PLASMA ASSISTED DEPOSITION OF TaB_2 COATINGS BY MAGNETRON SPUTTERING SYSTEM

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In the present paper the results of TaB_2 coating deposition in cluster set-up comprising a low pressure planar magnetron and an inductive plasma source are presented. The system allows to control independently the fluxes of the deposited Ta and B atoms from the sputtered TaB_2 target, and the fluxes of argon ions and electrons from the inductive plasma. Low argon pressure in the chamber allows the deposition process in the collisionless regime, providing the composition of the deposited film which is very close to the stoichiometry of the sputtered target. The correlation of the TaB_2 coating structure with the substrate voltage in the range from -50 to +50 V is demonstrated.

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INTRODUCTION

The DC magnetron sputtering is a efficient tool for the formation of nanocomposite coatings [1-4]. The sputtering of coatings can be realized in pure argon and the coating containing elements of sputter target can be formed [1]. It is known [5], that main parameters in DC magnetron sputtering, which affect the mobility of atoms and hence define the growth mechanism and the film structure are:

- the substrate heating, i.e. ratio T_s/T_m (where T_s and T_m – substrate temperature and the melting point of the film material, respectively);
- the ion bombardment of the growing film.

The energy ε_b, delivered to the growing coating by the ion bombardment, which has a crucial effect on the structure, microstructure, elemental and phase composition and physical properties [6]. The value of the ε_b controlled by three parameters: (1) the substrate bias U_s, (2) the substrate ion current density j_i and (3) the deposition rate of coating a_D.

The transition metal diboride films are actively investigated owing to their high physical and mechanical characteristics. The tantal diboride films were subjected the most detailed study of their structure, composition and properties in the work [7]. Thus overstoichiometric films with growth texture (00.1) and columnar structure exhibit the superhardness effect of 48.5 GPa compared to films not possessing such structures.

The effect of the bias potential and substrate temperature on the structure, composition and mechanical properties of transition metals diboride (HfB_2, TaB_2) films deposited by RF-magnetron sputtering of targets in argon was studied in our previous papers [8, 9]. It was shown that the formation of coating of various structures – from an amorphous to nanocrystalline occurs depending on the substrate temperature and applied bias potential.

The aim of this work is a comparative analysis of the bias potential effect applied to the substrate and the additional argon ion bombardment from ICP discharge at unbalanced DC magnetron sputtering system of TaB_2 on the structure and the properties of the films.

1. EXPERIMENTAL SETUP

The tantalum diboride coatings were deposited on AISI 302 stainless steel substrates in the experimental multifunctional cluster ion plasma system with parameters corresponding to the demands of industrial operation. The main purpose of this system is synthesis and processing of complex composite (including nanocomposite) coatings and structures, based on TiN, AlN, TiO_2, Al_2O_3, ZrO_2, Ta_2O_5 and their combinations. The research results of the different module components investigations and technological module operation of high quality complex coatings were published previously in the works [10-14].

The basic novelty of the present work is the investigation of the argon ion flow with different energy and ion current density influence on the structure and mechanical properties of tantalum diboride coatings.

The multifunctional cluster set-up is schematically shown in the Fig. 1. The system consists of low-pressure magnetrons 2 (photo on Fig. 2,b) located on the butt end of chamber, the RF inductive source of argon plasma 3 located inside the chamber and the ion source 7 located on lateral flange of the chamber. The relative location of these components has been chosen to provide the possibility of the simultaneous action on the processed surface of the flows of metal atoms and ions of rare gas.

The RF inductive coupled plasma (ICP) source (3), (see Fig. 1 and photo on Fig. 2,a) produced a plasma stream, consisting of slow ions of argon with energy 20…40 eV and electrons. In such source plasma is concentrated in discharge chamber made from ceramic tube (see Fig. 2,a). At the source exit the perforate metal screen is erected to restricts the plasma and provide a pressure drop between the source volume and the technological chamber.

The plasma source was placed inside the vacuum chamber, that allows to choose the optimum relation between the distance from the magnetron (2) and plasma source (3) to samples on the substrate holder (9) (see Fig. 1). The ICP source was supplied by the RF power up to 1 kW (frequency 13.56 MHz) from the RF generator (4), which connected to the inductive coils through the RF matchbox (5).

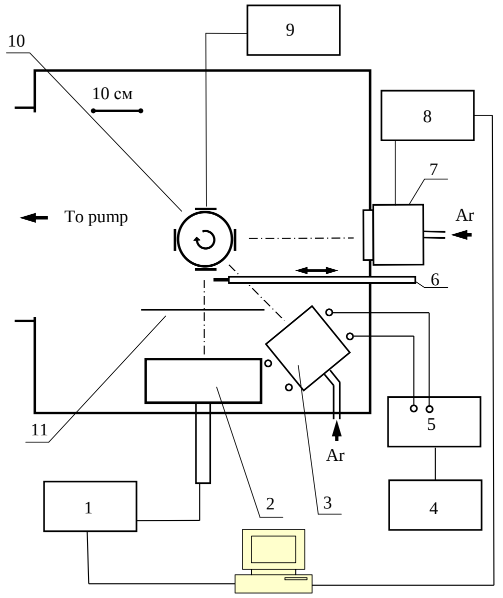
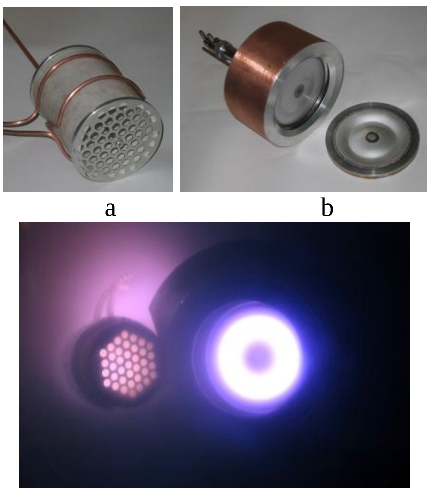


Fig. 1. Scheme of the cluster set-up for complex composite compounds synthesis. 1 – DC magnetron power supply; 2 – magnetron; 3 – RF ICP source; 4 – RF generator; 5 – RF matchbox; 6 – probe; 7 – ion source; 8 – DC power supply; 9 – power supply for samples polarization; 10 – samples rotation system; 11 – shutter

The multichannel ion source "Radical M" (7) produced the argon ion beam with the mean energy 0.5…1 keV [15], directed to the processed samples and applied for cleaning and activation the sample surface before the coating process.

Using the pulsed or DC power supply (9) for the work peaces polarization, it is possible to apply a constant or impulses voltage with different duty cycle to the rotated substrate holder (11).



c

Fig. 2. The photo of ICP source (a), magnetron (b) and the photo of inside chamber during the process (c)

2. TECHNOLOGICAL REGIME

The key novelty of the present system comparing to the known designs is the operating pressure range (0.4…2) mTorr, where motion of ions and sputtering atoms is free fall. It allows to increase the distance magnetron-substrate holder up to 30…40 cm, significantly increase the deposition area and operate with ICP and "Radical" ion sources.

In the Fig. 3 the current-voltage characteristics (CVC) of magnetron for the tantalum and sintered TaB_2 powder targets are shown. As can be seen from the figure CVC determines the main parameters of magnetron discharge – target voltage U_m and total discharge current I_m, for deposition technological regimes. In the Fig. 3 the basic technological regime in our experiments is demonstrated by area between shaded lines.

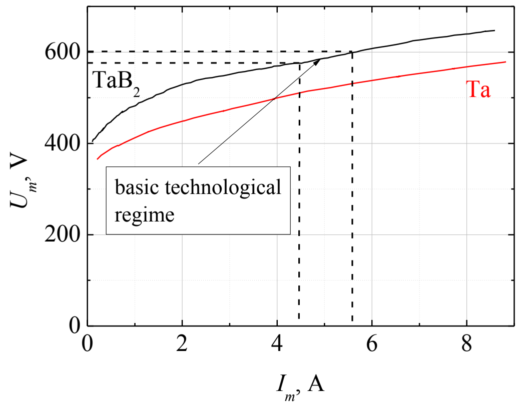


Fig. 3. Current-voltage characteristics of magnetron and the basic technological regimes (shaded area) *for deposition TaB_2 coating. Argon pressure p = 0.8 mTorr*

The second important parameter for deposition is the ion current density j_i to the substrate holder. In the Fig. 4 the radial distributions of j_i are presented separately for the magnetron plasma and ICP discharge. The distributions were measured by flat probe (7) (see Fig. 1) at potential (–30) V.

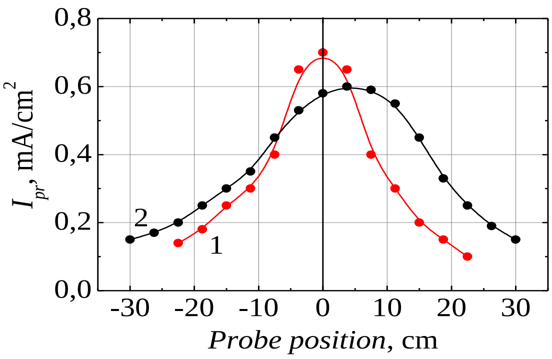


Fig. 4. Radial distributions of ion current density to the substrate holder for magnetron plasma (1) and ICP (2)

A detailed study of the dependencies of ion bombardment on the parameters of the magnetron and the ICP discharges given in the work [12].

The main parameters during the technological processes were monitoring by PC and the typical time dependences of these parameters and technological steps are presented in the Fig. 5.

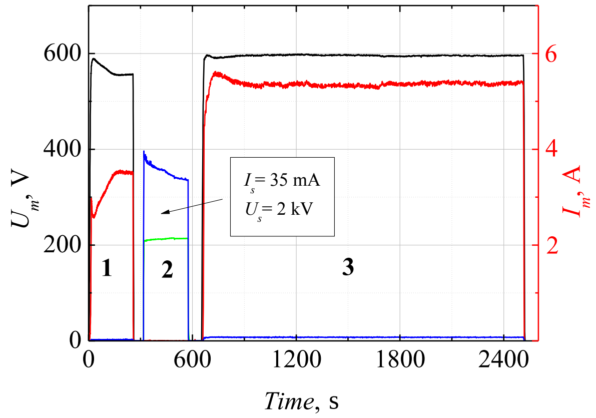


Fig. 5. Technological process. 1 – target trenning; 2 – samples cleaning; 3 – film deposition

3. EXPERIMENTAL RESULTS

The tantalum diboride coatings were deposited on substrates with the magnetron discharge power 2.5…2.8 kW, argon pressure in the working chamber was 0.6 mTorr. The substrate temperature was varied from 200 to 300°C. Deposition was carry out as on the grounded metal substrate holder, as well as upon application of a positive or negative bias potential. Specimens were disposed at a distance of 20 cm from the target, sputtering was carried out within 30 min. Magnetron target training and sample cleaning by ion beam were performed directly before deposition within 3 min.

X-ray diffraction researches of the material structure were carried out on an automated diffractometer DRON-3. The CuKa radiation (wavelength 0.154 nm) and the Bragg-Brentano focusing method θ–2θ (2θ – Bragg angle) were used in the shooting. The values of current and voltage on the X-ray tube were 20 mA and 40 kV. Shooting of specimens was carried out with horizontal slits of 4 mm on the tube and of 1 mm on the detector in continuous registration mode with a rate of 1°/min in a 2θ angle range from 25° to 60°. Calculation of the nanocrystallites size was performed by approximation method.

The effect of the bias potential on the structure of tantalum diboride films deposited with ICP source was studied. X-ray diffraction peak profile analysis (see Fig. 6) of films prepared at different bias potentials applied to the substrate shows that the textured films with growth texture (00.1) are formed when the substrate holder is grounded and the positive (+50 V) or negative (–50 V) bias potential is applied to the substrate. Thus, the degree of the films texture increases at the application of a negative bias potential (–50 V), that lead to an increase in crystallite size from 24 to 47 nm.

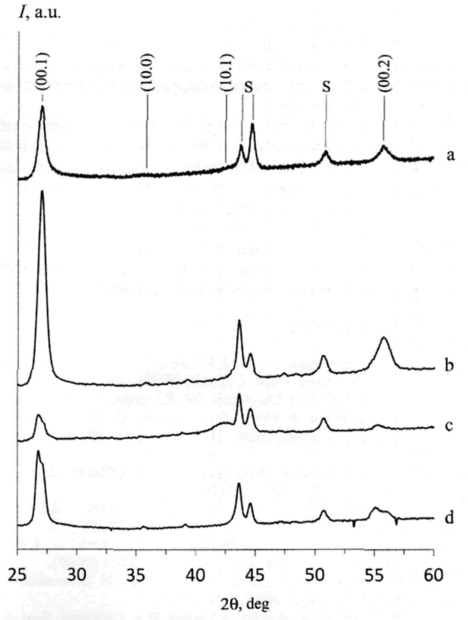


Fig. 6. The diffraction patterns of the specimens for various bias potentials applied to the substrate: specimen 1 (grounding, gap) (a); specimen 2 (–50 V) (b); specimen 3 (floating potential) (c); specimen 4 (+50 V) (d)

CONCLUSIONS

The effect of the bias potential and ion current from ICP source on the structure TaB_2 films deposited in cluster set-up system with unbalanced magnetron and ICP discharge were studied.

Nanocristalline TaB_2 films with various degree of texture by plane (00.1) were formed at the change in applied bias potential.

It was shown that the value of the bias potential applied to the substrate is crucial to the films structure formation, which determines respectively their physical and mechanical properties.

REFERENCES

1. J.Musil. Low-pressure magnetron sputtering // *Vacuum*. 1998, v. 50, № (3-4), p. 363-372.
2. R.D. Arnell, P.J. Kelly. Recent advances in magnetron sputtering // *Surf.Coat.Technol.* 1999, v. 112, p. 170-176.
3. I. Safi. Recent aspects concerning DC reactive magnetron sputtering of thin films: a review // *Surf.Coat. Technol.* 2000, v. 127, p. 203-218.
4. P.J. Kelly, R.D. Arnell. Magnetron sputtering: a review of recent developments and applications // *Vacuum*. 2000, v. 56, p. 159-172.
5. J. Musil, J. Vlček, P. Baroch. Magnetron discharges for thin films plasma processing, Chapter 3 // *Materials Surface Processing by Directed Energy Techniques*. Ed.: Y. Pauleau. Elsevier Science Publisher B.V., Oxford, UK. 2006, p. 67-106.
6. Musil. Flexible Hard Nanocomposite Coatings // *RSC Advances*, 2015, DOI: 10.1039/C5RA09586G.
7. P.H. Mayrhoffer, S. Mitterer, J.G Wen, J.I. Greene, I. Petrov. Nanoscale effects on ion conductance of layer-by-layer structures of gadolinia-doped ceria and zirconia // *Appl.Phys.Lett.*, 2005, v. 86, p. 131906.

8. A.A. Goncharov, V.A. Konovalov, S.N. Dub, V.A. Stupak, V.V. Pepukhov. Structure, composition, and physicomechanical characteristics of tantalum diboride films // *Phys. Met. Metal*. 2009, v. 107, p. 285-290.
9. A.A. Goncharov, G.K. Volkova, V.A. Konovalov, V.V. Pepukhov. Effect of underlayer on orientation and structure of thin films obtained by high-frequency magnetron sputtering of tantalum diboride target // *Met. Phys. Adv. Technol*. 2006, v. 28, p. 1621-1628.
10. S.V. Dudin, V.I. Farenik, A.N. Dahov, J. Walkowicz. Development of arc suppression technique for reactive magnetron sputtering // *Physical Surface Engineering.* 2005, v. 3, № 3-4, p. 211-215.
11. J. Walkowicz, A. Zykov, S. Dudin, S. Yakovin, R. Brudnias. ICP enhanced reactive magnetron sputtering system for syntesis of alumina coating // *Tribologia.* 2006, № 6, p. 163-174.
12. A.V. Zykov, S.D. Yakovin, S.V. Dudin. Synthesis of dielectric compounds by DC magnetron // *Physical Surface Engineering.* 2009, v. 7, № 3, p. 195-203.
13. S. Yakovin, S. Dudin, A. Zykov, V. Farenik, Integral cluster set-up for complex compound composites syntesis // *Problems of Atomic Science and Technology. Series:"Plasma Physics"*. 2011, № 1, p. 152-154.
14. I. Denysenko, S. Dudin, A. Zykov, N. Azarenkov and M. Yu. Ion flux uniformity in inductively coupled plasma sources // *Phys. Plasmas*. 2002, v. 9, № 11, p. 4767-4775.
15. Yu.P. Maishev. Ion sources and ion-beam equipment for deposition and etching of materials // *Vacuum technique and technology*. 1992, v. 2, № 3-4, p. 53-58.

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НАНЕСЕНИЕ ПОКРЫТИЙ TaB_2 МЕТОДОМ МАГНЕТРОННОГО РАСПЫЛЕНИЯ С ПЛАЗМЕННЫМ АССИСТИРОВАНИЕМ

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Представлены результаты нанесения покрытий TaB_2 в кластерной установке, включающей плоский магнетрон низкого давления и индукционный источник плазмы. Система позволяет контролировать независимо друг от друга как потоки осаждаемых атомов Та и В из распыляемой мишени TaB_2, так и потоки ионов аргона и электронов из индукционной плазмы. Низкое давление аргона в камере позволяет проводить процесс напыления в бесстолкновительном режиме, обеспечивая состав осаждённой плёнки, очень близкий к стехиометрическому составу распыляемой мишени. Показана взаимосвязь структуры покрытия TaB_2 с напряжением смещения на подложке (в диапазоне от -50 до +50 В) и с плотностью ионного тока.

НАНЕСЕННЯ ПОКРИТТІВ TaB_2 МЕТОДОМ МАГНЕТРОННОГО РОЗПИЛЮВАННЯ З ПЛАЗМОВИМ АСИСТУВАННЯМ

С. Яковін, О. Зиков, С. Дудін, В. Фаренік, О. Гончаров, І. Шелест, В. Кузнєцов

Представлено результати нанесення покриттів TaB_2 у кластерній установці з плоским магнетроном низького тиску та індукційним джерелом плазми. Система дозволяє контролювати незалежно один від одного як потоки осаджуваних атомів Та й В з мішені TaB_2, так і потоки іонів аргону і електронів з індукційної плазми. Низький тиск аргону в камері дозволяє проводити процес нанесення в режимі без зіткнень, забезпечуючи склад синтезованою плівки, дуже близький до стехіометричного складу мішені. Показано взаємозв'язок структури покриття TaB_2 з напругою зсуву на підкладці (в діапазоні від -50 до +50 В) і з щільністю іонного струму.