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DOI: 10.15199/40.2023.3.1

The erosion resistance of Cr/CrN multilayer coating deposited using industrial Arc-PVD process for compressor blades application

Odporność erozyjna wielowarstwowych powłok Cr/CrN osadzonych metodą Arc-PVD w warunkach przemysłowych do zastosowania na sprężarki silników lotniczych

This study presents the results of a study on the feasibility of using a commercial Cr/CrN multilayer coating to protect the surface of aircraft engine compressor blades made of stainless steel. The research was conducted for coatings composed of 10 and 14 alternating layers of chromium and chromium nitride, respectively. The both variants of coating was manufactured in lon Galenica Company. Studies of its microstructure showed that their thickness did not exceed 3 μ m. Studies of the erosion resistance of both coating variants showed about 50% less weight loss of the samples compared to the material without a protective coating. The highest erosion resistance was found for the 10-layer variant.

<u>Słowa kluczowe</u>: PVD coatings, Cr/CrN, Arc-PVD, erosion resistance, compressor blade

1. Introduction

Surface hardening processes such as carburizing [1] and diffusion boriding [2] have been widely used in aviation technology for many years. The most critical part of a turbojet engine is the compressor. Compressor blades are made of materials that do not provide efficient corrosion resistance, so they must be protected by coating designed to increase susceptibility to corrosion and erosive wear. In order to adequately protect compressor blades, the coating used should consist of multiple thin layers, which will provide resistance to aggressive media and wear under complex operating conditions [3].

The development of PVD coating technology has resulted in increased durability of tooling materials, and is now becoming more W artykule przedstawiono wyniki badań dotyczących możliwości zastosowania komercyjnej wielowarstwowej powłoki Cr/CrN do ochrony powierzchni łopatek sprężarki silnika lotniczego wykonanych ze stali nierdzewnej. Przeprowadzono badania powłok złożonych z 10 i 14 naprzemiennych warstw chromu i azotku chromu, wykonanych w firmie lon Galenica. Analiza mikrostruktury powłok wykazała, że ich grubość nie przekraczała 3 µm. Badania odporności erozyjnej obu wariantów powłoki wykazały o ok. 50% mniejsze spadki masy próbek w porównaniu z materiałem bez powłoki ochronnej. Największą odporność erozyjną stwierdzono w wypadku wariantu 10-warstwowego.

<u>Keywords</u>: powłoki PVD, Cr/CrN, Arc-PVD, odporność erozyjna, łopatka sprężarki

widely used for aerospace technology as well. Currently, the most commonly used titanium nitride-based coatings are characterized by relatively high hardness and wear resistance. TiN coatings produced by Arc-PVD method are characterized by a columnar microstructure, where grain boundaries exhibit micro-cracks due to compressive stresses created during deposition. In addition, these coatings exhibit a cathodic nature to most steels, which results in pitting corrosion [4]. An Arc-PVD TiN coating applied to a blade made of El962-type martensitic steel shows structural leakage, as observed after a 48-hour corrosion test in a salt chamber. This resulted in corrosion products visible as infiltration on the surface of the blade, as well as breaks in its continuity especially in the area of the blade's leading edge. The use of TiN coatings as a pro-

Otrzymano / Received: 27.10.2022. Przyjęto / Accepted: 5.01.2023



Fig. 1. The microstructure of two types of obtained multilayer Cr/CrN coatings with 10 (a) and 14 sublayers (b)

Rys. 1. Mikrostruktura dwóch rodzajów wielowarstwowych powłok Cr/CrN złożona z 10 (a) oraz 14 podwarstw (b)

tective layer for the blade is therefore not a proper protection of the substrate, due to the leakage of the coating, which results in the occurrence of pitting corrosion in the areas of defects. The solution to this problem can be the design of a sealed TiN coating or the application of a sealing interlayer to prevent corrosion by chloride ions. A significant improvement in the corrosion resistance of the protective layer was achieved by producing an additional 3 μ m thick platinum layer [5].

Sand and dust carried with the air hit the surface of the compressor blades, especially the leading edge, at different angles and with different energy levels. Large particles hit the blades mainly at 90° and are crushed at the leading edge into dust, which abrades the blade material even more intensively at the leading edge at small angles of 20–30°. The variation of abrasive impact angles indicates that it is necessary to use multilayer coatings that show better resistance than single-layer coatings. The erosion resistance of PVD-produced Ti/TiN multilayer coatings was found during abrasive impact tests. Reduced erosion wear was achieved by using a multilayer coating with a total thickness of about 25 µm built from alternating soft layers, made of titanium, and hard layers, containing titanium nitrides. Manufactured coatings based on multilayer structure with different hardness values significantly reduced erosion wear in laboratory tests [6, 7].

Arc-PVD multilayer coatings consisting of chromium nitride and chromium as an intermediate layer show high erosion resistance. Protection of aircraft engine compressor blades with coatings produced by the PVD arc method is the subject of many works. In addition to Ti-derived and Ti/TiN coatings, chromium nitride coatings of multilayer structure are also studied [8]. The properties of chromium nitride coatings make them gain more corrosion resistance than other nitride thin films such as TiN. This is due to the high adhesion to the substrate, achieved by using a layer of pure chromium as an interlayer, and also due to the dense fine-crystalline structure, which is less porous than titanium nitride. The Cr zones result in very good adhesion for the subsequent layers applied, allowing Cr/CrN coatings up to several μm thick to be deposited. In addition, it is possible to increase the tightness of the coating itself by adding interlayers of chromium or aluminum. Chromium coatings deposited by the ARC-PVD arc method show the highest adhesion to the steel substrate, reaching up to 80 N, and, moreover, provide an excellent bonding for subsequent layers of much harder chromium nitride in multilayer Cr/CrN coatings.

Coatings consisting of alternating zones of hard nitride and soft chrome adapt to current operating conditions. The relatively soft chromium zones in the chrome leading edge area suffer less damage, while the hard nitride zones on the trailing edge provide effective erosion protection. The properties of the Cr/CrN multilayer coatings have been confirmed by engine tests conducted, which also showed significantly inferior protective properties of Ti/TiN coatings [9]. In the industrial application the Cr/CrN coatings were applied for protection of different parts against wear [10] and corrosion resistance [11]. They can be uses for manufacturing of coatings for die casting moulds [12]. It might be also used for compressor blades in jet engines [13]. The multilayer Cr/CrN coatings were commercially produced for this type of application by lon Galenica Company. In presented article the potential using of commercially available coatings for compressor blades were initially investigated.

2. Experimental

The aim of this study was to determine the influence of deposition conditions on the microstructure and properties of a Cr//CrN-type coating for potential use on aircraft engine compressor blades, based on multilayer arc deposition (Arc-PVD) coatings produced under industrial conditions at Ion Galenica Company. Two coating variants differing in the number of layers were used for comparison. The test material was 1H13 grade steel (1.4006). The samples were in the form of flat sheets with maximum dimensions of $30 \times 10 \times 0.5$ mm. The specimens were cut to the appropriate size, and then they were ground using F500-grade waterproof abrasive paper with a carbide-silicon grain bonded with synthetic resin. The samples prepared in this way formed the substrate for the deposited coatings. This stage consisted of the process of applying two coatings:

- Cr/CrN with a multilayer structure amounting to 10 \times Cr/CrN,
- Cr/CrN with a multilayer structure amounting to $14 \times$ Cr/CrN.

Accordingly placed samples were placed in the Domino L device of the manufacturer Oerlikon Balzers GmbH. Samples intended for microstructure and chemical composition studies were cut transversely to the surface on which the coating was deposited, inlaid in a metallographic press and polished. The cross sections of PVD coating was observed by using a ZEISS Crossbeam 350 scanning electron microscopes (SEM, Jena, Germany). In the article the average thickness of coatings and sublayers was presented. The EDS area and linear mapping were analysed with X-ray energy dispersion spectrometer (EDS) from Ultim Max Oxford Instruments (AZtec 5.1 Oxford Instruments NanoAnalysis software; Abingdon, United Kingdom). The beam accelerating voltage during microscopic observation was 15 kV. Adhesion testing of the obtained coatings was carried out by scratch test using the RST CSM Revetest Scratch Tester. The erosion resistance test was carried out on a bench equipped with an Air Jet Erosion Tester TR-470. This device consists of an abrasive feeder, a jet nozzle and an acceleration section. The abrasive used was electrocorundum, grain size F240, fed at a pressure of 0.6 kg/cm² and a quantity of 3.5 g/min. The test used grips to adjust the angle of impact of the abrasive particles: 30°, 60° and 90°. To determine the degree of erosion wears, the samples were weighed before and after 2, 4, 6, 8, and 10 minutes of the test.

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Fig. 4. Linear analysis of the relative concentration of Fe, Cr and N on the cross-section of 10 × Cr/CrN (a) and 14 × Cr/CrN coatings (b) Rys. 4. Wyniki liniowej analizy stężenia względnego Fe, Cr i N na przekroju 10- (a) i 14-warstwowej powłoki Cr/CrN (b)

3. Results and discussion

3.1. Microstructure and chemical composition of Cr/CrN coatings

The coating, which consisted of 10 layers Cr/CrN (marked '10 × Cr/CrN'), had a total thickness of 2.59 μ m (Fig. 1a). The coating showed very good adhesion to the substrate and no visible pores or microstructural defects in the form of micro-droplets, which is a characteristic phenomenon of Arc-PVD technology. Microscopic examination of the 10×Cr/CrN coating showed a multilayer structure, with a single chromium layer averaging 306 nm and a chromium nitride layer averaging 232 nm. Relative concentration ana-lysis from the coating

area showed chromium and nitrogen in the coating area and iron below (Fig. 2). There was a striation in the concentration of chromium and nitrogen in the coating, which was further confirmed by linear chemical composition analysis (Fig. 4b). The Cr/CrN sublayers as well as total thickness of obtained coating were much more lower in comparison witch coating investigated by Naveed, Obrosov, and Weiss [13]. The phase composition of this type of coating was identified in a previously published paper [14].

The coating, consisting of 14 Cr/CrN layers, had an average thickness of 1.9 μ m. The coating showed very good adhesion to the sample substrate and no visible pores or other microstructural







Fig. 6. Weight loss during erosion testing of $10 \times$ Cr/CrN and $14 \times$ Cr/CrN coatings at impact angles of 30° (a), 60° (b), and 90° (c)

Rys. 6. Spadek masy próbek z 10- i 14-warstwowymi powłokami Cr/CrN przy kątach oddziaływania strumienia 30° (a), 60° (b) i 90° (c)

defects (Fig. 1b). Analysis of relative elemental concentrations indicates, as in the case of the 10-layer coating, an elevated concentration of chromium and nickel in the coating and iron below (Fig. 3). Similarly, the banded concentration of chromium and nitrogen in the PVD coating was also found in both the maps and the results of linear chemical composition analysis (Fig. 4b). The thickness of produced $14 \times$ Cr/CrN coating was smaller than typically proposed for this application [15]. From the other hand Smolik et al. [16] proposed the 16-layer coating with total thickness about 5.6 µm deposited on titanium alloy.

3.2. Results of scratch test

The highest value of the critical load L_{C1} for the 10 × Cr/CrN coating is 18.17 N, which indicates the occurrence of cohesive failure (Table 1). The absence of an L_{C2} emission signal indicates that the coating has not delaminated from the substrate (Fig. 5). For the 14 × Cr/CrN coating, the critical load of L_{C1} was 12.24 N, delamination of the coating occurred at a force of 17.20 N, and complete removal of the coating from the sample surface occurred at a force of 17.70 N.

Table 1. Values of measured critical forces for 10 \times Cr/CrN and 14 \times Cr/CrN coatings

Tabela 1. Wartości zmierzonego obciążenia krytyczr	1ego dla 10- i 14-warst-
wowej powłoki Cr/CrN	

Coating	<i>L</i> _{C1} [N]	L _{C2} [N]	L _{C3} [N]
$10 \times Cr/CrN$	18.17	-	-
$14 \times Cr/CrN$	12.24	17.20	17.70

3.3. Erosion resistance

The erosion mechanism of surface of compressor blades is very complex according to different angles of solid particle impact [6–8, 17]. The PVD multilayer coatings such as TiN/Ti are characterized by better erosion resistance than other types of coatings produced by electroplating or slurry methods [18–20].

Both types of Cr/CrN multilayer coating were subjected to an erosion resistance test for angles of 30°, 60° and 90° to simulate the impact of sand particles on different areas of a compressor blade with complex geometry. Due to the presence of the coating, it was not possible to perform measurements according to ASTM G73 and only comparative tests with uncoated material. The results obtained for all analysed abrasive impact angles indicate a linear weight loss of samples with both coatings and the substrate material itself (Fig. 6). The highest intensity of erosive wear was observed - as expected - for the substrate: 1H13 steel. When Cr/CrN coatings were applied, erosion wear was lower. The lowest values were found for the 10-layer Cr/CrN coating. Significantly, the measurement of total weight loss showed no significant differences for higher powder impact angles, i.e. 60° and 90°, between the two types of coatings and the substrate material (Fig. 6b, c). However, the use of both 10-and 14-layer Cr/CrN coating was found to provide a weight loss reduction of about 50% compared to the uncoated sample.

4. Summary

The study of the microstructure of the coatings made it possible to demonstrate the multilayer structure of the produced coatings. The Arc-PVD technology enables to form multilayer structure of coating for compressor blades application [21]. For the 10-layer coating, the total film thickness averaged 2.59 µm, while the thickness of a single chromium interlayer averaged 306 nm, and the chromium nitride layer averaged 232 nm. The coating growth rate was 51 nm/min. For the 14-layer coating, the average thickness was 1.09 μ m, giving a coating growth rate of 23 nm/min for the same layer application length. Due to the small thicknesses of the interlayers, it was not possible to study their chemical composition, but only the mixed region composed of Cr and CrN compounds, obtaining average values. The different kinetics of coating growth has to do with the cyclic switching of process parameters, and thus with the lower stability of deposition conditions and finally – influence of coating microstructure [22–24].

The adhesion strength of X-Cr/CrN coatings to the 1H13 steel substrate was carried out using the standard sample scratching method, which is used to test PVD coatings measuring several micrometers in thickness. The results showed that for the $14 \times Cr/CrN$ coating, the critical force was 12.24 N, while tests of the $10 \times Cr/CrN$ sample showed no loss of adhesion to the substrate, and no acoustic emission signal was recorded. According to ref. [25, 26] it might be concluded that multilayer structure improves the scratch resistance in comparison with single-layer coatings. The measured good adhesion of obtained coating is the important factor for improving of erosion resistance of coating [13].

Conducting the test allowed us to conclude that the erosion wear resistance of the $10 \times Cr/CrN$ -coated samples is higher than that of the $14 \times Cr/CrN$ coating and the uncoated sample. The modelling of multilayer structure of Cr/CrN coatings conducted by Ratajski et al. [10] showed that their geometry (the thickness of the individual layers of the module) determines the their mechanical properties. The presence of more Cr bands in the coating may have contributed to faster erosion through peeling and undercutting of successive thin layers [27].

The initial results of developed coating showed that chromium nitride-based coatings can provide protection for aircraft engine blades against the damaging effects of environmental factors, i.e. sand [3, 6–9, 17–21]. In further research it will be necessary to perform corrosion tests on the developed coatings [13, 28]. The results obtained can provide a basis for conducting further research on coating modification at lon Galenica company and their application in Polish aerospace industry.

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