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COMPLEX COATINGS OBTAINED BY A TWO SOURCE BEAM EVAPORATION SYSTEM

Key words: electron beam evaporation system, multi-component coatings, gradient coatings, multi-layer coatings.

Abstract: The article presents the results of work on the verification of the technological capabilities of the EB-Dual BEAM device in the field of complex coatings, including gradient coating Ti↑-Zr↓, multi-pattern coating Ti-Zr, and multi-layer coating Ti/Ti-Zr/Zr. The obtained coatings were subjected to an analysis of changes in the chemical composition by the EDS method as a function of distance from the surface and observations of brittle fracture using scanning microscopy in order to analysis their internal structure. It has been shown that the use of two independent electron guns provides the ability to differentiate the chemical composition of two-component coatings independently from the character of these changes, i.e. linear changes, and step changes.

Powłoki złożone wytwarzane metodą EB PVD z wykorzystaniem dwóch dział elektronowych

Słowa kluczowe: odparowanie, materiałów, wiązka elektronów, powłoki wieloskładnikowe, powłoki gradientowe, powłoki wielowarstwowe.

Streszczenie: W artykule przedstawiono wyniki prac dotyczące weryfikacji możliwości technologicznych urządzenia EB-Dual BEAM w zakresie wytwarzania powłok złożonych, w tym: gradientowych Ti↑-Zr↓, wieloskładnikowych Ti-Zr oraz wielowarstwowych Ti/Ti-Zr/Zr. Wytworzone powłoki poddano analizie zmian składu chemicznego metodą EDS w funkcji odległości od powierzchni oraz obserwacjom przełomów z wykorzystaniem mikroskopii skaningowej, w celu oceny ich budowy wewnętrznej. Wykazano, że wykorzystanie dwóch niezależnych dział elektronowych zapewnia możliwość różnicowania składu chemicznego powłok dwuskładnikowych niezależnie od charakteru tych zmian, tj. zmiany liniowe, zmiany skokowe.

Introduction

Surface engineering plays an important role in the development of technology, especially in the field of the development of innovative construction and technological solutions, including in the area of the automotive industry [1], aviation industry [2], or the tool industry [3]. The advanced material solutions of complex coatings, including multi-component coating [4], multilayer coating [5] and gradient coating [6], have a special meaning. A characteristic feature of complex coatings is the ability to design properties as a function of their thickness, e.g., by changing the chemical composition or structure in gradient coatings, or by changing the amount, thickness, and the order of component layers in multilayer coatings. As a result, there is the possibility of creating multilayer coatings that combine properties in which the simultaneous occurrence is not possible to obtain in monolayers, e.g., high hardness and a reduced state of internal stresses [7] (TiB_2-TiC), high hardness and heat resistance [8] (TiAlN-CrN), and high hardness and a low coefficient of friction (TiAlN-WC/C). In the case of multi-component and composite coatings, by proper selection of the chemical composition and phase composition of the coatings, it is possible to shape their operational properties [9], e.g., high hardness and increased temperature resistance (CrAlN, CrTiAlN) or reduced friction coefficient at elevated temperatures (TiAlSiN). Another, very promising, direction of development is the doping of simple nitrides, such as TiN and CrN, with the addition of other metals (Al, Si, V) and deposition of complex nitride coatings [10] (Ti-Al-N, Cr-Al-N, Ti-Si-N, Cr-Si-N , Cr-VN), which are characterized by higher thermal resistance, which, for example, for multi-component coatings (Ti, Cr, Al), N is close to 1000°C.

A special role in this area is played by the method of the evaporation of materials by electron beam – EBPVD (electron beam physical vapour deposition) [11–12]. The fundamental phenomenon in the EB-PVD method is bringing the material placed in the crucible to evaporation state by high energy electron beam bombardment. Focusing the electron beam on a small area of material enables obtaining high temperatures in the bombardment area and, as a result, the evaporation of the infusible materials, including metals and ceramic materials Al_2O_3. It is these capabilities that make the EB-PVD method increasingly used both independently and in hybrid surface treatment processes for the creation of functional coatings composed on the basis of ceramic materials, such as thermal barrier coatings [13], friction wear resistant coatings [14], and erosion wear resistant coatings [15]. The authors' own experience shows that the use of the EB-PVD method in the configuration with the arc evaporation method allows the deposition of functional nanostructured coatings, including both composite coatings and multilayer coatings [16–17].

1. Deposition of complex coatings with the use of one source beam evaporation system

In order to determine the possibility of producing composite coatings (multi-component, multi-layer, gradient) by the electron beam method, the analysis of the process of the evaporation of the material with an electron beam is of particular importance. In the area of electron beam interaction with the bombarded material, the four zones should be distinguished as shown in Figure 1 [18]. Zone 1 is the zone where the electrons directly penetrate the material. In this zone, the most intense evaporation occurs and the sublimation of the bombarded material is possible. In Zone 2, the temperature exceeds the material evaporation temperature, but the evaporation intensity is much lower than in Zone 1 and decreases along with the distance from the beam incident area. The temperature of the bombarded material in Zone 3 is still higher than melting point but lower than evaporation temperature, while in Zone 4, called "the intensive cooling zone," the temperature is lower than the melting temperature. The intensity of the material evaporation in the process as a result of bombardment with the electron beam is determined by the size of Zones 1 and 2, which form the so-called "evaporation pond". On the other hand, Zones 3 and 4 protect the crucible from overheating or melting, which, as a result of diffusion or mixing processes, could cause contamination of the evaporated material with the material of the crucible.

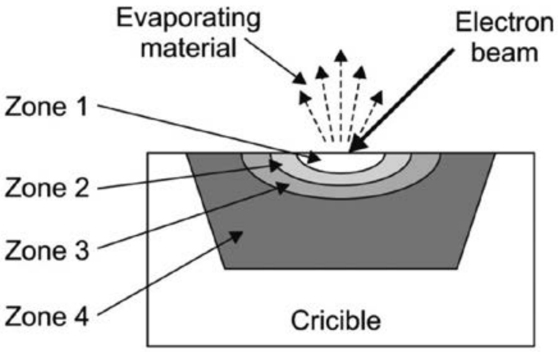


Fig. 1. The electron beam interaction zones with the evaporated material [18]

In order to ensure high efficiency, stability, and repeatability of the evaporation process, it is necessary to create a time-stable "evaporation pond". The time required for this will be significantly dependent on the type of the material to be evaporated (thermal conductivity, melting temperature), the power of the electron gun, and the cooling parameters in the crucible. For example, in the case of titanium bombarded with a beam I_e = 150 mA, creating a stable "evaporation pond" with a diameter of 25 mm requires time t_{Ti} = 20 s. However, in the case of tungsten bombarded with a beam of I_e = 250 mA, creating a stable "evaporation pond" with a diameter of only 10 mm requires time $t_W \approx$ 1 min. The creation of multicomponent or gradient coatings requires that several different materials be allowed to evaporate at the same time in order to mix them. However, in the case of multilayer coatings, it is required to provide alternating evaporation of different materials with the shortest possible time delay in order to ensure maximum adhesion between the component layers in the coating.

The analysis indicates that the deposition of complex coatings, including multilayer, multi-component, and gradient coatings for technological systems equipped with one electron gun, is difficult and requires special technological operations, including multi-position crucibles or specially prepared multi-component targets.

1.1. Electron beam evaporation process with the use of multi-position modular crucible

In practice, there are two different solutions with a multi-position modular crucible are used, i.e. multi-position rotary (Fig. 2a) and multi-position stationary crucible (Fig. 2b). The multi-position rotary crucible is adapted to work with a stationary electron beam deflection system. Materials placed in individual sockets of the crucible are delivered to the evaporation area by the appropriately programmed rotation of the crucible (Fig. 4a). The evaporation of the material from the multi-position stationary crucible is achieved by programmed

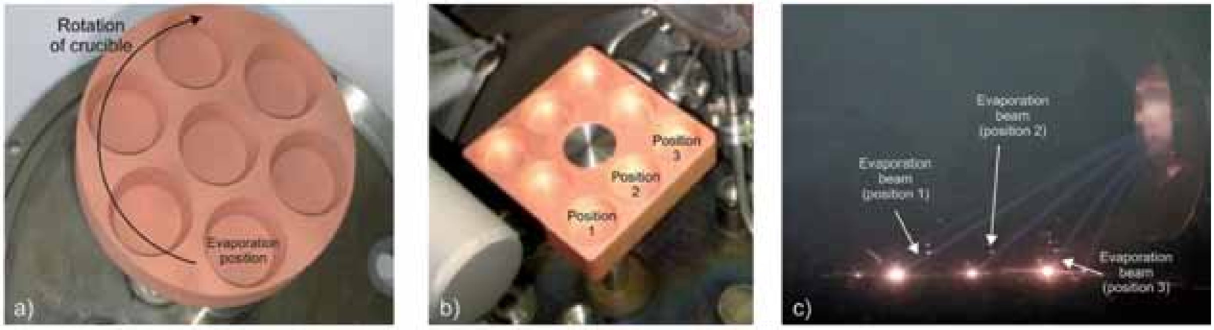


Fig. 2. **Electron beam evaporation of material with the use of multi-position crucibles: (a) multi-position rotary crucible, (b) multi-position stationary crucible, (c) positioning the electron beam in different seats of the multi-position crucible [18]**

switching of the electron beam between subsequent sockets (Fig. 2c). However, the deposition of multilayer coatings is very difficult and, due to the time needed to prepare the material for evaporation in some cases, it does not provide the required adhesion between the component layers of the coating.

The authors' experiences indicate additional difficulties resulting from different thermal properties of the evaporated materials. In the case of a common cooling system for all sockets in a multi crucible, it is virtually impossible to obtain an efficient and stable evaporation process for materials with different thermal conductivity. The constructional solutions shown in Figure 2 also do not allow for evaporation of sublimation materials, e.g., Cr, which require a change of position in relation to the electron beam during the evaporation process.

In order to create technical possibilities to differentiate the cooling intensity in individual slots of a multi-position crucible, a multi-position modular crucible project was developed [19]. The concept of the multi-position modular crucible was created by splitting the multi-position crucible into four modules according to the division of materials with different thermal conductivity (Fig. 3).

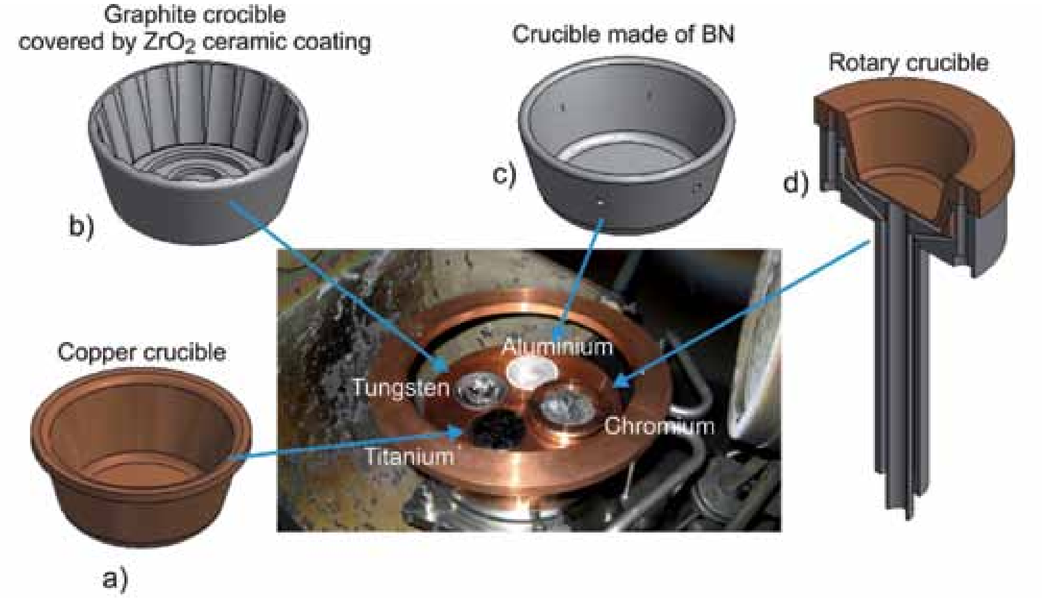


Fig. 3. **Multi-position modular crucible: (a) copper crucible, (b) graphite crucible covered by ZrO_2, (c) crucible made by BN, (d) rotary crucible**

In order to evaporate materials with a low coefficient of thermal conductivity (e.g.,Ti), intensive heat dissipation from the walls of the crucible is required; therefore, the typical copper crucible cooled directly with water was designed (Fig. 3a). Because of the high thermal conductivity of copper (401W/(mK)), the heat is effectively removed from the melted charge heated to the temperature of 1700– 1800ºC. This enables carrying out long-term deposition processes without the threat of crucible overheating. For the high-melting materials (e.g., W, Mo) with high thermal conductivity and high melting point (2600–3400ºC) that require the crucible with limited thermal conductivity, a graphite crucible (Fig. 3b) with an inner surface of the walls coated with ZrO_2 ceramic coating with a thickness ≈150 μm was designed. The low coefficient of thermal conductivity, high melting temperature (3600°C), and additional thermal barrier on the inner surface (NiCoCrAlY/ZrO_2+Y_2O_3) allows the function of the crucible at temperatures above 3500ºC. For the materials with a high coefficient of thermal conductivity (e.g., Al, Cu) and low melting point that require a crucible with low thermal conductivity, the designed crucible is made of ceramic material (BN) shown in Fig. 3c. For the sublimating materials (e.g., Cr, Mg) that require the use of a crucible with constructions that allow for the continuous movement of the evaporated areas of the material in the electron beam operation area, a rotary water-cooled crucible was designed (Fig. 3d). The crucible rotation allows for the even evaporation of material from the entire surface of the charge. Positioning the slot intended for evaporating in relation to the beam is performed by rotating of the multi crucible, as in the conventional multi-rotating crucible.

The presented solution definitely facilitates the creation of multilayer coatings but still makes impossible the creation of multi-component coatings.

1.2. Electron beam evaporation process with the use of multi-component tablets

Technological solution used to prepare the multicomponent coating by the electron beam method implemented using a single electron gun is the evaporation of specially prepared dough into the crucible in the form of tablets [20], with a strictly defined chemical composition, which we want to reproduce in the coating. The experiences of the authors in this respect relate to the deposition of Cr-Ni coatings in the process of the evaporation of tablets with chemical composition Cr 80% – Ni 20% made by the Pulse Plasma Sintering method [21]. Figure 4 shows the results of material research that showed significant heterogeneities in the chemical composition and microstructure of the produced Cr-Ni coatings.

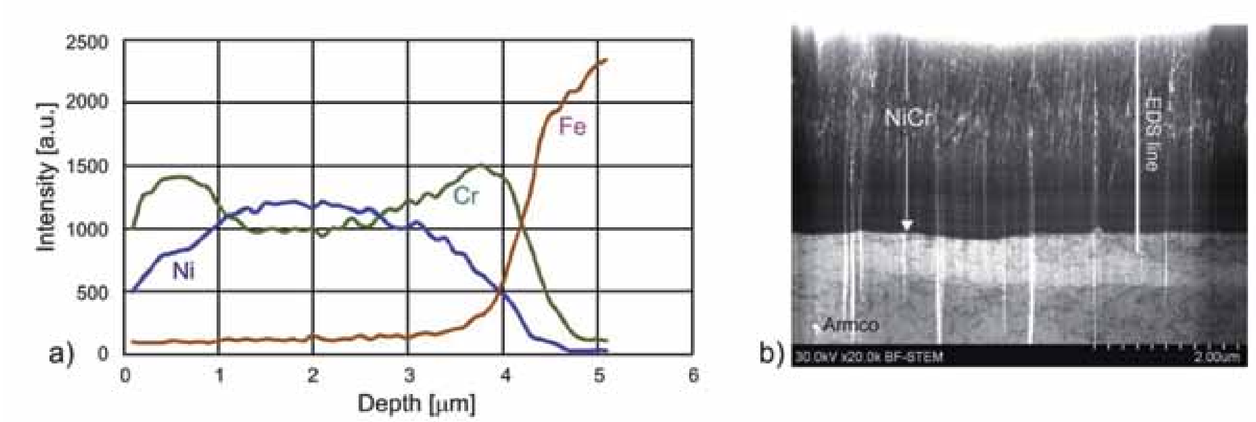


Fig. 4. **The results of chemical changes as a function of distance from the surface (s) and studies of the microstructure (b) Ni-Cr coating produced by electron beam evaporation in the evaporation process of the tablet 80% Cr – 20% Ni produced by Pulse Plasma Sintering method [19]**

The coating deposition process was carried out in an argon atmosphere at a pressure of $p_{Ar} = 3.5 \times 10^{-4}$ mbar. With decreasing pressure, the evaporation temperature of chromium is decreased, and the evaporating temperature of nickel remains constant [22]. As a result, chromium evaporation is much more intense compared to the intensity of nickel evaporation in the first phase of the process (Fig. 4a). With the loss of chromium, the intensity of nickel evaporation increased. The effect of this is an increase in the nickel content in the Cr-Ni coating deposited in the middle of its thickness. In the final stage, the intensity of chromium evaporation increased again. The observed changes in the chemical composition in the Cr-Ni coating, as a function of the distance from the surface, indicate the stage character of the process of the evaporation of multi-component materials in which the intensity of evaporation of individual components changes. This is confirmed by the analysis of the microstructure of the tested Cr-Ni coating by the scanning electron microscopy method supported by the ionic sample preparation technique (STEM + FIB – Scanning Transmission Electron Microscopy + Focus Ion Beam), the results of which are shown in Figure 4b. The microstructure investigations confirmed both a significant enrichment of the microstructure in chromium in the initial evaporation stage and a nanomultilayer structure of the Cr-Ni coating in this area. This allows the conclusion that the evaporation rate of the individual components of composite material are subject to cyclical changes both on the macro scale and on the micro scale. Changes in the intensity of the evaporation of individual components on a macro scale, illustrated by the distinct banding of the coating, are probably caused by the mutual changes in the amount of the constituent elements in the whole volume of the crucible during the evaporation process. Changes in the intensity of the evaporation of individual components on the micro scale, which was evidenced by a clear multilayer structure with a nanometric thickness of individual component layers, may be caused by temperature fluctuations directly on the surface of the evaporating material. These fluctuations result, e.g., from the discontinuity of the scanning process of the electron beam, the discontinuities of the cooling process, or temporary changes in the pressure of the process atmosphere.

The authors' experience indicates that problems with the stability of the evaporation of individual components increase with the increase in the amount of elements included in the multi-component material that is evaporated. This is confirmed by the experiment carried out with the alloy Ni_3Al + 1.5% Hf [23]. In the process of electron beam evaporation (I_e = 130 mA, p_{Ar} = 5x10^{-5} mbar), the vapour pressure of Hf was so small that the evaporation process was absent and 100% Hf remained in the crucible.

Experiments carried out have shown that the use of multi-component targets for deposition of multi-component coatings by the electron beam method is very difficult. In order to obtain the expected chemical composition and microstructure in the coating, it is necessary to know the vapour pressure of the elements that are part of the evaporated material and their mutual influence on the evaporation rate.

The analysis carried out in the area of complex coating creation allowed the authors to formulate the conclusion that the necessary condition to ensure the highest possible deposition, homogeneity, and repeatability of the chemical composition and microstructure of multicomponent, multi-layer, and gradient coatings, produced by the electron beam evaporation method, is to ensure stable conditions of the evaporation process, through the following:

1. Enabling the evaporation of several single-component targets at the same technological time and ensure uniform mixing of their pairs;
2. Adapting the cooling rate of the evaporated materials to their thermal properties;
3. Adapting the intensity of energy supply to the evaporated materials through an intelligent system of scanning the target surface with an electron beam; and,
4. Ensuring the precision stabilization of atmospheric parameters of the process on the basis of digital PID auto-tuning procedure [24].

The adopted assumptions were used to develop the construction of a technological device for the deposition of coatings by the electron beam evaporation method, ensuring effective deposition of complex coatings. The subject of the article is a description of the construction and technological verification of the device in the field of multi-component, multi-layer, and gradient coatings deposition.

2. Deposition of complex coatings with the use of two source beam evaporation system

2.1. Two source beam evaporation system

The EB-Dual BEAM device used in the research for the deposition of electron beam evaporation coatings using two electron guns was designed and built at the Institute for Sustainable Technologies – National Research Institute in Radom (ITeE – PIB). The device has a vacuum chamber with the process zone in the shape of a horizontal cylinder and dimensions: diameter ϕ = 500 mm, length L = 600 mm. The device is equipped with a modern pump system composed of a Leybold DIP 8000 diffusion pump assisted by a Roots pump, which provides the possibility of obtaining a pressure of 10^{-7} mbar in the chamber. The process atmosphere complex was composed using the multi-channel gas atmosphere dosing system from MKS Instruments, enabling the flow control of 4 process gases at the same time, and based on the Pfeiffer pressure control system, using 4 measuring probes and providing pressure control in the range 10^{-9} mbar < p = 1000 mbar. The construction of the chamber allows the mounting of various positioning systems for coated elements, from simple tables with one axis of rotation compatible with the central axis of the chamber, to complex systems of multi-axis rotation. Two electron guns were installed in the process chamber with the voltage Ue = 60 kV and the maximum current of the electron beam Je = 1A, which allow independent evaporation of two different materials at the same time. In addition, the device is equipped with a modern computer system enabling the control of the operating parameters of all functional units of the device. The EB-Dual BEAM device and its components is shown in Fig. 5.

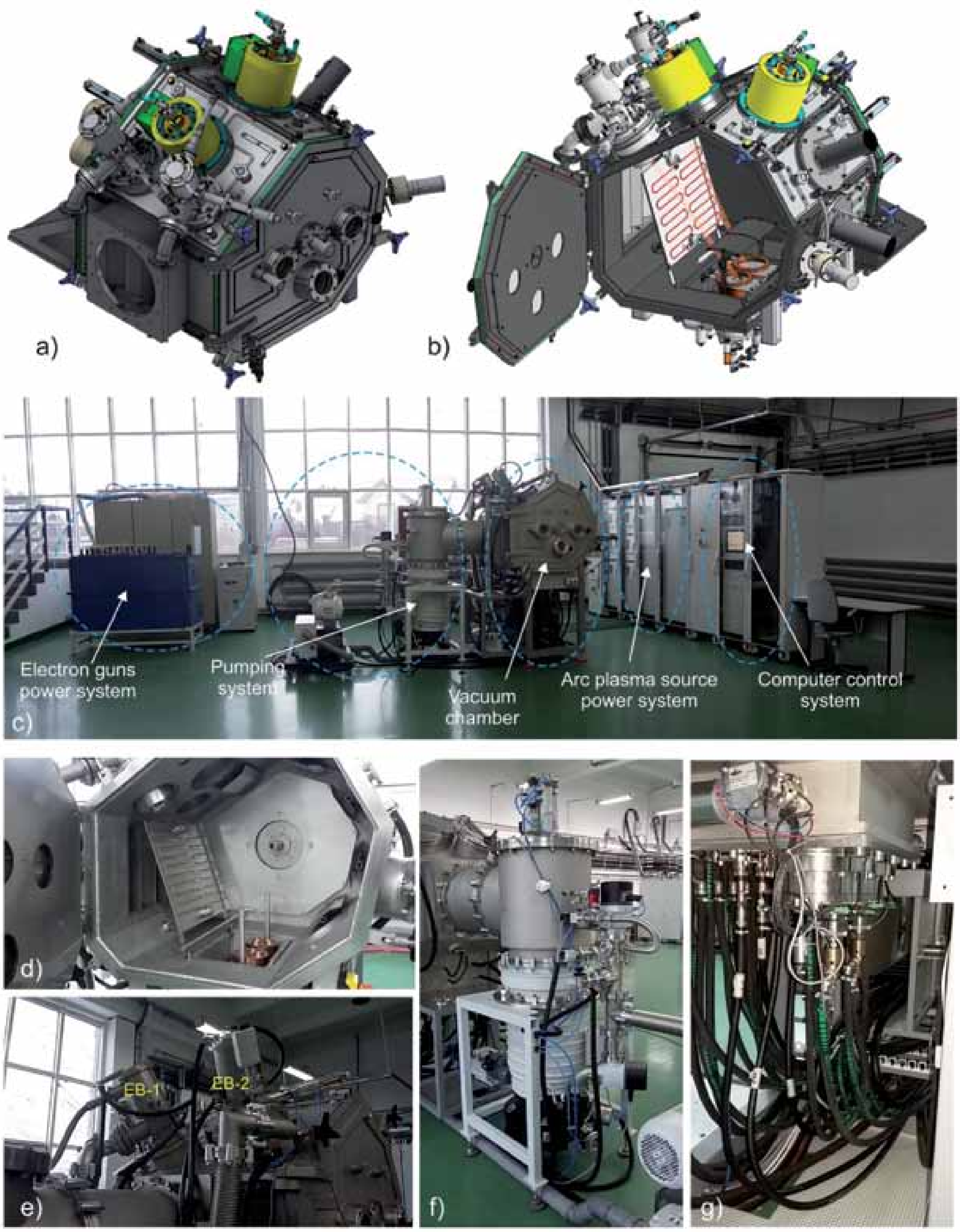
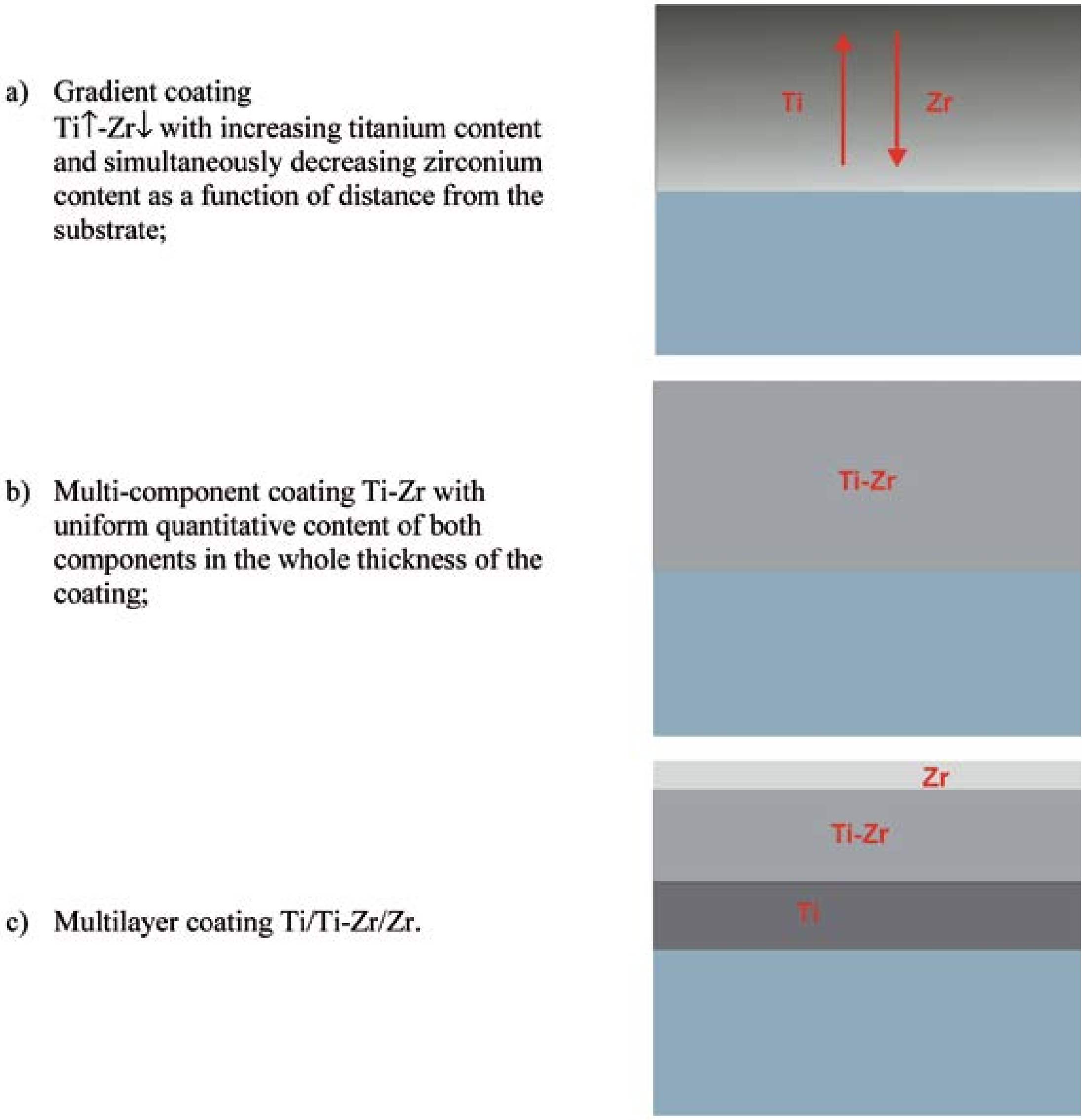


Fig. 5. The EB-Dual BEAM technological device designed and built at ITeE – PIB in Radom (Poland), adapted to the process of evaporation materials by electron beam, using two electron guns: (a, b) technical design, (c) view of the device with the description of individual functional systems, (d) the inside of the process chamber, (e) view of two electron guns mounted on the process chamber, (f) pumping system, (g) water cooling system of the process chamber

2.2. Experimental procedure

In order to verify the technological capabilities of the EB-Dual BEAM device in the field of composite coatings creation the three different deposition processes of complex coatings composed of Ti and Zr, shown in Fig. 6a, b, c, were developed.

The deposition processes of selected coatings were carried out in three stages: 1 – preheating with resistance heaters, 2 – ion etching using arc plasma source, and 3 – deposition using two electron guns EB1 (Ti) and EB2 (Zr). The intensity of Ti and Zr evaporation is regulated by changing the value of the current of both electron beams, i.e. J_e1 (Ti) oraz J_e2 (Zr). The total deposition time of the each coatings was the same and was $t_D = 8$ min. Selected coatings were made on two types of substrate, i.e. Armco and monocrystalline Si plates with a surface roughness of $Ra \leq 0.05$ μm. The parameters of deposition processes of selected composite coatings are presented in Tables 1–4, respectively. The location of the electron guns, evaporated materials, and the samples inside the process chamber are presented in Figure 7.


a) Gradient coating Ti↑-Zr↓ with increasing titanium content and simultaneously decreasing zirconium content as a function of distance from the substrate;

b) Multi-component coating Ti-Zr with uniform quantitative content of both components in the whole thickness of the coating;

c) Multilayer coating Ti/Ti-Zr/Zr.

Fig. 6. **Types of complex coatings selected for testing: (a) gradient coating Ti↑-Zr↓, (b) multi-composition coating Ti-Zr Ti-Zr, (c) multi-layer coating Ti/Ti-Zr/Zr**

Table 1. Deposition parameters of gradient coating Ti↑-Zr↓

No.	Stage	Parameters		
1	Heating	J_R = 120 A; t_R = 60 min		
2	Ion Etching	J_{ARC} = 80 A; U_{BIAS} = −1000 V; p_{Ar} = 7x10^{-4} mbar; t_{IE} = 3 min		
3	Deposition: total time = 8 min	t_D [min]	J_e1 (Ti) [mA]	J_e2 (Zr) [mA]
		0.5	25	150
		0.5	30	147
		0.5	35	144
		0.5	40	141
		0.5	45	138
		0.5	50	135
		0.5	55	132
		0.5	60	129
		0.5	65	126
		0.5	70	123
		0.5	75	120
		0.5	80	117
		0.5	85	114
		0.5	90	111
		0.5	95	108
		0.5	100	105

Table 2. Deposition parameters of multi-composition coating Ti-Zr

No.	Stage	Parameters		
1	Heating	J_R = 120 A; t_R = 60 min		
2	Ion Etching	J_{ARC} = 80 A; U_{BIAS} = −1000 V; p_{Ar} = 7x10^{-4} mbar; t_{IE} = 3 min		
3	Deposition: total time = 8 min	t_D [min]	J_e1 (Ti) [mA]	J_e2 (Zr) [mA]
		8	100	140

Table 3. Deposition parameters of multi-layer coating Ti-Zr

No.	Stage	Parameters		
1	Heating	J_R = 120 A; t_R = 60 min		
2	Ion Etching	J_{ARC} = 80 A; U_{BIAS} = −1000 V; p_{Ar} = 7x10^{-4} mbar; t_{IE} = 3 min		
3	Deposition: total time = 8 min	t_D [min]	J_e1 (Ti) [mA]	J_e2 (Zr) [mA]
		1.25	100	60
		1.00	100	140
		1.25	100	60
		1.00	100	140
		1.25	100	60
		1.00	100	140
		1.25	100	60

In order to assess whether the obtained changes in the chemical composition are consistent with the intentions shown in Figure 6, complex coatings selected for testing (gradient coating Ti↑-Zr↓, multi-composition coating Ti-Zr and multi-layer coating Ti-Zr) were subjected to an analysis of chemical composition as a function of distance from the surface by the EDS method. The changes in the chemical composition were analysed on samples made of Armco iron. The analysis of structure of the tested coating was performed on monocrystalline silicon samples in the form of brittle fracture, which were subjected to observations using scanning microscopy.

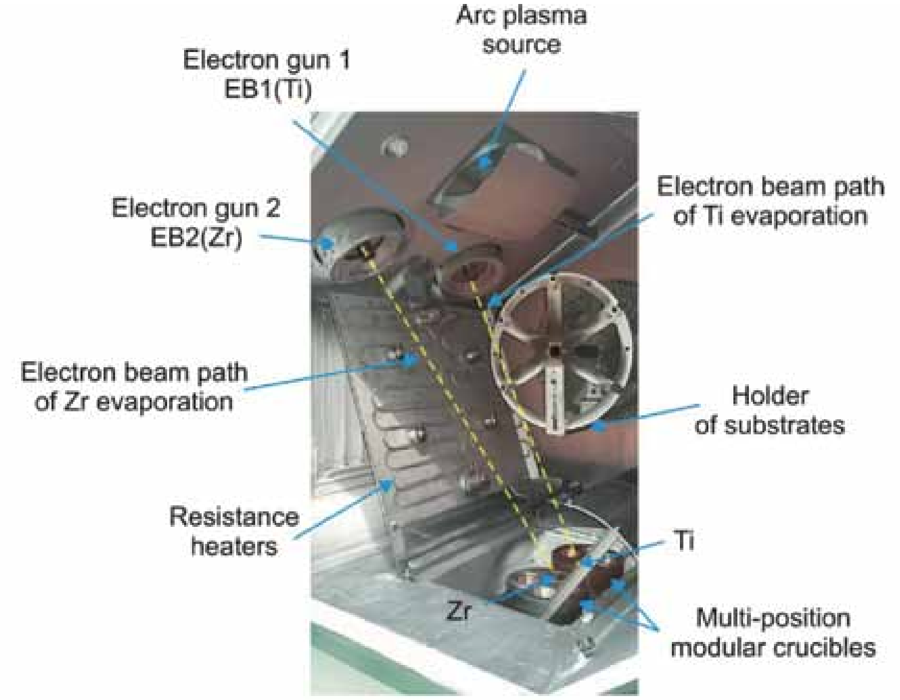


Fig. 7. View of the inside of EB-Dual BEAM process chamber

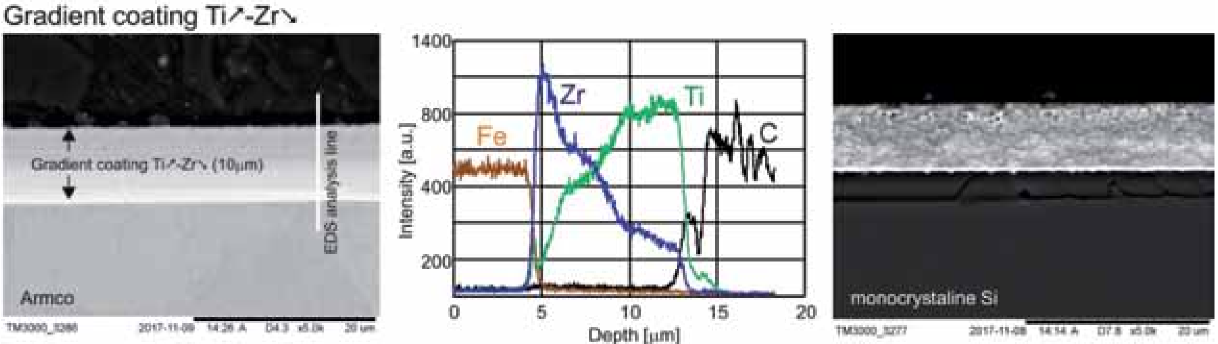
2.3. Results and discussion

Figure 8 shows cross sections and profiles of changes in chemical composition as a function of distance from the substrate for three types of tested complex coatings, *i.e.* gradient coating Ti↑-Zr↓, multi-composition coating Ti-Zr, and multi-layer coating Ti/Ti-Zr/Zr. The results of changes in the chemical composition (Figs. 8b, e, h) showed that, independently from the character of the changes (linear, step), the use of two independent electron guns enables effective control of changes in the chemical composition of two elements.

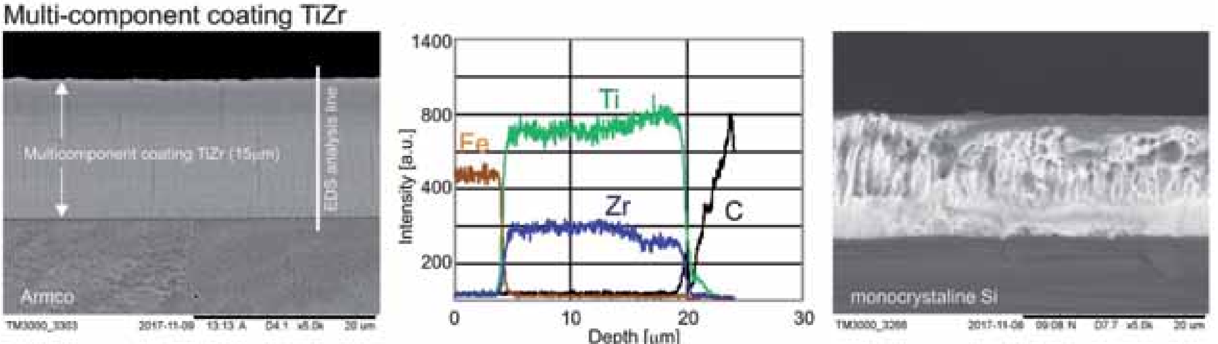
Microscopic analysis of brittle fracture tested coatings showed that the microstructure of gradient coating Ti↑-Zr↓ (Fig. 8c) and the microstructure of multi-composition coating Ti-Zr (Fig. 8f) change as a function of distance from the substrate. In both cases, two zones with different microstructure are visible. With the increase of titanium content (Ti) and decrease of the zirconium content (Zr) in the gradient coating Ti↑-Zr↓, a porous zone was observed. A similar phenomenon was observed in the multi-composition Ti-Zr coating, in which, despite the stable electron beam operation parameters, i.e. electron beam currents J_e(Ti) = 100 mA and J_e(Zr) = 140 mA, there was an increase in Ti content and a decrease in Zr content the surface zone.

In the opinion of the authors, the observed increase in the porosity of the Ti-Zr coating occurs for specific, relative proportions of the chemical composition of Ti_x-Zr_y, which is similar for both types of coatings: gradient coating Ti↑-Zr↓ i multi-composition coating Ti-Zr for which the coating growth is much faster. Under such conditions, thermodynamic diffusion of the surface was too small to provide a suitable density in the coating. Nevertheless, the explanation of the problem requires thorough investigations of the phase structure and a quantitative analysis of the chemical composition in selected areas of the deposited coating.

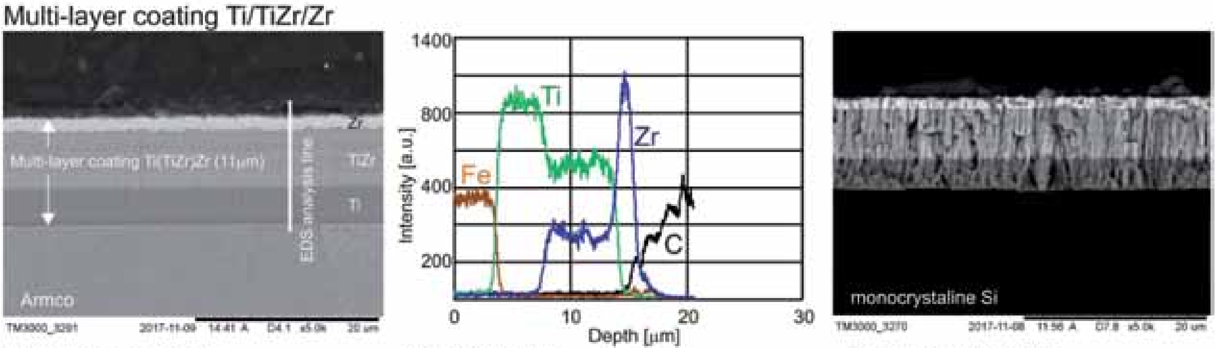
The multi-layer coating Ti-Zr shown in Figures 8g,h,i, is characterized by a very dense column microstructure in the entire thickness. It is characteristic that the individual columns of grains grow in the entire thickness of the coating, which indicates very good cohesion between the component layers of the multilayer coating.



a) metallographic cut b) EDS analysis c) fragile breakthrough



d) metallographic cut e) EDS analysis f) fragile breakthrough



g) metallographic cut h) EDS analysis i) fragile breakthrough

Fig. 8. The results of studies on changes in chemical composition as a function of distance from the surface and the results of microscopic observation of three different layers of complex prepared by EB-PVD method with use of two electron gun: (a,b,c) gradient coating Ti↑-Zr↓, (d,e,f) multi-composition coating Ti-Zr, and (g,h,i) multi-layer coating Ti/Ti-Zr/Zr

Conclusions

The article presents the results of work on the verification of the technological capacity of the device EB Dual-BEAM in the creation process of complex coatings, including gradient coating Ti↑-Zr↓, multi-composition coating Ti-Zr, and multi-layer coating Ti/Ti-Zr/Zr. It has been shown that the use of two independent electron guns provides the possibilities of the differentiation of the chemical composition of two-component coatings, regardless of the character of these changes, i.e. linear changes, and step changes.

At the same time, it has been shown that the condition necessary to ensure the homogeneity of the chemical composition is the stability of the evaporation parameters of individual elements. For this purpose, it is necessary to ensure stable conditions for the supply of energy to the evaporated material through a complex selection of the following process parameters:

– Enabling the stability of the process of supplying energy to the evaporated materials through the operating parameters of the electron gun and the intelligent system of scanning the target surface with an electron beam;

- Enabling the stability and adjusting the speed of cooling process of the steamed materials for their thermal properties; and,
- The precision stabilization of atmospheric parameters of the process on the basis of a digital PID auto-tuning procedure.

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