JOURNAL OF NANO- AND ELECTRONIC PHYSICS
Vol. **10** No 1, 01010(6pp) (2018)

ЖУРНАЛ НАНО- ТА ЕЛЕКТРОННОЇ ФІЗИКИ
Том **10** № 1, 01010(6cc) (2018)

The Influence of Layers Thickness on the Structure and Properties of Bilayer Multiperiod Coatings Based on Chromium Nitride and Nitrides of Transition Metals Ti and Mo

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(Received 25 July 2017; published online 24 February 2018)

The influence of the layers thickness of bilayer multi-period coatings of the CrN_x/MoN_x and CrN_x/TiN_x systems on their phase-structural state, substructure, stress-strain state and mechanical properties was studied using methods of precision structural analysis in combination with computer simulation of implantation processes during particle deposition. It is established that a two-phase structure of CrN and γ-Mo_2N phases of the structural type NaCl is formed in the multi-period coatings of the CrN_x/MoN_x system with a nanometer thickness of the layers. Because of the small difference in periods (less than 0.5 %) for $\Lambda < 20$ nm, the layers form a coherent interlayer interface. The use of small $U_b = -20$ V during deposition makes it possible to avoid significant mixing at interlayer (interphase) boundaries even at the smallest $\Lambda = 10$ nm. Nitride layers formed under conditions of vacuum arc deposition are under the action of compressive stresses.

In the CrN_x/TiN_x system, because of the relatively large discrepancy between periods (more than 2.5 %), during the formation of the same structural components in the layers (CrN and TiN phases of the structural type NaCl), the epitaxial growth with period adjusting does not occur, even for the smallest $\Lambda = 10$ nm. The action of the deformation factor at the interphase boundary allows achieving an ultrahard state (with a hardness of about 50 GPa), which causes a relatively low friction coefficient.

The obtained results on the formation of phase-structural states with the nanoscale thickness of layers of multi-period nitride coatings are explained from the position of minimization of surface energy and deformation energy.

Keywords: Vacuum arc, Multi-period coatings, CrN, MoN, TiN, Layer thicknessm, Bias potential, Phase composition, Structure, Computer simulation, Hardness, Friction coefficient.

DOI: 10.21272/jnep.10(1).01010

PACS numbers: 81.07.Bc, 61.05.cp, 61.82.Rx
61.50.Ks, 62.25. – g

1. INTRODUCTION

Nitrides of transition metals such as TiN [1], ZrN [2], MoN [3], CrN [4], TiAlN [5] and TiZrSiN [6] are widely used as protective coatings for cutting tools, parts and mechanisms of various functional purpose because of their high hardness and wear resistance [7, 8]. In addition, coatings such as CrN also have a high resistance to oxidation, wear and corrosion [9, 10]. Nevertheless, CrN has a rather high friction coefficient (COF) (0.4-0.8) in air [5]. The transition to multielement nitride coatings based on five to six transition metals makes it possible to slightly increase the tribological characteristics [11]. However, for this type of structure, the optimization possibilities are very limited.

Thus, the mechanical properties of nitrides of transition metals, which are in the form of a single-phase coating, in most cases cannot fully satisfy the requirements for their functional characteristics [12]. The transition to a multi-layered structure of coatings allows not only to reproduce the advantages of each component, but also to achieve higher properties, which is one of the most effective methods for improving the characteristics of coatings. It was shown that multilayer coatings have higher hardness and viscosity as compared to monolayers.

To achieve high mechanical properties, it is necessary to select the systems used, taking into account the phase composition [12], structure, substructure and stress-strain state [13]. This selection process is based on the results of structural engineering [14]. So it is assumed that in the case of the CrN base, it is advisable to use MoN as the second layer in the multi-period system [15]. The creation of such a multi-period (CrN/MoN) structure is one possible way to reduce the friction coefficient of coatings, for example from 0.6-0.8 (present in CrN) to 0.3-0.4 (present in MoN [16]), which can lead to a significant increase in the service life of such a coating. Also for the mechanical properties, the macrostress state of the layers is of great importance. As shown in [17], compressive stresses of – 2 GPa in the Mo-N layers and tensile stresses 0.15 GPa in Cr-N layers take place during magnetron sputtering. The hardness of such coatings does not exceed 26 GPa. The thickness of the layers in the period is also an important parameter determining the operability of a multi-period coating. Thus, for coatings obtained by magnetron sputtering (where the low degree of ioniza-

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tion and the average energy of the particles are relatively small), an increase in hardness with decreasing layer thickness was established [e.g. 18]. However, even in this case, there is a critical thickness below which the hardness of the multi-period coating falls. For most systems, this critical layer thickness is 5-8 nm [19].

In this study aim was to study the structure, substructural characteristics, stress-strain state, mechanical and tribological properties of nanolayer multi-period coatings, depending on the thickness of the nanolayers. It was expected that intensive ion bombardment using vacuum arc method [20] compared with electron-beam [21] and magnetron sputtering [22] will increase the adhesion, residual stresses and nanohardness of coatings, but may lead to interphase mixing at the boundaries of the layers.

In addition, the systems selected in this study are interesting in comparison because the CrN_x/MoN_x system is characterized by a small difference in the periods of the crystal lattices of the constituents (less than 0.5 %), and the CrN_x/TiN_x system is characterized by a relatively large corresponding difference (more than 2.5 %).

2. EXPERIMENTAL DETAILS

Multi-period nanolayer coatings were deposited in the modernized vacuum-arc unit "Bulat-6" using a rotary device with two evaporators [23]. Cr, Ti and Mo are metal targets of purity more than 99.95 %. Active gas is nitrogen (purity 99.9 %).

Coatings were applied to the surface of samples (20 × 20 × 2 mm) made of 12X18H10T steel by rotating the substrate holder with alternate deposition from two evaporators. To reduce the effect of mixing during the deposition of multi-period structures, a low bias potential was used, with the value $U_b = - 20$ V. Deposition was carried out at an operating pressure of the nitrogen atmosphere $P_N = 3$ mTorr. For the CrN_x/MoN_x system, the bilayer period (Λ) was 300, 150, 80, 40, 20, 10 nm. Coatings of the CrN_x/TiN_x system were obtained with $\Lambda \approx 150$, 20 and 10 nm. The total thickness of the coatings was about 10 μm.

The phase composition, structure, and substructural characteristics were studied by X-ray diffractometry (DRON-4) using Cu-Kα radiation. The determination of structural and sub-structural parameters, as well as of the stress-strain state, was carried out according to standard techniques for film samples [13]. To decode the diffractograms, the tables of the international diffraction data center Powder Diffraction File were used.

Microindentation was carried out at the «Micron-gamma» device at a load up to $F = 0.5$ N with a Berkovich diamond pyramid with an angle of sharpening 65°, with automatic loading and unloading for 30 seconds. The study of the wear resistance characteristics was carried out on a «Micron-friction» device by rotating a diamond indenter with a radius of curvature of approximately 500 μm along the coating circle. In the automatic mode, the load level (P) and the frictional force between the diamond surface and the coating (F) were recorded. The friction coefficient (f) was determined as a result of the ratio of the frictional force to the load.

To understand the spatial distribution of radiation-stimulated changes in the boundary (interlayer) regions during the deposition of high-energy particles, computer simulation was used. For this purpose, a program based on the approximate method of double collisions, SRIM [24] was used in the work.

3. RESULTS AND DISCUSSION

In the formation of multi-period systems in the first case (CrN_x/MoN_x) one can expect a combination of CrN-inherent high resistance to oxidation, wear, corrosion) with low friction coefficient present in MoN in a wide temperature range (~ 0.18 of self-lubrication) and low wear rate [25].

Figure 1 shows an electron microscopic "cross section" image of a multilayer coating of CrN_x/MoN_x with layer thickness $\Lambda \approx 300$ nm.

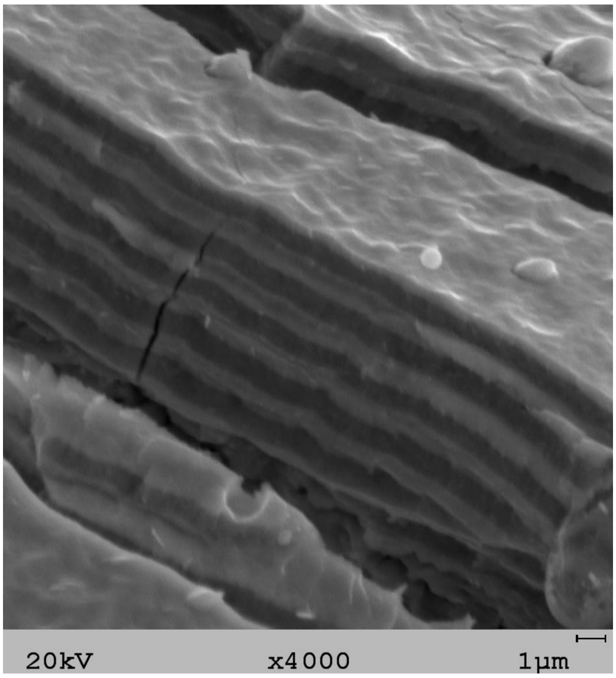

Fig. 1 – A scanning electron microscope image of the cross section of the CrN_x/MoN_x coating with layer thickness $\Lambda \approx 300$ nm

Since heavier atoms with a large atomic number Z give more vivid shades of gray in the SEM images, the MoN layers with a large average Z are visible, as the lighter ones. The CrN layers have a lower average atomic number, which leads to a darker color of the layers. Note the good planarity of the layers and their equal thicknesses (Fig. 1).

Analysis of the results of X-ray diffraction studies of multi-period compositions CrN_x/MoN_x showed the formation of phases with an isostructural crystal lattice CrN (JCPDS 77-0047) and γ-Mo_2N (JCPDS 25-1366) (structural type NaCl) in the layers. Figure 2 shows the most typical spectrum for this series of coatings (at $\Lambda \approx 300$ nm) and its division into components. Spectrum 1, taking into account the isolation of the component profiles, shows the presence of 2 phases (CrN and γ-Mo_2N) corresponding to the layers. A texture formation with the axis [311] is observed in the γ-Mo_2N

layer, and the formation of a significant texture is not observed in the CrN layer at the used bias potential – 20 V. The lattice period in γ-Mo$_2$N layers was 0.424 nm, and in CrN was 0.417 nm. Such an increase in the period in thick layers (where mixing is negligible) can be determined by the action of compressive stresses [13]. The calculations show that a γ-Mo$_2$N develops higher compression voltages of – 4.77 GPa, while in the CrN layers, the stresses are less significant and amount to – 1.68 GPa. The compressive stress in the coating is caused by high-energy ion bombardment during deposition [6]. Such stresses have a favorable effect on the increase in hardness and adhesion of the coating.

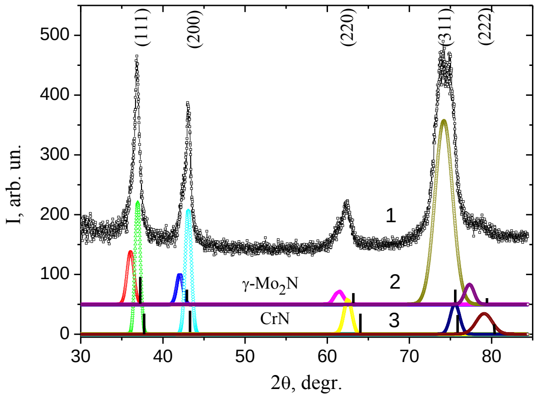

Fig. 2 – The area of the diffraction spectrum of CrN$_x$/MoN$_x$ (1), as well as the γ-Mo$_2$N spectra of the component (2) and the CrN component (3), obtained after division of complex diffraction profiles. The vertical lines show the positions and relative intensities of the table spectra of γ-Mo$_2$N and CrN phases (JCPDS 25-1366 and JCPDS 77-0047)

At the substructural level, there are also significant differences in the layers:

– in γ-Mo$_2$N layers, the average crystallite size is 180 nm, and the micro strain $<\varepsilon>$ reaches 1.52 %;

– in the CrN layers, the average size of the crystallites is less (about 27 nm), and the micro strain is much less than 0.47 %. In the latter case, the small values of the micro strain are apparently determined by partial relaxation due to the nanoscale grains scanning along the boundaries.

An increased period (typical for γ-Mo$_2$N with more than 80 % filled with octahedral interstitial sites) was observed in vacuum-arc coatings earlier in the formation of the Mo-N system under conditions of high nitrogen pressure and a low bias potential [26]. It should be noted that for the formation of CrN, a single layer exhibits the face-centered-cubic phase (JCPDS 77-0047), whereas the MoN single layer demonstrates the hexagonal δ-MoN structure (JCPDS 89-5024) occurs in the N$_2$ atmosphere only at high pressures of 2 Pa and at a bias voltage of – 200 V [5].

In contrast to the large thickness of the layers (when the interlayer boundary has a small effect on the phase composition and the stress state), at a small thickness of the layers, there is no noticeable allocation into two phases on the diffraction spectra. The average value of the period calculated from the peak positions is 0.422 nm. This value exceeds the table values for γ-Mo$_2$N (period 0.416 nm, JCPDS 25-1366) and for CrN (period 0.414 nm, JCPDS 77-0047). Such an increase in the period corresponds to a compression macro strain of – 1.4 % or (with an elastic modulus of 316 GPa and a Poisson's ratio of 0.29) compressive stresses of – 3.4 GPa.

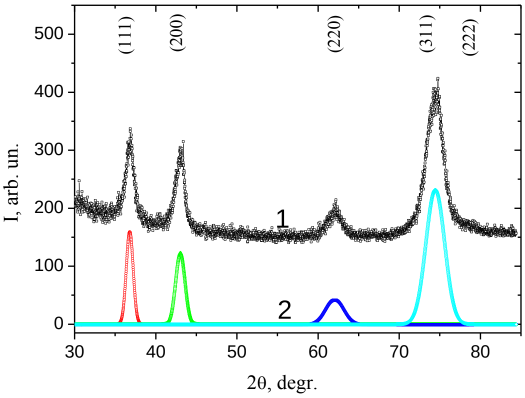

Fig. 3 – The area of the diffraction spectrum of CrN$_x$/MoN$_x$ (1), as well as the component spectra after division of diffraction peaks of complex shape (2)

The obtained results can be interpreted as a result of the "adjustment" (epitaxy) of the lattice for a single crystal structure (the structural type of NaCl) with a small discrepancy between the periods [14]. In the CrN$_x$/MoN$_x$ system, a γ-Mo$_2$N phase is formed in the Mo-N layers with a crystalline cell parameter close to the cubic phase of CrN. The reason for the appearance of such epitaxy at low thicknesses is the determining factor for minimizing the interphase energy between the layers. At large layer thicknesses, the difference in the periods leads to the determining contribution of the deformation factor (due to the difference in periods) and does not allow epitaxial growth.

At the substructural level for this type of coating, the average crystallite size is 15.4 nm, and the micro strain $<\varepsilon>$ reaches 1.04 %, which is somewhat less than in the thick γ-Mo$_2$N layers, where it is 1.52 %.

The diffraction spectra obtained from the coatings of the CrN$_x$/TiN$_x$ system with $\Lambda \approx 150$ and 10 nm are shown in Figures 4 (a) and 4 (b), respectively.

It can be seen that for the case of a relatively large layer thickness (Figure 4a) and for a small thickness (Figure 4b), two diffraction spectra appear which correspond to CrN (JCPDS 77-0047) and TiN (JCPDS 38-1420) phases with cubic lattices structural type of NaCl. The lattice periods are increased in comparison with the tabulated values, and for the CrN phase crystallites are 0.4171 nm ($\Lambda \approx 150$ nm) and 0.4174 nm ($\Lambda \approx 10$ nm), and for the TiN crystallites the phases are 0.4260 nm ($\Lambda \approx 150$ nm) and 0.4258 nm ($\Lambda \approx 10$ nm). The values obtained correspond to the action of compression strain in the layers, about 0.7 %. It should be noted that as the layer thickness decreases, the lattice period of the TiN phase decreases and the CrN phase increases. Such an effect can be associated with the initial stage of lattice epitaxy, which, however, because of the large difference in the periods, does not go over to full growth epitaxy.

Also the peculiarity of thin layers is the appearance of a preferential orientation of the crystallites with the [100] axis perpendicular to the growth plane (Fig. 4b – can be seen in an increase in the relative intensity of the peaks from the (200) plane in the angular range $2\theta = 40...45°$, both from TiN, so and CrN phases). As the Λ increases, the axis of the axial texture appears [111].

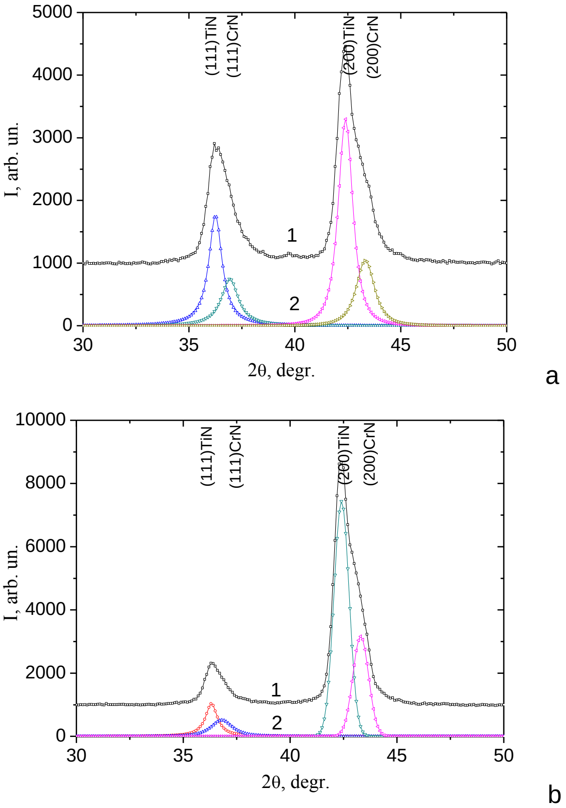

Fig. 4 – The areas of the diffraction spectra of the CrN_x/TiN_x coatings obtained at $U_b = -20$ V and $\Lambda \approx 150$ nm (a) and $\Lambda \approx 10$ nm (b). 1 – initial spectrum, 2 – after division of complex diffraction profiles

The study of the functional characteristics of the coatings obtained consisted of a study of hardness and tribological characteristics.

Hardness is the most universal indicator of the mechanical properties of the surface. The results of hardness measurements are shown in Figure 5.

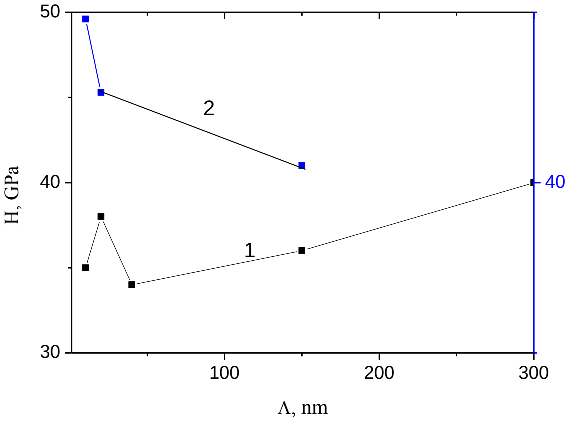

Fig. 5 – Dependence of hardness H on Λ in multi-period coatings CrN_x/MoN_x (1) and CrN_x/TiN_x (2)

It can be seen that the hardness of the multi-period coatings of the CrN_x/MoN_x system is lower than CrN_x/TiN_x. This is due to the higher hardness of the TiN component compared to MoN. It should be noted here that the dependence of H on Λ is different in the region of small periods ($\Lambda < 20$ nm). It is seen that for this critical region $\Lambda < 20$ nm for the CrN_x/MoN_x system there is a relative decrease in hardness, which reaches 10.4 %. In the CrN_x/TiN_x system such a drop is not observed, and the hardness reaches 49.7 GPa (Fig. 5, curve 2).

Thus, the state that determines the stability of the structure and properties of multi-period composite coatings is the state of interphase boundaries [16]. To assess the depth of impact during layers deposition, computer simulation of implantation processes was carried out in the work. Simulation of processes that can occur at the boundary at the nanometer scale of layers is based on radiation-stimulated effects upon deposition. The simulation showed that with the small potential $U_b = -20$ V used, the maximum depth of action, both in the CrN_x/MoN_x system and in the CrN_x/TiN_x system, does not exceed 1 nm. Thus, radiation-stimulated mixing at the boundaries of layers cannot be a determining factor of the structural state and properties even at the smallest critical thickness $\Lambda < 20$ nm.

The tribological characteristics of coatings showed quite predictably the interrelation with the structure of coatings and their hardness. As a rule, coatings with the highest possible hardness, adhesion and minimum friction coefficient possess the least wear. [27]

The study of the wear resistance characteristics (a typical profilogram is shown in Figure 6) showed that the friction coefficient of coatings with the highest hardness ($\Lambda < 20$ nm) for systems "diamond indentor - multi-period coating" is rather low (0.12-0.13) (for the CrN_x/TiN_x system) and 0.12-0.14 (for the CrN_x/MoN_x system). For the system "steel ShKh-multi-period coating" the friction coefficient is much larger and is 0.32-0.41.

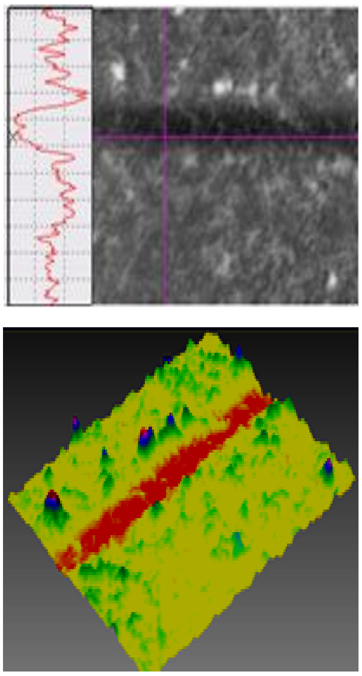

Fig. 6 – A photo of the friction surface, a scanning curve of the groove depth and a 3D groove profile of the CrN_x/MoN_x coating

At the same time, a smooth decrease of the frictional force is observed at the frictional force variations obtained for the pair "diamond indentor - multi- period coating" with the increase in the number of rotations, which is associated with an increase in the contact area and, correspondingly, a decrease in the contact load level. For the system "steel ShKh - multi-period coating", a more significant decrease in the frictional force at the first turns is observed. This is due to the fact that the steel has a much lower hardness and therefore, at the first turns, the contact area increases and, correspondingly, the frictional force decreases due to a sharp decrease in the load per unit area.

4. CONCLUSIONS

In the multi-period coatings of the CrN_x/MoN_x system with a nanometer thickness of layers, a two-phase structure is formed from CrN and γ-Mo_2N phases of the structural type NaCl. Because of the small difference in periods (less than 0.5 %) at $\Lambda < 20$ nm, the layers form a coherent interlayer interface (with matching lattice periods) to minimize the interfacial energy (due to the less favorable thermodynamic state in the Mo-N layer, the crystal structure is reproduced crystal structure, as in the CrN layers (such as NaCl)). The use of small $U_b = -20$ V in deposition avoids significant mixing at interlayer boundaries even at the smallest $\Lambda = 10$ nm. The nitride layers formed under vacuum arc deposition are under the action of compressive stresses.

In the CrN_x/TiN_x system, epitaxial growth does not take place due to the relatively large discrepancy of the periods (more than 2.5 %) during the formation of the same structural constituents in the layers (CrN and TiN phases of the structural type NaCl) with the adjustment of the periods (due to the large deformation factor) even at the smallest $\Lambda = 10$ nm. The action of the deformation factor on the interphase boundary (preventing the penetration of defects and formation of nanocracks) makes it possible to achieve a superhard state (with a hardness of about 50 GPa), which results in a relatively low friction coefficient: 0.12-0.13 for the system "diamond indentor - multiperiodic coating" and 0.32-0.34 for the system "steel ShKh - multi-period coating".

Вплив товщини шарів на структуру і властивості бішарових багатоперіодних покриттів на основі нітриду хрому і нітридів перехідних металів Ti і Mo

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Методами прецизійного структурного аналізу в поєднанні з комп'ютерним моделюванням імплантаційних процесів при осадженні прискорених частинок досліджено вплив товщини шарів двошарових багатоперіодних покриттів систем CrN_x/MoN_x і CrN_x/TiN_x на їх структурно фазовий стан, субструктуру, напружено-деформований стан і механічні властивості. Встановлено, що в багатоперіодних покриттях системи CrN_x/MoN_x нанометровою товщиною шарів формується двофазна структура з CrN і γ-Mo_2N фаз структурного типу NaCl. Через малу різницю в періодах (менше 0,5 %) при $\Lambda < 20$ нм шари формують когерентний міжшаровий інтерфейс. Використання при осадженні низької $U_b = -20$ В дозволяє уникнути істотного перемішування на міжшарових (міжфазних) границях навіть при найменшому $\Lambda = 10$ нм. Нітридні шари, які формуються в умовах вакуумно-дугового осадження знаходяться під дією напружень стиснення.

В системі CrN_x/TiN_x через відносно велику невідповідності періодів (понад 2,5 %) при формуванні однотипних структурних складових в шарах (CrN і TiN фази структурного типу NaCl) епітаксіального зростання з пристосуванням періодів не відбувається навіть при найменшому $\Lambda = 10$ нм. Дія при цьому деформаційного фактора на міжфазній границі дозволяє досягти надтвердого стану (з твердістю близько 50 ГПа), що обумовлює відносно низький коефіцієнт тертя.

Отримані результати по формуванню структурно фазових станів при нанорозмірній товщині шарів багатоперіодних нітридних покриттів пояснені з позиції мінімізації поверхневої енергії та енергії деформації.

Ключові слова: Вакуумна дуга, Багатоперіодні покриття, CrN, MoN, TiN, Товщина шару, Потенціал зсуву, Фазовий склад, Структура, Комп'ютерне моделювання, Твердість, Коефіцієнт тертя.

Влияние толщины слоев на структуру и свойства бислойных многопериодных покрытий на основе нитрида хрома и нитридов переходных металлов Ti и Mo

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Методами прецизионного структурного анализа в сочетании с компьтерным моделированием имплантационных процессов при осаждении ускоренных частиц изучено влияние толщины слоев бислойных многопериодных покрытий систем CrN$_x$/MoN$_x$ и CrN$_x$/TiN$_x$ на их фазово-структурное состояние, субструктуру, напряженно-деформированное состояние и механические свойства. Установлено, что в многопериодных покрытиях системы CrN$_x$/MoN$_x$ с нанометровой толщиной слоев формируется двухфазная структура из CrN и γ-Mo$_2$N фаз структурного типа NaCl. Из-за малой разницы в периодах (менее 0,5 %) при $\Lambda < 20$ нм слои формируют когерентный межслоевой интерфейс. Использование при осаждении малого $U_b = -20$ В позволяет избежать существенного перемешивания на межслоевых (межфазных) границах даже при самом малом $\Lambda = 10$ нм. Формируемые в условиях вакуумно-дугового осаждения нитридные слои находятся под действием напряжений сжатия.

В системе CrN$_x$/TiN$_x$ из-за относительно большого несоответствия периодов (более 2,5 %) при формировании однотипных структурных составляющих в слоях (CrN и TiN фазы структурного типа NaCl) эпитаксиального роста с подстраиванием периодов не происходит даже при самом малом $\Lambda = 10$ нм. Действие при этом деформационного фактора на межфазной границе позволяет достичь сверхтвердого состояния (с твердостью около 50 ГПа), что обуславливает относительно низкой коэффициент трения.

Полученные результаты по формированию фазово-структурных состояний при наноразмерной толщине слоев многопериодных нитридных покрытий объяснены с позиции минимизации поверхностной энергии и энергии деформации.

Ключевые слова: Вакуумная дуга, Многопериодные покрытия, CrN, MoN, TiN, Толщина слоя, Потенциал смещения, Фазовый состав, Структура, Компьютерное моделирование, Твердость, Коэффициент трения.

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