that in fact the secondary α_{II} -particles contained a larger amount of β -stabilizing elements as compared to the globular particles of α_{I} -phase (Table 3). This can be obviously explained by the fact that the secondary α -phase formed upon continuous cooling via decomposition of metastable β -solution enriched by these alloying elements. Besides, the essential difference in the angles of XRD peaks of alloys with iron (curves 1 and 3 in Fig. 4) and chromium (curve 2 in Fig. 4) can be explained both by the different nature of these elements and their different concentrations in the phases (Table 3). Obviously, this difference leads to a different effect of iron and chromium on the interplanar spacing in the α_{I} - and α_{II} -phases, i.e., on the lattice parameters a and c (Fig. 5). As seen, alloying with iron led to more pronounced changes of a as compared to c . At the same time, alloying with chromium caused significant changes of c in the α_{I} - and α_{II} -phases (Fig. 5b).

The presence of all reflexes of α and β phases and small changes in their intensity in the X-ray diffraction patterns of titanium alloys (Fig. 4) indicate the absence of too sharp textures, that was confirmed by texture studies (Fig. 6). The pole figures of alloys with iron and chromium are rather different. The more pronounced textures in both phases are observed in the alloy #3 with higher Fe content (Fig. 6a, b). These textures have almost axial symmetry with a weakly expressed rotational axis of order 4 (L_4) [31]. In this alloy, the residual stresses and the CSR sizes in both α - and β -phases are practically identical. In the alloy #4 with Cr, the (002) α texture is noticeably more intense, however, more spread relatively pole figure center (Fig. 6c), while the texture of β -phase looks rather similar (Fig. 6d). Both textures retain axial symmetry; in the α -phase the L_4 axis is more pronounced, whereas in the β -phase it is rather weak, as in the alloy #3. It is very likely that this difference in texture of the α -phase in alloys #3 and #4 is the result of the above mentioned features of the formation of primary and secondary components of this phase. Taking into account works [11, 12, 16] where the features of phase and structural

¹ It is possible that when measuring the chemical composition of the α_{II} -phase, the "response" from the surrounding β -phase also had a certain effect; however, a similar difference in the composition of the primary and secondary α -phases was observed in other titanium alloys [2, 3, 30].

Table 3 Local chemical compositions of α -phase particles (wt.%)

Phase	Alloy #3 (Ti–Al–Mo–Fe)				Alloy #4 (Ti–Al–Mo–Cr)			
	Al	Mo	Fe	Ti	Al	Mo	Cr	Ti
α_I	5.30	1.81	0.75	Balance	5.93	1.27	0.6	Balance
α_{II}	4.42	3.36	1.46	÷	4.82	5.36	2.49	÷

Fig. 5 Lattice parameters (a) a and (b) c for alloys #2, #3, #4

transformations in titanium alloys with iron and chromium were studied in detail, it should be concluded that the different XRD results for Fe- and Cr-containing alloys can be

associated predominantly with a significant difference in the diffusion mobility of these elements in the titanium matrix [30, 31].

The results of XRD study of phase composition of the alloys in different conditions are summarized in Table 4. It is clearly seen that in as-annealed and finally aged conditions all alloys had stable $\alpha + \beta$ phase composition. At the same time, after heating to high temperature upon either STA or RHT treatments with subsequent quenching the phase compositions of alloys were different. For instance, alloys #1 and #2 with comparatively low amount of β -stabilizing elements after solid solution treatment in furnace and quenching had $\alpha_I + \alpha'$ composition, whereas after RHT they were in single-phase α' condition. Alloys #3 and #4 with higher content of β -stabilizers after heating in furnace and quenching had $\alpha + \beta_m$ composition, whereas after RHT they comprised of α'' -martensite. Final aging all water quenched alloys resulted in either $\alpha_I + \alpha_{II} + \beta$ (after STA), or $\alpha_{II} + \beta$ (after RHT) phase composition.

These results allow to determine the following sequences of phase transformations:

	Initial state	Heated to high temperature	As-quenched	After final aging
Alloys #1 and #2	STA $\alpha + \beta \rightarrow$	$\alpha_I + \beta_m \rightarrow$	$\alpha_I + \alpha' \rightarrow$	$\alpha_I + \alpha_{II} + \beta$
	RHT $\alpha + \beta \rightarrow$	$\beta_m \rightarrow$	$\alpha_I \rightarrow$	$\alpha_{II} + \beta$
Alloys #3 and #4	STA $\alpha + \beta \rightarrow$	$\alpha_I + \beta_m \rightarrow$	$\alpha_I + \beta_m \rightarrow$	$\alpha_I + \alpha_{II} + \beta$
	RHT $\alpha + \beta \rightarrow$	$\beta_m \rightarrow$	$\alpha'' \rightarrow$	$\alpha_{II} + \beta$

Heating on the 1st stage of STA hardening—solid solution treatment at temperatures approximately by 30–45 °C below T_β (see Table 1) formed in the alloys microstructural states consisting of remnants of primary α_I -particles surrounded by metastable β_m -matrix (Fig. 7a). Subsequent aging caused β_m decomposition into fine $\alpha_{II} + \beta$ mixture (Fig. 7b) where the thickness of α_{II} did not exceed 100–150 nm (Fig. 7c), and a length not more than several μm (Fig. 7c, d), which ensured the strengthening effect [2, 31]. After RHT and aging by the same regimes completely β -transformed microstructure formed; the average size of β -grains increased from a few μm in initial state (Fig. 3d, e) to 20–30 μm , and fine $\alpha + \beta$ mixture formed inside them (Fig. 7e, f). It is worthwhile to note that after strengthening heat treatment the final α_{II} -particles are noticeably finer in the alloys #3 and #4 which contain a higher amount of

Fig. 6 Normalized pole figures (a, c) (002) α , and (b, d) (200) β of alloys (a, b) #3, and (c, d) #4 in as-annealed state

β -stabilizing elements as compared to the alloys #1 and #2 (compare Fig. 7f, c), because of finer α'' -martensitic needles as compared with α' -ones [22, 28, 32].

Tensile Properties

The results of tensile tests of all program materials are presented in Table 5. Data for the Ti–1.5Al–1Fe–7.2Cr alloy (lines 17–19) with somewhat higher chromium content [23, 24] were added for comparison to illustrate the influence of C_{Mo} above 10 wt.%. Typical examples of engineering tensile stress–strain curves for alloys #3 and #4 are given in Fig. 8: these curves clearly demonstrate that hardening heat treatment leads to a significant increase in strength and a decrease in the ductility of the alloys.

An analysis of the results in Table 5 shows that all alloys have a very high balance of mechanical characteristics in the as-rolled state, as well as after all types of heat treatment.

For example, the alloy #1 with lower content of β -stabilizing elements ($C_{Mo}=3.8\%$) has strength and ductility (pp. 1–4 in Table 5) rather comparable with those of widely used Ti6Al–4 V alloy [2]. An increase in iron content in the alloys of this system leads to a significant increase in strength, while ductility remains at rather attractive level (pp. 5–12 in Table 5). The substitution of iron by chromium in the alloy with the highest content of β -stabilizers (alloy #4) provides the best balance of mechanical characteristics (pp. 13–16 in Table 5). The effect of C_{Mo} values on the tensile properties of the studied alloys can be evidently illustrated by the dependencies shown in Fig. 9. In all cases, the strength dependencies have a maximum at concentrations 8–10 wt.%. As for the ductility characteristics, they gradually decrease with C_{Mo} , with the exception of alloy #4, which shows local maximum, probably due to the fact that it contains chromium instead of iron, unlike other alloys.

Table 4 Phase composition of studied alloys at different stages of heat treatments

Alloys ##	Heat treatment (Table 2)	Phase composition		
		After annealing	In as- quenched state	After final aging
1	1.1 Annealing	$\alpha + \beta$	...	...
	1.2 STA	...	$\alpha_I + \alpha'$	$\alpha_I + \alpha_{II} + \beta$
	1.3 RHT	...	α'	$\alpha_{II} + \beta$
2	2.1 Annealing	$\alpha + \beta$	...	...
	2.2 STA	...	$\alpha_I + \alpha'$	$\alpha_I + \alpha_{II} + \beta$
	2.3 RHT	...	α'	$\alpha_{II} + \beta$
3	3.1 Annealing	$\alpha + \beta$	...	...
	3.2 STA	...	$\alpha_I + \beta_m$	$\alpha_I + \alpha_{II} + \beta$
	3.3 RHT	...	α''	$\alpha_{II} + \beta$
4	4.1 Annealing	$\alpha + \beta$	...	...
	4.2 STA	...	$\alpha_I + \beta_m$	$\alpha_I + \alpha_{II} + \beta$
	4.3 RHT	...	α''	$\alpha_{II} + \beta$

In as-rolled state (Fig. 9a) the highest strength was obtained for the alloy #3 ($C_{Mo}=9.8\%$), while in all others cases (after annealing—Fig. 9b, and strengthening of both kinds—Fig. 9c, d) maximum strength had the alloy #4 ($C_{Mo}=8.6\%$). At the same time, the better balanced characteristics of ductility and strength in all cases were obtained for the same alloy #4. This phenomenon could be also explained by the different diffusional mobility of iron and chromium in titanium matrix [1, 28, 29], which in many cases has rather significant effect on structural and phase transformations and, thus, on the mechanical properties of titanium alloys. In particular, iron is prone to local fluctuations enriched with this element up to the precipitation of very fine particles of intermetallic compounds [1, 2], that can cause local embrittlement, especially along grain boundaries [16]. Keeping in mind the differences in chemical composition (Table 3) and lattice parameter c of the secondary α_{II} -phase in the alloy #4 (Fig. 5), in this sense, chromium seems to be a more preferable element, which has a slightly lower diffusion mobility and is not prone to local fluctuations; due to this, the ratio between iron and chromium was shifted toward the latter element, when creating a new metastable β -alloys of the Ti–Fe–Cr–(Al) system [11, 12, 23].

The above data on tensile testing allow to conclude that the experimental alloys of both alloying systems investigated in the present work have rather good balance of strength and ductility, competitive with conventional commercial alloys in all studied states: as-rolled, annealed, and thermally hardened. In the latter condition, better results were obtained when special non-equilibrium continuous rapid heat treatment was applied. It should be mentioned that we used only one, not optimal regime of RHT, otherwise very detailed

additional studies for tailoring thermo-kinetic and final aging regimes would be required [12, 14, 15, 17, 32, 33].

Deformation and fracture mechanisms

First of all, it should be mentioned that despite of essential difference in the content of β -stabilizing elements in the alloys #1, #2, and #3, there were no noticeable differences in their fracture surfaces. Due to this we present and analyze here only fracture surfaces of tensile tested specimens of alloys #3 and #4 as typical examples (Fig. 10). In as-rolled state all alloys, regardless the content of alloying elements, had similar fracture surfaces: at the macro-level a lamination along microstructural elements was observed (compare Figs. 3a, 10a, c), while at the micro-level fracture surfaces had a dimpled relief indicating the ductile nature of fracture (Fig. 10b, d). The same situation was observed in the specimens tested after annealing (Fig. 10e), and, of course, the fracture was also ductile (Fig. 10f). The annealing did not cause recrystallization of the β -phase grains which retained the shape elongated in the rolling direction. The surface dimples indicate that fracture was still ductile (compare Fig. 10f with b, d). The lamination cannot be explained by the crystallographic texture of β -phase (see Fig. 6b, d). Obviously, the lamination is a result of secondary cracking (perpendicular to the fracture surface which was formed by main crack growth) along high-angle boundaries between neighboring non-recrystallized β -grains elongated in the rolling direction.

Subsequent STA hardening with furnace heating led to the formation of $\alpha_{II} + \beta$ matrix with much higher strength in comparison with both residual primary α_I -phase and initial (discussed above for annealed states) β -matrix. As a result, the fracture surfaces (Fig. 10g–j) became more flat (not laminated) as compared to as-rolled (Fig. 10a–d) or annealed (Fig. 10e, f) states; nevertheless, on the micro-level similar ductile dimples (somewhat smaller in size— $2 \div 5 \mu\text{m}$ in STA state vs. $5 \div 12 \mu\text{m}$ in as-rolled or annealed ones) were observed (Fig. 10h, j), which can be explained by finer α_I -phase remnants in comparison with dimensions of initial α_I -particles in annealed state.

The application of RHT completely changed the character of fracture surfaces (Fig. 10k–o). Due to the $\alpha + \beta \rightarrow \beta_m \rightarrow \alpha_I + \alpha_{II} + \beta$ phase transformations initial (formed upon thermomechanical processing) grains recrystallized.² A well-developed relief of fracture surface (Fig. 10k, m) illustrates the change of β -grain structure and cracking along grain boundaries. Nevertheless, local zones with ductile dimples were also observed along with

² As it was shown earlier [34, 35], such fast recrystallization due to short exposure at temperatures of single-phase β -field does not cause principal changes in the initial crystallographic texture.

Fig. 7 Microstructure of alloys (a–c) #2, and (d–f) #4 after (a) WQ, (b–d) STA, and (e, f) RHT and aging. (a, b, d, e) SEM, (a) BSE, (b, d, e) SE; (c, f) TEM

Table 5 Tensile properties (strain rate $8 \times 10^{-4} \text{ s}^{-1}$) of studied alloys in different states

##	Alloy ##/C _{Mo}	State/treatment	YS, MPa	UTS, MPa	El _{unif.} , %	El _{tot.} , %	RA, %
Ti–Al–Mo–Fe							
1	1/3.8	As-rolled	946	983	6.5	14.2	36.7
2		Annealed	944	987	7.6	16.5	49.0
3		STA	952	992	4.3	11.5	38.6
4		RHT	981	1010	2.9	10.4	36.9
5	2/6.5	As-rolled	1012	1034	1.9	10.7	30.3
6		Annealed	988	1022	4.8	16.2	42.6
7		STA	1114	1182	2.1	8.7	24.8
8		RHT	1195	1267	1.4	7.9	26.4
9	3/9.8	As-rolled	1098	1187	2.1	9.8	32.5
10		Annealed	998	1065	7.8	14.6	42.4
11		STA	1340	1363	3.3	7.5	22.1
12		RHT	1400	1428	1.8	6.9	21.5
Ti–Al–Mo–Cr							
13	4/8.6	As-rolled	1143	1168	2.7	13.2	34.2
14		Annealed	1098	1121	12.4	18.6	48.6
15		STA	1365	1412	3.5	8.8	27.5
16		RHT	1488	1512	1.9	7.9	27.8
Ti–1.5Al–1Fe–7.2Cr [23, 24]							
17	5/12.5	As-rolled	1027	1085	...	10.3	26.7
18		Annealed	938	952	...	18.6	42.4
19		STA	1312	1328	...	9.5	28.8

cleavage facets (Fig. 10l, n). Thus, the cracks propagated either along grain boundaries (as it was shown in several works, the weakest links in the microstructures of this type are the zones near the α -lathes along grain boundaries [2, 16, 36–38]) or through appropriately oriented grains, depending on specific crystallographic orientation of individual grains. Careful observation of cleavage facets revealed distinctive traces (indicated by letters A, B, and C in Fig. 10o) that can be identified as evidences of rupture between the grain boundary α -phase and adjacent packets of secondary α_{II} -particles which had different spatial orientations.

The microstructures close to the fracture surfaces of the same (from Fig. 10) specimens are presented in Fig. 11. As can be seen, elongated in tension (and plastic flow) direction particles of α -phase and β -phase interlayers are observed near the fracture surface in as-rolled (Fig. 11a, c) and annealed (Fig. 11b, d) materials. At the same time, it is clearly seen that the β -phase appears to have significantly higher ductility, as its tips formed the ridges of the dimples (indicated by the letter A in Fig. 11a, b). Absolutely the same situation was observed in the alloys after annealing (Fig. 11c, d). It should also be noted that in both as-rolled and annealed states, neither pores nor cracks were observed in the fractured specimens either near the fracture surface or at some distance from it.

In STA hardened state the microstructure of fractured specimens was essentially different. First of all, a lot of

pores and cracks (the latter, obviously, formed as a result of merging of neighboring pores) were observed close to the fracture surface (Fig. 11e, g). These pores and cracks appeared on the interphase boundaries between the particles of residual α_I -phase and strengthened $\alpha_{II} + \beta$ matrix (Fig. 11f). The latter structural element had higher strength than initial β -phase and also formed ridges of dimples (indicated by A in Fig. 11h).

After RHT there were no remnants of primary α_{II} -particles, and all alloys had the highest strength compared to other treatments applied in the present work. The microstructure near fracture surface has evidences of plastic flow—elements of microstructure elongated in tension direction (Fig. 11i–l). In the alloy #3 with iron as alloying element pores and cracks in highly deformed zone close to the fracture surface appeared mainly on the boundaries of β -grains or coarse packets of secondary α -needles. These particles were observed in the zone close to the fracture surface in the alloy #4 with chromium, in which some relatively coarser secondary α_{II} -particles were formed during both rolling (Fig. 3e) and aging (Fig. 6e) (Fig. 11k, l). Obviously, pores and cracks nucleated exactly at these particles, while dimple ridges (especially for coarser dimples) probably can be associated with β -grain boundaries (Fig. 11l).

Thus, an analysis of the above observations allows to draw the following conclusions. First of all, the program alloys with all employed microstructural states had ductile

Fig. 8 Engineering tensile stress–strain curves of alloys (a) #4, (b) #3 after different heat treatments

character of fracture at least on micro-level. The highest level of plastic deformation (values of El and RA—see lines 1, 2; 5, 6; 9, 10; 13, and 14 in Table 5) occurred when the strength and ductility of the constituent phases were the closest—this situation took place in as-rolled and annealed conditions, when the α - and β -phases were in equilibrium (or close to equilibrium) states and had comparable sizes of particles. In this case, both phases deformed “cooperatively,” and intense plastic flow in the direction of applied stress occurred. When an ultimate elongation was reached in less ductile phase (α -phase due to the lower number of slipping planes [1–3]), pores/cracks formed in it, and then more ductile β -phase also ruptured, and ductile dimples formed (e.g., see a dimple indicated by A in Fig. 11a). The main crack propagated by merging adjacent ductile micro-dimples, while the high-angle boundaries between neighboring unrecrystallized grains

served as sites for the propagation of secondary cracks that formed the stratified relief at the “macro-level.”

After the initial β -phase and some part of initial α -particles transformed during STA hardening into much stronger fine $\alpha_{II} + \beta$ mixture, this structural matrix component of the alloys became the strongest one, but also the least ductile in comparison with the remnants of the primary α_I -phase. In the process of plastic flow of such a material, the greatest local stresses arise at the interfaces between softer α_I -phase and stronger $\alpha_{II} + \beta$ -matrix, as a result a plenty of pores appear, finally causing fracture. Harder $\alpha_{II} + \beta$ -matrix provides elevated strength of the alloys (which increases with higher content of β -alloying elements), and a mismatch in the strength and ductility of $\alpha_{II} + \beta$ -matrix and α_I -phase particles leads to a noticeable drop in ductility (lines 3, 7, 11, 15 in Table 5). After strengthening RHT treatment the softer particles of primary α_I -phase completely disappeared and the interior structure of the β -grains and their size changed, that resulted in the highest level of strength of the alloys (lines 4, 8, 12, and 16 in Table 5). However, relatively coarse layers of the α -phase formed along β -grain boundaries during the process of complete phase recrystallization (Fig. 7e, f), which became the sites of much higher local stresses at the interface between them and the $\alpha_{II} + \beta$ -matrix as compared to another structural conditions [2, 36]. The cracks nucleated at significantly higher stresses (compared to other structural states) fast propagated along the layers of grain boundary α -phase due to above mentioned mismatch in strength and ductility between adjacent matrix and this phase and long effective dislocation slip length in the latter [2].

Conclusions

Summarizing the obtained results, it is possible to conclude that the proposed compositions of titanium alloys of Ti–Al–Mo–Fe and Ti–Al–Mo–Cr systems allow to provide a high, reliable, and competitive with commercial alloys balance of mechanical properties in hot deformed, annealed and thermally strengthened states. Since relatively cheap master alloys are needed in the production of these alloys (for instance, those used in steel-making industry, as ferromolybdenum [4]), and comparatively simple melting and thermomechanical processing³

³ To obtain sufficient ductility, all titanium alloys require at least two-stage heavy hot deformation, first at temperatures above and then below T_β [2, 36], which may cause some increase in costs.

Fig. 9 Influence of content of β -stabilizing elements on tensile properties of alloys studied in states: (a) as-rolled, (b) annealed, (c) STA-strengthened, (d) RHT-strengthened. Arrows and numbers indicate

positions of studied alloys. Data for alloy #5 (Ti–1.5Al–1Fe–7.2Cr ($C_{Mo}=12.5\%$)) in (a)–(c) were taken from [24]

procedures are employed, the program alloys ensure high cost efficiency. The following conclusions can be drawn:

1. Annealing at temperatures slightly below the boundary between the $\alpha + \beta$ and β fields allowed to form a well-transformed microstructure of a globular type. In the stable state all alloys had two-phase $\alpha + \beta$ composition; in the alloys with higher content of alloying elements ($C_{Mo} \geq 6.5\%$) of both systems (either Fe or Cr) relatively fine secondary α -particles were observed after thermo-mechanical processing due to cooling conditions. This secondary α_{II} -phase is enriched by β -stabilizing elements in comparison with the primary α_I -phase, which caused some increase in both a and c lattice parameters. Most pronounced difference in c parameter was observed in the alloy with Cr. Probably this particular feature of this alloy caused noticeable difference in the crystallographic texture of α -phase, which had lower sharpness and symmetry as compared with the texture of β -phase in the same alloy and the textures of both α - and β -phases in all alloys with Fe. A distinctive feature of the investigated titanium alloys is the different c/a
2. All alloys in annealed state had a good balance of strength and ductility. Strength level (UTS) for alloys of Ti–Al–Mo–Fe system gradually increased from 987 MPa (for the alloy with $C_{Mo}=3.8\%$) to 1065 MPa (for $C_{Mo}=9.8\%$), while elongation slightly decreased from 16.5% to 14.6% for the same alloys. The alloy of Ti–Al–Mo–Cr system with $C_{Mo}=8.6\%$ had the best strength–ductility combination: UTS = 1121 MPa, El = 18.6%.
3. The phase and microstructural transformations in the program alloys were studied by X-ray diffraction and SEM, depending on the content of alloying β -stabilizing elements and the method of thermal hardening—equilibrium conditions of heating in furnace or non-equilibrium heating by RHT technique. After water quenching transition martensitic phases appeared, which type (hexagonal α' , or orthorhombic α'') was determined by the presence of residual α_I -particles (heating method and peak temperature). After STA (solid solution treatment), some

Fig. 10 Fracture surfaces of alloys (a, b, e, f, g, h, k, l) #3, (c, d, i, j, m–o) #4. States: (a–d) as-rolled; (e, f) annealed; (g–j) STA hardened; (k–o) RHT hardened. Letters A, B, and C in (o) indicate the traces of subgrains with different orientations of intragrain α_{II} -phase packets. SEM, SE

Fig. 11 Microstructure of alloys (a, b, e, f, i, j) #3, (c, d, g, h, k, l) #4 near the fracture surface in states: (a, c) as-rolled, (b, d) annealed, (e–h) STA hardened, (i–l) RHT hardened. A in (h) indicates the

ridges of a ductile dimple. SEM: a–d, g (overview), h–j SE, e, f, g (the inset) BSE. The specimens were strained along the horizontal direction

remnants of primary α_I -phase particles were present, whereas after RHT (rapid heat treatment) the materials have undergone complete phase transformation.

4. The application of STA strengthening heat treatment (furnace heating with subsequent quenching) caused transformation of initial β -phase (without changes in

grain structure) and a part of initial α_1 -phase into martensitic α' or α'' -phases which, in turn, formed a fine two-phase mixture $\alpha + \beta$ phases after final aging. As a result, the strength increased to 992 MPa (the alloy with $C_{Mo} = 3.8\%$), and even to 1363 MPa (the alloy with $C_{Mo} = 9.8\%$) with some decrease in ductility. For this STA hardening, the best balance of mechanical properties was obtained on the Cr-containing alloy ($C_{Mo} = 8.6\%$): UTS = 1412 MPa, El = 18.8%.

5. The RHT led to complete phase recrystallization which resulted in a slight increase in β -grain size and the formation of a completely β -transformed fine $\alpha + \beta$ intragranular microstructure. After final aging, this condition had an attractive balance of high strength and acceptable ductility. The strength level achievable with RHT is determined by the total content of β -stabilizing elements in the alloy, and its maximum (above 1500 MPa) corresponds to $C_{Mo} \approx 8\text{--}9$ wt.%. Better combination of strength and ductility was obtained in the alloy with Cr, obviously due to lower diffusion mobility of chromium and its lower tendency to segregate in the titanium matrix.
6. A detailed study of fracture surfaces and adjacent microstructure allowed to identify phases and/or interphase boundaries which were weak links and sites for the nucleation of cracks.

Acknowledgements This work was supported by funding provided by National Academy of Sciences of Ukraine as part of the Project # III-09-18 (State Registration # 0118U001036).

Conflict of interest The authors declare that they have no conflict of interest.

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