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V–Cd nanoparticle-formed alloys: fabrication, phase composition and structure

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Abstract: The results of the study of targeted sputtering and deposition of ultrafine vanadium and cadmium particles on substrates that are not heated and shifted with respect to the substrate plasma currents are revealed. As a result of the conducted studies, coatings were obtained in the range with a concentration of cadmium from 9.6 to 88.6 at.%. The critical size of vanadium particles capable of forming alloys with cadmium is 0.6 nm. The concentration limit for the presence of solid solutions of cadmium in vanadium is the cadmium content of ~37 at.%, at a higher cadmium content the film coating is represented by a mixture of cadmium phases and a solid solution of cadmium in vanadium. The dependence of the lattice parameter of α-vanadium on the content of cadmium in it corresponds to the expression: a [nm] = $8\cdot10^{-4}C_{\rm Cd} + 0.3707$, where $C_{\rm Cd}$ is the concentration of cadmium, at.%. On the surface of the sample in the region of solid solutions (31.6 at.% Cd), the presence of threadlike crystals of cadmium was found, the reason for the appearance of which is the lattice pressure of the matrix metal. Annealing of films rich in cadmium (69.5 at.%) in vacuum is accompanied by cracking of the coating and the formation of pores. The latter can be used as a method for obtaining porous vanadium.

Keywords: vanadium, cadmium, nanoparticles, alloy, solid solution, lattice parameter, coating, threadlike crystals

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Сформированные наночастицами сплавы V–Cd: получение, фазовый состав и структура

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Аннотация: Приведены результаты исследований распыления и осаждения ультрадисперсных частиц ванадия и кадмия на необогреваемые и перемещаемые относительно потоков плазмы подложки. Были получены покрытия в интервале концентраций кадмия от 9,6 до 88,6 ат.%. Критическим размером частиц ванадия, способных к образованию сплавов с кадмием, определена величина 0,6 нм. Концентрационной границей существования твердых растворов кадмия в ванадии является содержание кадмия ~37 ат.%, при большей его доли пленочное покрытие представлено смесью фаз кадмия и твердого раствора кадмия в ванадии. Зависимость параметра решетки α-ванадия от содержания кадмия в нем соответствует следующему выражению: a [нм] = $8\cdot10^{-4}C_{\text{Cd}}$ + + 0,3707, где C_{Cd} – концентрация кадмия, ат.%. На поверхности образца в области твердых растворов (31,6 ат.% Cd) обнаружено наличие нитевидных кристаллов кадмия, причиной появления которых является решеточное давление матричного металла. Отжиг богатых по содержанию кадмия пленок (69,5 ат.%) в вакууме сопровождается растрескиванием покрытия и образованием пор. Последнее может быть использовано как метод получения пористого ванадия.

Ключевые слова: ванадий, кадмий, наночастицы, сплав, твердый раствор, параметр решетки, покрытие, нитевидные кристаллы

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Introduction

To date, there is a problem of obtaining intermetal alloys with very large differences in physical properties, such as melting point, vapor pressure, density, crystal lattice type, etc. At the same time, the production of such alloys requires new properties and technologies to obtain potential materials with unusual properties. In our case, the choice of alloy is due to the fact that the distillation of the highly volatile cadmium from the V—Cd alloys allows the recovery of porous vanadium, which can be used as a catalyst or special filter in another technical field.

One of the ways to compensate for the differences in the properties of the metals, especially in their melting temperatures, in the production of solid solution alloys is to use the size factor. The significant decrease in melting temperature (from a few tens to hundreds of degrees) of various metal dispersion particles, with a simultaneous decrease in droplet size, has sparked the interest of researchers in this area of materials science. Here and now, numerous studies have been carried out on the influence of particle size on melting temperature [1—8], thermodynamics of small formations [9—13] and material structure, preparation of alloys at “room temperature”, synthesis of intermetallic compounds [14—16] and others. This research led to the introduction of the concept of “thermofluctional melting” to the scientific community. The latter means that a very small particle remains in a quasi-liquid state up to a certain critical size, beyond which it crystallizes.

The author of the paper [13], in his consideration of the thermodynamics of small systems, has noted some important points, in particular: for sufficiently small systems, the phase state and phase transitions are meaningless; for very small and isolated systems, it becomes impossible to define the concept of appropriate temperature; the melting and boiling temperatures of nano-sized liquid drops of matter are always lower than the corresponding values in the bulk phase of the same material, etc.

There is no information in the literature about the existence of vanadium and cadmium alloys or intermetallic compounds. Vanadium melting point [17] is 1910 °C, cadmium — 321 °C; vanadium boiling point [18] (at which vapor pressure equals atmospheric pressure) is 3392 °C, cadmium — 766 °C.

Instrumental determination of small particle size and melting temperature is very difficult, but alloy formation, a solid dissolution of two metals, proves the presence of ultradisperse particles that can coalesce in a quasi-liquid state at low temperature. In this context, we have studied the double V—Cd alloy by atomized metal deposition to determine the critical sizes of the nanoclusters, the concentration limits of the V—Cd solid solutions, and the phase composition and structure of the film coatings.

Materials and methodology

One of the ways to form nanometer size (<100 nm) ultra-dispersed particle flows consists in metal atomization in a low-pressure plasma. Studies were performed using an ion-plasma unit with oppositely placed planar DC magnetrons with water-cooled targets on the walls of the vacuum chamber. The unit design and the sample atomizing technique were described in detail earlier [15].

The substrate temperature did not exceed 100 °C during sample formation. The ratio of metal concentrations in the alloy samples was changed by the atomization rate of magnetron sputtering targets. The ratio of deposited components was controlled by the weight method (by the amount of atomized and deposited metal during the coating formation) and in parallel by the method of Rutherford backscattering of protons at the tandem accelerator UKP-2-1 of the Institute of Nuclear Physics (Almaty).

Metals used in experiments were with major element content, wt.%: vanadium — 99.6; cadmium — 99.99. Targets are made of each of the metals in the disk form, 40 mm in diameter and 4 mm thick.

Sample preparation included sequential operations of vanadium and cadmium target sputtering and deposition of atomized particles on unheated substrates that move relative to the plasma streams in the form of island films — nanometer sublayers — to the proposed total coating thickness. The sublayer thickness was calculated by dividing the total coating thickness by the number of substrate passes relative to the metal-containing plasma flows.

The use of the thickness of the sublayer as a dimensional factor is due to the fact that the change in the melting temperature of the film with decreasing thickness is similar to that of small particles [13], which form according to the “island” principle.

The critical size of the metal particles was determined by reducing the thickness of the sublayer through alternate confinement of niobium and second metal atomizers as a result of each intersection of a metal-containing plasma stream through the moving substrate. The fluxes with dissimilar metal particles were separated by 180° relative to each other, which excluded the formation of solutions when the fluxes touched or crossed. Moreover, the substrate took 3 s to move from the center line of one magnetron atomizer to the second, which was sufficient for crystallization of very small particles, assuming the existing process. The thickness of the sublayer at which the solid solution appears in the coating was considered to be the critical size of the metal particles.

The element concentration at which phases other than the solid solution appeared was considered to be the existence limit of the alloy.

Polycor substrates (α-Al_2O_3) and monocrystalline silicon were used for the coatings. *X*-ray diffractometer “D8 Advance” (Bruker, Germany) with CuK_α-radiation ($\lambda = 0.154051$ nm) and graphite monochromators were used for the *X*-ray diffraction study of the “V—Cd” system. The lattice parameter value was calculated as the average of all diffraction lines from a given phase.

High-temperature anneals were performed in a vacuum high-temperature furnace based on the URVT-2500.

Results and discussion

The results of determining the critical dimensions of the vanadium and cadmium sublayers by alternating short-range (with a small number of lattice periods) layers and reducing the layer thickness of each metal for a system with a concentration of ~25 at.% Cd are shown in Table 1.

As a result of the obtained samples diffractometrical research it was established (see Table 1) that with the cadmium vanadium capillary-porous system (CPS) thickness of 0.6 and 0.3 nm respectively in the coating of this composition cadmium vanadium solid solution was obtained with a body-centered cubic (BCC) structure and lattice parameter, $a = 0.3931 \pm 0.0009$ nm. So that the vanadium nanoparticles critical size capable of coalescing with cadmium is 0.6 nm. As expected, the critical size of the cadmium particles can be adjusted towards higher values, since increasing the CPS while maintaining the critical size of the vanadium particles with the existing method of coating production of the given composition takes the system out of the range of existence of solid solutions due to alloys with higher concentration of the alloying element. For example, the critical size of cadmium nanoparticles in the formation of our alloys with refractory metals such as niobium [15] and tantalum [19] corresponds to values of 3.2 and 2.1 nm, respectively.

In order to determine the solid solutions concentration boundaries in the V—Cd system taking into account the size factor, 11 coating samples with cadmium concentration from 9.6 to 88.6 at.% have been formed. The coatings composition and the phases detected are given in Table 2.

Fig.1 shows some V—Cd system coatings diffractograms with different cadmium concentrations.

Cadmium and amorphous vanadium phases were detected in the sample with cadmium content of 88.6 at.% (spectrum *1* in Fig. 1). In the concentration interval of 42.0—80.2 at.% Cd (spectrum *2*), the cadmium phase coexists with cadmium-vanadium solid solution with BCC structure. Cadmium as a separate phase is not observed in coatings with <31.6 at.% Cd (spectrum *3*). Moreover, in this case the cadmium-vanadium solid solution has a predominant orientation (111). At concentrations <14.7 at.% (spectrum *4*), the coatings are represented by cadmium-vanadium solid solutions with the lattice parameter of the solid solution

Table 1. **Thickness of short-period metal layers and lattice parameters of detected phases in vanadium films containing ~25 at.% Cd**

Таблица 1. Толщина короткопериодных слоев металлов и параметры решетки обнаруженных фаз в пленках ванадия, содержащих ~25 ат.% Cd

Layer thick-ness, nm		Phases lattice parameters, nm		
V	Cd	V	Cd	
		a	*a*	*c*
16.0	8.0	0.3491 ± 0.0018	0.2981 ± 0.0002	0.5620 ± 0.0003
12.1	6.0	0.3815 ± 0.0012	0.2980 ± 0.0003	0.5621 ± 0.0002
9.3	4.8	0.3868 ± 0.0008	0.2980 ± 0.0002	0.5622 ± 0.0003
6.2	3.0	0.3925 ± 0.0007	0.2982 ± 0.0002	0.5620 ± 0.0003
3.4	1.7	Amorphous	Amorphous	
0.6	0.3	Cadmium vanadium solid solution, *a* = 0.3931 ± 0.0009 nm		

changing towards the increase due to a considerably larger cadmium atoms radius (0.1727 nm) in comparison to that of vanadium (0.1489 nm) [20]. Change of the lattice parameter of cadmium-vanadium solid solution as well as the solution coexisting with cadmium is shown in Fig. 2.

From data of Fig. 2 one can see that up to ~37 at.% Cd concentration the α-vanadium lattice parameter changes linearly with increase of Cd content in solution, the saturated solid solution with the average parameter *a* = 0.4001 nm is allocated into a separate phase (coexisting with cadmium) for higher

Table 2. **Coatings composition and phases detected**

Таблица 2. Состав покрытий и обнаруженные в них фазы

Coatings composition, at.%		Phases detected
V	Cd	
11.4	88.6	Cd + V amorphous
19.8	80.2	Cd + cadmium vanadium solid solution
30.5	69.5	Cd + cadmium vanadium solid solution
35.0	65.0	Cd + cadmium vanadium solid solution
43.3	56.7	Cd + cadmium vanadium solid solution
47.7	52.3	Cd + cadmium vanadium solid solution
57.0	43.0	Cd + cadmium vanadium solid solution
68.4	31.6	Cadmium vanadium solid solution (texture (111))
73.5	26.5	Cadmium-vanadium solid solution
85.3	14.7	Cadmium-vanadium solid solution
90.4	9.6	Cadmium-vanadium solid solution

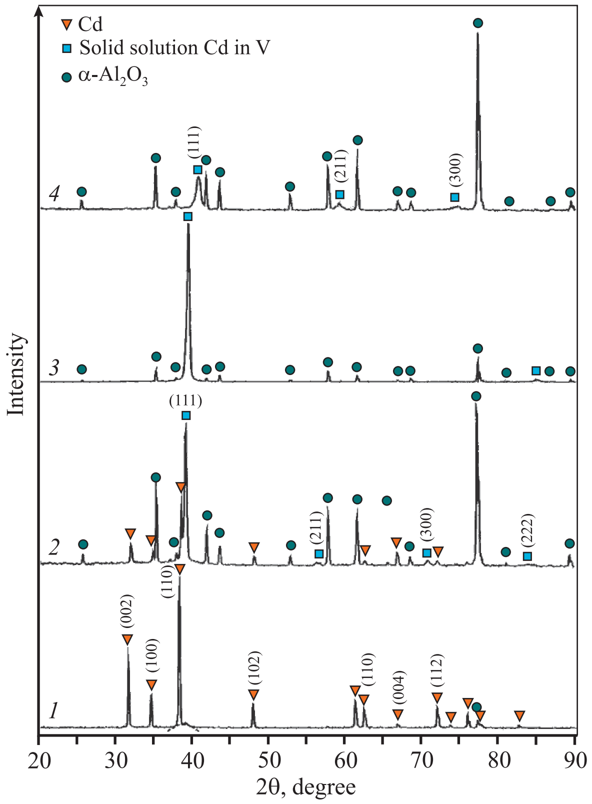

Fig. 1. V–Cd system coatings diffractograms with different cadmium contents

Cd, at.%: ***1*** – 88.6; ***2*** – 43.0; ***3*** – 31.6; ***4*** – 14.7

Рис. 1. Дифрактограммы покрытий системы V–Cd с различным содержанием кадмия

Cd, ат.%: ***1*** – 88,6; ***2*** – 43,0; ***3*** – 31,6; ***4*** – 14,7 values of Cd content than mentioned above. That is, the concentration boundary of existence of cadmium solid solutions in vanadium corresponds to ~Cd 37 at.%. Dependence of the α-vanadium lattice parameter before its saturation on the cadmium content in it corresponds to the following equation: a [nm] = $8\cdot10^{-4}C_{\text{Cd}} + 0.3707$, where C_{Cd} — cadmium concentration, at.%.

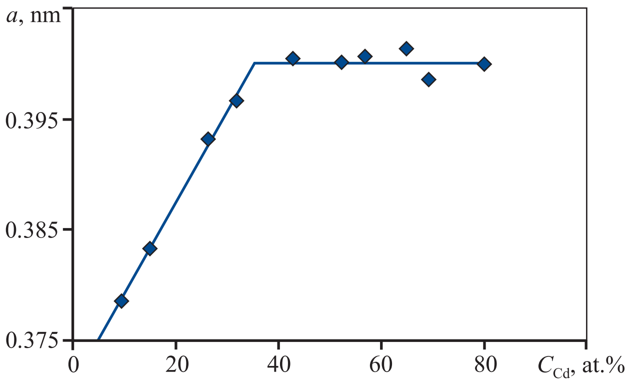

Fig. 2. Cadmium–vanadium solid solution lattice parameter dependence on the cadmium content in the coating

Рис. 2. Зависимость параметра решетки твердого раствора кадмия в ванадии от содержания кадмия в покрытии

SEM-researches of a primary sample with 31.6 at. % cadmium concentration (Fig. 3) have revealed the filamentous crystals of various shapes appearance up to 30 μm long and 2 μm thick as well as crystals resembling sprouting plants on the coating surface besides formations of 1—2 μm.

The mechanism of such formations on the samples surface is apparently similar to the phenomenon of similar tin crystals formation in the form of whiskers and protrusions on Lu_4Sn_5 surface at storage in the air during several days where the filamentous crystals growth initiating force are compressive matrix compound stresses [21]. The surface of the sample annealed at 300 °C in vacuum losing a significant portion (by preliminary data, 30—40 wt.% [22]) of cadmium by evaporation) is represented by melted grains of irregular shape that differ in size.

A similar study of a sample with 69.5 at.% cadmium content characterized by coexisting phases of cadmium and cadmium-vanadium solid solution showed a different surface topography (Fig. 4). The initial sample has no pronounced grain formations. After annealing, the surface is represented by "shatt" formations with a porous structure inside the film due to cadmium evaporation.

In the coating sample diffractometric study with

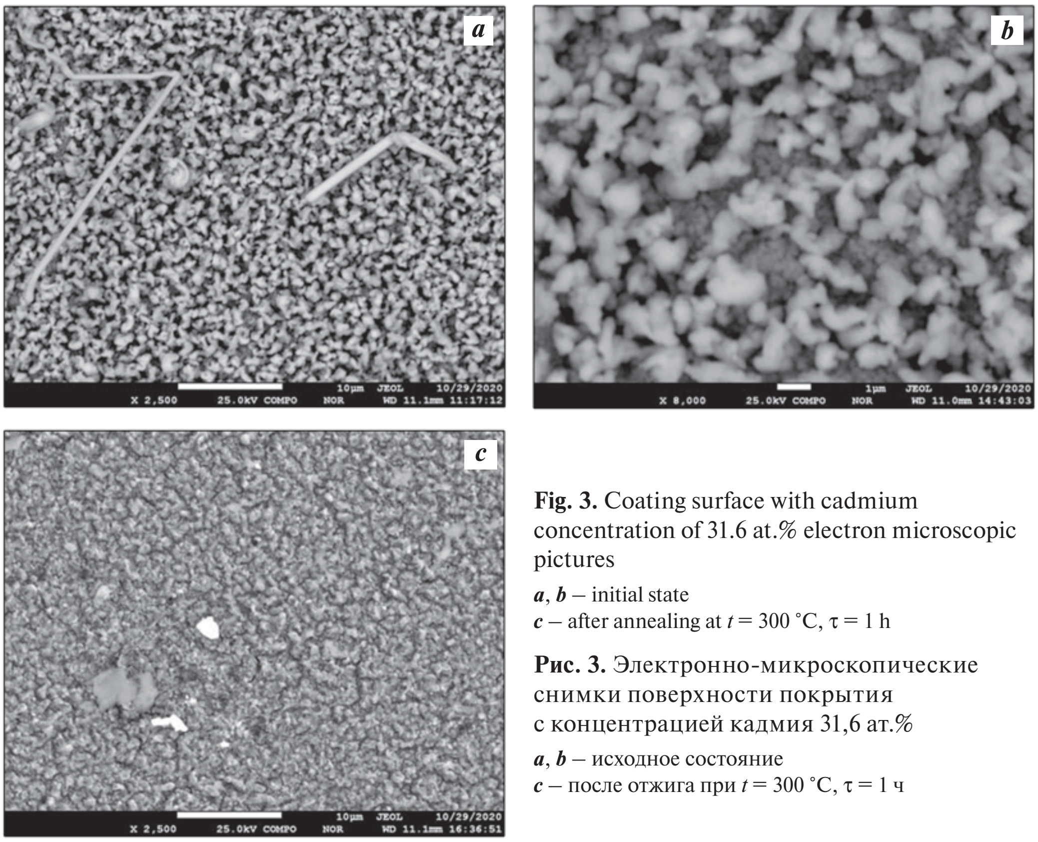

Fig. 3. Coating surface with cadmium concentration of 31.6 at.% electron microscopic pictures

a, ***b*** – initial state
c – after annealing at t = 300 °C, τ = 1 h

Рис. 3. Электронно-микроскопические снимки поверхности покрытия с концентрацией кадмия 31,6 ат.%

a, ***b*** – исходное состояние
c – после отжига при t = 300 °C, τ = 1 ч

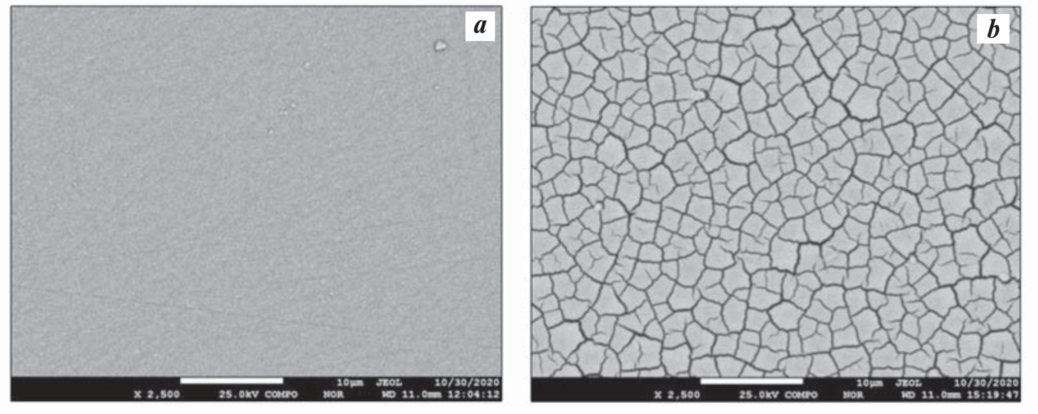

Fig. 4. Coating surface with 69.5 at.% cadmium concentration electron-microscopic pictures
a – initial state; ***b*** – after annealing at t = 300 °C, τ = 1 h

Рис. 4. Электронно-микроскопические снимки поверхности покрытия с концентрацией кадмия 69,5 ат.%
a – исходное состояние; ***b*** – после отжига при t = 300 °C, τ = 1 ч

9.6 at.% cadmium content (Fig. 5) annealed at 400 °C for 1 h, the reflexes of the phase which can be assigned to cubic syngony are observed. Reflex (111) of this phase exactly coincides with reflex (104) (d_{hkl} = 0.2552 nm), and reflex (220) coincides with polycor reflex (211) (d_{hkl} = 0.1546 nm). The assumed lattice parameter of this cubic phase a = 0.4393 ± 0.0008 nm suggests that the compound is a complex cadmium-vanadium oxide with the $V_xHf_{1-x}O$ formula, since cadmium oxide CdO (ICPDS card number 1011051) with lattice parameter a = 0.4730 nm is known. The measured d_{hkl} values of the presupposed phase and the corresponding Miller indices are given below:

d_{hkl}, nm	0.2552	0.2195	0.1546	0.1269
hkl	(111)	(200)	(220)	(222)

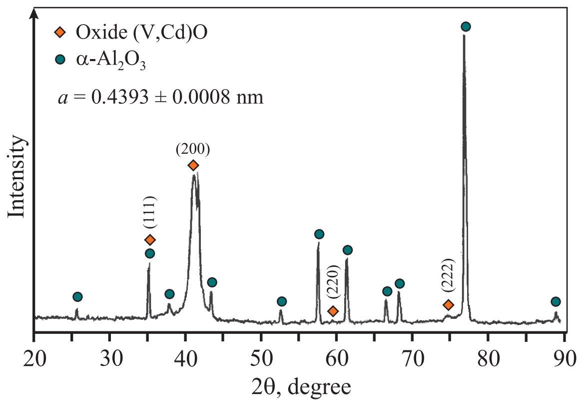

Fig. 5. Coating sample containing 9.6 Cd at.% diffractogram after annealing (t = 400 °C, τ = 1 h)

Рис. 5. Дифрактограмма образца покрытия с содержанием 9,6 ат. % Cd после отжига (t = 400 °C, τ = 1 ч)

Oxide formation after the sample extraction from vacuum volume testifies, in our opinion, the developed structure presence in the film depth. Judgment on the presence of through pores in this case is not possible, but the probability of their presence is very high. This requires additional studies along with examination of the cadmium evaporation degree from the films of similar composition and from cadmium-vanadium solid solution.

At the same time, coatings with high cadmium content and subsequent evaporation from their mixtures with cadmium-vanadium solid solution can be used to obtain materials with highly developed surface.

Conclusion

As a result of this research the possibility of obtaining ultradisperse vanadium and cadmium particles which can form alloys and compounds in the quasi-liquid state was confirmed. Formation of coatings with ultradisperse metal particles allows obtaining alloys (solid solutions) at temperatures below 100 °C. The critical size of vanadium particles capable of coalescence with cadmium was determined to be 0.6 nm. For cadmium, an underestimated formations critical size was obtained as compared to the confirmed data from earlier studies, 0.3 nm.

The concentration boundary of the cadmium-vanadium solid solutions existence is ~Cd 37 at.% content, provided that the parameter of the α-vanadium volume-centered cubic lattice linearly increases because of larger cadmium atomic radius in relation to vanadium. At higher (up to 80.2 at.%) cadmium content, the film coating is represented by a mixture of cadmium and cadmium-vanadium solid solution phases.

Electron-microscopic studies revealed the presence of filamentary crystals and cadmium formations on the sample surface in the solid solutions field (31.6 at.% Cd). We assume by virtue of the study results of other authors that they appeared due to the lattice pressure of the matrix metal.

Annealing of samples in vacuum is accompanied by diffusion of cadmium to the phase interface and evaporation, which reduces its concentration in the coating. Annealing of cadmium-rich films in the region of cadmium phases and solid solutions (69.5 at.% Cd) is associated with coating cracking and pore formation. After further investigation, it can be used as a method for the recovery of porous vanadium.

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