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PHYSICS OF STRENGTH AND PLASTICITY

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Microstructure and Mechanical Properties of a New Ti–1.5Al–1Fe–7.2Cr Alloy Produced with Conventional Cast and Wrought Approach

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The ability to obtain a new cost-efficient titanium β-metastable alloy Ti–1.5 (% wt.)Al–1Fe–7.2Cr using conventional cast and wrought technological approaches is investigated. The microstructure and phase composition are characterized in the as-cast, hot-deformed and aged states. The aging behaviour of the state water-quenched from the temperature of single-phase β-field alloy is studied by exposure at 673 K and 773 K, and compared with previously obtained data for the Ti–11V–7Cr–4Al alloy. The mechanical properties are determined by tensile testing of the as-deformed, annealed at the temperature of the two-phase $\alpha + \beta$-field, and strengthened by STA treatment (solid solution treatment, water quenching and aging) states. As confirmed, under all the test conditions proposed, the new alloy has an attractive balance of high strength and ductility, which are competitive with those for other titanium alloys of the same metastable β-type.

Key words: cost efficient titanium alloy, thermomechanical processing, heat treatment, aging behaviour, microstructure, mechanical properties.

Вивчено можливість отримання нового економно легованого титанового

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сплаву Ti–1,5% мас. Al–1Fe–7,2Cr метастабільного β-класу з використанням традиційних технологічних процесів вакуумного плавлення і гарячої деформації. Мікроструктура і фазовий склад були вивчені в литому, гарячедеформованому і зістареному станах. Поведінку при старінні стану, загартованого у воду від температури однофазної β-області, вивчено при витримці при 673 К і 773 К і зіставлено з раніше отриманими даними для сплаву Ti–11V–7Cr–4Al. Механічні властивості визначені шляхом випробування на розтягування в деформованому стані, що відпалюють при температурі двофазної α + β-області і після зміцнюючої термічної обробки. Встановлено, що у всіх досліджених структурних станах новий сплав має привабливий баланс високих значень міцності і пластичності, які є конкурентоспроможними з властивостями інших більш легованих титанових сплавів того ж метастабільного β-класу.

Ключові слова: економно легований титановий сплав, термомеханічна обробка, термічна обробка, поведінка при старінні, мікроструктура, механічні властивості.

Исследована возможность получения нового экономно легированного титанового сплава Ti–1,5 (% масс.) Al–1Fe–7,2Cr метастабильного β-класса с использованием традиционных технологических процессов вакуумной плавки и горячей деформации. Микроструктура и фазовый состав были изучены в литом, горячедеформированном и состаренном состояниях. Поведение при старении состояния, закалённого в воду от температуры однофазной β-области, изучено при выдержке при 673 К и 773 К и сопоставлено с ранее полученными данными для сплава Ti–11V–7Cr–4Al. Механические свойства определены путём испытания на растяжение в деформированном состоянии, отожжённом при температуре двухфазной α + + β-области и после упрочняющей термической обработки. Установлено, что во всех исследованных структурных состояниях новый сплав имеет привлекательный баланс высоких значений прочности и пластичности, которые конкурентоспособны со свойствами других более легированных титановых сплавов того же метастабильного β-класса.

Ключевые слова: экономно легированный титановый сплав, термомеханическая обработка, термическая обработка, поведение при старении, микроструктура, механические свойства.

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1. INTRODUCTION

Titanium alloys are very important structural materials for many critical applications due to their high specific strength, which is well-balanced with other mechanical properties, such as excellent corrosion resistance and good ductility [1–3].

The greatest advantages over other structural materials titanium alloys provide is a high strength state, which is only achievable by strengthening through heat treatment of the higher-alloyed transition near beta (C_{Mo} = 11–12%) and martensitic (C_{Mo} = 8–9%) type alloys [4, 5]. The main disadvantage of these materials is that they are relatively expensive due to both the high cost of individual raw materials, and to the complex equipment required for ingot melting and further processing. Therefore, there have been numerous studies aimed at cost reduction. One approach is the development of new compositions of low-cost (or cost-efficient) titanium alloys, based on the use of cheap alloying elements and master alloys [6–9]. It is first necessary to mention the TIMETAL-LCB alloy developed using Fe–Mo master alloy typically used in steel production [7]. Another successful approach has been to produce low-cost Ti–Al–Fe–Cr alloys containing high amounts of relatively cheap Fe and Cr [8, 9]; however, all developed compositions to date have corresponded to the higher-alloyed metastable β-alloy type. In our previous work [10], where an elemental powder metallurgy approach was employed, we showed that the composition of an alloy of this type can be changed to a lower content of β-stabilizing elements, such as iron and chromium, to 1 and 7% mass, respectively, which corresponds to a molybdenum equivalent of C_{Mo} = 11.5% mass. Such a reduction in the content of β-stabilizing elements allows both a reduction in the cost and specific weight of the alloy, in addition to simplification of the smelting process (due to faster melt homogenization) and shortening of the isothermal exposure during heat treatments due to the high diffusibility of Cr and especially Fe.

Therefore, the present study focused on evaluation of the microstructural features and tensile properties of the same Ti–1.5Al–1Fe–7.2Cr alloy, but obtained using conventional cast and wrought technologies.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

An ingot with dimensions of 25×13×170 mm^3 was melted with a laboratory scale arc-melting furnace under an Ar atmosphere using titanium sponge, aluminium (99.99%), iron (99.5%), and chromium (99.9%) as starting materials. After melting, several alloy specimens were cut for characterization of the initial as-cast state and study of the aging behaviour. As the last aim, specimens were solution treated at 1173 K for 3.6 ks, and then water quenched to retain the metastable β-state. Subsequent aging was studied at two temperatures (673 and 773 K) after different durations of isothermal exposure by Vickers hardness measurements and changes in the phase composition determined by XRD measurements.

The main part of the ingot was subjected to free hammer-forging at 1193 K with a total reduction in area of 20%, then rolled at 1123 K in mills having rollers with cylindrical grooves for a total reduction in area of 60%. The obtained rod with a diameter of about 10 mm was cut into 10 mm long specimens for microstructure and phase content characterization, and 52 mm long specimens for tensile tests. Both types of specimens were studied and tensile tested in 3 different states: i) as-rolled, ii) annealed at 1073 K for 3.6 ks, and iii) after STA treatment, which included solid solution treatment at 1123 K for 1.8 ks, water quenching (WQ) and then aging at 773 K for 18 ks. The temperature for the solid solution treatment was selected based on resistometric data around the temperature of polymorphous $\alpha + \beta_0 \to \beta$ metastable transformation completion (the so-called beta transus temperature, T_β, Fig. 1) obtained by the in situ method described in Ref. 11. The temperature and duration of aging were selected based on the results of the aging behaviour investigation (see Part 3.1).

Electrical resistivity (used for the aging behaviour study) was measured by the direct current (100 mA) four-probe method at room temperature (RT) and at liquid nitrogen (LN) temperature (ρ_{RT} and ρ_{LN}, respectively). The thermoelectric force was cancelled by reversing the polarity of the DC supply in the measurement circuit. Resistivity measurements at LN and RT were performed to confirm the formation and disappearance of the athermal ω-phase [12]. The microstructure at different stages of processing and treatment, and the fracture surfaces of specimens after tensile tests, were studied using scanning electron microscopy (SEM; Tescan Vega 3). The tensile properties were investigated according to the ASTM E8 standard using a tensile tester (Instron 3376) with specimens having a gauge diameter of 4 mm and a gauge length of 40 mm.

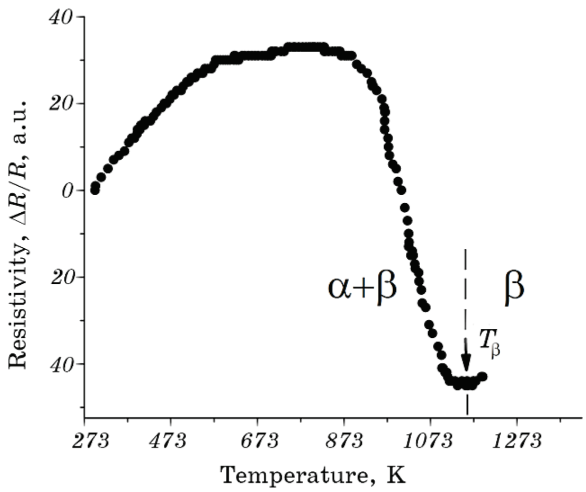


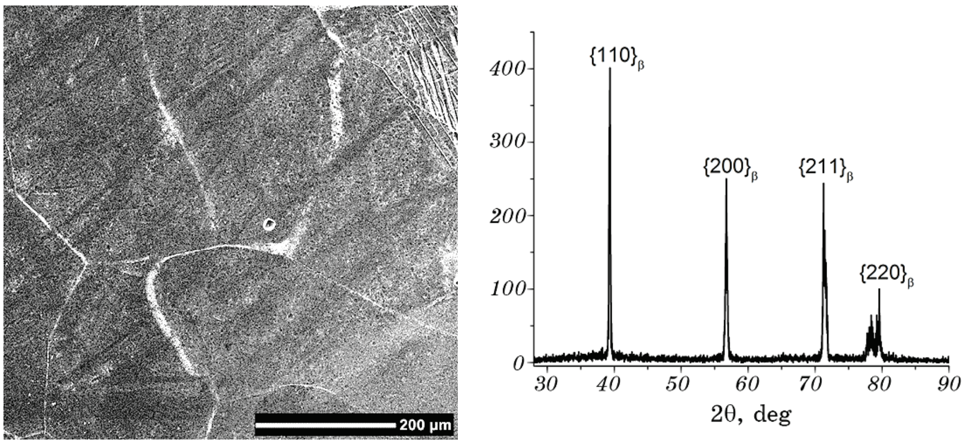
Fig. 1. Resistivity *in-situ* curve of Ti–1.5Al–1Fe–7.2 alloy annealed 1073 K, 7.2 ks, and continuously heated with rate 1 K·s^{-1}.

3. RESULTS AND DISCUSSION

3.1. As-Cast State and Aging Behaviour of Solution-Treated and Quenched Alloy

In the as-cast state, the alloy was characterized by a single-phase β-state with average β-grain sizes of about 300 μm (Fig. 2). This is typical for the as-cast state of Ti-based alloys of β-metastable class alloys [1, 2, 6]. Therefore, even this as-cast state could be employed for further study regarding the influence of isothermal exposure on the aging behaviour. However, taking into account previous data obtained with a similar composition alloy obtained by the elemental powder metallurgy approach [10], and keeping in mind the aim to eliminate the possible influence of structural and chemical inhomogeneity of the initial cast state, water quenching was applied after exposure at 1173 K for 3.6 ks (single-phase β-field) to form and retain a uniform single-phase metastable β-state. The water quenched specimens were then subjected to isothermal exposure for various durations at two temperatures of 673 and 773 K to study the aging behaviour. Changes in resistivity at both LN and RT with respect to the duration of isothermal exposure, the resistivity ratios (LN to RT) and Vickers hardness with isothermal aging at 673 and 773 K were investigated and the results are presented in Fig. 3.

Figure 3 shows that the resistivity of the alloy measured at both temperatures decreased significantly after a relatively short exposure of 0.06 ks, and at the same time the hardness increased significantly



a *b*

Fig. 2. Microstructure (SEM) (*a*), and XRD pattern (*b*) of Ti–1.5Al–1Fe–7.2 alloy in as-cast state.

from an initial 284 *HV* to 480 *HV*. After aging for 0.06 ks, the temperature dependence of the resistivity changed from negative to positive, because the resistivity ratio became less than 1. This can be explained by isothermal ω-phase precipitation, as evidenced by the results of XRD measurements (Figs. 4 and 5).

A second clear decrease in resistivity occurred at 30 ks, which also led to an increase of hardness. The reason for this phenomenon is completely different; in the XRD spectrum, the peaks for the isothermal ω-phase disappeared, while the intensity of the α-phase peaks increased, which indicated an intensive process of $\beta_{metastable} + \omega \rightarrow \alpha$ decomposition. The dispersion of α-phase precipitates at these temperatures and via such a reaction/mechanism is very high, which ensures strengthening of the β-matrix, and thus an increase in the hardness of the titanium β-alloys [6, 7, 11, 13].

It should also be noted that XRD results revealed the first appearance of α-phase after exposure for 1.2 ks. However, the resistivity changes typically caused by α-precipitation were not observed, as a re-

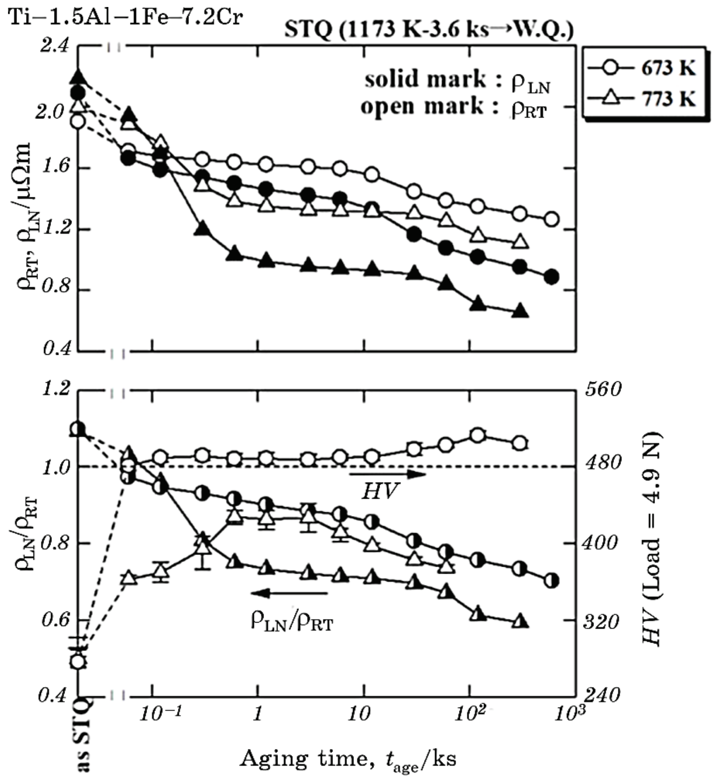


Fig. 3. Changes in ρ_{LN}, ρ_{RT}, ρ_{LN}/ρ_{RT} and *HV* of Ti–1.5Al–1Fe–7.2Cr alloy during isothermal aging at 673 K and 773 K.

sult of some balance at the intermediate stage of dissolving one phase (ω) while precipitating another (α). In the range of isothermal exposure, 1.2–3.0 ks, the hardness remained at almost the same level and then decreased, which may be associated with the simultaneous occurrence of two parallel processes; a reduction of the amount of ω-phase and coarsening of the precipitated α-phase.

It is interesting to compare the results obtained in the present study with similar results obtained previously for another metastable β-alloy containing the isomorphic β-stabilizing element vanadium, *i.e.*, Ti–11V–7Cr–4Al, which is characterized by a molybdenum equivalent of 16.2% mass [14]. Resistivity changes and hardness data for both alloys are compared in Fig. 6. Metastable β-phase decomposition in the alloy began without any incubation period; at about 100 ks at 673 K,

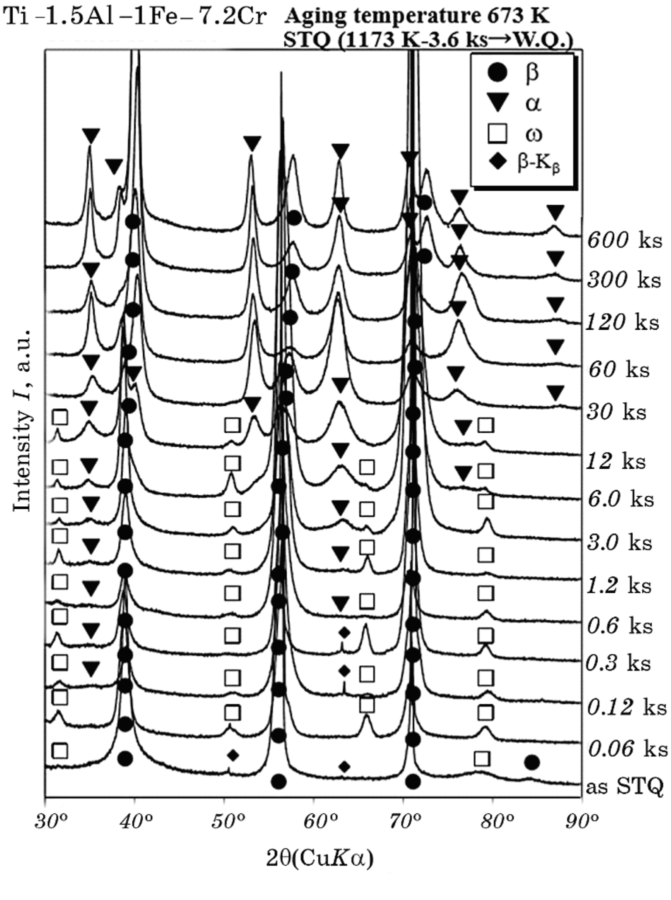


Fig. 4. Change in XRD patterns of Ti–1.5Al–1Fe–7.2Cr alloy during isothermal aging at 673 K.

and at 1 ks at 773 K. At the same time, decomposition of the metastable β-phase of the present Ti–11V–7Cr–4Al alloy began without any measurable preparatory stage.

The simplest explanation for this difference in aging behaviour would be the difference in the content of the stabilizing β-phase elements because a higher content of these elements increases the β-phase stability. However, we consider that the difference in the diffusibility of iron and vanadium should also be taken into account [1–3, 11, 13] because it is well known that the presence of highly mobile iron atoms leads to significant diffusive phase transformations in titanium alloys. Therefore, it is important to note that the highest levels of hardness in the Ti–1.5Al–1Fe–7.2Cr alloy was reached just after about 100 ks and 1 ks at 673 and 773 K, respectively, whereas in the Ti–11V–7Cr–4Al alloy after the same exposure, only the decomposition process began. Therefore, the former alloy requires shorter isothermal expo-

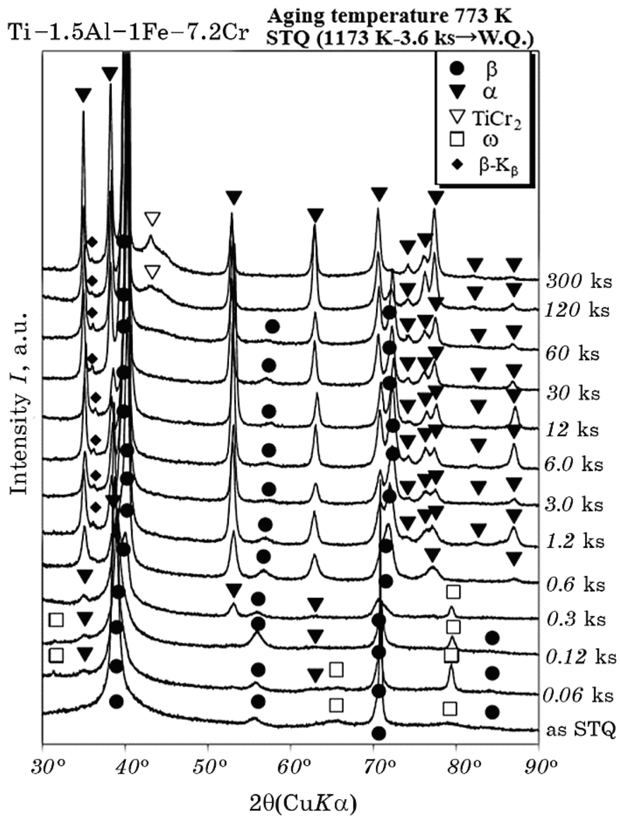


Fig. 5. Change in XRD patterns of Ti–1.5Al–1Fe–7.2Cr alloy during isothermal aging at 773 K.

sure times for heat treatments such as aging.

3.2. Microstructural Evolution upon Heat Treatment and Influence on Tensile Properties

The hot deformation route employed resulted in the transformation of the equiaxed single-phase β-structure into a laminated two-phase microstructure consisting of β-grain-elongation along the flow direction of the matrix with fine α-precipitates inside, and some α-lamellae partially covering the β-grain boundaries (Figs. 7, *a*, *b*). This indicates that hot deformation caused plastic deformation of the primary equiaxed β-grains into grains elongated along the longitudinal axis of the rod-like grains, which for this type of thermomechanical processing, have a spindle-shaped form.

Subsequent annealing at the temperature of the two-phase α + β field (1073 K) did not lead to recrystallization of the β-grain structure, although the phase composition became stable, and the amount of α-phase increased and the size of α-precipitates upon annealing (as a result of metastable β-phase decomposition) became slightly smaller than the primary (formed by thermomechanical processing) particles (compare Figs. 7, *b*, *c*).

Application of STA hardening treatment at the first stage (solid solution treatment at 1123 K) led to the partial dissolution of the α-phase

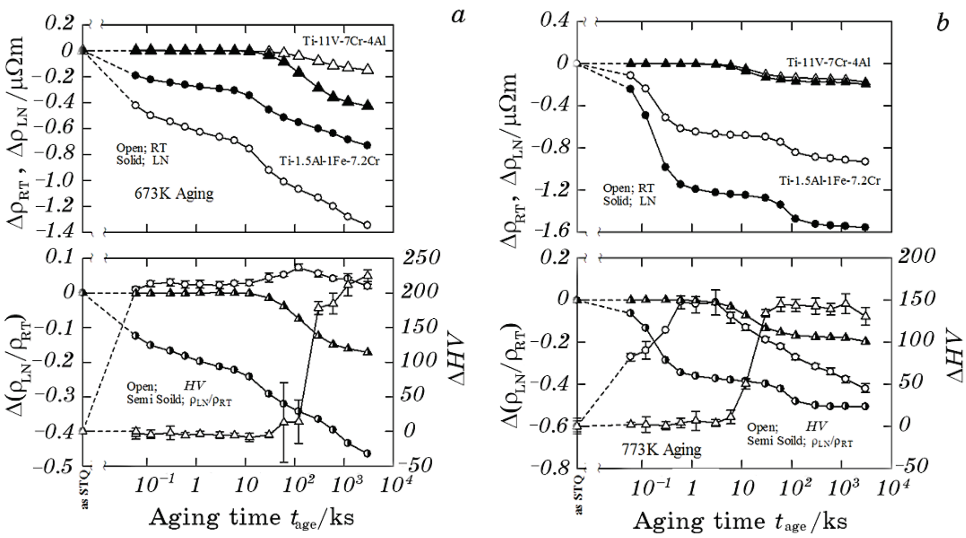
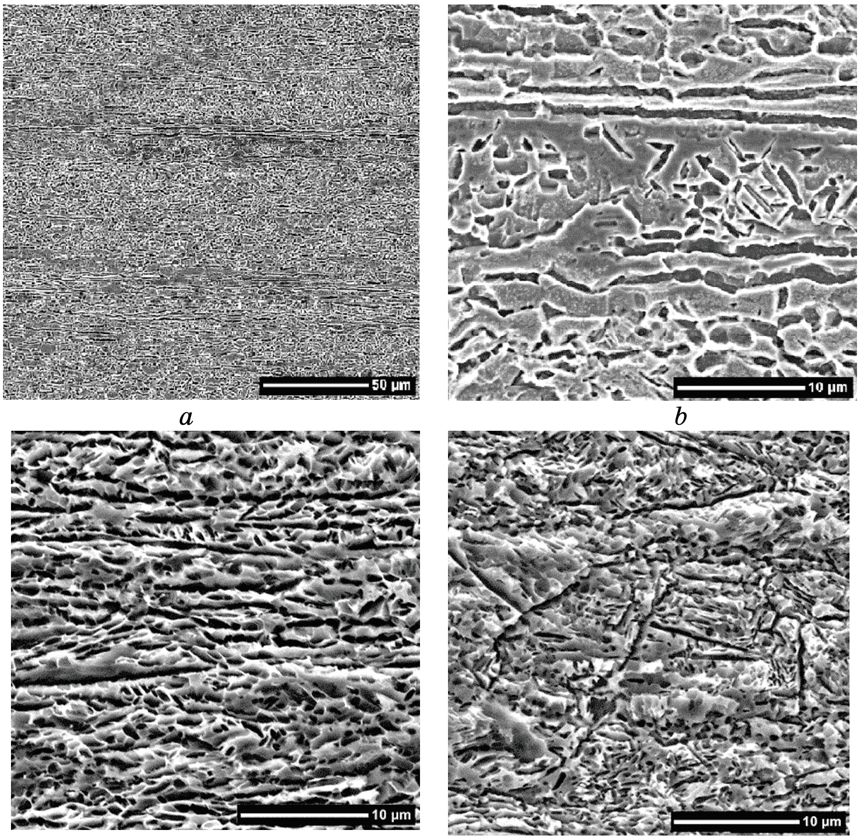
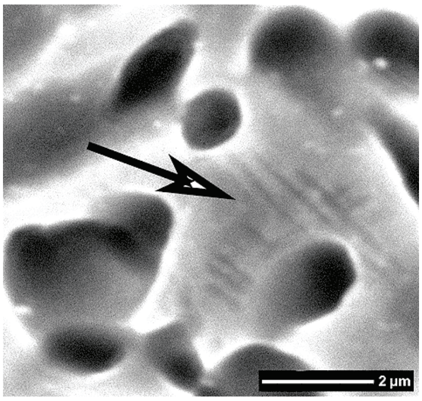


Fig. 6. Comparison of changes in ρ_{LN}, ρ_{RT}, ρ_{LN}/ρ_{RT} and *HV* of Ti–1.5Al–1Fe–7.2Cr and Ti–11V–7Cr–4Al alloys during isothermal aging at 673 K (*a*) and 773 K (*b*).



a *b* *c* *d*



e

Fig. 7. Microstructure of Ti–1.5Al–1Fe–7.2Cr alloy studied in the longitudinal direction in states: as-deformed (*a*, *b*), after annealing 1073 K, 7.6 ks (*c*), after STA: 1123 K, 1.8 ks, WQ + + 773 K, 18 ks (*d*, *e*). Arrow in Fig. 5, *e* indicates particles of secondary α-phase precipitated on aging. SEM.

(first of all the smallest particles), fixation of an elevated (as compared with annealed state) amount of metastable β-phase with water quenching, and its decomposition during the final aging with the formation of very fine secondary α-precipitates inside the β-matrix (Fig. 7, *e*).

It should be noted that solid solution treatment before quenching in some locations caused partial β-grain recrystallization with the formation of finer grains (Fig. 7, *d*). This can be explained by a much finer α + β-microstructure in the locations of tips (or spikes) of the spindle-shaped grains, where stresses accommodated during deformation can be essentially higher than those across the main body of the β-grains. Typical engineering stress-strain curves of tensile tested Ti–1.5Al–1Fe–7.2 alloy specimens with different microstructures are shown in Fig. 8. The mechanical properties obtained are listed in Table 1, where they are compared with the data previously obtained for the alloy of the same composition, but produced by a blended elemental powder metallurgy approach.

Analysis of the obtained tensile properties and comparison with similar data obtained using the elemental powder metallurgy approach [9] allows the following conclusions to be drawn. First, it should be noted that even in the deformed (as-rolled) state, the alloy had a good combination of strength and ductility (curve *1* in Fig. 8 and No. 1 in Table 1). Further annealing improved the ductility characteristics (both elongation and a reduction in area) without a significant decrease in strength (curve *2* in Fig. 8 and No. 2 in Table 1). This seemingly unexpected result could be explained by two factors: (i) the removal of residual stresses due to deformation, and (ii) an increase in the amount of α-phase. As expected, the hardening heat treatment significantly increased the strength and decreased the ductility (curve *3* in Fig. 8 and

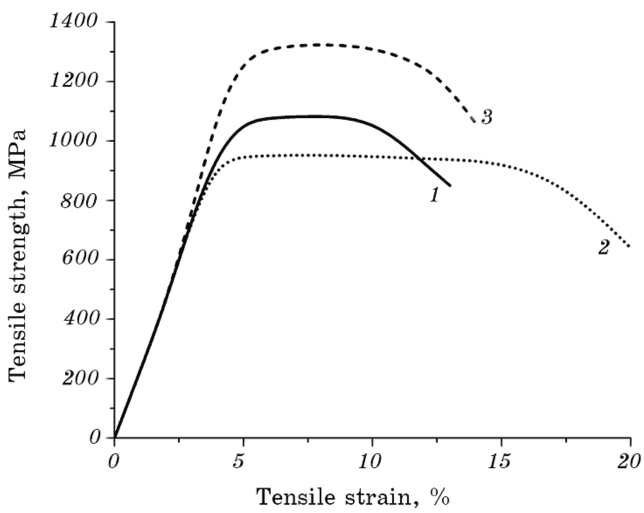


Fig. 8. Typical engineering stress-strain curves of Ti–1.5Al–1Fe–7.2Cr alloy in states: *1*—as-rolled, *2*—annealed at 1073 K, 3.6 ks, *3*—STA: 1123 K, 1.8 ks, WQ + 773 K, 18 ks.

TABLE 1. Tensile properties of Ti–1.5Al–1Fe–7.2Cr alloy in different microstructural states.

No.	State	YS*, MPa	UTS**, MPa	El***, %	RA****, %
	Cast and wrought—present study				
1	As-rolled	1027	1085	10.3	26.7
2	Annealed 1073 K, 3.6 ks	938	952	18.6	42.4
3	Annealed 1073 K, 3.6 ks + + STA: 1123 K, 1.8 ks, WQ + + 773 K, 18 ks	1312	1328	9.5	28.8
	Prepared with Powder Metallurgy approach—from [9]				
4	As-sintered	1014	1018	1.9	20.6
5	Hot pressed: 1223 K, 70% reduction + annealed 1073 K, 3.6 ks	896	969	14.73	35.11

*YS—yield strength, **UTS—ultimate tensile strength, ***El—relative elongation, ****RA—reduction in area.

No. 3 in Table 1). A comparison of the present material obtained by a standard metallurgical approach with the same alloy produced by blended elemental powder metallurgy and subjected to the same annealing (see No. 2 and No. 5 in Table 1) shows that cast and wrought metal has a better balance of strength and ductility, which is due to a finer β-grain structure, a lack of residual porosity and a lower impurity content (primarily oxygen).

Figure 9 shows typical examples of fracture surfaces of the tensile tested specimens. The initial as-rolled state is characterized by secondary brittle cracking along β-grain boundaries (Figs. 9, *a*, *b*); however, at the microscopic level, ductile dimples are observed (Fig. 9, *c*). Annealing at the temperature of the α + β field led to a decrease in the number of brittle cleavages along β-grain boundaries (Figs. 9, *d*, *e*) due to formation of a stable phase state; the ductile dimples became deeper, which indicated an increase in the ductility of the material (Fig. 9, *f*). Application of STA hardening slightly changed the nature of the fracture surfaces; in addition to cracking along the grain boundaries, intragranular sub-boundaries appeared (Fig. 9, *g*) and the depth of the dimples decreased (Fig. 9, *h*).

Therefore, the new low-cost titanium alloy of metastable β-type Ti–1Al–1Fe–7.2Cr obtained with conventional cast and wrought techniques exhibits characteristics that are attractive and competitive with other alloys of this class [3–9] in terms of a balance of strength and ductility in thermomechanical processing and annealing, and in

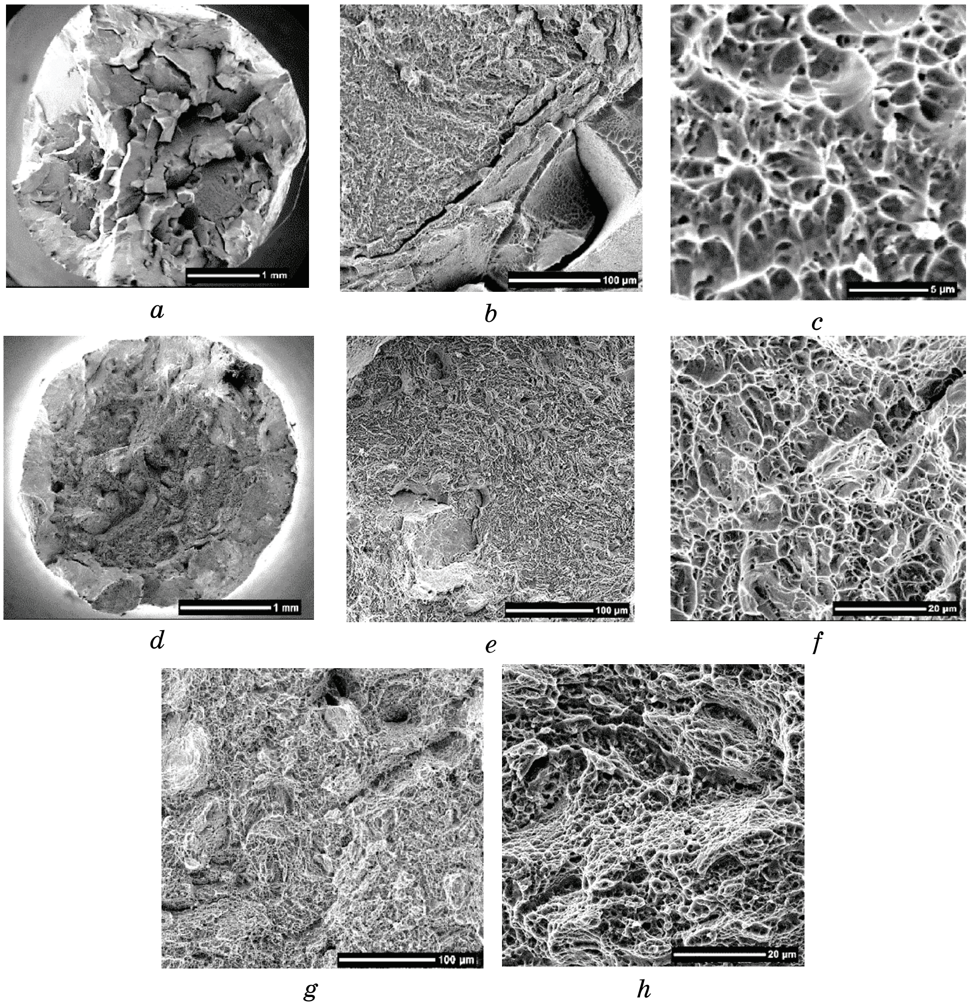


Fig. 9. Fracture surfaces of Ti–1.5Al–1Fe–7.2Cr alloy in states: as-deformed (*a*–*c*), after annealing 1073 K, 7.2 ks (*d*–*f*); after STA: 1123 K, 1.8 ks, WQ + + 773 K, 18 ks (*g*, *h*). (SEM).

terms of the STA hardened state.

4. CONCLUSIONS

1. The possibility of obtaining a new cost-efficient titanium β-metastable Ti–1.5Al–1Fe–7.2Cr alloy by conventional cast and wrought technologies was studied. The microstructure and phase composition of the as-cast, solid solution-treated and water quenched,

aged, hot deformed, and annealed states were investigated.
2. The behaviour of the metastable β-phase during aging with different durations of isothermal exposure was studied at two temperatures of 673 K and 773 K, and the results obtained were compared with another typical β-metastable titanium alloy, Ti–11V–7Cr–4Al. Decomposition of the alloy of the metastable β-phase occurred much faster and without any incubation period.
3. When the alloy was subjected to hot deformation with a total degree of approximately 80%, followed by annealing, an ultimate tensile strength of around 950 MPa was achieved with a relative elongation of 18.6% and a reduction in area of 42.4%. Subsequent strengthening by heat treatment elevated the strength to 1328 MPa, while maintaining the ductility characteristics at a good level.
4. The proposed composition of the titanium metastable β-alloy should ensure a good balance of strength and ductility after various processing and treatment steps. The mechanical properties are attractive, especially taking into account the lower content of alloying elements, which reduces both the cost and the specific weight of the alloy, simplifies the smelting process (due to faster melt homogenization), and reduces the duration of isothermal exposure necessary for heat treatment.

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