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Cr-CrN AND CrN-TiC MULTILAYER COATINGS MANUFACTURED BY MEANS OF VACUUM ARC METHOD

Keywords

Chromium nitride, multilayer coatings, arc-vacuum method.

Abstract

The issue of this paper is the testing of properties of multilayer coatings composed on the basis of chromium nitride, multilayer Cr-CrN coating and multilayer CrN-TiC coating manufactured by means of the arc-vacuum method on 4H13 steel foundation. The multilayer structure of the obtained coatings has been determined by means of scanning microscopy and spectral analysis of the chemical composition change in the depth function (GDOES). Besides, their hardness and Young module has been measured (NanoHardnessTester) and the adhesion has been tested by means of scratch-test (REVETEST). In the scratch-tests the load values of the intender determining the moment of cracks initiation and cohesion failure were determined. They define the moment of adhesion failures initiation and determine the moment of complete coating's removal from the foundation surface in the scratch area. Thanks to the testing, it was possible to define the influence of the multilayer structure on the mechanical properties of manufactured multilayer compositions and the mechanism of their devastation in the scratch test.

Introduction

The fast development pace of many modern industry branches in the last decades was, first of all, determined by the capacities of surface engineering [1–3]. Thanks to new studies in the field of thin coatings material engineering and to new technological studies in this area, the advanced mechanical systems are adjusted to work in harder and harder maintenance conditions, i.e. with high mechanical and thermal loads, intensive abrasive wear and corrosive operation of environment. According to the author, these new tasks can be undertaken and solved exclusively by taking advantage of capacities provided by new coating materials, among which multilayer coatings are of special importance. The introduction of multilayer structure causes, according to the multistage mechanism of multilayer coatings destruction [4–6], an increase of resistance against cracking. The separation zones between subsequent composition layers are places in which the directions of microcracks propagation change or undergo dispersion. This phenomenon will diminish the possibility of microcracks propagation into the coating, thus prolonging the way of a single crack and, at the same time, reducing its energy. In author's previous studies [7–9] he demonstrated, on the example of multilayer $TiC/Ti(C_xN_{1-x})/TiN$ coating, the possibility of synergic [10] cooperation between its particular component layers, as a result of which there is a significant increase of maintenance durability of the manufactured multilayer coating. The confirmation of the influence of the coating's multilayer structure on their maintenance durability is the large quantities of data provided in literature [11–14].

Chromium nitride – CrN is one of the most important materials applied in surface engineering. This results, first of all, from the fact that it is, at the same time, characterised by very good tribological properties [15–16], mechanical properties [17–18] and chemical properties [19–21]. Compared to TiN and Ti(C,N) [22–23] chromium nitride is characterised by lower thermal conductivity ($\lambda_{Cr\text{-}N} = 0.183$ [$Wcm^{-1}K^{-1}$]), greater resistance to oxidation in raised temperature ($T^{O2}_{CrN} = 720^{o}C$) and by greater plasticity [12]. Chromium nitride also has a very good corrosive [24] and chemical resistance [25]. A favourable phenomenon resulting from the application of CrN coatings is also the possibility of reducing, to a great extent, the application of lubricants in cold working [25]. The application area of CrN as an antiwear coating comprises the following: cutting tools for non-ferrous metals working [26], plastic working dies [27], aluminium pressure casting dies [28] and hot working dies [29–31]. The outcomes of numerous scientific studies [30–34] revealed the great effectiveness of CrN as an anti-wear coating in applications in which the protection of surface material against temperature influence is significant.

According to the authors, it seems important to extend knowledge on the optimisation of functional properties of coatings composed on the foundation of chromium nitride – CrN, including above all multilayer coatings.

1. Selection of materials for testing

The issue of the paper is the manufacture of multilayer coatings composed on the basis of chromium nitride, i.e. multilayer Cr-CrN coating and multilayer CrN-TiC coating, manufactured by means of the arc-vacuum method, and testing their properties.

According to numerous literature records [35–39], multilayer Cr-CrN coatings are characterised by a very good crystallographic matching of subsequent constituent Cr and CrN layers (Fig.1) [35] and by the creation of crystalline transient layer with the thickness of tens of nanometers [39]. It ensures the good connectivity of layer in particular separation zones, and as a result, good maintenance properties: adhesion [36], abrasive wear resistance [37] and corrosive resistance [38].

In this paper [40], it has also been revealed that the presence of constituent layers with a greater plasticity in the multilayer coating significantly increases coating's abrasive wear resistance. The outcomes of experimental testing indicate, in this case, an important role of constituent layers of metallic chromium which, thanks to their capability of plastic deformation, limit the operation of hard particles in the friction area.

The manufacture of a multilayer CrN-TiC coating is a result of a different opinion in the process aimed at the improvement of the tribological properties of coatings composed on the basis of CrN. According to the authors of this paper, hard constituent layers of titanium carbide will not undergo plastic deformation, as it is in case of metallic chromium layer in multilayer Cr-CrN coating, but thanks to a great hardness and abrasive wear resistance [41–42], they should ensure a high abrasive wear resistance of the whole multilayer composition.

At the same time, the titanium carbide is characterised by a considerably lower thermal conductivity than chromium nitride, (λ_{CrN} = 0.183 [$Wcm^{-1}K^{-1}$] λ_{TiC} = 0.068 [$Wcm^{-1}K^{-1}$]); therefore, the presence of this phase in the structure of PAPVD layer will limit the temperature influence on the foundation material. It seems that it can significantly contribute to the improvement of the effectiveness of coatings composed on the basis of CrN in limiting the external temperature influence on the foundation material which, as the author revealed in his works [30–34], is a frequent objective of applying chromium nitride coatings. The literature records [43–45] confirm the very good properties of multilayer coatings composed with the participation of TiC phase, e.g. TiN/TiC [43], Ti_2B/TiC [44], TiC/TiCN/TiN [45].

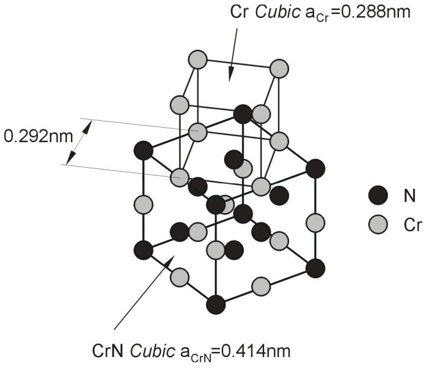


Fig. 1. Schematic presentation of crystallographic matching of CrN and Cr network cells [35]

2. Manufacture of Cr-CrN and CrN-TiC multilayer coatings

Multilayer coatings based on chromium nitride, i.e. Cr-CrN and CrN-TiC selected for testing were obtained at Plasma Technology Centre ITeE-PIB in Radom by means of arch-vacuum method [41, 46] with the use of MZ383 device by Metaplas Ionon company.

The following scheme of reciprocal system of particular constituent layers of multilayer coatings selected for testing was adopted. In the case of Cr-CrN multilayer coating, the first constituent layer deposited directly on the foundation was CrN 1µm thick. As next 5 two-layer Cr-CrN complexes were deposited, in which Cr thickness amounts to 0.2 µm and the thickness of CrN is 0.8µm (Fig. 2a). In case of CrN-TiC coating, in order to minimise the degree of crystallographic mismatch of both materials, additional constituent TiN layers separating the constituent CrN and TiC layers were introduced into coating's multilayer structure (Fig. 2b).

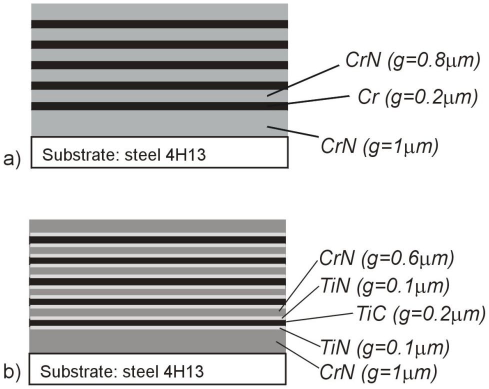


Fig. 2. Structure of multilayer coatings on the basis of chromium nitride selected for testing: a) CrN/(Cr/CrN)x5, b) CrN/(TiN/TiC/TiN/CrN)x5

Multilayer coatings selected for testing were obtained on 4H13 steel foundation according to parameters displayed in Table 1. The differentiation of thickness in the manufactured constituent layers was achieved by choosing the deposition process time.

Table 1. Technological parameters of manufacturing constituent layers materials Cr, CrN, TiN, TiC in the investigated multilayer coatings

Material	Atmosphere	Pressure p [mbar]	Polarisation voltage U_{bias} [V]	Foundation temperature T [°C]	Deposition speed V [nm/min]
Cr	100% Ar	$3.0x10^{-5}$	-200	450	50
CrN	100% N_2	$3.5x10^{-2}$	-200	450	60
TiN	100% N_2	$1.2x10^{-2}$	-200	450	30
TiC	100% C_2H_2	$3.0x10^{-3}$	-200	450	50

3. Testing methodology

In order to determine the multilayer structure of the two multilayer coatings obtained, microscope observations of their section were carried out (*Neophot32 optical microscope, SEM Hitachi S2606*) and a quality analysis of their chemical composition was conducted by means of the GDOS method (*spectrometer Jobin Yvon 10000 RF*).

In order to determine the influence of multilayer structure on mechanical properties of the manufactured coatings, the testing of hardness and Young module was carried out by means of penetration method (*NanoHardnessTester by CSEM*). The measurements were conducted with a Berkovitch indenter. To eliminate the influence of foundation material on measurement outcomes the range of indenter penetration was reduced to the depth $g \leq 0.1$ of coating thickness.

Adhesion testing of the obtained multilayer coatings was carried out by means of scratch test (*REVETEST by CSEM*) in the scope of indenter load with force $Fn=0 \div 100$ N, using Rockwell indenter. For each scratch the changes of friction rate values μ between the indenter and the scratched surface and the intensity of acoustic signal – AE were observed. The values of indenter load were identified which determine the moment of initiating cohesive cracks and failures (Fc1), the moment of initiating adhesive failures (Fc2) and the moment of complete removal of the coating from the foundation surface in the scratch area (Fc3).

4. Testing results

4.1. Testing of CrN/(Cr/CrN)x5 multilayer coating

The outcomes of CrN/(Cr/CrN)x5 multilayer coating testing are presented in Fig. 3 and Fig. 4. As the quality analysis of chemical composition changes (fig. 3) and metallographic testing (Fig. 4a) revealed, the created multilayer coating consists of alternately laid CrN and Cr layers whose total thickness amounts to $g_{Cr\text{-}CrN} = 3.5$ μm. Measurements carried out by means of penetration method indicated that the multilayer CrN/(Cr/CrN)x5 coating is characterised by a lower hardness ($HV_{Cr\text{-}CrN} = 1800$) and a lower value of Young module ($E_{Cr\text{-}CrN} = 280$ GPa) than CrN coating ($HV_{CrN} = 2100$, $E_{CrN} = 320$ GPa) [47].

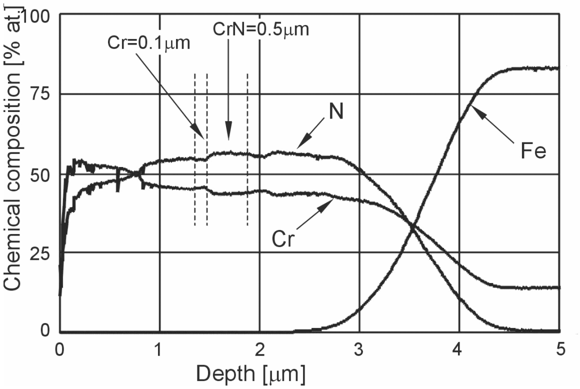
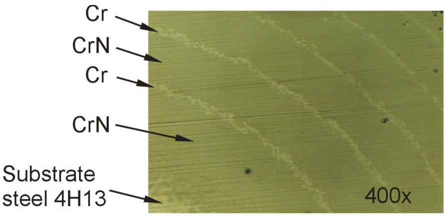


Fig. 3. The results concerning chemical composition changes in the depth function of CrN/(Cr/CrN)x5 multilayer coating

a)



b)

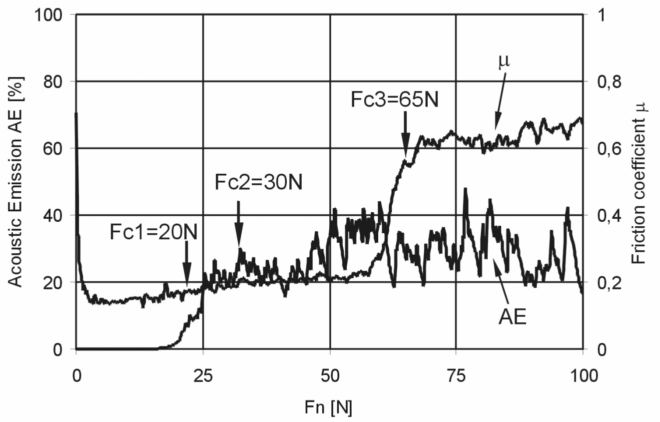


Fig. 4. Outcomes of CrN/(Cr/CrN)x5 multilayer coating testing: a) multilayer structure, b) scratch test results

According to the results of adhesion testing (Fig. 4b), despite a very good crystallographic matching between constituent CrN layers and metallic chromium, the first cohesive failures in the multilayer Cr-CrN coating are generated already at indenter load Fc1 ≈ 20N. First adhesive failures were already observed at an indenter load Fc2 ≈ 30N, whereas a complete removal of coating from the scratch area took place for Fc3 ≈ 65N.

4.2. Testing of CrN / (TiN/TiC/TiN/CrN)x5 multilayer coating

The results of testing conducted for CrN/(TiN/TiC/TiN/CrN)x5 multilayer coating are presented in Fig. 5 and Fig. 6.

As the quality analysis of chemical composition changes (Fig. 5) and metallographic testing (Fig. 6a) revealed in the created multilayer coating, the subsequent CrN composition layers, about 0.7μm thick, are separated by three-layer TiN/TiC/TiN complexes the thickness of which the TiC thickness amounts to about 0.3 μm and is TiN thickness is about 0.2 μm. The total thickness of CrN/(TiN/TiC/TiN/CrN)x5 coating amounts to $g_{Cr\text{-}CrN} = 8$ μm.

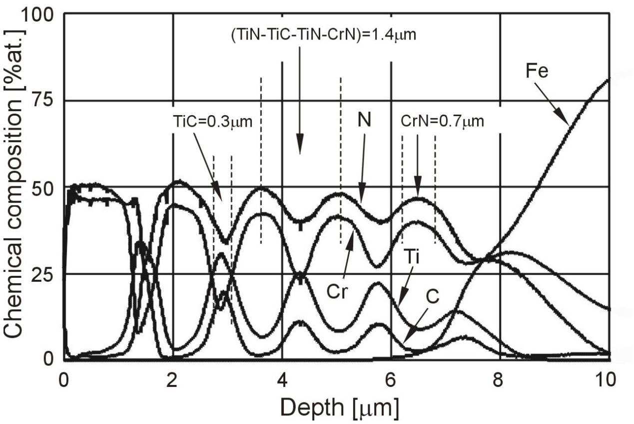


Fig. 5. Outcomes of testing concerning chemical composition changes in the depth function of CrN/(TiN/TiC/TiN/CrN)x5 multilayer coating

In connection with introducing the hard TiC phase to multilayer composition CrN / (TiN/TiC/TiN/CrN)x5, the multilayer coating is characterised by significantly greater hardness ($HV_{CrN\text{-}TiN\text{-}TiC} = 2700$) and Young model value ($E_{CrN\text{-}TiN\text{-}TiC} = 390$GPa) than the CrN coating.

The conducted adhesion testing (Fig. 6b) revealed that both the first cohesive and first adhesive failures are generated in multilayer

CrN/(TiN/TiC/TiN/CrN)x5, similarly, as in CrN / (Cr/CrN) layer at indenter loads Fc1≈20N and Fc2 ≈ 27N. The complete removal of coating from the scratch area in the case of CrN/(TiN/TiC/TiN/CrN)x5 coating occurred already at indenter load Fc3 ≈ 35N.

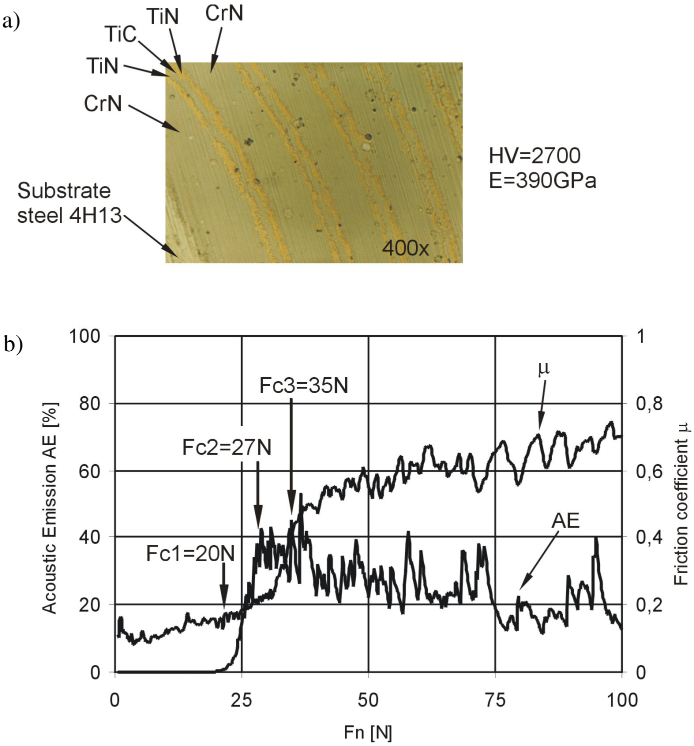


Fig. 6. Results of CrN/(TiN/TiC/TiN/CrN)x5 multilayer coating, a) multilayer structure, b) adhesion testing results by means of scratch test

Fig. 7 presents the outcomes of failures observation of the investigated multilayer coating in cohesive failures initiation zone during the adhesion testing by means of scratch test conducted with the use of scanning microscope. It can be clearly seen that the coating's cohesion has been disturbed mainly in the separation zone between CrN layer laying directly at the surface and the first TiN/TiC/TiN complex. As the microscope observation revealed, in the majority of cases cracks were generated in the separation zone between CrN and TiN constituent layers laying closest to the foundation and spread in the direction of coating surface. No cracks have been observed for which the place of generation would be the coating surface. This indicates that, in the process of generating the coating's failures in the scratch test, the state of internal stresses in the multilayer coating plays a decisive role. It seems that substantially lesser meaning in this process have defects occurring on the coating's surface, i.e. roughness, microdrops and local microdefects.

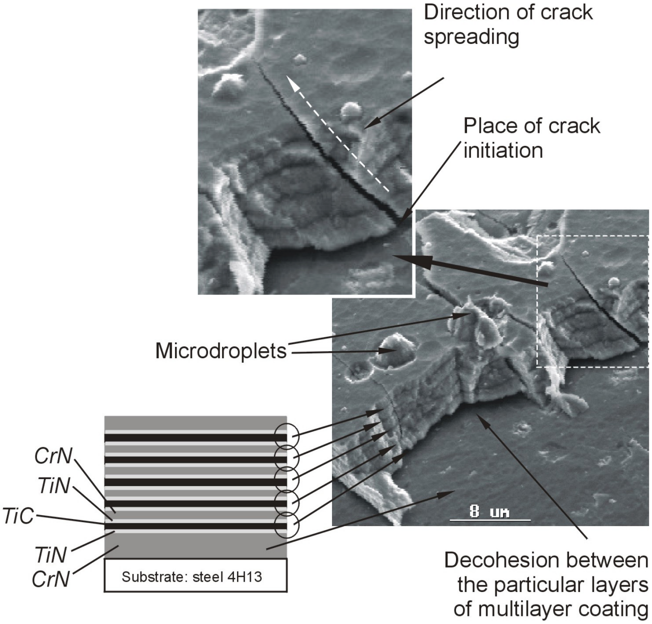


Fig. 7. The outcomes of the analysis of CrN/(TiN/TiC/TiN/CrN)x5 multilayer coatings failures occurred during adhesion testing by means of crack test

5. Discussion

Within the conducted testing, two multilayer coatings were designed and created on the basis of chromium nitride, i.e. CrN/(Cr/CrN)x5 and CrN/(TiN/TiC/TiN/CrN)x5 on the foundation of 4H13 steel. The method applied for their manufacture was the arch-vacuum method. The conducted analysis of chemical composition changes in the thickness function and microscope analysis confirmed the fact of obtaining designed multilayer structures in the created coatings.

In the case of CrN/(Cr/CrN)x5 coating the testing of hardness and Young module carried out by means of penetration method revealed that it is characterised by slightly lower values of these parameters, compared to values achieved for CrN monolayer coating. According to the authors of this paper, it is the result of minor participation of the Cr phase in the total volume of CrN/(Cr/CrN)x5 coating. As the GDOES analysis indicated, the thickness of metallic chromium layers amounted to about Cr ≈ 0.1 μm; therefore, the total participation of Cr phase in the created multilayer coating 3.5 μm thick amounted solely to 0.5 μm (14%). Scratch tests conducted for CrN/(Cr/CrN)x5 coating received on the foundation of 4H13 steel 40HRC thick revealed that the obtained parameters of adhesion assessment, i.e. Fc1 = 20 N, Fc2 = 30 N and Fc3 = 65 N, are similar to parameters achieved for CrN coatings obtained on WCL steel ≈ 55HRC thick [48] ($Fc1_{CrN}$ = 17 N, $Fc3_{CrN}$ ≈ 64 N), and they prove a very good adhesion to the surface.

In the case of CrN/(TiN/TiC/TiN/CrN)x5 coating, the testing of hardness and Young module showed that it is characterised by significantly higher values of these parameters, compared to values achieved for CrN monolayer coating. Here GDOES analysis revealed that the thickness of a single TiN/TiC/TiN three-layer complex amounts to ≈ 0.7 μm; hence, the complete participation of TiN and TiC phases in the created multilayer coating 8 μm thick amounted to 3.5 μm (43%). The assessment parameters of coating adhesion CrN/(TiN/TiC/TiN/CrN)x5 in the scratch test, i.e. Fc1 = 20 N, Fc2 = 27 N and Fc3=35 N, revealed that, despite similar values of Fc1 and Fc2 as for CrN / (Cr/CrN)x5 coating, it undergoes a considerably faster complete abrasion in the scratch area. The conducted analysis of failures of CrN/(TiN/TiC/TiN/CrN)x5 coating in the scratch area revealed that, already for the indenter load of about 20 N, a loss of its cohesion in the separation zone occurs between the CrN layer placed directly at the foundation and the TiN layer which is in its contact. Cracks generated in the separation zone propagate in the direction of coating surface, causing that in the range of indenter loads 20÷27 N an adhesive failure of the multilayer coating's constituent layers occurs outside the CrN layer deposited directly on steel foundation. As a result, after the load of 27 N had been extended, the indenter affects exclusively CrN layer located directly at the foundation whose thickness, according to GDOS analysis, is <1 μm. In the end effect the foundation in the scratch area is completely revealed already for indenter load Fc3 = 35 N.

According to the authors, the obtained results of adhesion testing indicate that, in the scratch test of CrN/(TiN/TiC/TiN/CrN)x5 coating, an increased state of stresses in CrN-TiN separation zone occurs. The local increase of stresses can be the result of the non-uniform distribution in multilayer coating thickness function, both internal stresses in the coating and stresses introduced to the coating by means of indenter. According to the analysis carried out within this paper, it appears that the degree of crystallographic mismatch between CrN and TiN is greater than between CrN and Cr in the CrN/(Cr/CrN)x5 coating. At the same time, the separation zone of CrN-TiN has a pseudo-diffusive nature [46], i.e. the gradient of changes of Cr and Ti in the separation zone is forced by the parameters of technological process. Additionally, due to small thickness of TiN (0.2 μm) layers, the state of stresses in CrN-TiN zone can influence the TiC

(0.3 μm), which is in touch with TiN. As a result, one can presume that the state of stresses in CrN-TiN separation zone is considerably higher than in CrN-Cr separation zone.

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Powłoki wielowarstwowe Cr-CrN i CrN-TiC wytwarzane metodą łukowo-próżniową

Słowa kluczowe

Azotek chromu, powłoki wielowarstwowe, metoda łukowo-próżniowa.

Streszczenie

Przedmiotem artykułu są badania właściwości powłok wielowarstwowych skomponowanych na bazie azotku chromu, tj. powłoki wielowarstwowej Cr-CrN oraz powłoki wielowarstwowej CrN-TiC, wytworzonych metodą łukowo-próżniową na podłożu ze stali 4H13. Z wykorzystaniem mikroskopii skaningowej oraz spektralnej analizy zmian składu chemicznego w funkcji głębokości (GDOES) określono budowę wielowarstwową otrzymanych powłok, dokonano pomiarów ich twardości i modułu Younga (NanoHardnessTester) oraz zbadano adhezję metodą scratch-test (REVETEST). W testach zarysowania wyznaczono wartości obciążenia wgłębnika określające moment inicjowania pęknięć i zniszczeń kohezyjnych, określające moment inicjowania zniszczeń adhezyjnych oraz określające moment całkowitego usunięcia powłoki z powierzchni podłoża w obszarze zarysowania. Przeprowadzone badania pozwoliły określić wpływ budowy wielowarstwowej na właściwości mechaniczne wytworzonych kompozycji wielowarstwowych oraz mechanizm ich niszczenia w teście zarysowania.