Structure of gradient coatings deposited by CAE-PVD techniques

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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 Si₃N₄. 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.

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
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 \( \pm 0.02 \) µm.

Observation of structure and topography of surfaces 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 µm 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/mmm 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

<table>
<thead>
<tr>
<th>Material</th>
<th>Roughness [µm]</th>
<th>Coating thickness [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sialon substrate</td>
<td>0.12</td>
<td>-</td>
</tr>
<tr>
<td>TiAlN coating</td>
<td>0.21</td>
<td>0.6</td>
</tr>
<tr>
<td>AlSiCrN coating</td>
<td>0.25</td>
<td>0.8</td>
</tr>
</tbody>
</table>

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
4. Conclusions

The results of the investigations of the structure of wear-resistant 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.

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References