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I.V. SERDIUK^{1,*}, **R.V. KRYVOSHAPKA**^{1,**},
V.O. STOLBOVYI^{1,2,****}, and **N.O. SERDIUK**^{3,****}

¹ National Science Centre 'Kharkiv Institute of Physics and Technology'
of the N.A.S. of Ukraine,

1 Akademichna Str., UA-61108 Kharkiv, Ukraine

² Kharkiv National Automobile and Highway University,
25 Yaroslava Mudrogo Str., UA-61002 Kharkiv, Ukraine

³ Kharkiv National Medical University,
4 Nauky Ave., UA-61022 Kharkiv, Ukraine

* iraserduk@kipt.kharkov.ua, ** kryvoshapkarv@gmail.com,

*** stolbovoy@kipt.kharkov.ua, **** natashaserduyk23@gmail.com

TANTALUM COATINGS: APPLICATIONS, TECHNIQUES, AND PROPERTIES

The present study considers the methods of fabrication and investigation, properties and areas of application of tantalum coatings. These coatings attract attention due to their properties: high wear resistance and corrosion resistance, thermal stability, chemical inertness and biocompatibility. The presented structural, mechanical, tribological, electrical, corrosion, and biological properties make it possible to establish the relationship not only between the technological parameters of obtaining tantalum coatings (argon pressure in the vacuum chamber, substrate temperature, distance between the substrate and the target, *etc.*) and specific properties, but also to investigate the influence of the structural and phase state on various properties. Currently, tantalum coatings are widely used in microelectronics, medicine, and for corrosion protection. The wide range of information presented in this article enables the prediction of the properties and modelling of the processes of obtaining tantalum coatings with the required characteristics. This, in turn, will make it possible to expand the field of applications of tantalum coatings and deepen knowledge on the relationship between properties.

Keywords: tantalum, nitrides, coatings, magnetron sputtering, cathodic vacuum arc deposition, investigation techniques.

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1. Introduction

Tantalum is a refractory metal with excellent properties, such as a high melting point, toughness, resistance to chemical attack, etc. Its properties make it useful for many applications, from electronics to biological systems. Recent advances in technologies demand coating solutions with required properties (high wear and corrosion resistance, mechanical toughness, thermal stability, chemical inertness). Tantalum coatings have these properties and are widely used in the microelectronic industry, medicine and in chemical systems [1–54].

Bulk tantalum has a bcc crystallographic structure, known as α -phase, with the lattice constant of 3.306 Å. This crystallographic structure is tough and ductile, with a Knoop hardness of 300–400, and a relatively low electrical resistivity (13.5 $\mu\Omega$ cm) [14].

The behaviour of thin coatings is quite different from that of the bulk materials. The grain size, film texture, stress state, and defect density play a very important role in determining the mechanical and tribological properties of thin coatings. It is observed that the microhardness of thin coatings increases when the grain size decreases. A tetragonal structure, known as β -phase, with parameters $a = 10.194$ Å and $c = 5.313$ Å has been identified in tantalum coatings [14]. This metastable phase is hard with Knoop hardness values more than 900), brittle, and has an order of magnitude higher electrical resistivity than the α -phase. Metastable β -phase converts to the α -phase upon heating to 750° [15].

In the present article, we report on the structural, mechanical, tribological, electrical, corrosion, biological properties of tantalum coatings obtained different methods, the relationship between the technological deposition parameters (argon pressure in the vacuum chamber, substrate temperature, distance between the substrate and the target, etc.) and individual properties, the influence of the structural and phase state of tantalum coatings on various properties.

2. Methods, Substrates, and Investigation Techniques

2.1. Methods and Substrates

Our investigation showed that tantalum coatings were usually obtained by using such methods: magnetron sputtering [1–20, 36, 44–46], filtered cathodic vacuum arc deposition [21–25], vacuum plasma spraying [26, 27], plasma alloying technique [28], pulse metal vacuum arc source deposition [29], chemical vapour deposition [30].

Tantalum films were deposited onto a series of different substrates. They are: silicon substrates Si(100) [1, 2, 4, 8, 11, 14, 20, 25, 29, 36, 45], glass substrates [10, 15, 34, 44, 46], SiC substrates [30], SiO₂/Si substrates [2, 12, 14], ceramic substrates (3Y-TZP) [28], titanium alloy sub-

strates (Ti-6Al-4V, Ti-13Nb-13Zr alloys) [5, 17, 21–24, 26, 27], stainless steel substrates (AISI 316BL, SS304, AISI 317L) [3, 6, 8, 9, 13, 27, 31, 46], titanium substrates [3, 4, 18, 32], fused quartz substrates [16, 34], aluminium substrates [7, 36], Ta₂O₅ coated substrates [10], sapphire substrates [34], steel substrates (AISI 4340, A723 steel) [7, 14, 15], CoCrMo alloy substrates [19].

2.2. Investigation Techniques

2.2.1. Structural Characterization

Structural properties of the coatings (crystallographic orientations, phase composition, crystalline structures) were characterized using X-ray diffraction (XRD) with X-ray from a conventional CuK_α source [1–6, 8, 10, 11–19, 20–26, 28–30, 34, 36, 44–46].

Surface and cross-section characterizations of the coatings were carried out using scanning electron microscopy (SEM) [2–6, 8, 11–14, 18–20, 22, 23, 25–30, 32, 36, 44–46] and transmission electron microscopy (TEM) [1, 21, 23]. High-resolution micrographs of the sample surfaces and cross sections were produced using scanning electron microscopy. Samples were cleaved into smaller pieces for analysis, and cross-section images were obtained by viewing the fractured coating surfaces.

Coating thicknesses were determined from the cross-section images of SEM [4, 5, 18–20, 22, 23, 25–29], using a Tolansky multiple beam interferometer [10], from the cross-section transmission electron microscopy [21, 24], were measured using Rutherford back-scattering spectrometry (RBS) [12, 36], using surface profilometer [16, 44].

The chemical composition and element distributions were investigated by energy dispersive X-ray spectroscopy (EDX) analysis [4, 5, 26, 27, 30], X-ray photoelectron spectroscopy (XPS) [3–5, 13, 21, 24, 25], Auger electron spectroscopy (AES) [1, 11], Rutherford backscattering spectrometry (RBS) [11, 12, 36].

Surface topography and roughness of the coatings were examined using atomic force microscopy (AFM) [3–5, 9, 11, 17, 20, 21, 23, 25, 36, 46], a 3D laser scanning confocal microscope [22, 26], and white light scanning profiler [28].

2.2.2. Mechanical Testing

The mechanical properties (hardness H , Young's modulus E and the H/E ratio) were determined using nanoindentation measurements [3–5, 8, 12, 13, 16, 17, 19, 20, 22, 24, 26, 27, 34]. A pyramidal diamond indenter of the Berkovich type has been used with different curvature radii. The indentation depth was kept below 10 % of the coating thickness to eliminate substrate effects. Indentation tests at each load were repeated several ti-

mes and then averaged. Nanoindentation data have been analysed by the Oliver–Pharr method.

The coating residual stress s was measured using the surface profilometer [2, 5, 9, 11], a TEC X-ray stress analyser using Cr radiation [8], the traditional $\sin^2\psi$ analysis [17], and was calculated according to Stoney's formula [2]:

$$\sigma = \frac{E_{s_i} t_{s_i}^2}{6(1 - \nu_{s_i}) t_a} \left(\frac{1}{R_a} - \frac{1}{R_b} \right),$$

where E_{s_i} , ν_{s_i} , and t_{s_i} are the Young's modulus, Poisson's ratio, and thickness of the silicon substrate wafer, t_a is an average thickness, R_b and R_a are the wafer's curvature radius before and after the deposition of coatings.

The adhesion strength of the tantalum thin film layer (with a thickness of 360–390 nm) on the silicon substrate is evaluated by a semi-quantitative scotch tape test [2]. A 10 by 10 square grid cross-cut with an area of 1 mm² each has been made on the deposited tantalum thin film layer. Scotch tape is attached to the square grid and rubbed firmly to ensure good contact. After 90 s, the tape is detached from the substrate. Thin film delamination and peeling of Ta film from substrates are inspected according to the scale in ASTM standard D3359-09.

The bond strength of the thin tantalum coatings (with the thickness from 0.088 to 10 μm) was determined using a scratch test with a spherical diamond tip (indenter) of 4.6 and 200 μm radius [5, 14, 17, 19, 22, 23, 27, 28]. Normal load off indenter was increased linearly from 0 to 500 mN [5, 17, 24], from 0 to 30 N [14], from 0.1 to 50 N [17, 22, 23]. The scratch length was 5 mm.

The adhesion property of the thick tantalum coatings with a thickness of 100 μm was evaluated using two methods [8]. For the coating deposited on AISI 304 stainless steel, Rockwell-C adhesion tests were carried out at a 150-kg load using a 200 μm radius Rockwell-C indenter. For the coating deposited on A723 hard tool steel, the adhesion strength of the coating was evaluated by a Teer scratch tester using a Rockwell-C indent tip (tip radius 200 μm and conical angle 120°).

2.2.3. Tribological Characterization

Wear test was performed in the air or vacuum conditions to evaluate the tribological properties of the coatings. The wear resistance (the friction coefficient, wear rate, mass loss of samples) was determined using a dry ball-on-disk device at room temperature and relative humidity of $50 \pm 5\%$ conditions [5, 13]. During the wear test, balls with diameter from 4 and 5.5 mm were used as the friction partner. Counter-partners were GCr15 balls [5, 13]. The normal load was 2 N [5] and 10 N [13]. The average value of mass loss was used to analyse wear resistance.

2.2.4. Tribocorrosion Testing

Tribological tests of tantalum coatings were conducted using a ball-on-disk tribometer, where a stationary ball was pressed against a rotating disk [22, 28]. An alumina ball with a diameter of 4 mm [22] and a hardness of 16 GPa was chosen as the counter-face because of its high wear resistance.

The operating conditions of the tribological test were selected based on a simulated hip joint, defined in terms of contact pressure, speed, and lubrication. The test was conducted for 1 h at 37 ± 1 °C, 50% relative humidity and simulated body fluid (SBF) was used as a lubricant. The SBF was used as the test solution because its ionic composition is similar to human blood plasma. The SBF was prepared by dissolving an analytical grade mixture of NaCl, NaHCO₃, Na₂CO₃, KCl, K₂HPO₄×3H₂O, MgCl₂×6H₂O, CaCl₂, and Na₂SO₄ in distilled water. The SBF solution was adjusted to pH 7.4. The average wear volume of the disk was calculated by averaging the worn area measured at four positions at intervals of 90 degrees and multiplying by the length of the circumferential sliding path. The wear rates were calculated as the worn volume divided by the product of load and sliding distance (mm³/N m) based on Archard's equation [22].

The wear resistance in phosphate buffered saline solution was measured against Si₃N₄ balls with a diameter of 5.5 mm with a load of 5 N and sliding for 15 min [28].

2.2.5. Electrical Testing

The square resistance R_s of tantalum coatings at room temperature has been measured using four-point probe technique [2, 10–12, 14, 20, 25, 44–46].

The thin film sheet resistivity r was estimated by multiplying the square resistance with coating thickness t_c : $\rho = R_s t_c$.

To study the effect of high temperature on the stability of coatings and to determine the temperature coefficient of resistance (TCR), a thermal test was carried out. The temperature profile in the thermal cycling test was measured by varying the temperature from –65 to 165 °C [10].

The TCR values of coatings were calculated with the following equation [42]: $\text{TCR}(\text{ppm}/^\circ\text{C}) = (R_m - R_o) / R_o (1/\Delta T) \cdot 10^6$, where R_m and R_o were the resistivity of the coating at the measured temperature and room temperature (RT), respectively, and DT was the difference between the measured temperature and RT.

2.2.6. Electrochemical Testing

The corrosion resistance of tantalum coatings was determined using potentiodynamic and potentiostatic polarization tests [6, 7, 17, 18, 22, 23].

Potentiodynamic polarization measurement was carried out at different temperatures, such as room temperature [23], 25 °C [17], 37 °C [18], 70 °C [6, 7]. Different electrolytes were used: H₂SO₄ [6, 7], phosphate buffered saline (PBS) [17, 23], Ringer's physiological electrolyte [18]. For the execution of electrochemical tests, a standard three-electrode flat cell (working electrode + counter electrode (a Pt wire) + reference electrode (Ag/AgCl saturated in KCl)) was utilized.

In the potentiodynamic tests, the initial potential was -0.25 V [6, 7, 17, 18, 23] versus open circuit potential (OCP) and the final potential was 0.25 V [17, 23], 1 V [18], 1.3 V [6, 7] versus SCE. The scan rate was 0.166 mV/s [18], 0.3 mV/s [17, 23], 1 mV/s [6], 2 mV/s [17].

The measured current-voltage data were plotted as Tafel plots in the form of log versus potential. The corrosion potential (E_{corr}), corrosion current density (I_{corr}), and polarization resistance (R_p) were calculated from the Tafel plot.

For the purpose of simulating actual working conditions of proton exchange membrane fuel cell (PEMFC), potentiostatic tests were conducted. At the anode, the applied potential was -0.1 V versus SCE purged with H₂, at the cathode, the applied potential was 0.6 V versus SCE purged with air, and the temperature and electrolyte were 70 °C and 0.5 mol dm⁻³ H₂SO₄, respectively [6, 7].

To study the electrochemical and passive behaviour of tantalum coatings, electrochemical impedance spectroscopy (EIS) were performed [18, 21–24]. A conventional three-electrode electrochemical cell was used with platinum and Ag/AgCl as auxiliary and reference electrodes, respectively. The working electrode surface area of 0.8 cm² was exposed to the test electrolyte via a glass cell affixed to the surface by an O-ring and clamp. A simulated biological fluid (SBF) solution was used as the test solution because its ionic composition is similar to human blood plasma. The pH of SBF solution was 7.4 ± 0.5.

Impedance measurements were conducted in the frequency range of 0.01–10⁵ Hz with a data density of 10 points per decade at their respective open circuit potentials. The measurements were carried out by applying sinusoidal wave perturbations of 10 mV RMS in amplitude. Impedance measurements were taken at interval of 1, 6, 12 and 24 hours, 7, and 14 days.

2.2.7. Biological Investigation

The investigation of biological properties of tantalum coatings, such as hydrophobic characteristics, cytocompatibility, and biocompatibility were presented in several articles [17, 21, 23, 24, 26, 27, 29, 30, 32].

The surface wettability of the samples was obtained by measuring the contact angle with sessile drop method at room temperature, using a Rame–Hart Instrument contact angle goniometer. In order to assess the

compatibility and bonding behaviour of the modified surface in the human body that contains mainly water and protein, two liquids of distilled water and fetal bovine serum were implemented [17, 21, 23]. The surface free energy was calculated by using the Owens–Wendt method [23].

The *in-vitro* cytocompatibility of the tantalum coatings was investigated using rat bone mesenchymal stem cells. The rat bone mesenchymal stem cells (rBMSCs) were seeded on samples in 96-well plates with 200 μ l α -MEM (with 10% fetal bovine serum, 1% antimicrobial of penicillin and streptomycin) in each well and cultured for 1, 4, and 7 days. For cell morphology observation, the cultured samples were rinsed in phosphate buffered saline (PBS) aqueous solution and then kept in 4% glutaraldehyde (in PBS) at 4 °C for 2 h. The samples were then washed in PBS, dehydrated in graded ethanol solution (30, 50, 70, 80, 90, 95, and 100%), and observed via scanning electron microscope [24]. The cell proliferation was evaluated according to the alamarBlue™ assay (AbD Serotec Ltd., UK). The fluorescence intensity of the reduced alamarBlue™ was examined using a fluorescence microplate reader at an extinction wavelength of 560 nm and an emission wavelength of 590 nm. Triplicate samples were used to achieve accuracy and reproducibility [24].

Biological properties of the tantalum coatings were determined using initial *in vitro* biocompatibility tests [26, 29, 32], histopathological observation and histomorphometric analysis [27], biomechanical testing [27]. An initial assessment of the *in vitro* biocompatibility of the coatings was made by examining the attachment and spread of osteoblast-like osteosarcoma MG-63 cells [26, 32] and human umbilical vein endothelial cells (HUVEC) on the specimen surface [29]. The specimens were examined by scanning electron microscopy to investigate morphology and accumulation of cells. The viability/proliferation of living cells was evaluated using fluorescent measurements with microplate reader.

2.2.8. Diffusion Barrier Characterization

The diffusion barrier properties of tantalum and tantalum nitride coatings were investigated in Refs. [1, 11, 25].

2.2.9. Hydrothermal Ageing

The protective effect of tantalum coatings was investigated by successive hydrothermal ageing of the specimens in an autoclave in water vapour at 134 °C and 3 bar [28]. Chemical composition and the progress of the phase transformation were observed with the ageing step 0, 5, 10, 20, 40, 80, and 100 hours.

3. Results and Discussion

3.1. Structural Characterisation of Tantalum Coatings

The tantalum coatings have been deposited on different substrates using sputtering systems (direct current magnetron sputtering (DC MS) [2, 4, 12–14, 16, 18, 20], r.f. magnetron sputtering (RF MS) [3, 10, 11, 19], modulated pulse power magnetron sputtering (MPP MS) [8], high power impulse magnetron sputtering (HiPI MS) [20], magnetron sputtering (MS) [5, 6, 9, 17]), filtered cathode vacuum arc deposition (FCVAD) [21-25], and double glow plasma surface alloys technique (DGPSA) [28]. The technological deposition parameters of tantalum coatings are presented in Table 1, namely the target-to-substrate distance L , the substrate temperature T_s , the deposition time t , the deposition pressure P , the base pressure P_b , the sputtering power range W , and the substrate bias U_s .

The structural properties of tantalum coatings are presented in Table 2, namely the film phase, the coating texture, and the coating thickness l .

The effect of substrate temperature on the phase content of tantalum coatings is usually attributed to an increased surface mobility of Ta atoms. Deposition of Ta atoms on cold substrates is likely to result in the formation of disordered metastable structures (β -Ta) rather than the more ordered b.c.c. structure (α -Ta). Increasing the substrate temperature to 400 °C changes the crystalline phase of the growing coating.

The crystallographic orientation of the tantalum coatings is highly dependent on the substrate: β -Ta phase was obtained on glass substrates, α -Ta phase was formed on SS316LVM substrates and a nanocrystalline β -Ta phase was formed on Ti substrates [3].

The negative bias voltage has a pronounced effect on the phase structure of the tantalum coatings. At the sputtering power 210 W, the low direct voltage ($U_s = -50$ V) resulted in a pure β -Ta coating, but the high pulse power voltage ($U_s = -1000$ V) leads to high ion energy, promotes b.c.c. structure growth. It is consistent with that there exists a condition in the deposition process (ion energy and ion flux) where the α -Ta can be synthesized [5]. At the sputtering power 2.5–3 kW the crystalline phase changed from a pure β -Ta phase when the bias voltage was 0 V to a mixed α -Ta and β -Ta phases when the bias voltage was in the range of –30 to –40 V, and finally to pure α -Ta phase when the negative bias voltage was –50 V or greater [8].

The phase contents of the tantalum thin coatings for different sputtering pressures and sputtering power are presented in Tables 3 and 4, respectively. The results of the measurement were taken from [2]. α -Ta, β -Ta and a mix of these two phases were randomly grown with no generic trend towards the used deposition parameters (pressure and power). The sputtering pressure and power have a mutual combinational effect on argon plasma parameters in the chamber and thus indirectly affect the phase formation.

The mean grain sizes of the tantalum coatings are presented in Table 5.

Table 1. Technological deposition parameters of tantalum coatings

<i>L</i> , cm	<i>T_s</i> , °C	<i>t</i> , min	<i>P</i> , Pa	<i>P_b</i> , Pa	<i>W</i> , W	<i>U_s</i> , V	Method	Refs.
30	≈60	10–50	0.4–2	10 ⁻⁵	100–250	no	DC MS	[2]
6	30–40	30	6.5	5·10 ⁻⁴		—	RF MS	[3]
13	RT	Adapted	0.67	1.3·10 ⁻⁸	200	—	DC MS	[4]
	20–40	300	0.45	5·10 ⁻⁴	210	-50	MS	[5]
	25–260					-1000		
12		270	0.67	10 ⁻⁴	2500 3000	0–100 -50	MPP MS	[8]
	25–200		0.67	6.7·10 ⁻⁴	20–40		MS	[9]
4	300		0.2	1.3·10 ⁻⁶		2500	RF MS	[10]
5			0.22–0.3	3·10 ⁻⁶	1000		RF MS	[11]
12.5	90		0.3–2.2	2.7·10 ⁻⁶			DC MS	[12]
4	200	240	0.16	6·10 ⁻³	2000		DC MS	[13]
5	Room		0.1–3	10 ⁻⁵	150		DC MS	[14]
	300							
	400		1.3–9.1				MS	[15]
			0.5		100		DC MS	[16]
5			1.5	10 ⁻⁵			MS	[17]
10		60	0.5			-50	DC MS	[18]
4.5	Room	120	1.33		150	0	RF MS	[19]
	200					78		
	400					90		
	600		0.24	10 ⁻⁴	50 150	no	HiPI MS pulsed DC MS	[20]
		5	0.35–0.4	10 ⁻²		-100	FCVAD	[21]
		30						
	Nonheated	30	0.4	10 ⁻²		-100	FCVAD	[22]
	300							
		5	0.32	1.5·10 ⁻³		0	FCVAD	[23]
						-100		
	350	30	0.27	3·10 ⁻⁴		-100	FCVAD	[24]
						-200		
	RT					0	FCVAD	[25]
1.8	750	10	35				DGPSA	[28]
	800							
	850							
	900							
5		10–40	0.67	10 ⁻⁶	150	0–300	RF MS	[36]
6.5	RT	25–110	0.3–6	7·10 ⁻⁵	100		DC MS	[44]

Table 2. Structural properties of the tantalum coatings

Phase	Diffraction peaks	l , μm	Substrate	Comments	Refs.
α -Ta	(110) (200) (211)	0.65–0.8	Silicon wafers Si(100)	$P_{\text{Ar}} = 1.5$ Pa Sputtering power 250 W	[2]
β -Ta	(004) (303) (304)	0.36–0.39		$P_{\text{Ar}} = 1.2, 1.7$ Pa Sputtering power 100, 200 W	
mixed α -Ta– β -Ta		0.36–0.39		$P_{\text{Ar}} = 0.4, 0.7, 0.9, 2$ Pa Sputtering power 150 W	
α -Ta	(110)	0.3	SS316LVM	$P_{\text{Ar}} = 6.5$ Pa	[3]
β -Ta	(330) (720)	1	Glass		
	(330)	0.3	Ti		
α -Ta	(110) (200) (211)	0.4	15 nm f.c.c.- TaN seed layer	$P_{\text{Ar}} = 0.67$ Pa Sputtering power 200 W	[4]
β -Ta	(002) (004)	0.4	Ti Si(100) DLC		
α -Ta	(110) (211) (220)	3.5	Ti6Al4V Si wafers	$P_{\text{Ar}} = 0.45$ Pa $U_s = -1000$ V	[5]
β -Ta	(202) (005) (303)	5.1		$P_{\text{Ar}} = 0.45$ Pa $U_s = -50$ V	
α -Ta	(110)	3	SS316L		[6]
α -Ta	(110) (211) (220)	4.2–4.5	AISI 304 Si(100)	$P_{\text{Ar}} = 0.67$ Pa $U_s = -50, -70, -100$ V	[8]
mixed α -Ta– β -Ta				$P_{\text{Ar}} = 0.67$ Pa $U_s = -30, -40$ V	
β -Ta	(002) (513)			$P_{\text{Ar}} \leq 0.67$ Pa $U_s = 0$ V	
	(002) (513) (330)	$P_{\text{Ar}} = 0.67$ Pa Floating bias voltage			
α -Ta	(110) (220)	100	AISI 304 A723 steel	$P_{\text{Ar}} = 0.67$ Pa $U_s = -50$ V	[10]
α -Ta		0.1–0.4	Corning glass7059 Ta_2O_5 -coated substrates	$T_s > 300$ °C	
mixed α -Ta– β -Ta				$T_s = 150–300$ °C	
β -Ta				$T_s < 150$ °C	
β -Ta	(002) (202) (212) (413)	0.1	Si(100) wafers	$P_{\text{Ar}} = 0.22–0.3$ Pa	[11]

Continued of the Table 2.

Phase		Diffraction peaks	$l, \mu\text{m}$	Substrate	Comments	Refs.
β -Ta		(002)	0.5—0.65	Si(100) wafers	$P_{Ar} = 0.3\text{--}2.2 \text{ Pa}$ $T_s = 90 \text{ }^\circ\text{C}$	[12]
mixed	α -Ta	(110) (211)	50	AISI 304	$P_{Ar} = 0.16 \text{ Pa}$ $T_s = 200 \text{ }^\circ\text{C}$	[13]
	β -Ta	(002) (513)				
α -Ta		(110) (220)	0.02, 0.07, 0.3, 4, 5.8	AISI 4340 Si SiO ₂ /Si	$P_{Ar} = 0.1\text{--}3 \text{ Pa}$ $T_s = 400 \text{ }^\circ\text{C}$	[14]
		(110) (211) (321)	10.1 41			
mixed α -Ta- β -Ta		(222)	21.6 41		$P_{Ar} = 0.1\text{--}3 \text{ Pa}$ $T_s = 300, 350 \text{ }^\circ\text{C}$	
β -Ta		(002) (004) (006)	4		$P_{Ar} = 0.1\text{--}3 \text{ Pa}$ $T_s = \text{RT}$	
β -Ta		(002)	50 0.65	Steel Glass	$P_{Ar} = 1.3 \text{ Pa}$	[15]
		Random	0.34 0.47 0.51 120		$P_{Ar} = 2.6, 3.9, 5.2, 9.1 \text{ Pa}$	
β -Ta		(200) (400)	0.25 0.35 0.5	Si(111) oxidized	$P_{Ar} = 0.5 \text{ Pa}$	[16]
mixed	α -Ta	(110) (310)	1.6	Ti6Al4V	$P_{Ar} = 1.5 \text{ Pa}$	[17]
	β -Ta	(002) (202)				
α -Ta		(110)	0.77	Ti	$P_{Ar} = 0.5 \text{ Pa}$ $U_s = -50 \text{ V}$	[18]
α -Ta			0.8 1.5	CoCrMo alloy Si wafers	$P_{Ar} = 1.33 \text{ Pa}$ $T_s = 400, 600 \text{ }^\circ\text{C}$	[19]
mixed α -Ta- β -Ta					$P_{Ar} = 1.33 \text{ Pa}$ $T_s = \text{RT}, 200 \text{ }^\circ\text{C}$ $U_s = 0 \text{ V}$	
β -Ta					$P_{Ar} = 1.33 \text{ Pa}$ $U_s = -78, -90 \text{ V}$	
α -Ta		(110) (211)	0.3	Si wafers	$P_{Ar} = 0.24 \text{ Pa}$ Duty cycle (%) 88, 75, 50, 25, 10, 6.25, 5	[20]
mixed α -Ta- β -Ta						
β -Ta		(002) (330) (513)				

Phase		Diffraction peaks	$l, \mu\text{m}$	Substrate	Comments	Refs.
β -Ta			0.7 1	Ti6Al4V	$P_{Ar} = 0.35\text{--}0.4 \text{ Pa}$ $U_s = -100 \text{ V}$	[21]
amorphous Ta			1.1	Ti13Nb13Zr	$P_{Ar} = 0.4 \text{ Pa}$ $U_s = -100 \text{ V}$ Nonheated substrate	[22]
mixed	α -Ta	(110)	0.64		$P_{Ar} = 0.4 \text{ Pa}$ $U_s = -100 \text{ V}$ $T_s = 300 \text{ }^\circ\text{C}$	
	β -Ta	(002)				
mixed	α -Ta	(110) (220) (310)	0.088	Ti6Al4V	$P_{Ar} = 0.32 \text{ Pa}$ $U_s = -100 \text{ V}$	[23]
	β -Ta	(002) (513)				
β -Ta		(002) (513)	0.11		$P_{Ar} = 0.32 \text{ Pa}$ $U_s = 0 \text{ V}$	
mixed	α -Ta	(110) (200)	0.5	Ti6Al4V	$P_{Ar} = 0.27 \text{ Pa}$ $U_s = -100 \text{ V}$	[24]
	β -Ta	(002) (410) (202) (411) (513)	0.23		$P_{Ar} = 0.27 \text{ Pa}$ $U_s = -200 \text{ V}$	
β -Ta			0.075	Si wafers	$U_s = 0 \text{ V}$ $T_s = \text{RT}$	[25]
α -Ta		(110) (200) (211) (220)	0.59	3Y-TZP ceramic specimens	$P_{Ar} = 35 \text{ Pa}$ $T_s = 750, 800, 850, 900 \text{ }^\circ\text{C}$	[28]
α -Ta		(110)	0.145	Si(100)	$U_s = -100 \text{ V}$	[36]
			0.057– 0.456	Si(100)	$U_s = -150 \text{ V}$	
			0.076– 0.456	Al substrate	$U_s = -(100\text{--}300) \text{ V}$	
β -Ta		(002)	0.558– 0.744	Si(100)	$U_s = 0 \text{ V}$	
			0.29		$U_s = -100 \text{ V}$	
			0.076– 0.152		$U_s = -300 \text{ V}$	
mixed α -Ta– β -Ta			0.186– 0.372	Si(100)	$U_s = 0 \text{ V}$	
			0.186– 0.744	Al substrate		
			0.16–0.48	Si(100) Al	$U_s = -50 \text{ V}$	
			0.218	Si(100)	$U_s = -100 \text{ V}$	
			0.29	Al		
			0.87–0.107	Si(100)	$U_s = -200, -250 \text{ V}$	

End of the Table 2.

Phase	Diffraction peaks	l , μm	Substrate	Comments	Refs.
β -Ta	(002) (212) (413)	0.21–0.29, 0.94	Glass substrate	$P_{\text{Ar}} = 0.3\text{--}6$ Pa	[44]
	(410) (413)	0.93		$P_{\text{Ar}} = 0.3$ Pa	
mixed α -Ta– β -Ta		1.3		$P_{\text{Ar}} = 0.9$ Pa	
β -Ta	(330) (720)	0.152– 0.388	BK7 glass 304 steel	$T_s = \text{RT}$	[46]

Table 3. Phase contents, compressive residual stress and sheet resistivity of the tantalum coatings at different sputtering pressure [2]

Sputtering pressure, Pa	α -Ta, %	β -Ta, %	Stress, MPa	Resistivity, μWcm
0.4	16	84	≈ -615	≈ 173
0.7	99.8	0.2	≈ -1160	≈ 71.8
0.9	22.7	77.3	≈ -380	≈ 173
1.2	0	100	≈ -300	≈ 254
1.5	100	0	≈ -400	≈ 67.9
1.7	0	100	≈ -90	≈ 250
2.0	72	28	≈ -525	≈ 86.1

Table 4. Phase contents, compressive residual stress and sheet resistivity of the tantalum coatings at different sputtering power [2]

Sputtering power, W	α -Ta, %	β -Ta, %	Stress, MPa	Resistivity, μWcm
100	0	100	≈ -460	≈ 850
150	49	51	≈ -200	≈ 300
200	0	100	≈ -180	≈ 250
250	100	0	≈ -500	≈ 70

3.2. Mechanical Properties of Tantalum Coatings

Table 6 shows the mechanical properties of the tantalum coatings, namely the values of nanohardness H , elastic modulus E , the ratio of H/E , roughness R_a , and coating thickness l .

The coating that is deposited on the grounded substrate ($U_s = 0$ V) exhibited strong tetragonal (002) and (513) reflections, corresponding to a pure β -Ta phase, and showed hardness of 17.6 GPa. The coating deposited at a floating bias voltage also exhibited a β -Ta phase with intense (002) and (513) reflections, while a broad (330) peak appeared, indicating that a

polycrystalline structure had developed. The hardness of these coatings is 19.5 GPa. When the coatings contain a mixture of α and β phases, deposited at -30 V and -40 V bias voltages, the hardness of the coatings dropped rapidly as the volume fraction of the α -Ta phase increased. As the negative bias voltage was increased to above -50 V, the hardness of the tantalum coatings was in the range of 10–11 GPa, corresponding to a pure α -Ta phase formation [8].

The hardness H and modulus E behaviour of the tantalum coatings shown in Table 7 can be explained in terms of the variation in microstructure of the β -Ta films with different sputtering pressure of argon. The

Table 5. Grain size of the tantalum coatings

Phase	Grain size, nm	l , μm	Comments	Method	Refs.
α -Ta	4.3	3.5	$P_{\text{Ar}} = 0.45$ Pa; $U_s = -1000$ V	MS	[5]
β -Ta	16.3	5.1	$P_{\text{Ar}} = 0.45$ Pa; $U_s = -50$ V		
β	90	0.1	$P_{\text{Ar}} = 0.22\text{--}0.3$ Pa	RF MS	[11]
β -Ta	22.5–49.2	0.5–0.65	$P_{\text{Ar}} = 0.3\text{--}2.2$ Pa $T_s = 90$ °C	DC MS	[12]
α -Ta	5–40	0.01–10.1	$P_{\text{Ar}} = 0.1\text{--}3$ Pa $T_s = 400$ °C	DC MS	[14]
β -Ta	22	0.25 0.35 0.5	$P_{\text{Ar}} = 0.5$ Pa	DC MS	[16]
α -Ta	12–14	0.3			
mixed α -Ta- β -Ta	13–14				
β -Ta	14–20				
β -Ta	3	0.075	$U_s = 0$ V $T_s = \text{RT}$	FCVAD	[25]
α -Ta	66 ± 7.6	0.59	$P_{\text{Ar}} = 35$ Pa $T_s = 750, 800, 850, 900$ °C	DGPSA	[28]
α -Ta	18–19	0.114–0.456	$U_s = -100, -150$ V	RF MS	[36]
mixed α -Ta- β -Ta	24–32	0.186–0.369	$U_s = 0$ V		
	7–12	0.076–0.152	$U_s = -250, -300$ V		
β -Ta	86–128	0.554–0.738	$U_s = 0$ V		
	62	0.29	$U_s = -100$ V		
β -Ta	20–30	0.152–0.388	BK7 glass substrate 304 steel substrate	DC MS	[46]
	100–140				

Table 6. Mechanical properties of the tantalum coatings

Phase	H , GPa	E , GPa	H/E	l , μm	R_a , nm	Comments	Refs.
α -Ta	6.8 ± 0.3	138 ± 8.7	0.04	0.3	4–10	$P_{Ar} = 6.5$ Pa	[3]
α -Ta	7 ± 0.8			0.4		$P_{Ar} = 0.67$ Pa; Sputtering power 200 W	[4]
β -Ta	14 ± 0.6						
α -Ta	6.5 ± 1.1	123 ± 11	0.053	3.5	35.2	$P_{Ar} = 0.45$ Pa $U_s = -1000$ V	[5]
β -Ta	16.1 ± 4	199 ± 40	0.08	5.1	1.84	$P_{Ar} = 0.45$ Pa; $U_s = -50$ V	
α -Ta	11	239	0.046	4.2–4.5		$P_{Ar} = 0.67$ Pa $U_s = -50, -70,$ -100 V	[8]
$(\alpha + \beta)$ -Ta	12.5–17	220–230	0.054–0.077			$P_{Ar} = 0.67$ Pa $U_s = -30,$ -40 V	
β -Ta	17.6–19.5	210–215	0.082–0.093			$P_{Ar} = 0.67$ Pa $U_s = 0$ V	
β -Ta				0.1	0.54	$P_{Ar} = 0.22$ – 0.3 Pa	[11]
β -Ta	11.3–16.4	197.6–210.9	0.057–0.078	0.5–0.65		$P_{Ar} = 0.3$ – 2.2 Pa $T_s = 90$ °C	[12]
$(\alpha + \beta)$ -Ta	7.141	220	0.032	50		$P_{Ar} = 0.16$ Pa $T_s = 200$ °C	[13]
β -Ta	11–18	140–250	0.055–0.072	0.25–0.5		$P_{Ar} = 0.5$ Pa	[16]
$(\alpha + \beta)$ -Ta	14 ± 1.2	210 ± 15	0.067	1.6	5.2	$P_{Ar} = 1.5$ Pa	[17]
α -Ta	14.9 ± 0.4	211 ± 8	0.071	0.8–1.5		$P_{Ar} = 1.33$ Pa $T_s = 400,$ 600 °C	[19]
β -Ta	18.7 ± 0.7	229 ± 14	0.082	0.8–1.5		$P_{Ar} = 1.33$ Pa $U_s = -78, -90$ V	
α -Ta	12–13	178–181	0.067–0.072	0.3	0.38–1	$P_{Ar} = 0.24$ Pa Duty cycle (%) 88, 75, 50, 25, 10, 6.25, 5	[20]
$(\alpha + \beta)$ -Ta	14.5	180	0.08				
β -Ta	16–18	174–180	0.092–0.1				
amor- phous Ta	20	160	0.125	1.1		$P_{Ar} = 0.4$ Pa $U_s = -100$ V non-heated substrate	[22]
$(\alpha + \beta)$ -Ta	13	128	0.1	0.64		$P_{Ar} = 0.4$ Pa $U_s = -100$ V $T_s = 300$ °C	

nanohardness varies from 16.4 to 11.3 GPa with sputtering pressure and is correlated with grain size, consistent with Hall–Petch behaviour [12].

The hardness obtained by Saha *et al.* [16] shows that the 250 nm thick coatings exhibit maximum hardness (≈ 18 GPa) while the hardness of the 350 nm coatings is the lowest (≈ 11 GPa) with those of the 500 nm coatings lying in between (≈ 15 GPa). These results are correlated with the occurrence of (202) texture in these coatings. The 350 nm coatings exhibit the highest value for the ratio of $I(202)/I(200)$ and the lowest hardness. It can be concluded that the (200) growth texture produces a harder coating than the (202) texture. The coatings with high hardness values (16–18 GPa) exhibit only the (200) growth texture.

Stress development in the tantalum coatings is influenced mostly by the sputtering pressure [2]. As the pressure increases from 0.4 Pa to 2 Pa in Table 3, the compressive stress for major β -Ta coatings decreases, but at 0.7 Pa, 1.5 Pa, and 2 Pa the compressive stress suddenly intensifies. It is suspected that high α -Ta phase content promotes the build-up of compressive stress within the thin coating. Major α -Ta phase growth might contribute as the primary factor on compressive stress development.

As DC sputtering power increases from 100 to 200 W, the compressive residual stress decreases, as can be seen in Table 4, while a further increase of sputtering power to 250 W, a sudden increase of compressive stress is measured [2].

The thick tantalum coatings with a thickness of 100 μm have a residual stress of -2.1 GPa [8]. The low residual stress in the thick tantalum coating can possibly be attributed to the low ion energy bombardment from the plasma and the releasing of the residual stress by the annihilation of the defects as the deposition time and the coating surface temperature were increased.

A compressive in-plane residual stress is considered advantageous as it tends to improve the mechanical properties and hardness of the coatings, but an extraordinarily high compression can have a negative effect. It can reduce the adhesion of protective coatings to the substrate. Hee *et*

Table 7. Properties of β -Ta coatings as a function of sputtering pressure P_{Ar} [12]

P_{Ar} , Pa	H , GPa	E , GPa	H/E	Grain size, nm	Stress, MPa	Thickness, nm	r , μWcm
0.3	16.4 ± 0.1	209.3 ± 2.1	0.078	22.5 ± 3.5	-1358	485	172 ± 1.1
0.5	16.2 ± 0.1	209.9 ± 1.3	0.077	23.0 ± 3.7	-1045	505	163 ± 9
1.1	15.6 ± 0.2	210.9 ± 2.8	0.074	26.1 ± 3.3	-641	601	169 ± 7
1.6	14.6 ± 0.2	210.3 ± 2.1	0.070	27.3 ± 4.5	-216	611	172 ± 7
1.9	13.4 ± 0.2	206.7 ± 2.8	0.065	30.4 ± 3.4	179	616	163 ± 6
2.0	12.9 ± 0.4	207.7 ± 2.2	0.062	37.1 ± 6.8	544	644	167 ± 7
2.2	11.3 ± 0.5	197.6 ± 5.1	0.057	49.2 ± 9.7	1149	638	177 ± 7

al. [17] note that the residual stress of tantalum coating with titanium buffer underlayer is reduced by 38% compared to that of the pure tantalum coating, which is believed to favour the coating adhesion. Texture has a different effect in polycrystalline materials, as it has been proven by the anisotropic nature of mechanical properties [36]. The (002) texture of pure tantalum coatings induced a higher compressive stress.

3.3. Tribological Properties of the Tantalum Coatings

Adhesion of the coating is one of the key factors in the service life of the coating products [5, 38, 39]. The adhesion property of tantalum coatings was evaluated by the scratch test. With the increase in load, different failure events are occurring along the length of the scratch. These events include fracture, delamination, spallation, and even detachment from the substrate. To evaluate the coating adhesion, several critical loads (L_{ci}) are determined. A minimum critical load (L_{c1}) makes the initial coating fracture, and the second critical load (L_{c2}) makes the continuous perforation and detachment of the coating from the substrate. Critical load values of tantalum coatings deposited on different substrates are presented in Table 8. The critical load is mainly determined by the change in friction, and the failure mechanism of each scratch was observed by scanning electron microscope [17].

The bad adhesion between tantalum coating and substrate may be related to the growth process of coating under the condition of high energy ion bombardment, which induces the substrate deformation and weakens the coating-substrate adhesion at high temperature, and the presence of the impurity (argon, oxygen), which can weaken the bond between atoms, is bad to adhesive [5].

The higher adhesion strength between the tantalum coating with titanium underlayer and the substrate, compared to the tantalum coating, was indicated by the higher critical load of 7 N. The second critical load was observed at 23 N with the evidence of coating detachment. This shows that the tantalum coating with a titanium underlayer can withstand a higher load with strong bonding between the coating and the substrate [17].

Lee *et al.* [42] reported that good cohesion and adhesion were observed in a-phase tantalum, and cracks and disbanding occurred more frequently in b-phase tantalum.

The tantalum coatings deposited on CoCrMo alloy substrate at room temperature, without bias voltage, consisting of a mixture of a and b phase, show poor adhesion, similar to the coatings deposited at 400 °C. In this case, the brittleness of b phase tantalum and its difference in hardness and Young's modulus with a phase tantalum are probably the main reasons causing the coating delamination. On the contrary, the single b-phase tantalum coating with a thickness of 0.8 μm deposited on CoCrMo substrate at a negative bias voltage of 78 V adhered well to the substrate [19].

Wear properties of tantalum coatings deposited on different substrates at various conditions are presented in Table 9. The evolution of friction coefficient of tantalum coatings contains several stages. First stage is running-in stage, when the friction coefficient values drastically increase. The friction coefficient is engendered by initial contact between counterpart and coating surface, so that an increase of friction force generates resulting from surface micro-asperities on the coating. Therefore, the quicker the termination of this stage is, the smoother the coating surface is. Second stage is a dramatic decrease of the friction coefficient. As the dry sliding continues, large products of friction test are accumulated and then entrapped in the coating by the friction force. This stage will release the friction force for a very short time, thus resulting in a transitory decrease in the friction coefficient value [40, 41]. The third stage is a comparatively stable sliding regime. This stage emphasizes that the quantity and dimensions of friction products achieve a dynamic equilibrium,

Table 8. Adhesion properties of the tantalum coatings

Phase	Critical load, N		Tip radius, μm	Load range, N	Substrate	Thickness, μm	Refs.
	L_{e1}	L_{e2}					
α -Ta	0.2		4.6	0–0.5	Ti6Al4V	3.5	[5]
β -Ta	0.32					5.1	
α -Ta	29		200	0–30	AISI 4340	10	[14]
β -Ta	24						
$(\alpha + \beta)$ -Ta	2.5	18.6	200	0–50	Ti6Al4V	1.6	[17]
amorphous Ta	3.5	17	200	0.1–50	Ti13Nb13Zr	1.1	[22]
$(\alpha + \beta)$ -Ta	1.5	7.5				0.64	
β -Ta	5		200	0.1–50	Ti6Al4V	0.088	[23]
$(\alpha + \beta)$ -Ta	10					44	
α -Ta (S1)	24			0–100	3Y-TZP	0.59	[28]
α -Ta (S2)	90.1						
α -Ta (S3)	74.8						
α -Ta (S4)	65.4						

Table 9. Wear properties of the tantalum coatings

Phase	Friction coefficient	Volume loss, $\text{mm}^3/(\text{Nm})10^{-4}$	Ball	Load, N	Substrate	Thickness, μm	Refs.
α -Ta	0.56		GCr15	2	Ti6Al4V	3.5	[5]
β -Ta	0.42					5.1	
$(\alpha + \beta)$ -Ta	0.58	18.9	Cr15	10	AISI 304	50	[13]

i.e. quantity of products ploughed in and expelled from the contact interface simultaneously is balanced [13].

The tribocorrosion properties of the tantalum coating are presented in Table 10. The friction properties were investigated in simulated body fluid [22] and phosphate-buffered saline solution [28]. As the samples, Ti13Nb13Zr alloy substrates and commercially available 3Y-TZP ceramic specimens were used.

Table 10. Tribocorrosion properties of the tantalum coatings

Phase	Friction coefficient	Wear rate, mm ³ /(Nm)	Ball	Load, N	Substrate	Thickness, μm	Refs.
amorphous Ta	0.35	6.8 · 10 ⁻⁷	Al	5	Ti13Nb13Zr	1.1	[22]
(α + β)-Ta	0.35	6.4 · 10 ⁻⁷				0.64	
α-Ta (S1)	0.35	4.92 · 10 ⁻⁵	Si ₃ N ₄	5	3Y-TZP	0.59	[28]
α-Ta (S2)	0.33	3.58 · 10 ⁻⁵					
α-Ta (S3)	0.4	6.5 · 10 ⁻⁵					
α-Ta (S4)	0.54	6.88 · 10 ⁻⁵					

Table 11. Electrical properties of the tantalum coatings

Phase	Resistivity, μWcm	Substrate	Thickness, nm	Refs.
α-Ta	67.9	Silicon wafers Si(100)	650–800	[2]
β-Ta	254			
(α + β)-Ta	71.8–173			
α-Ta	≈25	Glass	100–400	[10]
β-Ta	≈165	Ta ₂ O ₅		
β-Ta	≈169	Si(100)	100	[11]
β-Ta	≈163–177	Si(100)	500–650	[12]
α-Ta	≈13–20	AISI 4340	4000	[14]
β-Ta	≈150–160	Si(100)		
α-Ta	≈24–50	Si(100)	300	[20]
β-Ta	≈180–220			
β-Ta	≈166–317	Glass	210–930	[44]
(α + β)-Ta	≈16100–21900		280–940	
	≈145		1300	
β-Ta	≈225–250	Si(100)	100	[45]
β-Ta	≈600–1500	BK7 glass	152–388	[46]

The friction coefficient of the tantalum-coated Ti₁₃Nb₁₃Zr substrate remained around 0.35 in a relatively steady state, and it is approximately 27% lower compared to the bare substrate. The difference in the friction coefficient is probably due to the increased hardness to elastic modulus ratio H/E for the tantalum coating (≈ 0.1), as compared to H/E ratio of 0.04 for the bare Ti alloy substrate. The friction coefficient of the tantalum coatings on the non-heated and heated substrates shows no discernible difference. This result suggests that both amorphous and crystalline phases of the tantalum coatings take an effective role in protecting the surface from wear [22].

The wear rate of the tantalum-coated 3Y-TZP ceramic substrate dropped with the rise of the deposition temperature from 750 °C to 800 °C. However, the wear rate value increased with further rising temperature (900 °C). A possible reason is that the original pits on the substrate surface could be compensated gradually by the tantalum coating, leading to the dropped wear rate at 800 °C. Nevertheless, the higher deposition temperature would promote the surface roughening of the tantalum coating, which deteriorates the friction property [28].

3.4. Electrical Properties of the Tantalum Coatings

Electrical resistivity of the tantalum coatings deposited at different technological conditions on various substrates is presented in Table 11.

Sheet resistivity values of the tantalum coatings at different sputtering pressures are presented in Table 3. Pure 100% β -Ta phase coatings have the highest average resistivity of $\approx 254 \mu\Omega\text{cm}$, followed by the mixed-phase tantalum coatings with major percentage of β -Ta phase at $\approx 173 \mu\Omega\text{cm}$. Tantalum coatings with high percentage of α -Ta phase content give a smaller resistivity range of 71.8–86.1 $\mu\Omega\text{cm}$. The smallest sheet resistivity of 67.9 $\mu\Omega\text{cm}$ is measured from 100% α -Ta phase coatings, which is almost four times smaller than 100% β -Ta coatings. In general, tantalum coatings which possess high fraction of α -Ta phase with high intensity of (110) direction orientation yield small sheet resistivity [2].

Sheet resistivity values of the tantalum coatings at different sputtering powers are presented in Table 4. The smallest resistivity for 250 W is due to the high crystal growth quality of α -Ta phase with high intensity of (110) direction orientation. However, higher sheet resistivity of tantalum coatings for 200 W was obtained compared to 250 W, as a result of a major 100% β -Ta phase. High resistivity was also measured for 150 W albeit 49% α -Ta phase exist within the coating. This may be contributed by either high crystal degradation or the absence of strong (110) direction orientation. The largest sheet resistivity was measured from tantalum coatings deposited at 100 W, almost 15 times higher than 250 W. Very low crystal quality makes the tantalum coating to behave more like a thin film semi-

conductor layer than metal [2, 43]. High density of crystalline defects increases coating resistivity due to the increase in electron scattering, impeding electron conduction and current flow [44].

E.A.I. Ellis *et al.* observed essentially no change in resistivity (from $163 \pm 9 \mu\Omega\text{cm}$ to $177 \pm 7 \mu\Omega\text{cm}$, as can be seen in Table 7) over the entire deposition pressure range from 0.3 Pa to 2.2 Pa, despite significant differences in coating microstructure [12]. The average resistivity value of the tantalum coating was $169 \pm 5 \mu\Omega\text{cm}$ and did not vary systematically with deposition pressure.

Grosser *et al.* observed a strong increase in coating resistivity in the deposition pressure range up to 4 Pa [44]. By changing the deposition pressure, the resistivity of tantalum coatings can be tuned within two orders of magnitude, as with increasing pressure the coating microstructure changes from dense to open porous and the probability to incorporate gaseous impurities is substantially enhanced.

The temperature coefficient of resistance (TCR) of the α -phase tantalum coatings is $+1500 \text{ ppm}/^\circ\text{C}$ and the TCR of the β -phase tantalum coatings is $-160 \text{ ppm}/^\circ\text{C}$ [10].

3.5. Electrochemical Properties of the Tantalum Coatings

The potentiodynamic polarization testing results of the tantalum coating on different substrates are presented in Table 12. Electrochemical parameters extracted from potentiodynamic testing consist of corrosion current density i_{corr} , the corrosion potential E_{corr} , and polarization resistance R_p . The samples were immersed in $0.5 \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$ at 70°C [6], in phosphate buffered saline (PBS) aqueous solution at 25°C during 1 hour and 14 days [17], in Ringer's physiological solution (RPS) at 37° [18], in simulated body fluid (SBF) at 37°C [22], and in PBS at room temperature during 1 hour [23].

Potentiodynamic polarization curve of the tantalum coatings on different substrates can be divided into four regions: cathode region, anode region, passive region and transpassive region [6].

Yu *et al.* noticed that the corrosion current density of an uncoated SS316L substrate is $44.61 \mu\text{A}\cdot\text{cm}^{-2}$ versus $9.25 \mu\text{A}\cdot\text{cm}^{-2}$ for a tantalum-coated SS316L substrate in $0.5 \text{ mol}\cdot\text{dm}^{-3} \text{ H}_2\text{SO}_4$. A decrease of corrosion current density of coated substrate by 5 times shows a lower corrosion rate for tantalum-coated SS316L. The polarization resistance of uncoated SS316L is $403 \text{ W}\cdot\text{cm}^2$ versus $1002 \text{ W}\cdot\text{cm}^2$ for tantalum-coated SS316L in $0.5 \text{ mol}\cdot\text{dm}^{-3} \text{ H}_2\text{SO}_4$, an increase of about twice, showing better anticorrosion property of the substrate with the tantalum coating [6]. An uncoated SS316L steel substrate is corroded mainly on the grain boundary; the reason is that there are chromium carbides (Cr_{23}C_6) precipitated at the grain boundaries. In this way, the tantalum coating can play an anticorrosion

role on the bipolar plates used in the proton exchange membrane fuel cells (PEMFC).

At potentiostatic testing in simulative anode working conditions, the current densities for both uncoated SS316L substrate and tantalum-coated SS316L substrate are positive. The current density of uncoated SS316L is about $9 \cdot 10^{-3} \text{ A} \cdot \text{cm}^{-2}$, the current density of tantalum-coated SS316L is stabilized at about $2 \cdot 10^{-4} \text{ A} \cdot \text{cm}^{-2}$. The current density reduction by 45 times shows that the tantalum coating has well anticorrosion properties. It can be used as protective coating on SS316L substrate, which is used as an anode in PEMFC [6].

At potentiostatic testing in simulative cathode working conditions, both current densities of uncoated and tantalum-coated SS316L substrates are positive. The current density of uncoated SS316L is stabilized at about $8.9 \cdot 10^{-4} \text{ A} \cdot \text{cm}^{-2}$, the current density of tantalum-coated SS316L is about $6.5 \cdot 10^{-8} \text{ A} \cdot \text{cm}^{-2}$. A dramatic reduction of current density shows that a tantalum-coated SS316L steel substrate can be well used as a cathode in PEMFC [6].

The tantalum coatings with $\alpha + \beta$ dual-phase structure have improved corrosion properties compared to the bare Ti6Al4V substrate in phosphate buffered saline (PBS) aqueous solution. As noted in Ref. [17], the polarization testing results show that anodic oxide formed on the surface of the tantalum coating behaves as an effective dielectric layer. The tantalum-coated surface reduces the anodic dissolution at the active regions compared to bare Ti in a chloride solution [47]. The tantalum coating with b-

Table 12. Electrochemical parameters extracted from potentiodynamic testing of the tantalum coatings on different substrates

Phase	i_{corr} , $\text{A} \cdot \text{cm}^{-2}$	E_{corr} , mV	R_p , $\text{W} \cdot \text{cm}^2$	Substrate	T , $^{\circ}\text{C}$	Electro- lyte	Scanrate, mV/s	Thick- ness, μm	Refs.
α -Ta	$9.25 \cdot 10^{-6}$	-289.3	1002	SS316L	70	H_2SO_4	1	3	[6]
$(\alpha + \beta)$ -Ta	$3.41 \cdot 10^{-9}$	-172	$8.86 \cdot 10^6$	Ti6Al4V	25 ± 1	PBS 1 hour	2	1.6	[17]
	$2.85 \cdot 10^{-9}$	-89	$1.5 \cdot 10^7$				0.3		
$(\alpha + \beta)$ -Ta	$2.6 \cdot 10^{-9}$	-133	$1.25 \cdot 10^7$	Ti6Al4V	25 ± 1	PBS 14 days	2	1.6	
	$4.39 \cdot 10^{-9}$	-87	$9.26 \cdot 10^6$				0.3		
α -Ta				Ti	37	RPS	0.166	0.77	[18]
amor- phous Ta	$9.87 \cdot 10^{-9}$	-160	$9.33 \cdot 10^6$	Ti13b13Zr	37	SBF		1.1	[22]
$(\alpha + \beta)$ -Ta	$1.84 \cdot 10^{-9}$	-180	$2.18 \cdot 10^7$				0.64		
$(\alpha + \beta)$ -Ta	$9 \cdot 10^{-9}$	-45	$4.57 \cdot 10^6$	Ti6Al4V	RT	PBS	0.3	0.088	[23]
β -Ta	$42 \cdot 10^{-9}$	-168	$0.88 \cdot 10^6$					0.11	

phase structure stabilized the surface and reduced the dissolution of titanium [48]. Hee *et al.* [17] reported that the negligible capacitive behaviour of the tantalum coatings in the polarization testing suggests a relatively oxide-free and stable surface with minimal tendency for oxidation compared to that of the uncoated Ti6Al4V substrate. The polarization resistance values R_p of the tantalum-coated Ti6Al4V are higher compared with the bare Ti6Al4V, indicating a reduced dissolution of the metal in phosphate-buffered saline aqueous solution.

The effects of immersion time of the tantalum coatings in Ringer’s physiological solution at 37 °C on passive film resistance, passive film capacitance, and passive film thickness are presented in Table 13. Nanostructured tantalum coating with a-phase structure is almost free of severe coating defects. The passive film formed on the surface of nanostructured tantalum coating is more resistant and thicker than that formed on the surface of annealed tantalum. Tantalum coating with a-phase structure and (110) preferred crystallographic orientation showed a superior

Table 13. Passive film resistance and passive film capacitance of the tantalum coatings in Ringer’s physiological solution at 37 °C [18]

Phase coating	Passive film resistance, MW·cm ²	Passive film capacitance, μF·cm ⁻²	Passive film thickness, nm	Electrolyte	T, °C	Immersion time, hours	Substrate
α-Ta	1.344	44.52	1.5	Ringer’s physiological solution	37	1	Ti
	3.712	27.51	2.4			6	
	13.11	17.18	3.6			12	
	27.04	12.34	5.3			24	

Table 14. Electrical parameters calculated by fitting an equivalent electrical circuit on the electrochemical impedance spectroscopy data. Here, exponent n indicates a variation from ideal capacitor/resistor: $n = 1$ (0) for the pure capacitor (resistor)

Phase	Q_{dl}^* , μW ⁻¹ ·s ^{n} ·cm ⁻²	n_2	R_{ct}^* , kW·cm ²	Substrate	T, °C	Electrolyte	Immersion time, h	Refs.
β-Ta	2.28	0.84	366	Ti6Al4V	37	SBF	1	[21]
amorphous Ta	12.6	0.93	12380	Ti13Nb13Zr	37	SBF	336	[22]
(α + β)-Ta	9.81	0.94	30620					
(α + β)-Ta	11.2	0.92	2520	Ti6Al4V	RT	PBS	1	[23]
β-Ta	35.2	0.89	29.7					
(α+β)-Ta	10.7	0.95	4644	Ti6Al4V	37	SBF	168	[24]
	21.2	0.69	2396					

passive behaviour and high protection of titanium substrate in Ringer's physiological solution at 37 °C compared to pure tantalum [18].

Electrical parameters calculated by fitting an equivalent electrical circuit on the electrochemical impedance spectroscopy data are presented in Table 14. An equivalent electrical circuit consists of the resistance of the solution between the working and reference electrodes, the constant phase element (CPE) and resistance associated with the oxide film on the surface, the constant phase element Q_{dt} and resistance R_{ct} associated with the electrical double layer and charge transfer resistance. The constant phase element is indicative of the capacitive contribution and its deviation from ideal dielectric behaviour due to surface heterogeneities such as roughness, inhomogeneous surface, and absorption of ions or possible variations in the physical properties of the covering films [21–24, 49]. The exponent n indicates a variation from an ideal capacitor/resistor, where $n = 1$ for the pure capacitor, and $n = 0$ for the pure resistor.

The tantalum coating exhibits a somewhat protective effect because the pores are sealed by the precipitation of calcium phosphate in the electrolyte — simulated biological fluid (SBF) solution. A hydrated oxide film (Ta–OH group) was formed by a slow hydration of the tantalum oxide layer in the SBF solution [21]. This hydrated oxide film reacts with calcium ions and phosphate ions and builds up a layer of calcium phosphate [50, 51].

Tantalum coating on Ti13Nb13Zr substrate exhibits high values of the modulus of impedance and phase angle maximum of $\approx 84^\circ$, implying that the passive film formed on the tantalum coating is more protective than the uncoated Ti13Nb13Zr substrate. The tantalum coatings increase the charge transfer resistance R_{ct} by one order of magnitude higher than the value of an uncoated Ti13Nb13Zr substrate [22].

3.6. Biological Properties of the Tantalum Coatings

Surface wettability and surface biocompatibility are important parameters, which influence cell growth. Wettability and surface energy are useful to determine the cell adhesion and proliferation of implant materials [52]. Table 15 summarizes the values of contact angle for the uncoated and tantalum-coated substrates in contact with water and fetal bovine serum.

The contact angle values of the tantalum coatings in contact with the tested liquids are higher than the values of the uncoated Ti6Al4V substrate. The increased contact angle of the tantalum coatings indicates that the coated surfaces became more hydrophobic. A hydrophobic surface is the result of low surface energy, which provides less attraction to water molecules, thereby improving protein absorption [17, 53, 54].

Hee *et al.* [24] investigated cell morphologies of the bare Ti6Al4V substrate and tantalum-coated substrates after 1 day, 4 days and 7 days of culturing of the rat bone mesenchymal stem cells (rBMSCs). There were no

Table 15. The contact angle of the uncoated Ti6Al4V substrate and the tantalum coatings

Phase/substrate	Contact angle, °			Thickness, μm	Refs.
	Distilled water	Fetal bovine serum	Glycerol		
(α + β)-Ta	93	86	—	1.6	[17]
β-Ta	89.1 ± 2	73.7 ± 1	—	0.7, 1	[21]
(α + β)-Ta	102 ± 2	91 ± 3	72 ± 4	0.088	[23]
β-Ta	99 ± 2	79 ± 4	64 ± 1	0.11	
Ti6Al4V substrate	70.3 ± 2	77.2 ± 1	46 ± 2	—	
α-Ta (S1)	60 ± 1.5	—	—	0.59	[28]
α-Ta (S2)	28 ± 0.7				
α-Ta (S3)	43 ± 0.5				
α-Ta (S4)	46 ± 1.1				
3Y-TZP substrate	41 ± 1.8	—	—	—	

statistically significant differences in the proliferation of cells between the samples on the first day, but this changed dramatically after four days of culturing. The cells grown on the tantalum-coated substrates were slightly higher than the value of the bare Ti6Al4V substrate on the fourth day. The number of cells started to decrease when the incubation time was extended to 7 days. The average value of cells on tantalum-coated substrates was slightly less than on the bare substrate, because cells reached a certain stage of maturity during 7 days.

The quantitative alkaline phosphatase activity (ALP) of rBMSCs on the uncoated Ti6Al4V substrate and the tantalum coatings had the same value at 1 μM/μg after 7 days of incubation. The cell viability of all samples increased after culturing for 14 days. The formation of calcium phosphate promoted cell adhesion on the investigated surfaces [24]. The spreading of rBMSCs on tantalum coatings and the increased number of cells on the fourth day of culturing denotes good cytocompatibility of tantalum coatings.

Kuo *et al.* [26] obtained results from initial in vitro biocompatibility tests, showing that osteoblast-like osteosarcoma MG-63 cells had a significantly better attachment and spreading on the tantalum coatings than on the uncoated Ti6Al4V substrate. In MG-63 cell culture tests, the cells cultured on a tantalum coating began to spread on the surface after 3 h.

Leng *et al.* [29] noted that the tantalum coatings fulfil the requirements necessary for the application as the coatings for a blood-contacting device (stent). They are helpful for seeding endothelial cells.

4. Applications

Over the past decades, tantalum coatings have been widely used due to their excellent properties, such as high melting temperature, excellent thermal stability, high wear and corrosion resistance, and chemical inertness.

Tantalum coatings have been extensively used as elements for micro-electronic industry, such as interconnects, thin film resistors, electrodes, diffusion barriers for metallization Cu and Ag, microelectronic devices, elements for superconducting nanowire single-photon detectors, thin film heater elements, elements in a quantum metal field effect transistor, a seed layer and a capping layer of giant magneto-resistance sensor in data storage, electron-selective contact for crystalline silicon solar cells.

Tantalum coatings have potential as coatings for tribological resistance applications, *i.e.*, hard coatings for cutting tools, for austenitic steel substrates, for Ti6Al4V titanium alloy substrates and as coatings for corrosion protection of steel and ceramic materials subjected to mechanical wear in harsh chemical environments.

Besides that, tantalum coatings have been used for microlevel mechanical systems as strain gauges in micromachined sensors for harsh environmental applications, as candidates to improve different stainless steel bipolar plates' performance and durability for polymer electrolyte membrane fuel cells.

Due to good biocompatibility and chemical stability of tantalum, tantalum coatings attract much attention for biomedical applications, for implants, for bone filling materials, for fabrication of commercial artificial heart valves, for improvement biocompatibility and corrosion protection of biomedical alloys (Mg–Y–Re, Ti6Al4V, Ti6Al7Nb, Ti13Nb13Zr) and titanium, for achievement a specific biointerface that can reduce patient's healing time and prevent aseptic loosening.

5. Conclusions

A summary of the properties of tantalum coatings deposited on different substrates reveals a number of structural, mechanical, tribological, electrical, corrosion and biological features.

The investigation of the relationship between the technological deposition parameters (argon pressure in the vacuum chamber, substrate temperature, distance between the substrate and the target, etc.) and individual properties, and the influence of the structural and phase state on various properties of tantalum coatings makes it possible to predict the properties and model the processes of obtaining tantalum coatings with the required properties.

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I.V. Сердюк¹, Р.В. Кривошапка¹, В.О. Столбовий^{1,2}, Н.О. Сердюк³

¹ Національний науковий центр

«Харківський фізико-технічний інститут» НАН України,
вул. Академічна, 1, 61108 Харків, Україна

² Харківський національний автомобільно-дорожній університет,
вул. Ярослава Мудрого, 25, 61002 Харків, Україна

³ Харківський національний медичний університет,
проспект Науки, 4, 61022 Харків, Україна

ТАНТАЛОВІ ПОКРИТТЯ: ЗАСТОСУВАННЯ, МЕТОДИ ТА ВЛАСТИВОСТІ

У роботі розглянуто методи одержання і методики дослідження, властивості та сфери застосування танталових покриттів. Ці покриття привертають увагу завдяки своїм властивостям: високій зносостійкості, корозійній стійкості, термічній стабільності, хімічній інертності та біосумісності. Наведені структурні, механічні, трибологічні, електричні, корозійні та біологічні властивості дають змогу встановити взаємозв'язок не тільки між технологічними параметрами одержання танталових покриттів (тиском аргону у вакуумній камері, температурою підкладки, віддаллю між підкладкою та мішенню тощо) та окремими їхніми характеристиками, а й дослідити вплив структурно-фазового стану на їх різні властивості. На даний час танталові покриття широко застосовуються в мікроелектроніці, медицині та для захисту від корозії. Широкий спектр інформації, наведеної в цій статті, дає змогу прогнозувати властивості та моделювати процеси одержання танталових покриттів з необхідними характеристиками. А це, у свою чергу, уможливить розширення сфери застосування танталових покриттів і поглиблення знання про взаємозв'язок між властивостями.

Ключові слова: тантал, нітриди, покриття, магнетронне розпорошення, вакуумно-дугове осадження, дослідницькі методи.