

Structure of gradient coatings deposited by CAE-PVD techniques

L.A. Dobrzański ^{a, *}, M. Staszuk ^a, J. Konieczny ^a, J. Lelątko ^b

^a Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland ^b Institute of Materials Science, Faculty of Computer and Materials Science, University of Silesia, ul. Bankowa 12, 40-007 Katowice, Poland * Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received 17.04.2007; published in revised form 01.10.2007

Materials

ABSTRACT

Purpose: The main aim of this research was investigation of the gradient and multicomponent coatings structure deposited by cathode arc evaporation physical vapor deposition (CAE-PVD) on a SiAlON substrate. **Design/methodology/approach:** The structure of investigated coatings was characterized by scanning and transmission electron microscopy. Chemical composition was determined by energy dispersive spectroscopy (EDS) method. Investigation of surface roughness was done.

Findings: The results of the investigations of microstructure, chemical compositions and surface morphology of gradient TiAlN and multicomponent AlSiCrN coatings deposited by PVD techniques are given in the paper. The linear analyses of chemical compositions on cross-sections of the investigated coatings are given. In the gradient coatings the chemical composition changes continuously at the cross-section. The result of the investigations of thin foils performed witch use of transmission electron microscope and selected a diffraction are given also.

Research limitations/implications: In future the examination will progress for mechanical properties, e.g. microhardness, adhesion strength and abrasive wear resistance as well as for the technological machining test. **Originality/value:** Future examinations will progress for mechanical properties, e.g. microhardness, adhesion strength and abrasive wear resistance as well as technological machining tests.

Keywords: Tool materials; PVD coatings; TEM; SEM

1. Introduction

Sialons were worked out and put into industry at last decades of the 20th century. Moreover sialons combine advantages of oxide ceramic and oxygen-free ceramic which include Si_3N_4 . The tools which are made of them are used for rolling and milling of steel and hard to machine alloys such as: cast irons, improved thermal steels, nickel alloys, titan, aluminium and high-temperature creep resisting alloys [1-3].

Industrial applications of the gradient coatings synthesized

by physical vapor deposition (PVD) are increasing rapidly due to its advanced tribological properties, decrease of friction between tool and work-piece during working. Hard coatings of the metal nitrides increase life of elements coated by them. Deposition of wear resistance hard coatings based on transition metals nitrides characterizes the fast developing directions of research, stimulated by the growing service requirements of machines and equipment [3-8].

Gradient coatings are different multilayer coatings the chemical composition and properties of which are changing continuously on the cross-section.

It is possible to transfer metal locally into a vapour phase by means of cathode arc evaporation PVD process without necessity of totally melting the cathode. With use of this method of local arc evaporation one can compose easily any chemical composition of coatings. On the other hand, it has been proved that the gradient coatings have a useful columns structure without pores and discontinuity and good adhesion to the substrate. These coatings deposited for method CAE are widely used in industry cutting tools without application of cooling and lubricating liquids. The multiedge plates, which are covered with gradient coatings, have higher hardness of edges in comparison with plates, which are covered with single or multilayer PVD coatings [3-6, 9-16].

2. Methodology

Experiments were carried out on the multi-point inserts made from SiAlON with gradient and multicomponent PVD coatings, TiAlN and AlSiCrN type deposited by cathode arc evaporation process.

The roughness measurements of substrate surface and of the samples with wear-resistant coatings was measured by Surftronic 3+ profile measurement gauge. Investigation were made on gauge length of a test piece $L_c = 0.8$ mm with an accuracy of $\pm 0.02 \mu$ m.

Observation of structure and topography of surface coatings was carried out by using scanning electron microscopes ZEISS LEO 1525 and OPTON DSM 940. To obtain the topography and fracture images the Secondary Electrons (SE) and Back Scattered Electrons (BSE) were used with the accelerating voltage 15-20 kV. To linear profiles of chemical composition was made with use of Energy Dispersive Spectrometry (EDS) method and with utilization of detector RONTEC.

The investigations of structure and diffraction of thin foils were made with use of the JEOL 3010 transmission electron microscope at the accelerating voltage of 300 kV. The electron diffractions from TEM were solved with use of Eldyf computer program.

3.Results

The analysis of the chemical composition of coatings was made with use of X-ray energy dispersive spectrometry on cross sections as a function of the distance from substrate surface. The research confirm the presence of the elements suitable for the investigations coatings (Figs. 1, 2). In both coatings the gradient changed concentration was observed. For the sake of small thickness deposited coatings and diameter of incident electron beam about 1 μ m linear chemical analysis obtained by EDS method is only orientational.

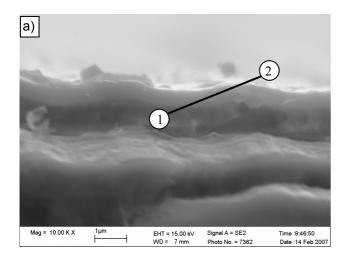
Examinations of thin foils parallel to surface coatings by means of transmission electron microscopy revealed that (Ti,Al)N phase in TiAlN coating with fine-graded structure and tetragonal crystal lattice from P42/mnm at the space group (Fig. 3). Parameters of the lattice are a=b=0.49454 nm and c=0.30342 nm. AlSiCrN coating (Cr,Al)N phase with cubic lattice (Fm3m type) also with fine-graded structure (Fig. 4) has been found. Parameters of this lattice are a=b=c=0.414 nm.

The roughness measurements have shown that the least parameter value R_a =0.12 exists for uncoated substrate. After deposition of PVD coatings the roughness increases (Tab. 1). Observation of the coatings surface topography by SEM has shown the occurrence of metal micro-drops due to intense local evaporation provoked by microarcs (Fig. 5). The size of the droplets is changing in a rather with range from decimal to a few micrometers.

Table 1.

Specification of the samples investigated in the work

Material	Roughness [µm]	Coating thickness [µm]
Sialon substrate	0.12	-
TiAlN coating	0.21	0.6
AlSiCrN coating	0.25	0.8



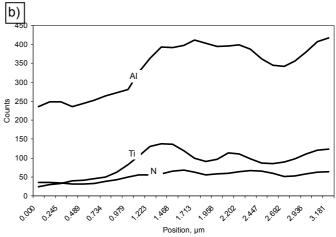


Fig. 1. a) SEM micrograph of structure in TiAlN sample; b) changes chemical composition in function of distance

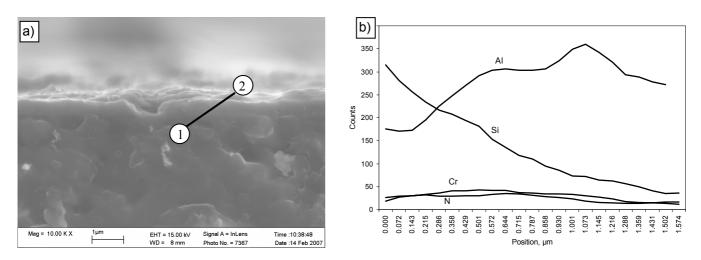


Fig. 2. a) SEM micrograph of structure in AlSiCrN sample; b) changes chemical composition in function of distance

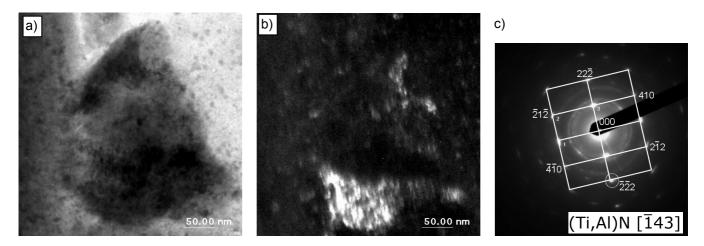


Fig. 3. Structure of TiAlN coating: a) bright field; b) dark field from figure a; c) diffraction pattern from area b and solution of the diffraction pattern

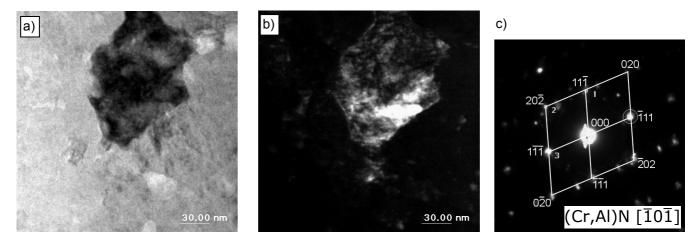


Fig. 4. Structure of AlSiCrN coating: a) bright field; b) dark field from figure a; c) diffraction pattern from area b and solution of the diffraction pattern

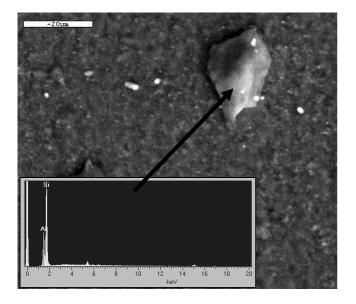


Fig. 5. Topography of the AlSiCrN coating surface and X-ray energy spectrum from area of drop

4.Conclusions

The results of the investigations of the structure of wearresistant gradient TiAlN and multicomponent AlSiCrN coatings deposited by cathode arc evaporation process are given in the paper. Fine-graded coating structure was confirmed by TEM studies. Electron diffraction of thin foils of investigated coatings shows, that (Ti,Al)N phase exists in TiAlN coating and (Cr,Al)N in AlSiCrN coating. Examination of chemical composition confirmed gradient changes of concentration of elements in investigation coatings.

Acknowledgements

The investigations were carried out within the projects financed by State Committee for Scientific Research (KBN) PBZ-100/4/T08/2004 headed by Prof. L.A. Dobrzański.

References

[1] L.A. Dobrzański, Engineering materials and materials design. Fundamentals of materials science and physical metallurgy, WNT, Warsaw, 2006 (in Polish).

- [2] H. Mandal, New developments in α -SiAION ceramics, Journal of the European Ceramic Society 19 (1999) 2349-2357.
- [3] M. Kupczyk, Surface engineering. Wear resistant coatings for cutting edges, Poznań University of Technology Publishing House, Poznań, 2004 (in Polish).
- [4] L.A. Dobrzański, K. Lukaszkowicz, J. Mikuła, D. Pakuła, Structure and corrosion resistance of gradient and multilayer coatings, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 75-78.
- [5] L.A. Dobrzański, K. Gołombek, J. Mikuła, D. Pakuła, Improvement of tool materiale by deposition gradient and multilayers coatings, Journal of Achievements in Materials and Manufacturing Engineering 19/2 (2006) 86-91.
- [6] L.A. Dobrzański, K. Gołombek, Gradient coatings deposited by Cathodic Arc Evaporation: characteristic of structure and properties, Journal of Achievements in Materials and Manufacturing Engineering 14 (2006) 48-53.
- [7] J. Łaskawiec, Surface engineering, Silesian University of Technology Publishing House, Gliwice, 1997 (in Polish).
- [8] Yin-Yu Chang, Da-Yung Wang, Chi-Yung Hung, Structural and mechanical properties of nanolayered TiAlN/CrN coatings synthesized by a cathodic arc deposition process, Surface & Coatings Technology 200 (2005) 1702-1708.
- [9] T. Burakowski, T. Wierzchoń, Engineering of metal surface, WNT, Warsaw, 1995.
- [10] L. Cunha, A.C. Fernandes, F. Vaz, N.M.G. Parreira, Ph. Goudeau, E. Le Bourhis, J.P. Riviere, D. Munteanu, F. Borza, Characterisation of TiC_xO_y thin films produced by PVD techniques, Journal of Achievements in Materials and Manufacturing Engineering 21/1 (2007) 35-38.
- [11] T. Wierzchoń, Structure and properties of multicomponent and composite layers produced by combined surface engineering methods, Surface and Coatings Technology 180-181 (2004) 458-464.
- [12] L.A. Dobrzański, L. Wosińska, K. Gołombek, J. Mikuła, Structure of multicomponent and gradient PVD coatings deposited on sintered tool materials, Journal of Achievements in Materials and Manufacturing Engineering 20/1-2 (2007) 99-102.
- [13] M. Kupczyk, Influence of coating thickness on tool life and wear, Surface and Coating Technology 60 (1993) 446-449.
- [14] Y. Wang, A study of PVD coatings and die materials for extend die-casting die life, Surface and Coatings Technology 94 (1997) 60-63.
- [15] M. Parlinska-Wojtan, A. Karimi, T. Cselle, M. Morstein, Conventional and high resolution TEM investigation of the microstructure of compositionally graded TiAlSiN thin films, Surface and Coatings Technology 177-178 (2004) 376-381.
- [16] L.A. Dobrzański, K. Lukaszkowicz, D. Pakuła, J. Mikuła, Corrosion resistance of multilayer and gradient coatings deposited by PVD and CVD techniques, Archieves of Materials Science and Engineering 28/1 (2007) 12-18.