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Effect of Rare-earth Element Addition on Structure of Heat-resistant Ti–6.5Al–4Zr–2.5Sn–2.4V–1Nb–0.5Mo–0.2Si Titanium Alloy

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Abstract. To guarantee the requirements for modern technology it is necessary to create new alloys with enhanced characteristics or upgrade existing ones. Especially it concerns high-tech industries, particularly aviation and rocket engineering. When designing and creating modern aircraft engines, a special place is devoted to heat-resistant titanium alloys. High strength characteristics and relatively low density make it possible to create alloys with outstanding specific parameters. However creation of new alloying systems is quite expensive and implies parallel development of technological process as well as various processing modes. There are several ways to solve this problem; one of them is modification of already existing alloy. Due to the unique combination of physical-chemical properties of rare earth metals adding them to existing alloying systems makes it possible to create alloys with increased characteristics. In particular, microalloying of titanium alloys with gadolinium leads to transformation of their microstructure and increase of some mechanical characteristics. Thus, microalloying of the high-temperature Ti-6.5Al-4Zr-2.5Sn-2.4V-1Nb-0.5Mo-0.2Si titanium alloy in as-cast state results in a change in the intragranular structure, but does not significantly affect the macrostructure of the ingots. At the same time, in deformed state increase of gadolinium content leads to refinement of the initial β-grain, growth and formation rates of the primary and secondary phases change, which in its turn affects the mechanical characteristics of the alloying system as a whole. The Rockwell hardness measurements carried out using the BUEHLER Macromet 5100T device show an increase of parameters in the alloy with gadolinium.

1 Introduction

To modify the existing alloys of different grades and create new alloys the lanthanides are used at present as additive agents at microalloying. Lanthanides or, as they are also called, rare-earth metals (REMs) [1-3] became widespread for microalloying due to the unique combination of physical-chemical properties they possess in full. They are obtained during reprocessing of the ore which contains minerals in form of mixtures consisting of REM oxides, phosphates, carbonates or silicates and also thorium, yttrium, scandium, titanium, niobium and tantalum similar to them by the properties [2-4]. REMs are many times more in the nature than zinc, copper, lead, tin and noble

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metals. Closeness of REMs in their physical-chemical properties is explained by the identical structure of the outer electron shells of their atoms. All of them possess the three outer-shell electrons; particular REMs differ in number of electrons on $4f$ level. These electrons are shielded from the common external influence by the surrounding closed shells and therefore have practically no effect on chemical activity of REMs. Slight differences in their properties are generally connected with the differences in the size of an atom nucleus [3-5].

All REMs possess an average refractoriness (cerium, Ce – 800°C; gadolinium, Gd – 1350°C; lutetium, Lu – 1700°C) [5-7]. In solid state they have typical for metals hexagonal closed-packed or cubic crystal structures [8]. The majority of them is polymorphous and differs by closeness of atomic and ionic radii (lanthanide compression) [8-10]. In their pure form REMs are plastic and can be obtained wire-shaped or sheet-shaped. REMs possess strongly negative electrochemical potential and therefore are good reducers. All of them are oxidized in air at the room temperature with different rapidity [8-10]. A set of such unique physical-chemical properties, close electronic and crystal structures makes it possible to predict improvement of parameters for titanium alloys. One of the main goals in development of heat-resistant titanium alloys is to increase operating temperatures for the components made of them as well as to improve lifetime, plastic fatigue strength and creep resistance. To solve these problems the required structure is needed that can be formed by alloying with rare-earth elements. Therefore in this work we considered the effect of gadolinium on structure and properties of heat-resistant alloy in as-cast and deformed conditions.

2 Materials and procedures

The ingots of a pilot Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy with different gadolinium content – without Gd (Composition 1), with 0.04% of Gd (Composition 2) and 0.18% of Gd (Composition 3) – were smelted to study the effect of Gd on structure formation and properties.

The pilot ingots were manufactured by triple remelting method. After a second remelting the ingots were subjected to mechanical treatment to remove corona, surface defects and pipe. The crystallizer with diameter of 160 mm was used for a third remelting which allowed improving chemical composition homogeneity and decreasing hydrogen and chlorides content. After a third remelting corona and pipe were removed, and then the ingots were turned for the further deformation.

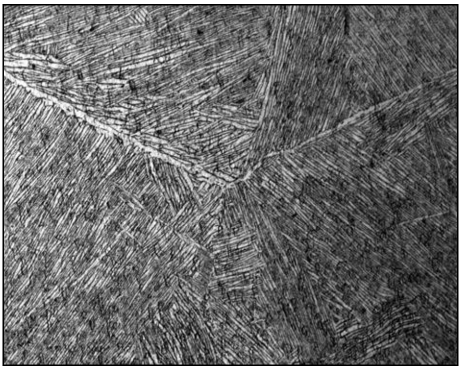
Metallographic investigations were carried out using the Axio Observer A1m optical microscope with the Image Expert Pro3 digital software intended for acquisition and analysis of the images. X-ray diffraction analysis was carried out using the DRON-4 and Dron-7 diffractometers in filtered CuK_{α} radiation.

Hardness was measured according to the Rockwell method in HRC scale using the BUEHLER Macromet 5100T device by diamond cone with a vertex angle of 120° and the load of 1500 N.

3 Results and discussion

3.1 Structure and phase composition of a pilot alloy in as-cast condition.

Structures of casted alloys with the three compositions are presented in Fig. 1; the samples were cut from the upper part of the ingots. It was stated that the additional Gd alloying resulted in a change of the intragranular casted structure – gradual reduction of α-phase plate’s length was observed and accompanied by gradual formation of a cage-netting structure similar to the Widmanstätten one with the growth of Gd content (Fig. 1).



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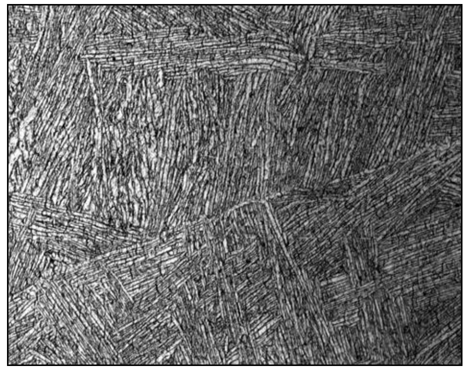

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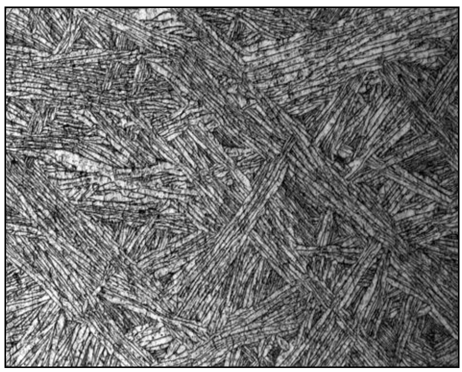


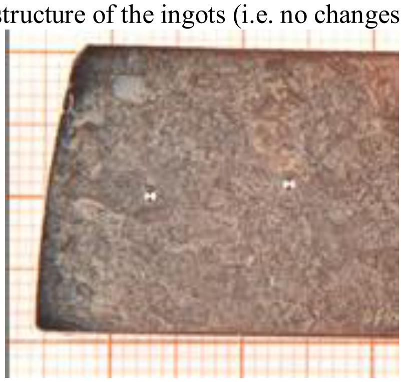
c)

x100

Figure 1. Microstructure of the samples made of Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy without Gd (a), with 0.04% of Gd (b) and 0.18% of Gd (c).

X-ray diffraction analysis of the samples cut from the ingots with the different chemical composition showed that the phase composition corresponded to near-α-titanium alloys: the substrate was presented by α-matrix with small (less than 5%) volume fraction of β-phase. No additional reflections corresponding to the other phases were identified.

Despite of changes in the intragranular structure we did not observed any visible changes in macrostructure of the ingots (i.e. no changes of grain size) under increase of Gd content (Fig. 2).



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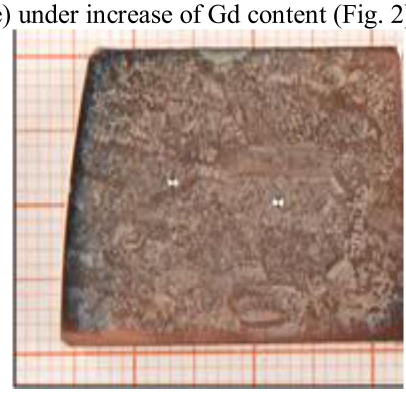

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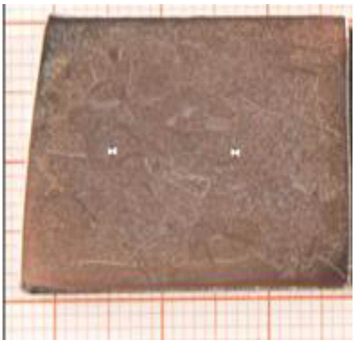


c)

Figure 2. Macrostructure of the ingots from Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy without Gd (a), and with 0.04% of Gd (b) and 0.18% of Gd (c).

To evaluate the influence of Gd content on the initial β-grain's size the samples cut from the upper part of the ingots were quenched from the temperature of 1060°C which is to be corresponded to a single β-phase region for all of the compositions according to preliminary evaluation based on reference data. The grain's size was evaluated by the Axio Observer A1m optical microscope with the use of the Image Expert Pro3 software; measurements were carried out in the two orthogonally related directions in more than 15 fields of view.

An average size of the initial β-grain equaled approximately 1700 μm in Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy without Gd; in the alloy with 0.04% of Gd the initial β-grains were greatly prolonged and had the average width of about 1550 μm and the length of more than 2000 μm; and at 0.18% of Gd the average size of the initial β-grain equaled about 1700 μm as in the alloy without Gd. Thus the conducted investigations showed that the additional alloying of Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy with Gd had no significant influence on the initial β-gran's size in as-cast conditions.

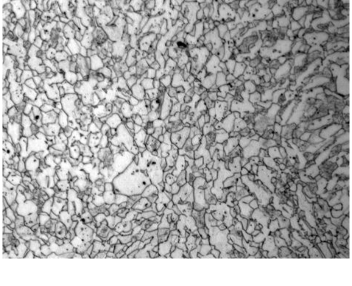
3.2 Investigation of deformed alloys

On the next stage of the work hot-rolled plates were made from the experimental ingots.

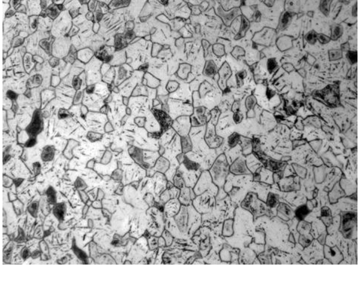
Rolling of the casted workpieces to the width of 13 mm was carried out at the temperature of 980°C after holding during 30 minutes at this temperature. Total degree of deformation equaled 70%. Rolled workpieces were air-cooled to the room temperature and subjected to mechanical treatment.

Samples cut from the rolled workpieces were used to determine polymorphic transformation temperature (PTT) by the trial quenching method. To this effect the samples were heated up to the temperature range of 960°−1060°C and water-cooled after isothermal holding during 30 minutes at these temperatures. Phase transition temperature was determined by metallographic method according to the occurrence of primary α-phase particles. The studies showed that PTT equaled 1000°C in Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy without Gd. Increase of Gd content resulted in gradual growth of PTT up to 1020° and 1040°C for the alloys with 0.04% and 0.18% of Gd, respectively (Figure 3).

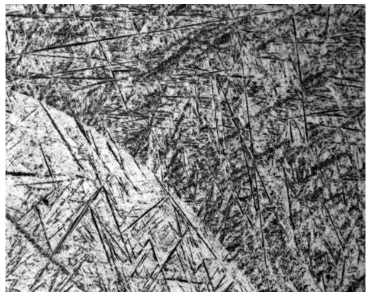
970°C



990°C



1010°C



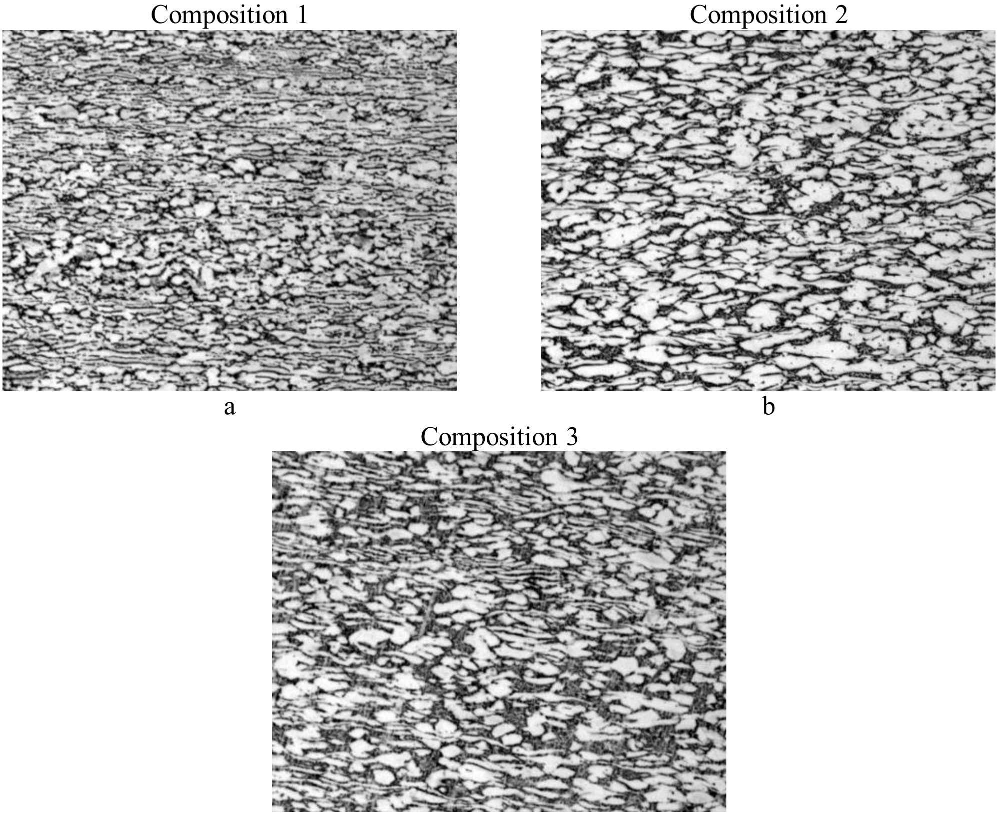
×200

Figure 3. Microstructure of the samples made of the alloy with Composition 3 after different quenching temperatures

Determination of PTT allowed developing proper modes of rolling to obtain plates with optimal structure from casted workpieces with the different compositions.

Structure of the rolled plate with Composition 1 presented typical structure of heat-resistant near-α-titanium alloys after rolling near PTT (Fig. 4 a). Additional alloying with Gd resulted in decrease of the primary α-phase volume fraction and increase of the secondary α-phase formed in β-phase during cooling from rolling temperature down to the room temperature (Fig. 4 b and c).

Composition 1 Composition 2



a b

Composition 3

c

×200

Figure 4. Microstructure of plates made of the pilot alloys with different chemical compositions after rolling

Rolling of the workpiece with Composition 2 began at the temperature by 40°C lower than PTT, and for the workpiece with Composition 3 it was by 60°C lower than PTT. Therefore at rolling temperature structure of the alloys with Compositions 2 and 3 consisted of greater quantity of α-phase than the alloy with Composition 1. Evidently the difference in structure is related to the presence of Gd. Cooling to the room temperature after rolling of the workpiece with Compisition 1 results mainly in the growth of existing primary α-phase particles, but in case of Compositions 2 and 3 decomposion of β-phase occurs with precipitation of dispersed secondary α-phase. It is formation of more dispersed structure that contributes to increase of workpieces hardness after hot plastic deformation: from 37 HRC units for Composition 1 up to 39.5 HRC units for Composition 3 (Table 1).

Table 1. Hardness of rolled plates made of the pilot alloys depending on Gd content

Gd content	Hardness, HRC units
Composition 1 (without Gd)	37.0
Composition 2 (0.04% Gd)	38.0
Composition 3 (0.18% Gd)	39.5

As it was mentioned above the additional alloying of heat-resistant Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy with Gd did not lead to any significant change of the initial β-grain size in as-cast condition. Therefore on the next stage of the work we evaluated the initial β-grain size after hot plastic deformation. Samples cut thereto from the rolled plates of different compositions were quenched from the temperature of 10°C higher than the corresponding PTT, i.e. from 1010°, 1030° and 1050°C for Compositions 1, 2 and 3, respectively. Measurements were made in the two orthogonally related directions in more than 20 fields of view.

Investigation carried out showed that the addition of Gd had an impact on the β-grain size in deformed semi-finished products: in the alloy containing 0.18% of Gd the grain size was 3.5 times less than in the alloy without Gd.

4 Conclusions

1. Alloying of the heat-resistant Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si titanium alloy with gadolinium has no influence on initial β-grain size in as-cast condition but contributes to transformation of intragranular structure and changes its type.
2. Addition of gadolinium results in gradual increase of polymorphic transformation temperature in the pilot alloy at the growth of Gd content: from 1000°C in the alloy without Gd up to 1040°C in the alloy containing 0.18% of Gd.
3. Addition of gadolinium to the Ti−6.5Al−4Zr−2.5Sn−2.4V−1Nb−0.5Mo−0.2Si alloy contributes to grain refinement in deformed state.

Acknowledgements

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