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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MULTICOMPONENT $Ti_{61}Cr_{10}Al_7V_{22}$ ALLOY

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The empirical and semi-empirical models were used to analyze the phase-structural state of lightweight alloys of the Ti-Cr-Al-V system. The composition of lightweight alloy $Ti_{61}Cr_{10}Al_7V_{22}$ (at.%) was selected for experimental study. Ingots of this alloy were obtained by argon-arc melting method, and they were subjected to homogenization, deformation by rolling and subsequent annealing at different temperatures. The influence of annealing temperature on the phase-structural state of the alloys, hardness, and mechanical properties during tensile tests at room temperature and 650 ºC has been studied experimentally. In cast condition and after deforming and annealing at 700…900 ºC the alloy has a single-phase state with bcc lattice. In the annealing state alloy has a high value of strength characteristics and the elongation to fracture.

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INTRODUCTION

The development of nuclear energy and its safety depend significantly on the creation of new structural materials that must have improved mechanical properties and corrosion resistance in operation, as well as high radiation resistance. In recent years, along with the improvement of austenitic and ferritic-martensitic reactor steels [1, 2], much attention has been paid to the development and research of a fundamentally new class of radiation-resistant materials – so-called high-entropy or multi-component concentrated alloys [1, 3–6].

Titanium-based alloys are of considerable interest to the nuclear power industry, as they are already used as structural materials for the manufacture of capacitors, steam turbine working blades, heat exchange equipment, and in the production of power fasteners for flange connections and connectors of various technological systems of reactor equipment for nuclear and fusion plants [7–9]. The advantages of titanium alloys are high specific strength, corrosion resistance in various environments, stability of mechanical properties, processability, nuclear properties, and the ability to weld with various types of welding. In recent years, in connection with the development of the latest nuclear power plants with fast neutron reactors, the possibility of using titanium alloys for the manufacture of fuel element cladding has been considered. Fast neutron reactors operate with a liquid metal cooling system. When melts of low-melting heavy metals are used as cooling environment, the main problem is corrosion compatibility and stability of mechanical properties of structural materials in interaction conditions with liquid metals [10, 11]. Lightweight titanium-based multicomponent alloys typically contain aluminum and vanadium in high concentrations, which allows for low densities. Finding a balance between density, mechanical properties, and corrosion resistance of lightweight alloys is a critical issue. To achieve a balance of these properties, it is reasonable to consider four component titanium alloys containing, in addition to aluminum and vanadium, a significant proportion of chromium, which is a corrosion-resistant element. In [12], the structure and mechanical properties of multicomponent titanium alloys $Ti_{60}Cr_{11}Al_7Nb_{22}$ and $Ti_{60}Cr_{11}Al_7Nb_{11}V_{11}$ (at.%) were studied. In the cast state, these alloys are single-phase and have a bcc lattice. The same state is realized after deformation and annealing at a temperature of 900 °C and above. However, at lower annealing temperatures (700…800 °C), precipitates of the brittle Laves phase are formed in the alloys, which leads to a decrease in the ductility of the alloys. The formation of the Laves phase is observed in many alloys containing niobium [13, 14].

The goal of this work is to create a lightweight titanium alloy based on a system Ti-Cr-Al-V that does not contain niobium and does not lose ductility when operating in the temperature range of 700…800 °C.

PREDICTION OF PHASE COMPOSITION AND LATTICE STRUCTURE OF Ti-Cr-Al-V ALLOYS

Since there are many possible variants of alloy constituents with 4 components, the issue of the phase structure formed in an alloy of a particular elemental composition is extremely important. Typically, the phase composition of alloys is determined by phase diagrams, which are traditionally obtained experimentally. This approach is poorly implemented for complex multicomponent systems. The prediction of the phase composition of multicomponent alloys can be made on the basis of a number of thermodynamic, dimensional, and electronic parameters. Currently, a significant number of empirical and semi-empirical criteria have been proposed that allow us to "predict" the structural state of a particular alloy with a certain degree of reliability. These criteria are described below.

Main calculation formulas and criteria. According to thermodynamics, in a multicomponent alloy in the equilibrium state realizes the phase composition, which corresponds to the minimum value of Gibbs free energy G:

$$G = H - TS\,, \qquad (1)$$

where H – enthalpy of the system; S – entropy of the system; T – absolute temperature.

As a result of mixing n components (e.g., alloying) there is a change in the free energy of the system (alloy)

$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}, \quad (2)$$

where ΔH_{mix} – mixing enthalpy; ΔS_{mix} – mixing entropy; T – absolute temperature in Kelvin.

In the ideal solution approximation, the mixing entropy can be represented as follows [13]:

$$\Delta S_{mix} = -R\sum_{i=1}^{n} c_i ln c_i, \quad R = 8.314\ \text{Jmol}^{-1}\text{K}^{-1}, \quad (3)$$

where R – gas constant; c_i – concentration of the i-th component in the alloy.

The mixing enthalpy for a multicomponent system can be calculated by the formula:

$$\Delta H_{mix} = \sum_{i=1, i\neq j}^{n} \Omega_{ij} c_i c_j, \quad \Omega_{ij} = 4\Delta_{mix}^{AB}, \quad (4)$$

where Δ_{mix}^{AB} – the mixing enthalpy of the binary equiatomic ab alloy, calculated on the basis of the Miedema model [25]; $c_{i(j)}$ – the concentration of the i(j) element of the alloy.

Since the choice of phases is based on the ratio between ΔH_{mix} and $T\Delta S_{mix}$ according to thermodynamics, it was proposed to use a dimensionless parameter Ω that takes into account the contribution of these parameters [18]. In this case, the temperature is fixed at the melting point of the alloy:

$$\Omega = \frac{T_m \Delta S_{mix}}{|\Delta H_{mix}|}, T_m = c_i (T_m)_i, \quad (5)$$

where T_m – the average melting point of the components; c_i – the concentration of the i-th alloy element.

The analysis carried out by various authors has shown that it is necessary to consider such parameters as atomic size factor δ and electronic characteristics of atoms – average values of valence electron concentration VEC and Pauling electronegativity Δχ - in addition to the considered thermodynamic parameters for more correct prediction of phase composition and crystal structure of phases. Expressions for these quantities are given below [16]:

$$\delta = 100 \times \sqrt{\sum_{i=1}^{n} c_i (1 - r_i/\bar{r})^2}, \bar{r} = \sum_{i=1}^{n} c_i r_i, \quad (6)$$

where r_i – atomic radius of the i-th alloy element; c_i – concentration of the i-th alloy element.

$$VEC = \sum_{i=1}^{n} c_i (VEC)_i, \quad (7)$$

where $(VEC)_i$ – the valence electrons concentration of the i-th alloy element; c_i – the concentration of the i-th alloy element.

Table 1

Criteria for determining the phase composition and crystal lattice of HEA

Criterion A: correlation between ΔS_{mix}, ΔH_{mix}, and δ			
A1	$-15 \le \Delta H_{mix} \le 5$ kJ/mol $0 < \delta < 5$	simple disordered solid solution (SDSS)	[15]
	$-20 \le \Delta H_{mix} \le 0$ kJ/mol $5 < \delta < 6.6$	disordered solid solutions (DSS)	[16]
A2	$-22 \le \Delta H_{mix} \le 7$ kJ/mol $0 \le \delta \le 8.5$ $11 \le \Delta S_{mix} \le 19.5$ J/Kmol	solid solution (SS)	[17]
A3	$-10 \le \Delta H_{mix} \le 5$ kJ/mol $\delta < 4$ $\Delta S_{mix} > 13.38$ J/Kmol	simple solid solution (SSS)	[18]
	$50 \le \Delta H_{mix} \le 0$ kJ/mol $4 \le \delta \le 20$ $4 \le \Delta S_{mix} \le 18$ J/Kmol	amorphous phase (AF)	[19]
Criterion B: correlation between Ω and δ			
B	$\Omega > 1.1$ $\delta < 6.6$	solid solution (SS)	[20]
Criterion C: correlation between VEC and δ			
C1	$\delta < 5$	FCC lattice	[21]
	$\delta > 5.4$	BCC lattice	
C2	VEC ≥ 8	FCC lattice	[22]
	VEC < 6.87	BCC lattice	
C3	4.33 < VEC < 7.55	BCC lattice	[23]
	7.80 < VEC < 9.50	FCC lattice	
Criterion D: correlation between Δχ and δ			
D	$0.03 < \Delta\chi < 0.06$ $1 < \delta < 6$	no intermetallic compounds (nC)	[24]
	$\Delta\chi \approx 0.09 \ldots 0.14$ $\delta \approx 6$	appearance of compounds (intermetallic, σ-phase) (aC)	

Electronegativity $\Delta\chi$ [17]:

$$\Delta\chi = \sqrt{\sum_{i=1}^{n} c_i(\chi_i - \bar{\chi})^2}\ , \bar{\chi} = \sum_{i=1}^{n} c_i\chi_i\ , \qquad (8)$$

where χ_i – the Poling electronegativity of the i-th alloy element; c_i – the concentration of the i-th alloy element.

There are several criteria for determining the phase composition of alloys according to the literature. These criteria have been verified experimentally, and their values differ from source to source, i.e., different authors give different ratios of parameters at which a solid solution is formed.

In addition to the criteria determining the phase composition (solid solution or solid solution + compound), criteria for predicting the type of solid solution crystal lattice or the type of compound that can be formed in the alloy (e.g., Laves phase, sigma phase) were proposed. The proposed criteria are summarized in Table 1, with criteria based on the same parameters grouped into separate groups.

Predicted calculations of the phase composition and crystal structure of alloys of the Ti-Cr-Al-V system. The atomic parameters of individual elements included in Ti-Cr-Al-V alloys, required for calculations of the phase composition and crystal structure, are given in Table 2.

Table 2

The main parameters of the Ti-Cr-Al-V system elements

Element	Atomic number	Atomic radius, Å	Pauling's electro-negativity	VEC
Cr	24	1.249	1.66	6
Al	13	1.432	1.61	3
Ti	22	1.462	1.54	4
V	23	1.316	1.63	5

Table 3 shows the results of calculations of the enthalpies of interaction of different atomic pairs in the corresponding binary alloys obtained using the Miedema theory [25].

Table 3

Values of binary mixing enthalpies for the elements of the Ti-Cr-Al-V system

Alloy	Δ_{mix}^{AB}, kJ/mol	Alloy	Δ_{mix}^{AB}, kJ/mol
Cr-Al	-20.325	Ti-Al	-40.481
V-Al	-26.936	Cr-Ti	-7.348
Cr-V	-1.937	Ti-V	-1.650

With the use of the parameters given in Tables 2 and 3, we calculated the criteria for determining the structural state of a significant number of Ti-Cr-V-Al alloys of various compositions. An example of such calculations for 4 alloys with a fixed content of aluminum and vanadium (Table 4) is given in Tables 5 and 6.

Table 4

Compositions of several studied alloys of the Ti-Cr-Al-V system (at.%)

Sample	Element			
	Ti	Cr	Al	V
#1	61	10	7	22
#2	56	15	7	22
#3	66	5	7	22
#4	51	20	7	22

Most of the criteria indicate that all alloys should be single-phase solid solutions, i.e., without intermetallic phases. According to criteria C2 and C3, all alloys have a bcc lattice, but alloy #3 may have a lattice structure according to criterion C1.

Alloy #1 with the composition 61Ti-10Cr-7Al-22V (at.%) was selected for further experimental studies based on the calculations and analysis of literature data on the effect of chromium, aluminum, and vanadium on the mechanical and corrosion properties of titanium.

METHODS OF ALLOY OBTAINING AND STUDY OF STRUCTURE AND PROPERTIES

Ingots of the multicomponent alloy were produced by arc melting of the components with a non-consumable tungsten electrode in high-purity argon. Before melting, for additional argon purification, the titanium getter was melted for several minutes. The purities of the alloying elements were higher than 99.9%. To ensure chemical homogeneity, the ingots were flipped over and remelted at least 5 times, and ingots' size was 12 × 12 × 30 mm. After that, the homogenization annealing was carried out at 1100 °C for 1 h in high purity argon, followed by quenching in the air. After that, ingots were rolled to 1 mm with interim annealing at 1100 °C. The specimens for microstructure were produced from the received tapes by spark cutting.

X-ray diffraction (XRD) patterns of the specimens were recorded using "DRON-4-07" diffractometer. The microstructure of as-cast samples was studied using a metallographic inversion microscope Olympus GX51 and scanning electron microscope JSM 7001F equipped with a system for energy-dispersive X-ray spectroscopy (EDS) INCA ENERGY 350. The microhardness was measured using a PMT-microhardness tester. The load on the indenter was 200 g, the loading duration was 15 s.

Table 5

Calculated parameters for alloys of the Ti-Cr-Al-V system

Alloy/ Parameter	VEC	δ	Δχ	ΔS_{mix}, J/kmol	ΔH_{mix}, kJ/mol	Ω
#1	4.35	5.6	0.047	8.74	-11.99	1.22
#2	4.45	6.07	0.050	9.38	-12.40	1.27
#3	4.25	4.99	0.044	7.84	-11.44	1.15
#4	4.55	6.43	0.051	9.85	-12.66	1.31

Table 6

Predicted phase-structural state of Ti-Cr-Al-V alloys calculated according to different criteria

Alloy/ Criterion	A1	A2	A3	B	C1	C2	C3
#1	DSS	DSS?	TZ	SS	bcc	bcc	bcc
#2	DSS	DSS?	TZ	SS	bcc	bcc	bcc
#3	DSS	DSS?	TZ	SS	fcc	bcc	bcc?
#4	DSS	DSS?	TZ	SS	bcc	bcc	bcc

TZ – transition zone; ? – the parameter values go beyond the limits specified by the criterion.

Nano-hardness measurements were carried out by Nanoindenter G200 with a Berkovich type indentation tip. The mechanical properties were studied using testing machine INSTRON-5581 under active stretching with speed 10^{-3} s^{-1} at 20 and 650 °C. Samples for investigation were in the shape of double blades with working part dimensions of 1 × 2 × 10 mm produced by spark cutting along the rolling direction and annealed in a vacuum at different temperatures. The yield strength $\sigma_{0.2}$, tensile strength σ_b and elongation to failure δ were investigated. More detailed preparation and research methods are described in [12].

EXPERIMENTAL RESULTS AND DISCUSSION

Microstructure. It was established experimentally that the ingot has a composition of 60.9Ti, 10.3Cr, 7.0 Al, 21.8 V (at.%), which coincides well with the calculated value. XRD patterns of samples are shown in Fig. 1. As-cast and annealed samples are single – phase and have bcc crystal lattice with a = 3.171…3.173 Å. When studying the rolled and annealed samples by scanning electron microscopy, it was found that the microstructure of all samples is single phase (Fig. 2).

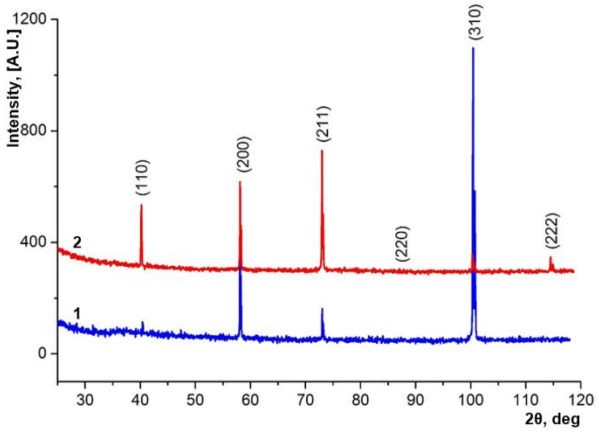

Fig. 1. Diffraction patterns of the alloy: 1 – as-cast state; 2 – after rolling and annealing at 900 °C/15 min

The average grain size in samples annealed at a temperature of 700 ºC for 1 h is about 15 μm, and in samples annealed at 900 ºC for 15 min it is about 60 μm.

Mechanical properties. Measurements of microhardness showed that the samples, both in the cast state and after deformation and annealing at 900 °C, are quite homogeneous, i.e., the deviations from the average value are very low.

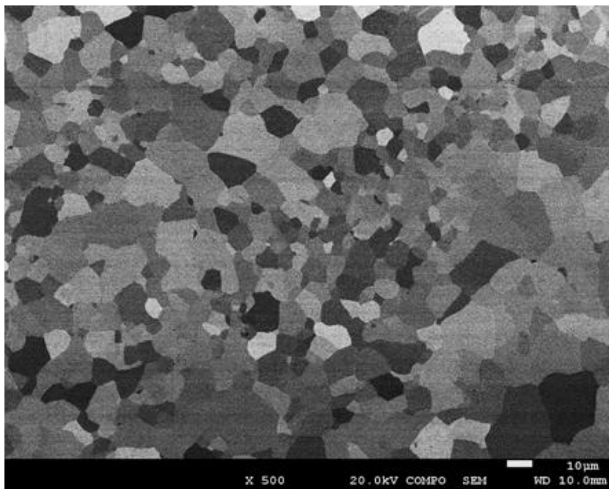

Fig. 2. Microstructure of $Ti_{61}Cr_{10}Al_7V_{22}$ alloy after rolling and annealing at 700 °C /1 h

Also, the microhardness values for these samples are similar and are (2960 ± 30) and (2990 ± 45) MPa for the as-cast alloy and the alloy after mechanical and thermal treatment, respectively. Wherein, nanohardness in the annealed sample is H = (4.7 ± 0.1) GPa, and the elastic modulus is E = (129 ± 3) GPa.

The results of mechanical tests at 20 ºC of alloy samples in different structural states (rolled, annealed after rolling at 700, 800 ºC/1 h, 900 ºC/15 min) in the form of true tensile curves are shown in Fig. 3. It can be seen that in the deformed state, the alloy has high strength characteristics $\sigma_{0.2}$ = 865 MPa, σ_b = 1184 MPa. At the same time, the ductility δ of the alloy is only about 2 percent. Annealing significantly increases the ductility of the alloy, while its strength characteristics, although reduced, remain at a fairly high level (Fig. 3). The alloy with the highest ductility is the one annealed at 900 °C for 15 min. In this condition, the total elongation is almost 30 percent, the proportionality limit is about 640 MPa, and the ultimate strength is about 750 MPa. A comparison with previously studied multicomponent titanium alloy $Ti_{60}Cr_{11}Al_7Nb_{11}V_{11}$ shows that the four-component alloy after annealing at 700 °C has a slightly higher tensile strength than the five-component alloy (947 MPa versus 900 MPa) and significantly higher ductility (more than 20 versus 7%).

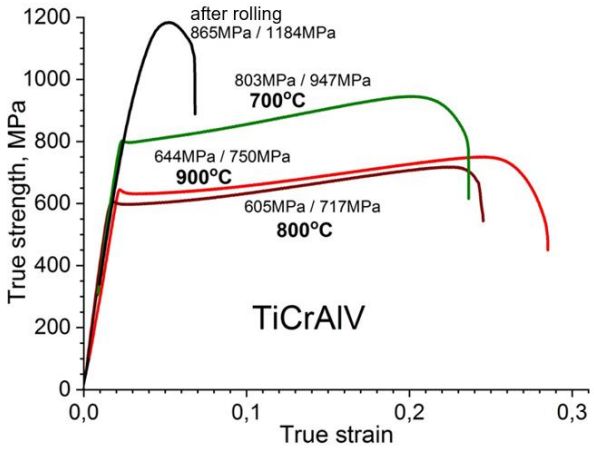

Fig. 3. True strain curves of $Ti_{61}Cr_{10}Al_7V_{22}$ alloy at room temperature for a deformed sample (rolled) and samples annealed at different temperatures (700, 800 °C/1 h, 900 °C/15 min). Numbers next to the curves: numerator – yield strength, denominator – ultimate strength

An unusual feature of the developed alloy is that all the characteristics (proportionality limit, tensile strength, and total elongation to break) at deformation temperatures of 20 and 650 °C are almost the same (Fig. 4), although the uniform strain at 650 °C is approximately two times lower than at 20 °C. That is, the formation of the neck during tension at 650 °C occurs much earlier than at 20 °C, which indicates a lower ability to strengthen at 650 °C. The fracture patterns shown in Fig. 4 reflect the plastic nature of fracture at both deformation temperatures.

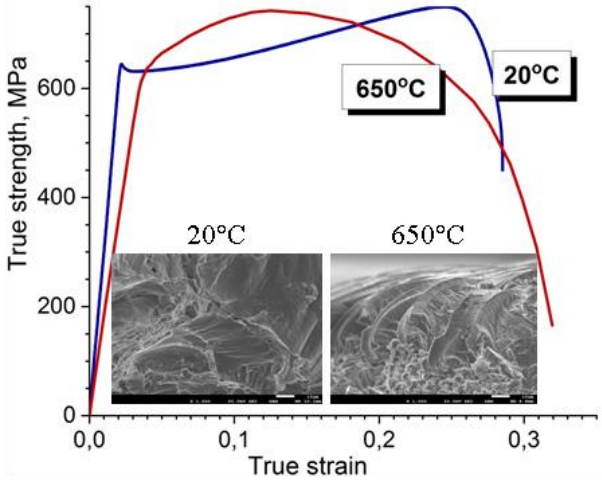

Fig. 4. True strain curves of 61Ti-10Cr-7Al-22V alloy at room temperature and 650 °C for samples annealed at 900 °C for 15 min (fractograms of fractured samples at different temperatures are shown in the inset)

CONCLUSIONS

1. In order to develop a new titanium alloy with enhanced mechanical properties at temperatures up to 650 °C, the phase composition and crystal structure of a series of Ti-Cr-Al-V alloys were calculated. On the basis of these calculations, the composition of a light concentrated alloy was chosen ($Ti_{61}Cr_{10}Al_7V_{22}$), which is most likely to be single-phase with a body-centered cubic lattice.

2. Experimentally it was revealed that after annealing at 700…900 °C chosen alloy $Ti_{61}Cr_{10}Al_7V_{22}$ remains single-phase bcc alloy with a lattice parameter a = 3.171…3.173Å.

3. It was found that annealed at 700 °C/1 h alloy has tensile strength at 20 °C about 950 MPa and elongation for rupture – more than 20%. After annealing at 900 °C/15 min the strength of alloy at 20 and 650 °C (operation temperature of new generation nuclear reactors) is practically identical (about 650 MPa) and elongation for rupture is 26…28%.

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МІКРОСТРУКТУРА ТА МЕХАНІЧНІ ВЛАСТИВОСТІ БАГАТОКОМПОНЕНТНОГО СПЛАВУ $Ti_{61}Cr_{10}Al_7V_{22}$

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Для аналізу структурно-фазового стану легких сплавів системи Ti-Cr-Al-V використовувалися емпіричні та напівемпіричні моделі. Для експериментальних досліджень було обрано легкий сплав $Ti_{61}Cr_{10}Al_7V_{22}$ (ат.%). Зливки цього сплаву були отримані методом аргонодугової плавки, після чого вони піддавалися гомогенізації, деформації прокаткою і подальшому відпалу при різних температурах. Експериментально досліджено вплив температури відпалу на структурно-фазовий стан сплавів, твердість та механічні властивості при випробуваннях на розтягування при кімнатній температурі та 650 ºС. У литому стані та після деформації і відпалу при 700…900 ºС сплав має однофазний стан з ОЦК-граткою. У відпаленому стані сплав має високі значення характеристик міцності та відносного подовження до руйнування.