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Phase composition, structure and stress state of magnetron sputtered W–Ti condensates

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The effects of elemental composition and deposition temperature on W–Ti ion-plasma condensate structure, substructure characteristics and stress state have been studied. The crystalline state of all phases formed during deposition has been found to be based on the α-W bcc crystalline lattice. In the initial stages of solid solution formation under relatively low content of substituting titanium atoms in the lattice (7 to 11 at.%), oriented crystallite growth takes place with (100) planes parallel to the condensate surface. At higher titanium atomic content in the lattice, strongly strained crystallites of (W_{1-x}, Ti_x) solid solution are formed with light preferential orientation of (110) plane parallel to the growth surface. In this case, the deposition temperature elevation from 300°C to 800°C results in concentration stratification of the formed solid solution followed by formation of two solid solutions with different stable elemental composition. The diffusion coefficient in nano-crystalline W–Ti condensates has been estimated and models describing the formed condensate structure state are proposed.

Исследовано влияние элементного состава и температуры осаждения ионно-плазменных конденсатов системы W–Ti на их структуру, субструктурные характеристики и напряженное состояние. Установлено, что кристаллическое состояние всех фаз, формируемых в процессе осаждения, базируется на ОЦК кристаллической решетке α–W. На начальных стадиях формирования твердого раствора при относительно малом содержании замещающих атомов титана в решетке (7–11 ат.%) происходит ориентированный рост кристаллитов с плоскостью (100), параллельной поверхности конденсации. При более высоком содержании в решетке атомов титана формируются сильнодеформированные кристаллиты (W_{1-x}, Ti_x) твердого раствора с небольшой степенью совершенства текстуры с плоскостью (110) кристаллической решетки, параллельной поверхности роста. В этом случае увеличение температуры осаждения от 300°C до 800°C приводит к концентрационному расслоению формируемого твердого раствора, что сопровождается образованием двух твердых растворов с различающимся стабильным элементным составом. Проведена оценка коэффициента диффузии в нанокристаллических конденсатах системы W–Ti и предложены модели для описания формируемого структурного состояния конденсатов.

A high interest in W–Ti film system is due first of all to the good promises of the application thereof in microelectronics, for instance, as diffusion barriers [1–4]. The best electrical characteristics for the system (the lowest electrical resistivity of barrier layer less than 55 μΩ·cm) are provided by condensates containing 70–55 at.% W and 30–45 at.% Ti [5]. Another promising field is the application of condensed W–Ti materials as binding layer in multi-layered wear-resistant coatings [6]. In this case, a substantial thickness of barrier layers (0.2 to 0.3 μm) in combination with W–Ti layer thickness of the order of micrometers in the multi-layered wear-resistant coatings imply a strong dependence of condensate functional characteristics on its structure and

phase state. It is well known that ion-plasma condensation results in formation of structure and phase state different from those expected for equilibrium formation conditions [7, 8]. Thus, the aim of this work was to study the formation regularities of structure-phase and stress states of W–Ti condensates as functions of their elemental composition and substrate temperature under deposition. Those problems are both of scientific importance in the field of physical material science of condensed systems and prectical one for development of a technique for structure based optimization of condensate functional characteristics.

In this connection, two characteristic concentration ranges were studied. The first is 88–86 at. % W and 12–14 at. % Ti. That concentration range, on the one hand, allows to obtain intense enough diffraction lines from solid solution lattice necessary to study the structure and substructure characteristics, and, on the other hand, allows to analyze the initial stages of solid solution formation due to relatively low concentration of titanium impurity atoms in tungsten basic bcc lattice (tungsten atomic concentration was five times so high as titanium one). The second range (60–52 at. % W and 40–48 at. % Ti), provides the best functional properties of the system, as it was found by several authors [4, 5], thus, it is of interest from practical point of view. Moreover, the concentration ranges were chosen according to phase diagram so that to get in the first case into solid solution area stable to spinodal decomposition (up to 14 at. % Ti), while in the second case, to get into the area of solid solution spinodal decomposition followed by concentration ordering [10].

The films were prepared by magnetron sputtering the composed target (cast tungsten disc (at least 99 % purity) of 55 mm in diameter and imposed Ti sectors) under operating pressure of argon atmosphere of 0.4 Pa. Application of composed target rather than sintered one is explained by the known fact that it provides purer condensate composition [5]. Before deposition, the substrates were hold in vacuum of $3 \cdot 10^{-4}$ Pa at 950°C for an hour to remove gaseous impurities from the surface. The deposition rate was near 0.7 nm·s^{-1}. As substrates, plates of glass ceramics and single-crystalline silicon were used. The coating thickness was 0.9 to 1.2 μm. The substrate temperature was varied from 200 to 800°C.

The X-ray diffraction was examined using a DRON-3 apparatus with Cu–K_α radiation in discrete regime of scattering registration with scanning step in the range $\Delta(2\theta)$ of 0.01 to 0.05°, depending on the half-width and intensity of diffraction lines. Exposure time was 20 to 100 s for a point. Diffraction profiles were separated using the software developed at NTU "KhPI". Volume fractions of phases in the film was calculated using a standard technique taking into consideration the integral intensity and reflectivity of several lines for each phase [11]. The structure perfection degree (ω) was estimated by measuring the half-maximum full width of diffraction line recorded in θ-scanning regime. For the analysis, diffraction lines from (110) and (200) crystallographic planes were chosen.

The residual elastic strain level and crystalline lattice parameters for non-strained state were calculated using $a - \sin^2\psi$ plots. The reflections from (321) planes at tilt angles ψ of 0, 19, 36.7, 41, 55, and 57.7° were considered. In bcc lattice inherent both to tungsten and tungsten based solid solution, the 19, 41, and 55° angles correspond to the angles between (110) and (321) planes with inversion, while 36.7 and 57.7°, to the angles between (100) and (321). At corresponding film growth texture, such choice of ψ angles allowed to separate the effects characteristic of different crystallite orientation systems. To study the strain distribution homogeneity over the condensate volume, the measurements were made both to towards $+\psi$ and $-\psi$ angles.

The linear approximation of experimental points in "a–$\sin^2\psi$"-plot gives strain value ε averaged over the coating thickness from the expression:

$$\varepsilon = \frac{a_{\psi=90} - a_{\psi=0}}{a_0} = \frac{a_\parallel - a_\perp}{a_0}$$

with $a_\perp$ is the lattice parameter in direction normal to reflecting plane ($\psi = 0°$); $a_\parallel$, the lattice parameter in the film plane ($\psi = 90°$); a_0, the lattice parameter in non-stressed state. From the "a–$\sin^2\psi$"-plots, the value a_0 was determined assuming isotropic symmetric plane-stressed state. It was calculated at ψ_0 value corresponding to unstrained section of straining ellipsoid:

$$\sin^2\psi_0 = \frac{2\nu}{1+\nu}.$$

The coating elemental composition was determined by X-ray fluorescent analysis using "SPRUT" apparatus (produced by "Ukrrentgen", Ukraine). The radiation from a "shoot-through" type X-ray tube with Ag anode at excitation voltage 42 kV was used as primary exciting radiation.

According to phase diagram [9], the W solubility in α–Ti does not exceed 0.2 at. %, while titanium atoms are completely soluble in tungsten bcc lattice by substitution type. In this connection, predominant formation of bcc lattice based solid solution should be expected in ion-plasma condensates. According to X-ray phase analysis data, the coatings consisting of almost pure tungsten α–W and solid solution (W_{1-x}, Ti_x) based on α–W bcc lattice are formed in the first concentration range (Fig. 1, curves *1*,). The relatively low dissolution of titanium atoms results in formation of not only texture with closest packed planes (110) parallel to growth surface inherent to ion-plasma condensates [12], but of another texture with (100) type planes parallel to growth surface. The perfection degree of texture with crystallite (100) planes parallel to the growth surface was substantially higher than that for (110) one, that is manifested as decreased ω (Table 1). The volume content ratio of (110) to (100) textured crystallites in the samples varied from 65/10 to 55/31 at the substrate temperature increasing from 700 to 800°C, respectively (Table 1).

The solid solution line maximum average positions obtained after separation of complex diffraction profile are close to one another both for T_s = 700°C and T_s = 800°C. This position corresponds to the lattice parameter $a_\perp$ = 0.322 nm. To compare the obtained lattice parameter with concentration of titanium atoms dissolved in solid solution lattice, it is necessary to take into account the effect of condensate stress state on the diffraction line positions. The results of such investigations will be given in the second part of the work. It should be noted that the coating elemental composition according to the X-ray fluorescent analysis data shows that the coating composition varies from about W/Ti = 86/14 to W/Ti = 90/10 as the substrate temperature increases.

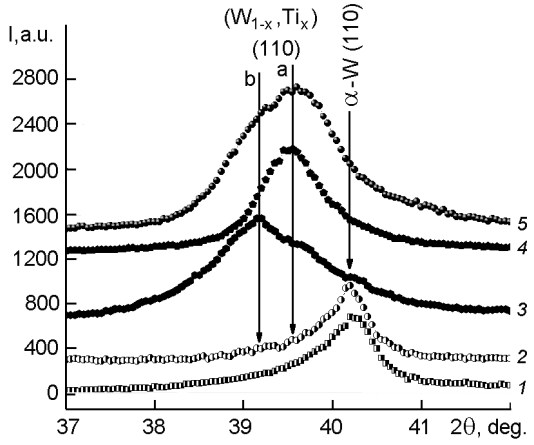

Fig. 1. Diffraction spectrum fragments for W–Ti films of the first concentration range (10 to 14 at. % Ti) prepared at substrate temperatures (T_s) 700°C (*1*); and 800°C (*2*); and for the films of the second concentration range (40 to 48 at.% Ti) prepared at T_s = 300°C (*3*), 500°C (*4*), 800°C (*5*). The letters *a* and *b* indicate the positions of two characteristic maxima for (W_{1-x}, Ti_x) solid solution.

Within the second concentration range, the coating main phase component is (W_{1-x}, Ti_x) solid solution (Fig. 1, curves *3*, *4*, *5*). The condensation temperature elevation results in increasing relative volume fraction of solid solution in the coating, while its dissociation into two characteristic compositions within the same type of bcc lattice

Table 1. Phase composition, crystallite texture degree (ω), substructure characteristics (microstrain ε, and crystallite size *L*) for W–Ti condensates in the first concentration range (10 to 14 at.% Ti)

T_s, °C	Phase	Texture		Content, vol.%	ε, %	L, nm
		plane	ω, degrees			
700	α–W	(110)	19	65	0.4	50
		(100)	5.5	10	0.15	40
	(W_{1-x},Ti_x)	(110)	>20	25	0.9	15
800	α–W	(110)	21	55	0.5	60
		(100)	12	31	0.2	50
	(W_{1-x},Ti_x)	(110)	>20	14	0.9	20

becomes just obvious from the diffraction line separation into two components (in Fig. 1, the positions are marked by arrows *a* and *b*). Such situation is inherent in solid solutions after concentration lamination due to spinodal dissociation mechanism [13]. The coherently bonded areas of solid solutions with different atomic ratios are formed in this case, this difference enhances then as a result of dissociation and atomic rearrangement due to ascending diffusion mechanism. In Fig. 2, one example of complex diffraction profile separation is shown, where two pronounced diffraction maxima of solid solution are seen (curves *2*, *3* in Fig. 2) in positions corresponding likely to two metastable concentration states. The curve *4* in Fig. 2 corresponds to α–W (110) diffraction line.

In Table 2, the data obtained by calculation of volume phase content in the films depending on condensation temperature are summarized. It is seen that the condensation temperature increase results in relative decrease of the pure α–W phase content and increasing content of (W_{1-x}, Ti_x) solid solution based thereon. A pronounced separation of (W_{1-x}, Ti_x) solid solution into two stable components is observed. According to X-ray fluorescence data, average atomic ratio changed from W/Ti = 52/48 and 53/47 at T_s = 300 and 500°C to W/Ti = 60/40 at T_s = 800°C.

The substructure characteristics analyzed using two reflection orders from {110}, {100} planes of tungsten and {110} of tungsten based solid solution evidenced micro-strains developed in the direction of film forming particle incidence (normally to growth plane): from 0.4 % (for T_s = 700°C)

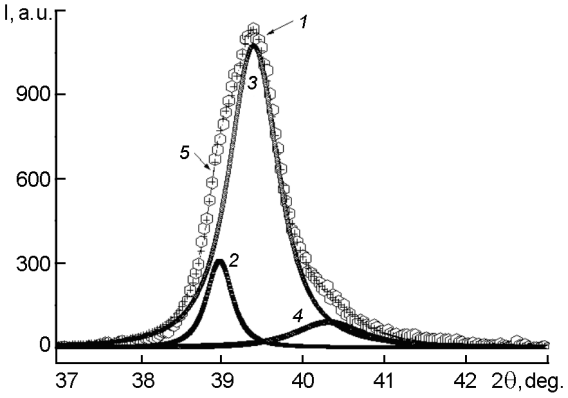

Fig. 2. The result of profile separation for X-ray diffraction line (*1*) into the components (*2*, *3*, *4*) and overall convolution line (*5*) for a condensate prepared at T_s = 800°C (C_{Ti}≈40 at.%).

to 0.5 % (for T_s = 800°C) for crystallites with predominant orientation (110), and from 0.15 % (T_s = 700°C) to 0.2 % (T_s = 800°C) for crystallites (100) oriented parallel to the growth surface in the samples with relatively low titanium atomic content (1st series) (Table 1). For α–W phase, crystallite size was found to be almost the same (40 to 60 nm) for both texture types. The solid solution crystallites were more strained. Their micro-strain level was about 0.9 %, while crystallite size did not exceed 15 to 20 nm. For the second sample series, it is just the solid solution crystallites (about 1.4 % for T_s = 300 and 500°C, and 1.2 to 1.3% for T_s = 800°C) that were most strained, too, but those had larger average sizes from 80 to 250 nm (Table 2). In α–W phase, the micro-strain level was relatively

Table 2. Phase composition, crystallite texture degree (ω), substructure characteristics (micro-strain ε, and crystallite size *L*) for W–Ti condensates in the second concentration range (40...48 at.% Ti)

T_s, °C	Phase	Texture		Content, vol.%	ε, %	L, nm
		plane	ω, degrees			
300	α–W	(110)	>20	15	0.4	6
	(W_{1-x},Ti_x), type *b**	(110)	20	60	1.4	80
	(W_{1-x},Ti_x), type *a*	(110)	>20	25	1.3	45
500	α–W	(110)	>20	8	0.1	8
	(W_{1-x},Ti_x), type *a*	(110)	21	92	1.4	150
800	α–W	(110)	>20	4	0.2	6
	(W_{1-x},Ti_x), type *a*	(110)	19	81	1.3	180
	(W_{1-x},Ti_x), type *b*	(110)	19	15	1.2	250

* — the type is indicated according to diffraction line position in Fig. 1.

low (0.1...0.4 %) for all the samples, while crystallite size being substantially less and did not exceed 5 to 8 nm.

Studying the macro-strained state by "a–$\sin^2\psi$"- method in the condensates of the first concentration range has shown that α–W phase crystallites were subjected to compressive macro-strains. The practically symmetric form of the plot obtained for macro-strain calculations by "a–$\sin^2\psi$"- method using scans both in $+\psi$ and $-\psi$ angle ranges indicates applicability of plane-stressed state model to the objects under study. The strain value was −0.5 to −0.7 % (the sign "−" indicates compressive strain) for (110) textured crystallites obtained in the 700 to 800°C condensation temperature range, while for (100) textured crystallites, it was a little lower (−0.2 to −0.5 %). The lattice parameter calculation for non-strained section has shown a value $a_0 = 0.31638$ to 0.31639 nm in α–W phase, that is close to the reference value for the bulk [12]. The lattice parameter of tungsten crystallites with (100) texture was increased a little: 0.3169 nm for $T_s = 700$°C, and 0.3173 nm for $T_s = 800$°C. According to data of [9] and assuming Vedard rule to be valid for the lattice parameter variation, the obtained parameter change in non-strained section indicates dissolution of 7 and 11 % titanium atoms, respectively in tungsten lattice.

In the second sample series with high content of titanium impurity atoms, the macro-strain could be determined by "a–$\sin^2\psi$"- method only for solid solution component. In the solid solution crystallites of condensate prepared at $T_s = 300$°C, the macro-strain was found to be compressive and to amount −1.8 %. In this case, the lattice parameter in non-stressed section was $a_0 = 0.3210$ nm that corresponds to composition $W_{0.39}Ti_{0.61}$. In condensates obtained at $T_s = 500$°C, the macro-strain value was −1.1 %, while parameter $a_0 = 0.3191$ nm corresponding to composition $W_{0.48}Ti_{0.52}$. At $T_s = 800$°C, compressive strains about −0.84 % were developed in a-type crystallites, while the lattice parameter $a_0 = 0.3180$ nm corresponded to $W_{0.62}Ti_{0.38}$ composition. Estimations made from b-type line positions gives characteristic composition $W_{0.40}Ti_{0.60}$ for the second solid solution component.

The results obtained show that in initial stages of solid solution formation, titanium addition into tungsten lattice promotes growing of textured crystallites with (100) planes parallel to the condensation surface. Such arrangement results in less strained basic bcc crystalline lattice, that is manifested in relatively low macro- and micro-straining of such crystallites. As the of atomic titanium content in bcc tungsten lattice increases up to 11 % (at $T_s = 800$°C), the crystallite (100) texture perfection is lost.

In the films containing about 10 to 14 at.% of Ti, the presence of several phases among which there are both almost pure tungsten phase and (W_{1-x}, Ti_x) solid solution with titanium content exceeding the average value for the film as a whole indicates inhomogeneity of elemental composition. The most probable cause of this fact is the limited titanium atomic diffusion length during the film growth. The diffusion length can be estimated under assumption that the impurity titanium atoms diffuse from the inter-crystallite area to the bulk of solid solution crystallite, the situation being close to reality for the case of magnetron sputtering from the composed target. As the substructure analysis has shown, for condensates prepared in the first concentration range (10 to 14 at % Ti) at T_s = 800°C, the solid solution crystallites had the average size of 20 nm. Using the estimating equation $\langle L\rangle = (D\tau)^{0.5}$ [13], where $\langle L\rangle$ is a half of the grain size assuming equiaxial grains; τ, the diffusion time which is taken 30 s according to the condensate growth rate, one can obtain the volume diffusion coefficient $D \approx 3\cdot10^{-14}$ cm$^2\cdot$s^{-1} for 20 nm size grains. This value agrees well with other results on atomic diffusion mobility in nano-crystalline condensates [13]. It is worth to note that in bulk microcrystalline state, such D value in W–Ti system may be attained only at temperatures exceeding 1000°C even for grain boundary diffusion [14]. Such an effect of diffusion coefficient increase in nano-materials is well known and is related to decreasing diffusion activation energy in the grains smaller than 40 nm as compared to micrometer size ones [15, 16].

The high diffusion mobility of titanium atoms in sub-surface layers, as well as secondary atomic sputtering from the surface are the main probable causes of atomic ratio variation in the film as condensation temperature increases. If at condensation temperatures 300 and 500°C, atomic concentration change does not exceed several per cent, such variation at $T_s = 800$°C is about 8 % higher that at $T_s = 300$°C (tungsten relative atomic content increases from 52 to 60 at.%, according to X-ray fluorescence

analysis). The elemental composition calculations basing on data on phase volume content in solid solutions of different compositions at such temperatures results in somewhat lowler (near 7 at.%) but as well rather high relative tungsten atomic concentration in the film under substrate temperature increase.

In the films with higher titanium atomic content (48 to 40 at. %), variations of both solid solution volume content and average concentration of its components are observed depending on the condensation temperature. According to X-ray diffraction data, two the most stable solid solutions contained about 60 to 61 % and 38 to 40 % of titanium, respectively. As these atomic ratios do not correspond to any special points in equilibrium phase diagram, appearance of such stable concentration states may be caused by concentration ordering and phase formation peculiarities in non-equilibrium nano-crystalline structures. To such processes, first of all, the spinodal type concentration stratification stimulated by limited diffusion may be attributed [10, 17].

At high average titanium atomic content in the film, its limited diffusion length influences very strongly, that results in formation of the practically impurity-free domains of α–W crystallites. It is of significance that as the condensation temperature increases, both specific volume and average size of such domains in the material decrease. As to the fact of forming such impurity-free domains under condensation, it is favored to a great extent by strong metallic bond inherent in tungsten atoms that results in development of high micro-straining in the crystalline lattice due to substitution of tungsten atoms by titanium ones having much larger atomic radius. As a result, additional strain energy occurring on the background of condensation compressive macro-stresses hinders the impurity titanium atomic diffusion from sub-boundary area into the crystallite bulk and causes multi-zone structure of titanium atomic distribution. The calculation for the condensates of the second concentration range (40 to 48 at.% Ti) deposited at the highest temperature $T_s = 800$°C shows that with obtained $D \approx 3 \cdot 10^{-14}$ cm^2/s and characteristic growth time of 250 nm size crystallites $\tau \approx 350$ s, we obtain diffusion length $<L> \approx 30$ nm which corresponds to the size of an external area (near grain boundary) with stable composition ($W_{0.4}$, $Ti_{0.6}$) most saturated by titanium atoms according to multi-zone grain structure model.

Thus, basing on the results of structure and substructure analysis of the condensates with high content of impurity atoms, the most likely scheme of grain structure is three-layered one including a 6 to 8 nm center area of almost pure α–W, followed by two periphery tungsten lattice based stable (W_{1-x}, Ti_x) solid solution areas with lower and higher titanium content, respectively. The zone of high titanium saturation is positioned closer to grain boundary, while the titanium-depleted zone being nearer to central grain part. In the last case, due to lesser micro-strain caused by impurity atoms, the diffusion length may be increased.

Thus, studies of the two concentration ranges of W–Ti system have shown that both at low (10 to 14 %) titanium atomic content and at about 1:1 W/Ti ratio, the phases based on α–W bcc crystalline lattice are formed in the condensate. The presence of few substituting titanium atoms in tungsten lattice (7 to 11 at.%) results in formation of predominantly oriented crystallites with (100) planes parallel to the condensate growth surface. The formation of (W_{1-x}, Ti_x) solid solution with a high content of titanium substituted tungsten sites in the lattice results in higher disorientation of crystallites and development of strong micro-strains up to 1.4 %. Though the condensation temperature elevation up to 800°C results in relative increase of (W_{1-x}, Ti_x) solid solution specific volume content as compared to pure α–W phase, however, this does not allow avoiding multi-zone structure inhomogeneity of the grains due to concentration stratification caused by limited diffusion mobility of titanium atoms.

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Фазовий склад, структура та напружений стан конденсатів системи W–Ti, одержаних магнетронним розпиленням

О.В.Соболь

Досліджено вплив елементного складу та температури осадження іонно-плазмових конденсатів системи W–Ti на їх структуру, субструктурні характеристики та напружений стан. Встановлено, що кристалічний стан усіх фаз, які формуються при осадженні, базується на ОЦК кристалічній решітці вольфраму. На початкових стадіях формування твердого розчину при відносно малому вмісті атомів титану у решітці (7...11 ат.%) відбувається орієнтований ріст кристалітів з площинами типу (100) паралельно поверхні підкладки. При більш високому вмісті атомів титану відбувається формування сильнодеформованих кристалітів (W_{1-x}, Ti_x) твердого розчину з невеликим ступенем досконалості текстури з площиною (110) кристалічної решітки, паралельною поверхні зросту. В цьому випадку збільшення температури осадження від 300°C до 800°C призводить до концентраційного розшарування твердого разчину і утворення двох твердих розчинів з різним стабільним елементним складом. Проведено оцінку коефіцієнта дифузії у нанокристалічних конденсатах системи W–Ti і запропоновано модель для опису структурного стану конденсатів, що формуються.