PHYSICAL METALLURGY AND HEAT TREATMENT / МЕТАЛЛОВЕДЕНИЕ И ТЕРМИЧЕСКАЯ ОБРАБОТКА

UDC 544.034
https://doi.org/10.17073/0021-3438-2023-1-75-83

Research article
Научная статья

Interdiffusion in refractory metal systems with a BCC lattice: titanium–tantalum and titanium–multicomponent (high-entropy) alloy

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Abstract: In this work, the interdiffusion features in multicomponent (high-entropy) alloys of refractory metals were studied. The following pairs were chosen as the diffusion study objects: titanium–equiatomic alloy (Hf–Nb–Ta–Ti–Zr–Mo) and titanium–tantalum for the sake of comparison. The article covers the issues of sample preparation, microstructure study, sample preparation methodology for diffusion research, and experimental results. Diffusion annealing was carried out for 12 h in a vacuum at a residual argon pressure of $6.65 \cdot 10^{-3}$ Pa and a temperature of 1200 °C. Particular attention was paid to the method of combining diffusion pairs (titanium with tantalum, titanium with alloy) by thermal cycling near the polymorphic transformation temperature in titanium (882 °C) within ± 50 °C. The behaviour of the most characteristic elements (Ta, Zr, Ti) in the weld area after the titanium and alloy diffusion pair joining was demonstrated. This is the first time that data on the dependence of the intensity of the corresponding spectral line for titanium and elements of a multicomponent alloy on the penetration depth were obtained. A change in the signal intensity for system elements was observed at a depth of 150–200 μm, whereas a sharp drop in the signal intensity was seen to occur at depths of about 50 μm. The effective value of the coefficient of diffusion of elements into titanium averaged over all elements of the alloying system (except for titanium) at a temperature of 1200 °C was calculated. The obtained value was compared to reference data: the self-diffusion coefficient in β-titanium and diffusion coefficients in titanium pairs with alloy doping elements.

Keywords: high-entropy alloys based on multiple refractory metals, interdiffusion parameters

Funding: This study was carried out with the financial support of a grant from the Russian Foundation for Basic Research (Project No. 20-03-00387).

Acknowledgments: The authors acknowledge V.K. Portnoi and E.A. Kolesnikov for their help with the experiments.

For citation: Razumovsky M.I., Rodin A.O., Bokstein B.S. Interdiffusion in refractory metal systems with a BCC lattice: titanium–tantalum and titanium–multicomponent (high-entropy) alloy. *Izvestiya. Non-Ferrous Metallurgy*. 2023; 29 (1): 75–83. (In Russ.).
https://doi.org/10.17073/0021-3438-2023-1-75-83

Взаимная диффузия в системах на основе тугоплавких металлов с ОЦК-решеткой: титан–тантал и титан – многокомпонентный (высокоэнтропийный) сплав

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Аннотация: Изучены особенности взаимной диффузии в многокомпонентных (высокоэнтропийных) сплавах на основе тугоплавких металлов. В качестве объектов диффузионного исследования были выбраны следующие пары: титан – эквиатомный сплав (Hf–Nb–Ta–Ti–Zr–Mo) и, для сравнения, титан–тантал. Рассмотрены вопросы приготовления образцов, исследования микроструктуры, методика подготовки образцов для изучения диффузии и экспериментальные результаты. Диффузионный отжиг был проведен в течение 12 ч в вакууме с остаточным давлением аргона $6{,}65 \cdot 10^{-3}$ Па при температуре 1200 °С. Особое внимание уделено методике соединения диффузионных пар (титана с танталом, титана со сплавом) путем термоциклирования вблизи температуры полиморфного превращения в титане (882 °С) в пределах ±50 °С. Показано поведение наиболее характерных элементов (Ta, Zr, Ti) в области сварного шва после соединения диффузионной пары титана и сплава. Впервые получены данные о зависимости интенсивности соответствующей линии спектра для титана и элементов многокомпонентного сплава от глубины проникновения. Изменение интенсивности сигнала для элементов систем наблюдается на глубине 150–200 мкм, а резкое падение интенсивности сигнала происходит на глубинах порядка 50 мкм. Рассчитано усредненное по всем элементам системы легирования сплава (за исключением титана) эффективное значение коэффициента диффузии элементов в титан при температуре 1200 °С. Проведено сравнение полученного значения со справочными данными: коэффициентом самодиффузии в β-титане, коэффициентами диффузии в парах титана с легирующими элементами сплава.

Ключевые слова: высокоэнтропийные сплавы на основе многих тугоплавких металлов, параметры взаимной диффузии

Финансирование: Работа выполнена при финансовой поддержке гранта Российского фонда фундаментальных исследований (проект № 20-03-00387).

Благодарности: Коллектив авторов выражает благодарность В.К. Портному и Е.А. Колесникову за помощь в проведении экспериментов.

Для цитирования: Разумовский М.И., Родин А.О., Бокштейн Б.С. Взаимная диффузия в системах на основе тугоплавких металлов с ОЦК-решеткой: титан–тантал и титан – многокомпонентный (высокоэнтропийный) сплав. *Известия вузов. Цветная металлургия*. 2023; 29 (1): 75–83. https://doi.org/10.17073/0021-3438-2023-1-75-83

Introduction

Achieving higher-than-average operating temperatures of gas turbine engines up to 1400 °C requires the use of alloys with a melting point exceeding 2000 °C. Refractory metal alloys are the most suitable for this purpose. In addition to their high melting points, these alloys usually contain more than 5 elements with a concentration of 5 to 35 % each, and hence are characterised by high configurational entropy. This is why they were called high-entropy alloys (HEA) [1, 2].

Since the increase in entropy is associated with an increase in the system disorder, the suggestion was made that the diffusion could be slowed down by the sluggish diffusion effect [3, 4]. There is very little data available on the dependence of the interdiffusion coefficients on the composition of the alloy in binary systems of refractory metals, even less for multicomponent alloys. The objective of this study was to select and analyse articles published in high-level influential journals (Acta Materialia, Journal of Alloys and Compounds, Intermetallics, etc.) in the 2018—2020 period. The articles were selected based on two criteria: 1) the subject matter of the study shall be multicomponent HEA; 2) the results obtained shall concern the diffusion mobility of the components.

Among the 47 articles selected, only 10 [5—14] met the first criterion, whereas only 3 articles [6; 9; 11] covered the problems of diffusion in the HEA of refractory metals with a BCC lattice. There are much more studies on diffusion in multicomponent HEAs with an FCC lattice than on those with a BCC lattice. The authors of [7] believe that “such studies still have a long way to go”.

The general conclusions may be formulated as follows: the slowing down of diffusion in high-entropy alloys (sluggish effect) is not uniformly present in all HEAs and depends on their composition; the results obtained by *X*-ray spectral microanalysis (EDX) proved to be the most informative; the sluggish diffusion effect is more pronounced for BCC structures than for FCC structures, although there have been much more studied conducted on FCC structures that are currently available; some authors suggest the possibility of the occurrence of a dissociative or interstitial mechanism in a BCC HEA, which is a consequence of the disordered structure, leading to increased, rather than sluggish, diffusion.

The main task of this study, therefore, is to carry out a comparative analysis of diffusion processes in diffusion pairs — one containing an alloy and the other containing single-component substances. The choice of the alloy composition — the multicomponent equiatomic alloy HfNbTaTiZrMo, is based on the analysis made in our review [15]

Sample preparation

To prepare the initial mixtures, «high-purity» class (99.96 %) powders of components (Hf, Nb, Ta, Ti, Zr, and Mo) with particle sizes of 40—80 μm were used. The powder mixture[1] was ground in a P-7 planetary mill (Fritsch, Germany) in an argon atmosphere at a powder-to-ball mass ratio of 1 : 8.

Hot isostatic pressing (HIP) was carried out in a laboratory gasostatic extruder at a temperature of 1200 °C for at least 2 h of exposure, and the powders were compacted in steel capsules that had been pre-evacuated during HIP.

X-ray spectral microanalysis (EDX) of the compacted samples was carried out using a scanning electron microscope of the brand LEO EVO 50 XVP (Carl Zeiss, Germany) with an INCA ENERGY (Oxford Instruments, UK) module.

The main phase obtained during the powder mixture grinding is a BCC solid solution with a lattice period $a \sim 0.34$ nm [16].

Microstructure of multicomponent alloys

In this work, thin, polished sections of pure titanium, tantalum, and an alloy based on multiple refractory metals were made. A mixture of hydrofluoric and nitric acids was used to reveal the resulting grain structure.

The structure was studied using an Axio Vert.A1 (Carl Zeiss, Germany) optical microscope and a JSM-6480LV (SEM) (JEOL, Japan) electron microscope equipped with an INCA ENERGY Dry Cool (Oxford Instruments, Great Britain) energy-dispersive spectroscopy module, as well as on a FEGFEINavaNanoSEM (Field Electron and Ion Company, FEI, USA) microscope fitted with a Bruker (Germany) module.

Fig. 1 shows a photograph of the homogeneous structure of one of the alloy samples.

Sample preparation method for diffusion studies

Diffusion welding was used for the diffusion contact of the samples. A stainless-steel clamp was placed in a quartz reactor in a vertical pipe furnace to provide and maintain the ~2 MPa pressure required for welding. The interior part of the reactor was argon-purged to exclude any oxidation.

The mode of preparation of diffusion pairs consisted in thermal cycling at approximately the titanium polymorphic transformation temperature ±50 °C. One cycle consisted in heating the samples to a temperature of 930 °C and cooling them down to a temperature of 830 °C with multiple cycling or to room temperature with a single cycling. The cooling procedure involved

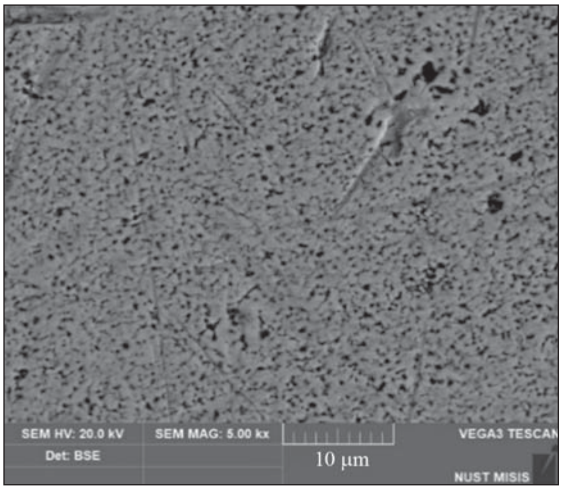

Fig. 1. SEM image of the alloy sample structure

Рис. 1. Электронное изображение (SEM) структуры образца сплава

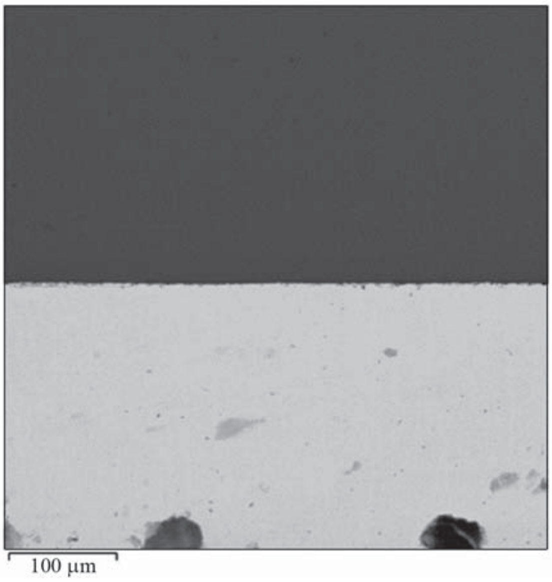

Fig. 2. SEM image of a welded joint between titanium and tantalum plates

Рис. 2. Электронное изображение (SEM) сварного шва между пластинами титана и тантала

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[1] Samples of a refractory-metal multicomponent alloy were obtained using equipment of Lomonosov Moscow State University.

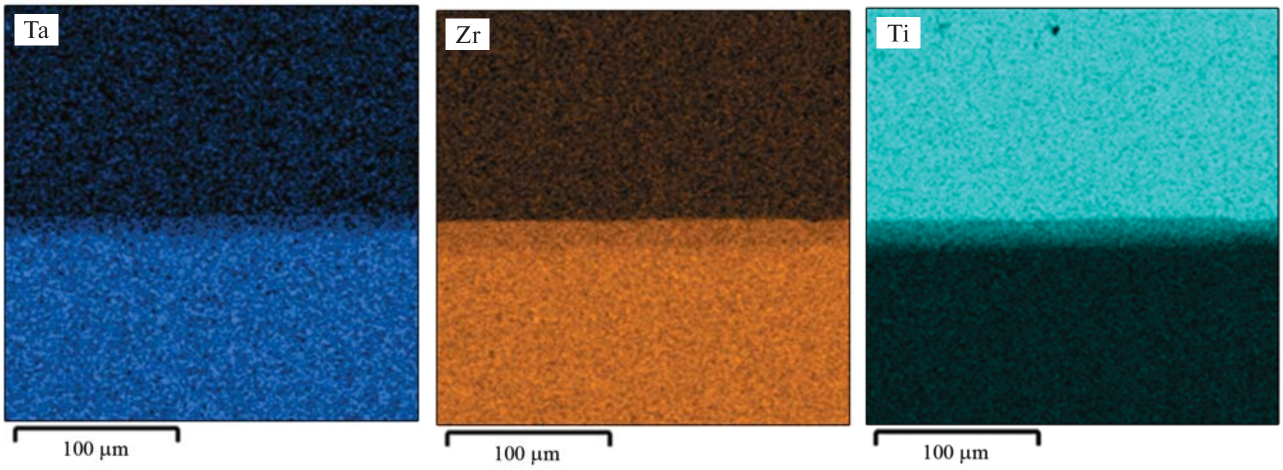

Fig. 3. SEM-EDS image showing element distribution in the welded titanium—alloy joint area

Рис. 3. Изображение SEM-EDS распределения элементов в области сварного шва титана и сплава

removing the quartz reactor containing the samples from the furnace, followed by holding at room temperature. A TVR A1 thermocouple was used to determine the change in temperature of the samples in the reactor.

After welding, the samples were specially processed to be able to examine the welded joint. For this, parts of the welded samples were removed by machining with grinding wheels, following which the welded joint areas were polished and then examined by *X*-ray spectroscopy. Fig. 2 and Fig. 3 show the SEM image of the weld and the element distribution in the welded joint area.

The size of the transition zone formed as a result of the fabrication of the diffusion pair is within a few micrometres and, apparently, is a consequence of phase recrystallisation. The obtained weld joints appeared continuous, with no pores or contraction cavities.

Experimental results and estimated diffusion coefficients

Diffusion annealing was carried out for 12 h in a vacuum with a residual argon pressure of $6.65 \cdot 10^{-3}$ Pa at a temperature of 1200 °C. After annealing, the samples were cut along the direction of diffusion penetration and the resulting surface ground and polished. Fig. 4 shows a typical polished surface. The lower, darker part is titanium, and the lighter part is the alloy. The dots show the areas of energy-dispersive analysis of the chemical composition. The distance between the dots is about 35 µm. Fig. 5 shows a typical spectrum.

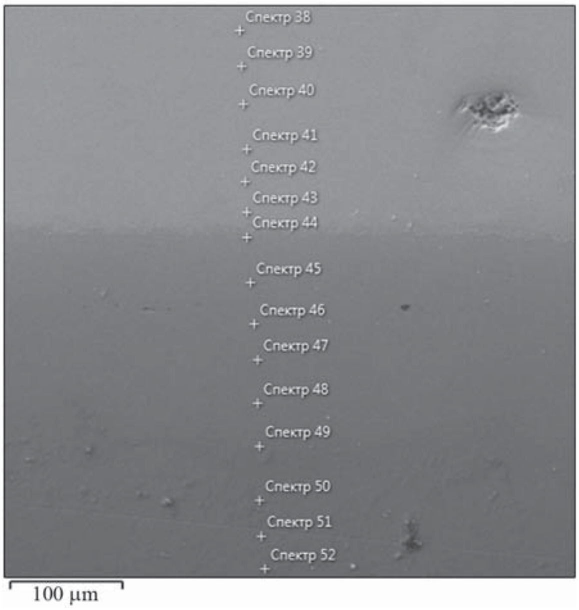

Fig. 4. EDX of an interdiffusion zone of a titanium—alloy pair (white crosses accordance each spectrum numbers from 38 to 52)

Рис. 4. РСМА зоны взаимной диффузии в паре сплав—титан

It is clear that interdiffusion is associated mainly with the penetration of the alloy components into titanium as the more fusible part of the system (Fig. 6); a change in signal intensity is observed at a distance of 150—250 µm for all elements. A sharp drop in intensity takes place at depths of ±50 µm.

Data on the recalculated concentrations of the components are given in the table.

Figure 7 shows data on the dependence of the intensity of the corresponding spectral line for titanium and other alloy components.

Fig. 8 shows the concentration distribution of all elements of the HEA doping system in the interdiffusion zone. For comparison, a line corresponding to the description using the erfc-shaped solution is shown [17; 18]:

$$C(x,t) = C_n + \frac{C_0 - C_n}{2}\left\{1 - \operatorname{erf}\left(\frac{x - x_0}{2\sqrt{Dt}}\right)\right\},$$

where C_n is the minimum concentration of the reference element; C_0 is the element concentration in the equiatomic alloy; D is the diffusion coefficient; t is the time; x_0 is the interface coordinate.

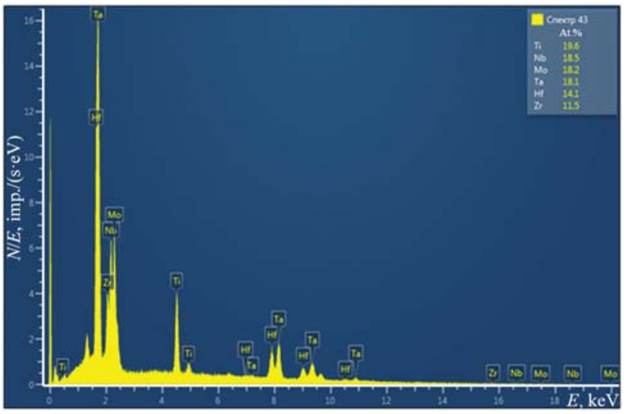

Fig. 5. Typical EDX spectrum in the interdiffusion zone of a titanium–alloy pair near the interface

Рис. 5. Типичный спектр РСМА в зоне взаимной диффузии пары титан–сплав вблизи поверхности раздела

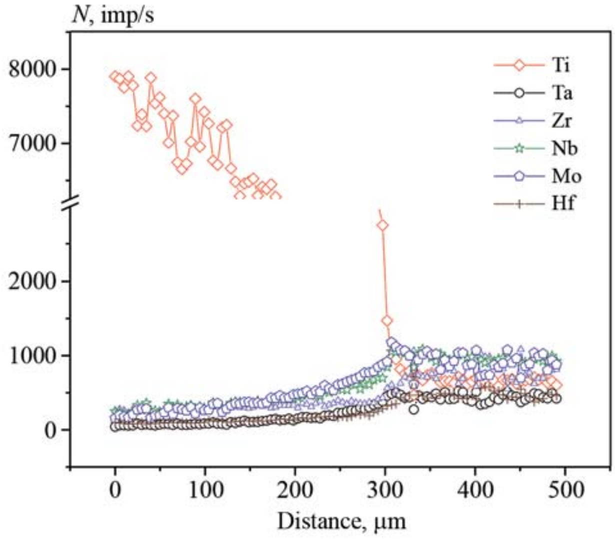

Fig. 6. Change in intensity of spectral lines in the interdiffusion zone of the titan–alloy pair

Рис. 6. Изменение интенсивности линий спектра в зоне взаимной диффузии в паре титан–сплав

Element concentration in the interdiffusion zone, at.%

Концентрации элементов в зоне взаимной диффузии, ат.%

Spectrum Ref. No.	Coordinate, µm	Ti	Zr	Hf	Mo	Ta	Nb
38	0	15.6	20.6	18.3	14.7	14.7	14.7
39	35	16.6	17.9	16.6	15.6	16.7	16.6
40	70	17.6	9.3	13.5	19.7	19.6	19.4
41	105	17.0	13.4	15.5	18.4	18.4	17.3
42	140	17.8	14.3	15.9	17.3	16.7	18.0
43	175	19.6	11.5	14.1	18.2	18.1	18.5
44	210	60.4	3.6	5.7	9.8	10.0	8.4
45	245	75.6	2.9	3.9	5.2	5.1	4.6
46	280	80.0	2.7	2.7	3.5	3.5	3.3
47	315	83.3	2.5	2.4	2.8	2.7	3.0
48	350	85.5	2.1	2.0	2.1	2.0	2.3
49	385	87.5	2.1	1.9	1.6	1.4	1.7
50	420	87.3	2.2	1.5	1.6	1.1	1.7

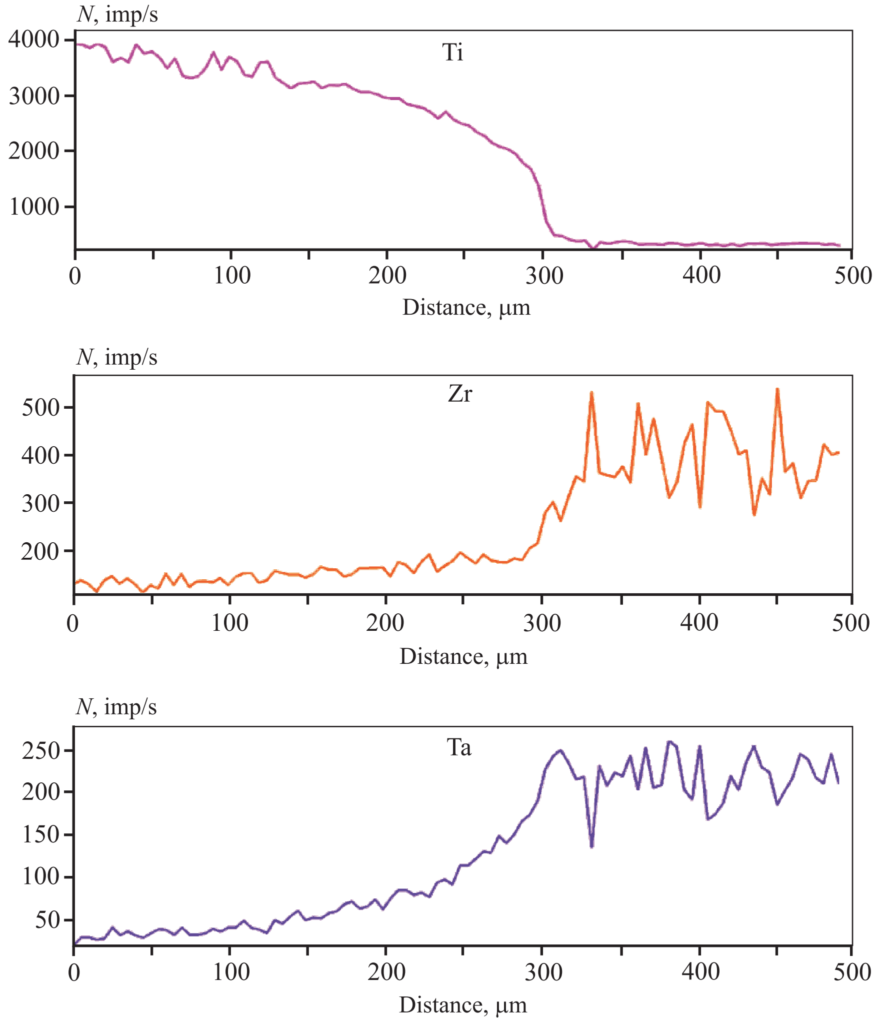

Fig. 7. Intensity profiles of some alloy components in the interdiffusion zone

Рис. 7. Профили интенсивности некоторых компонентов сплава в зоне взаимной диффузии

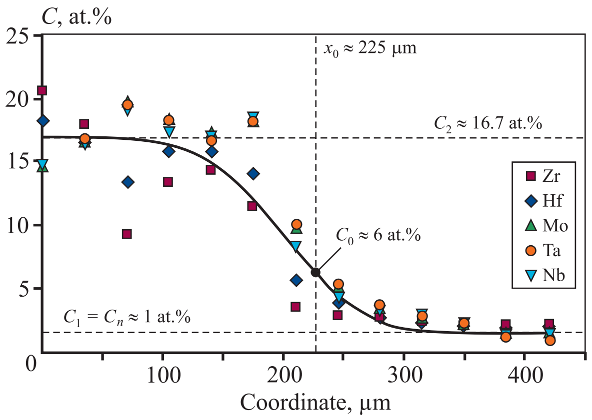

Fig. 8. Element distribution in the alloy–titanium diffusion zone

Рис. 8. Распределение элементов в диффузионной зоне пары сплав–титан

In Fig. 8, to the left of the x_0 plane is the alloy region and to the right is the titanium region.

Elements tend to have a similar behaviour that can be described well by the proposed solution. The experimental points for the titanium—HEA pair correspond to the diffusion coefficient $D = 3.0 \cdot 10^{-14}$ m^2/s.

Similar studies were carried out for the titanium—tantalum pair. Fig. 9 shows the photomicrograph and concentration profile of this pair.

The results presented show a significant difference between the diffusion processes in tantalum—titanium and alloy—titanium pairs. The data obtained for the titanium—tantalum pair correspond to the diffusion coefficient $D = 5.0 \cdot 10^{-16}$ m^2/s, which is approximately 2 orders of magnitude lower than for the titanium—HEA pair.

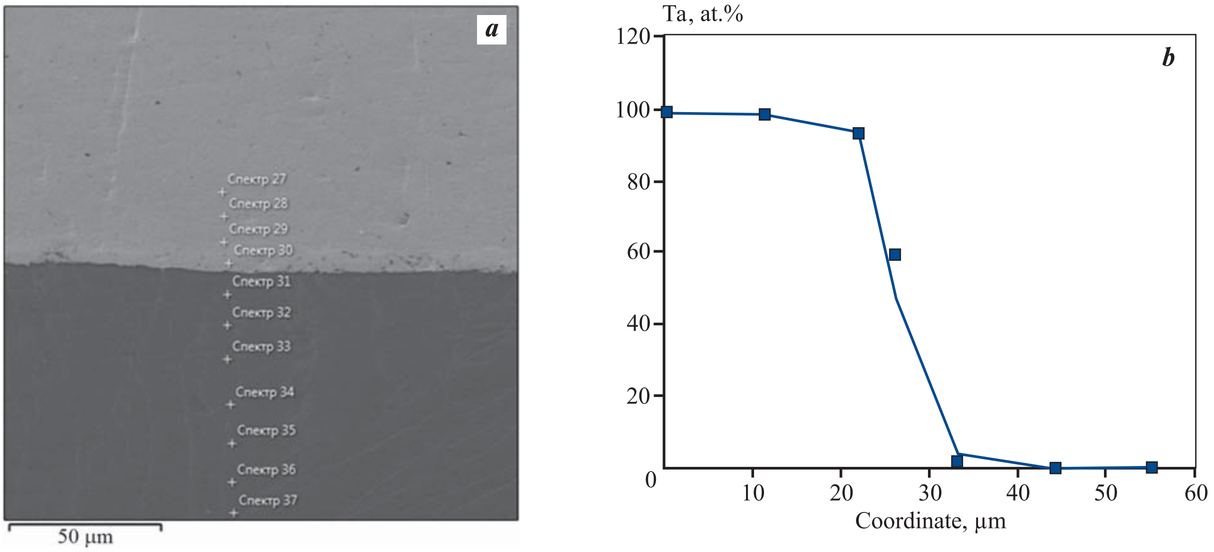

Fig. 9. Micrograph of the cut surface (***a***) and distribution of the Ta concentration in the Ti—Ta diffusion pair (***b***)

Рис. 9. Микрофотография поверхности среза (***a***) и распределение концентрации Ta в диффузионной паре Ti—Ta (***b***)

Conclusion

In this work, a technique for studying interdiffusion in pairs consisting of single-component (titanium—tantalum) and one and multiple (titanium—high-entropy alloy) refractory metals with a BCC lattice was developed and tested.

Diffusion annealing was carried out for 12 h in a vacuum with a residual argon pressure of $6.65 \cdot 10^{-3}$ Pa at a temperature of 1200 °C. The chemical composition in the interdiffusion zone was determined by energy-dispersive *X*-ray analysis. The obtained data on the dependence of the intensity of the corresponding spectral line for titanium and various elements of multicomponent alloys were recalculated in terms of the concentrations of the main elements in the diffusion zone.

Interdiffusion in the titanium—alloy pair is associated mainly with the penetration of the alloy components into titanium as a more fusible part of the system. A change in the signal intensity is observed at a distance of 150—250 μm for all elements. A sharp drop in the signal intensity takes place at a distance of around 50 μm.

The behaviour of the elements can be well described by the erfc-shaped solution, which indicates the absence of a noticeable concentration dependence of the interdiffusion coefficients. Under the given experimental conditions, the average diffusion coefficient for all elements (except titanium) is $D = 3.0 \cdot 10^{-14}$ m^2/s, which is approximately an order of magnitude less than the self-diffusion coefficient in β-titanium, which is equal to $2.0 \cdot 10^{-13}$ m^2/s [19]. Similarly, for comparison, it is possible to give the diffusion coefficient values in titanium at a temperature of 1200 °C of other refractory metals, namely: zirconium — D (Zr/Ti) = $2.6 \cdot 10^{-12}$ m^2/s; molybdenum — D (Mo/Ti) = $9.3 \cdot 10^{-13}$ m^2/s [20]; niobium — D (Nb/Ti) = $7.2 \cdot 10^{-13}$ m^2/s [21] (isotope method); tantalum — D (Ta/Ti) = $7.2 \cdot 10^{-13}$ m^2/s [22]; hafnium — D (Hf/Ti) = $1.0 \cdot 10^{-12}$ m^2/s [23].

Similar studies for a titanium—tantalum pair show a significant difference in the diffusion processes in tantalum—titanium and alloy—titanium pairs. A change in the signal intensity (and, accordingly, the concentration of components in the diffusion zone) takes place at a distance of 20 μm. The data obtained correspond to the interdiffusion coefficient $D = 5{,}0 \cdot 10^{-16}$ m^2/s, which is approximately 2 orders of magnitude lower than for the titanium—HEA pair. Note that tantalum and multicomponent alloys have significantly different melting temperatures (solidus and liquidus regions for alloys) of 3290 K for pure tantalum and 2100—2400 K for alloys [24].

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The article was submitted 19.04.2022, revised 28.06.2022, accepted for publication 30.06.2022

Статья поступила в редакцию 19.04.2022, доработана 28.06.2022, подписана в печать 30.06.2022