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Relationship between Structural Characteristics and Physical-Mechanical Properties of Hafnium-Diboride Films Deposited by RF Magnetron Sputtering

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A comparative analysis of the structure and physical-mechanical characteristics of hafnium-diboride films synthesized by means of RF magnetron sputtering system is performed. As shown, changing the design features of the magnetron sputtering system (MSS) changes the structure formation parameters and, accordingly, the properties of the synthesized films. Two series of samples are obtained: before and after making changes to the MSS design. In the first series of experiments, films with the growth texture of the (00.1) plane are formed at a positive bias potential applied to the substrate, have a strong growth texture and high physical-mechanical characteristics: nanohardness of 41 GPa and elastic modulus of 396 GPa. For the second series of samples, textured hafnium-diboride films with the most intense texture (00.1) are formed with a grounded-substrate holder, while the resulting films have the best physical-mechanical characteristics: nanohardness of 46 GPa and elastic modulus of 306 GPa. As shown, hafnium-diboride films with a strong growth texture normal to the (00.1) plane have the highest physical-mechanical properties, regardless of the deposition conditions during magnetron sputtering.

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Key words: coatings, films, magnetron sputtering, nanohardness, modulus of elasticity.

Проведено порівняльну аналізу структури та фізико-механічних характеристик плівок дибориду Гафнію, синтезованих за допомогою ВЧ-магнетронної системи розпорошення. Показано, що зміна конструктивних особливостей магнетронної розпорошувальної системи (МРС) змінює параметри структуроутворення та, відповідно, властивості синтезованих плівок. Одержано дві серії зразків: до та після внесення змін у конструкцію МРС. У першій серії експериментів плівки з текстурою росту площини (00.1) формуються за прикладеного до підкладинки позитивного потенціалу зміщення, мають сильну текстуру росту та високі фізико-механічні характеристики: нанотвердість у 41 ГПа і модуль пружності у 396 Па. Для другої серії зразків текстуровані плівки дибориду Гафнію з найбільш інтенсивною текстурою (00.1) формуються із заґрунтованим підкладинкотримачем; водночас, одержані плівки мають найліпші фізико-механічні характеристики: нанотвердість у 46 ГПа і модуль пружності у 306 ГПа. Показано, що плівки дибориду Гафнію з сильною текстурою росту, нормальною до площини (00.1), мають найвищі фізико-механічні властивості, незалежно від умов осадження під час магнетронного розпорошення.

Ключові слова: покриття, плівки, магнетронне розпорошення, нанотвердість, модуль пружності.

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1. INTRODUCTION

Film coatings of transition-metal diborides (CrB_2 , HfB_2 , TiB_2 , TaB_2 , etc.) having high hardness with a relatively small elastic modulus are promising for use as the heat-resistant and wear-resistant protective coatings in various industries [1–4]. Due to the high melting point, the most widely used method for applying such coatings is magnetron sputtering, mainly, on direct current and RF magnetron sputtering. By changing the parameters of magnetron sputtering (working pressure, discharge power, substrate temperature, distance between the target and the substrate, bias potential), it is possible to change the energy of particles, which participate in the deposition and formation of films on the substrate surface in a wide range. The review [5] provides a detailed analysis of the sputtering parameters and their influence on the formation of the structure and, accordingly, their physical-mechanical characteristics. This makes it possible to obtain coatings of various structural states, from amorphous to nanocrystalline ones, with the (00.1)-plane growth texture and the composition from substoichiometric MeB to superstoichiometric MeB_n . As shown, the superstoichiometry n values for transition-metal diboride films with the

(00.1)-plane growth texture vary widely from 2.07 to 4.5 for TiB_2 [6–11] and 2.7 to 3.84 for TaB_2 [10–12]. However, the best hardness values, more than 40 GPa, were possessed by superstoichiometric films with the B/Me ratio within 2–3, which was noted for titanium [6, 8, 13, 14] and tantalum [9, 10, 13, 15, 16] diborides.

A characteristic feature of transition-metal diboride films is that boron-superstoichiometric films with a strong (0001)-plane growth texture had higher hardness values of 43.9 GPa (HfB_2), 44 GPa (TaB_2), 43 GPa (CrB_2), 48.5 GPa (TiB_2) compared to stoichiometric films ($HV = 33$ GPa, 36 GPa, 37 GPa, and 35 GPa, respectively) [6, 9–17]. It should also be noted that, of this class of compounds, only vanadium-diboride films with a (0001)-plane growth texture did not show such high characteristics. This was associated with a high percentage of oxide phases in the film [18, 19].

Among all nanostructured coatings of transition-metal diborides, hafnium-diboride coatings are of particular interest. Hafnium diboride has the highest melting point among diborides, high chemical resistance, and high hardness. Functional coatings based on it are capable of operating under extreme conditions, while maintaining the above-mentioned properties. Some of the promising areas of application of these coatings are nuclear energy and aerospace, namely, the creation of hypersonic aircraft. The study of the properties of nanostructured coatings of hafnium diboride began relatively recently [20] and remains relevant to this day [21].

A comparative analysis of the influence of structural characteristics on the properties of transition-metal boride films showed that films with a strong growth texture by the (0001) plane and high hardness values are formed for various magnetron sputtering systems at various magnetron sputtering parameters, both with direct current and with RF sputtering [8, 22]. This is due to the design features of various magnetron systems.

In this work, a study was conducted of the relationship between the structure and physical-mechanical characteristics of hafnium-diboride films synthesized using an RF magnetron sputtering system.

2. EXPERIMENTAL/THEORETICAL DETAILS

The horizontal RF magnetron sputtering system (MSS) based on an unbalanced planar magnetron designed for the use of standard round targets with a diameter of 120–125 mm and a thickness of up to 10 mm was used for deposition of hafnium-diboride films. The schematic diagram of scattering unit is shown in Fig. 1 [18]. The magnetron magnetic field with strength of about $4 \cdot 10^3$ A/m on the target surface is created by a set of permanent ring magnets (CoSm) with a steel pole core. The source of RF power is a generator with a frequency of

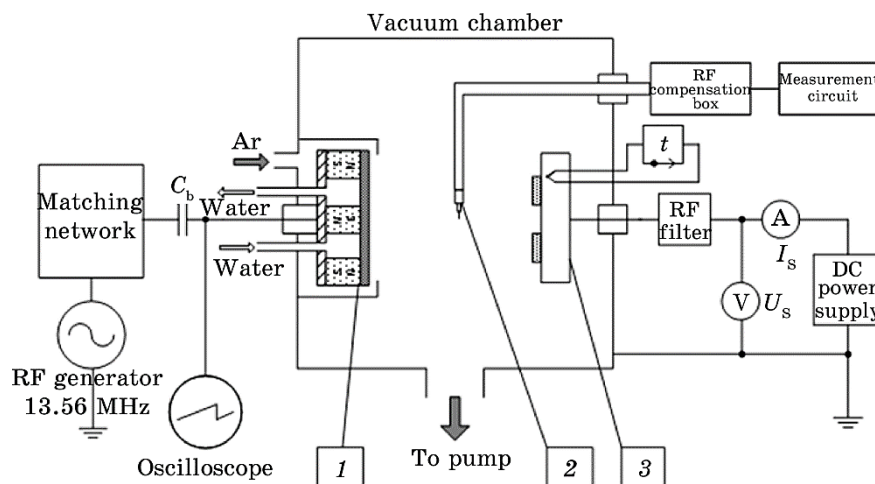


Fig. 1. Diagram of a magnetron sputtering system: 1—unbalanced magnetron, 2—Langmuir's probe, 3—substrate holder.

13.56 MHz and an adjustable power of up to 1 kW connected to the load, using a matching network, which is a L -circuit of reactive elements, and a blocking capacitor that ensures the occurrence of a negative self-bias on the target.

The substrate holder is electrically isolated from the installation body by ceramic insulators and designed for fastening plate substrates. A constant voltage source is connected to the substrate holder through an RF filter, allowing a bias potential in the range from -100 to $+100$ V to be applied to the substrate. The samples are heated, using a tungsten filament with a diameter of 2 mm; a chromel–alumel thermocouple provides temperature control up to 1100 K.

The sintered HfB_2 target was sputtered in Ar^+ plasma. Hafnium-diboride coatings were deposited on 12X18H10T stainless-steel substrates measuring $20 \times 10 \times 3$ mm, pre-polished to a surface roughness of $Ra = 0.25$ μm . The pressure of the residual gases in the chamber before spraying was of $3 \cdot 10^{-3}$ Pa. The duration of the deposition of each sample was of 60 min.

Two series of samples were obtained: the first series was obtained before modification of the magnetron sputtering system; the second series was obtained after modification of the MSS. The design changes were made in the modified installation: the designs of the magnetron and substrate holder were changed, the pumping system was changed, and a Langmuir's probe was installed to measure the parameters of RF plasma. In both cases, the working gas pressure during sputtering was of 0.65 Pa, the RF generator power was of 500 W, the bias potential applied to the substrate varied from $+25$ V to -25 V relative to ground

with a step of 25 V. The distance between the target and the substrate holder was $d_{s-t} = 60$ mm. The substrate temperature was maintained at 300°C.

Immediately before deposition, ion cleaning was carried out with argon ions for 3 min.

The thickness of the films was controlled by multibeam interferometry using an MII-4 interferometer; it was of approximately 1.5 μm .

X-ray diffraction studies of the structure of the material were performed on an automated diffractometer ДРОН-3. When recording, CuK_α radiation (wavelength of 0.154 nm) was used with Bragg–Brentano focusing θ – 2θ (2θ is the Bragg angle). The current and voltage values at the x-ray tube were of 20 mA and 40 kV. The samples were recorded with horizontal slits of 4 mm on the tube and of 1 mm on the detector in continuous recording mode at a speed of 1°/min with a range of 2θ -angles from 20° to 70°. The calculation of nanocrystallite sizes and microdeformations was carried out using the Scherrer's method.

Hardness and elastic modulus were measured by nanoindentation. For the first series of samples, nanoindentation was performed by means of the Nano Indenter II device from MTS Systems equipped with a diamond indenter sharpened in the shape of a trihedral pyramid (Berkovich's indenter). Nanoindentation of the second series of samples was performed by means of the Ultra-Nano Hardness Tester (UNHT) device from CSM Instruments equipped with a Berkovich's indenter too. Hardness and elastic modulus were found by analysing the indenter-unloading curve, using the Oliver–Pharr method [19].

3. RESULTS AND DISCUSSION

Two series of samples were obtained: the first series was obtained before modification of the magnetron sputtering system; the second series was obtained after modification of the MSS. The following design changes were made in the modified setup:

- the magnetron design was changed: the central iron core (pole) was replaced with a permalloy core, which significantly changed the magnetron balance (Fig. 2) and allowed increasing the concentration of the discharge plasma between the target and the substrate holder;
- the discharge gap (between the target and the grounded screen) was changed from 8 to 6.5 mm in order to reduce capacitive losses;
- the area of the substrate holder was increased in order to increase the discharge capacity;
- the pumping system was improved and the Langmuir's probe was installed to measure the parameters of the RF plasma.

Before replacing the magnetron core, the magnetron magnetic-field line configuration was simulated, using the ANSYS Mechanical APDL

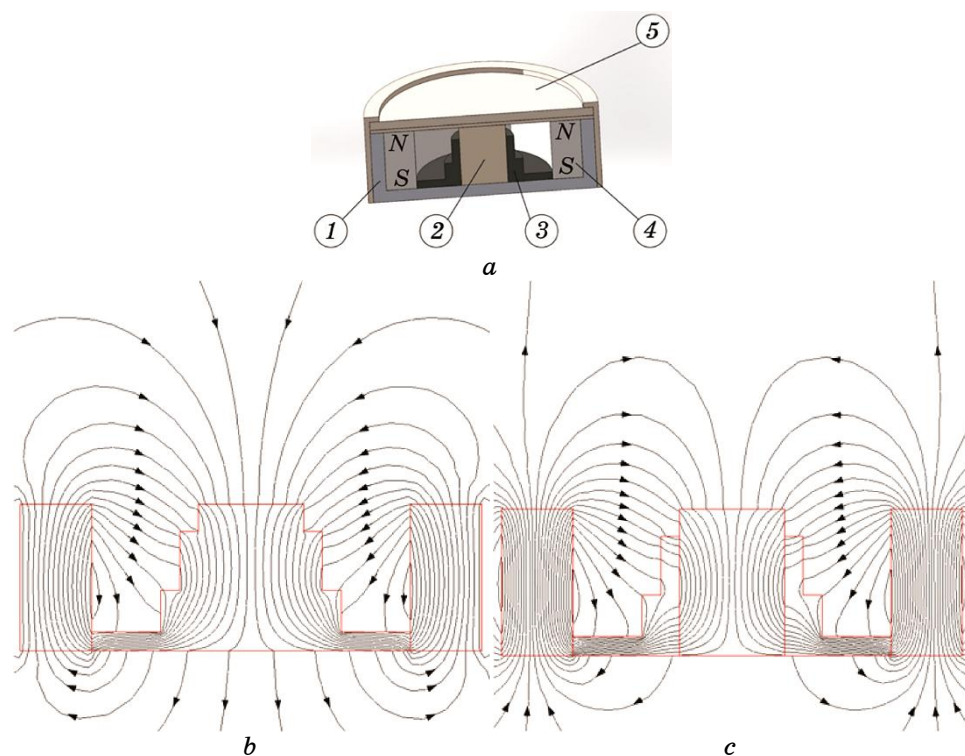


Fig. 2. Results of modelling the magnetic field of a magnetron: magnetron (1—stainless-steel body of the magnetron, 2—core, 3—steel core holder, 4—CoSm ring magnet, 5—HfB₂ target) (a); magnetic field of a magnetron with a steel core (b); magnetic field of a magnetron with a permalloy core (c).

package, the results of which are shown in Fig. 2. As can be seen, such a replacement leads to a change in the magnetron balance from type I to type II, according to Ref. [20]. Such a replacement leads to a significant change in the charged particle flows in the magnetron glow-discharge plasma during film deposition. Thus, when using a magnetron of the second type, there is an increase in the charged particle flows to the substrate (ions and electrons, depending on the bias potential on the substrate), and there is also an increase in the bias potentials on the surfaces coming into contact with the plasma discharge. This leads to an increase in the energy transferred by the charged particles to the growing film and, accordingly, to a modification of the structure and preferred orientation of the coating.

X-ray structural studies of the first series of samples showed (Fig. 3, a) that, with a grounded substrate holder, nanocluster films of hafnium diboride are formed. Applying a positive bias potential leads to the formation of films with a growth texture (00.1). Applying a nega-

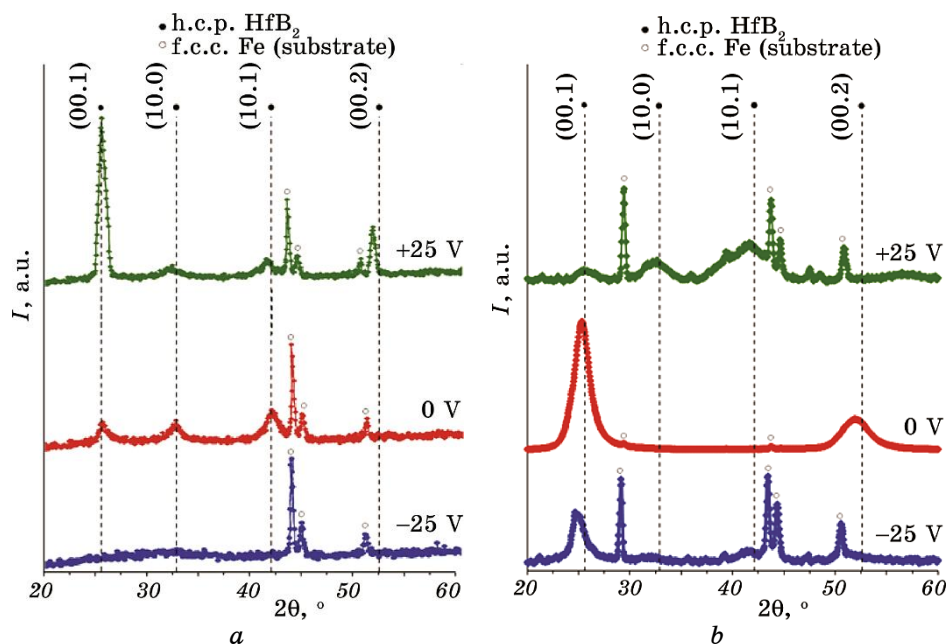


Fig. 3. Diffraction patterns of hafnium diboride films of samples obtained: in the first experiment [22] (a), in the second experiment (the diffraction pattern of the sample deposited with the substrate holder grounded is shown on a scale of 1:10) [18, 23] (b).

tive bias potential of -25 V leads to the formation of amorphous-like films. Changing the design features of the MSS leads to a significant change in the parameters of formation of the texture of the synthesized films.

For the second series of samples, with a grounded substrate holder, textured hafnium-diboride films with the most intense texture (00.1) are formed. Applying a negative bias potential of -25 V (Fig. 2, b) leads to a decrease in the growth texture by the (00.1) plane [22, 23]. Significant structural changes occur, when applying a positive bias potential of $+25$ V to the substrate holder (Fig. 2, b). Nanocluster films are formed [22, 23] with the growth texture being completely absent.

Calculation of the texture coefficient allows us to study the development of texture as a function of the change in bias voltage on the substrate. Texture coefficients for HfB_2 films were calculated, based on XRD studies of the obtained samples (Fig. 4), using the following formula [24]:

$$T_{(hk.l)} = \frac{I_{(hk.l)}}{I_{(00.1)} + I_{(10.0)} + I_{(10.1)}}, \quad (1)$$

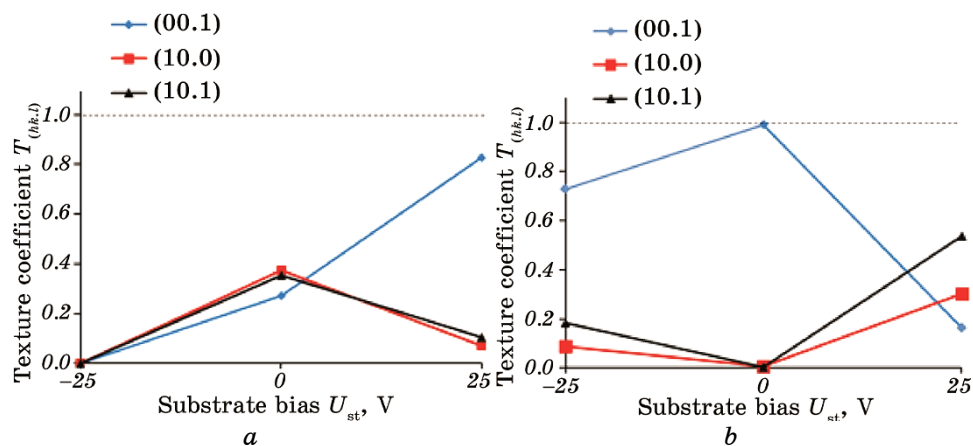


Fig. 4. Change in texture coefficients depending on bias voltage: for samples of the first series (a); for samples of the second series (b).

where $I(hk.l)$ represents the intensity of all the main orientations of the HfB_2 phase.

The sample from the second series obtained with a grounded substrate holder has the highest texture coefficient. In addition, the following have a high texture coefficient: the sample from the first series obtained with a bias potential of +25 V; the sample from the second series obtained with a bias potential of -25 V.

Based on the load-unload curves, the nanohardness and elastic modulus of the samples were calculated, using the Oliver-Pharr method (Fig. 5).

For the samples of the first series, the films with a strong growth texture by the (0001) plane had the best physical-mechanical properties: nanohardness of 41 GPa and elastic modulus of 396 GPa (Fig. 5, a). When the substrate holder is grounded, nanocrystalline films of hafnium diboride are formed, which leads to a decrease in hardness and elastic modulus (26 GPa and 254 GPa, respectively). Applying a negative bias potential of -25 V leads to the formation of amorphous-like films that ultimately leads to an even more-significant decrease in the hardness (13 GPa) and elastic modulus (187 GPa).

For the samples of the second series, the films with a strong growth texture by the (0001) plane also had the best physical-mechanical characteristics: nanohardness of 46 GPa and elastic modulus of 306 GPa (Fig. 5, b). For films with a less-pronounced growth texture, a decrease in the nanohardness value to 36 GPa and a decrease in the elastic modulus to 271 GPa are observed, which have already occurred previously for TaB_2 [25], TiB_2 [3, 26], CrB_2 [27] films. For hafnium-diboride nanocluster films obtained at a bias potential of +25 V, a significant

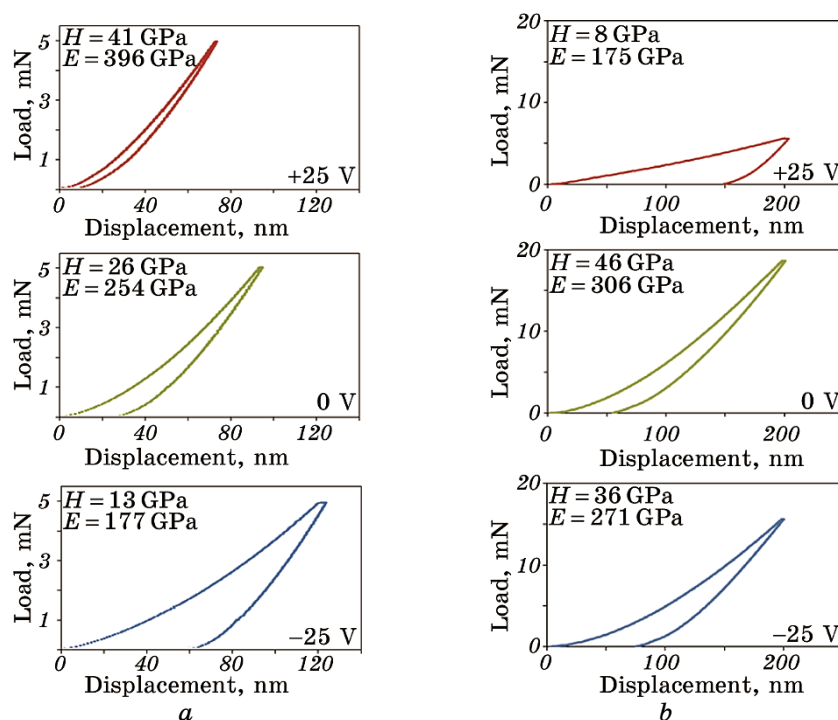


Fig. 5. Load–unloading curves, as well as nanohardness and elastic modulus obtained by nanoindentation of samples: first series (a); second series (b).

decrease in hardness and elastic modulus is observed (8 GPa and 175 GPa, respectively).

The calculations of the nanocrystallite sizes, carried out using the Scherrer's method, are presented in Table 1. A distinctive feature is that the nanocrystallite size of the highly-textured hafnium-diboride films from the first series is higher compared to similar highly-textured films from the second series.

Comparison of our results with those obtained by other authors, when depositing the transition-metal diboride films, using RF and DC magnetron sputtering systems, shows that each MSS has its own values of deposition parameters, at which films with the best physical-mechanical characteristics are formed.

Thus, in works [6, 13, 14], superstoichiometric-TiB_{2.4} films with the best physical-mechanical characteristics ($HV = 48.5$ GPa, $E \cong 480$ GPa) were obtained at a bias potential of -50 V, substrate temperature $T_s \cong 550^\circ\text{C}$ and current density $j = 1$ mA/cm². Highly-textured superstoichiometric titanium-diboride films with the composition $B/Me \cong 2.3\text{--}2.4$ having a nanohardness of 48.6 ± 3.1 GPa and elastic modulus of 562 ± 18 GPa were obtained by other authors [8, 32] at a

TABLE 1. Production modes, lattice parameters and mechanical properties of the studied HfB₂ coatings (*—JCPDS file, card No. 38-1398).

Sample series	Substrate bias potential U_s , V	Substrate temperature T , °C	Lattice parameters, nm			Nanocrystallite size D , nm	Nanohardness H , GPa	Modulus of elasticity E , GPa	Degree of texture
			a	c	c/a				
			3.1424*	3.476*	1.11*				
1	+25	300	3.230	3.400	1.05	25	41	396	strong
	0	300	3.160	3.460	1.09	10	26	254	—
	−25	300	3.179	3.560	1.10	—	13	177	—
2	+25	300	3.179	3.362	1.06	4	8	175	—
	0	300	3.210	3.494	1.09	8	46	306	relatively strong
	−25	300	3.182	3.379	1.06	15	36	271	strong

bias potential of -100 V and a substrate temperature of 175°C , which correlate with the above results. However, as shown in the works [34, 35] by Berger *et al.*, it is possible to form highly-textured titanium-diboride films with the best physical-mechanical characteristics ($H = 54 \pm 9$ GPa, $E = 600 \pm 85$ GPa) even at a positive bias potential of $+50$ V. In the work [17], highly-textured hafnium-diboride films of HfB_{2.4} with the best physical-mechanical characteristics ($HV = 44$ GPa and $E \cong 400$ GPa) were obtained by RF magnetron sputtering at a bias potential of $+50$ V and a substrate temperature of 500°C .

It is obvious that, regardless of the type of MSS (DC, RF) and its design features, there will always be a combination of deposition parameters (discharge power, working gas pressure, bias potential and temperature on the substrate, *etc.*) that will produce transition-metal diboride films with the highest degree of growth texture normal to the (00.1) plane and high physical-mechanical characteristics.

The analysis of the obtained results shows that the hardness of refractory compounds depends on their structural state. At the same time, the degree of texture affects the physical-mechanical characteristics of all transition-metal diborides having the AlB₂ structural type [4, 22].

4. CONCLUSION

The influence of structure on the physical-mechanical properties of hafnium-diboride films obtained by means of RF magnetron sputtering was studied. As shown, depending on the sputtering parameters, films are formed in various structural states from amorphous to nanocrystalline ones with a growth texture normal to the plane (0.01).

The values of hardness and elastic modulus varied from 8 GPa and 175 GPa to 46 GPa and 306 GPa, respectively. As shown, a change in the magnetron design leads to a change in the RF magnetron sputtering parameters. Hafnium-diboride films with the highest degree of growth texture normal to the plane (00.1) and high physical-mechanical properties (nanohardness of 41 GPa and elastic modulus of $\cong 400$ GPa) were obtained on unmodified MSS at a bias potential of +25 V. Modification of MSS led to a change in the values of the deposition parameters, as a result of which hafnium-diboride films with the highest degree of growth texture normal to the plane (00.1) and high physical-mechanical properties (nanohardness of 46 GPa and elastic modulus of 306 GPa) were obtained with a grounded substrate holder (bias potential of 0 V). A distinctive feature is that the size of nanocrystallites of highly-textured hafnium-diboride films from the first series is higher in comparison with similar highly-textured films from the second series, while the degree of texture for samples from the second series is higher, that probably leads to an increase in nanohardness to 46 GPa. It is obvious that the change in the design features of the MSS leads to a significant change in the parameters of the formation of the structural characteristics and texture of the synthesized films. The results obtained correlate with the results for highly-textured films of transition-metal diborides.

A comparative analysis of magnetron sputtering parameters for hafnium-diboride films with the data obtained for films of other transition-metal diborides showed that, regardless of the type of magnetron sputtering (DC, RF) and its design features, there will always be a combination of sputtering parameters (discharge power, working gas pressure, bias potential and temperature on the substrate, *etc.*), at which transition-metal diboride films with the highest degree of growth texture normal to the (00.1) plane and the best physical-mechanical properties are obtained.

Thus, it can be concluded that, if, at any sputtering parameters, highly-textured hafnium-diboride films are obtained, in particular, then, they will have *a priori* the highest physical-mechanical characteristics.

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