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PVD multi-layer hard coatings — Composition, structure and properties

Revêtements durs multicouches déposés par PVD — Composition, structure et propriétés

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Foreword

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, SC 9, *Physical vapor deposition coatings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Multi-layer hard coatings by physical vapor deposition (PVD), which possess high coating-substrate adhesion, high hardness and good wear resistance, are widely applied on tools and machine parts to improve their service life. Based on the chemical compositions, the mainstream PVD multi-layer hard coatings in the market involve transition metal nitrides and carbides, such as Ti/TiN, TiN/CrN, CrN/AlCrN, TiC/TiCN and CrAlN/AlCrTiSiN. To date, there has been no standard to qualify the composition, structure and properties of these multi-layer hard coatings, which has limited their further development.

This document defines the measurement and evaluation of the composition, microstructure, surface quality, thickness, hardness and tribological properties (such as friction and wear performance) of multi-layer hard coatings. The methods are for the purpose of coating development. Where standards for quality assurance in production exist, they are referred to in this document.

PVD multi-layer hard coatings — Composition, structure and properties

1 Scope

This document specifies the evaluation standard of the composition, structure and properties of multi-layer hard coatings by common physical vapor deposition (PVD), indicating a vacuum deposition method that produces a material source by evaporation, sputtering or related non-chemical ways.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4545-1, *Metallic materials — Knoop hardness test — Part 1: Test method*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 9220, *Metallic coatings — Measurement of coating thickness — Scanning electron microscope method*

ISO 14577-1, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 20808, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of friction and wear characteristics of monolithic ceramics by ball-on-disc method*

ISO 26423, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of coating thickness by crater-grinding method*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Samples for composition, structure and properties evaluation

Samples for the composition, structure and properties evaluation should be coated in the same batch as the products requiring the composition, structure and properties evaluation. The samples should be polished to a mirror finish (R_{pk} < 0,05 µm) before being coated and cleaned using ultrasonic agitation, which immerses them in the correct solution to remove hydrocarbons and other surface contaminants.

5 Testing of composition, structure and properties

5.1 Testing of chemical composition

The chemical composition of PVD multi-layer hard coatings is decided by many factors, including the composition of the evaporator source, the energy density of incident atoms/ions, the deposition pressure and the bias voltage. Various elements in the evaporator source can segregate during deposition, which results in different contents in the coatings. Testing methods that can be used to characterize the chemical compositions of PVD multi-layer hard coatings are energy dispersive spectrometer (EDS), electron probe micro analysis (EPMA), X-ray photoelectron spectrometer (XPS), auger electron spectrometer (AES), secondary ion mass spectrometry (SIMS), X-ray fluorescence (XRF) and glow discharge optical emission spectroscopy (GDOES). The details are shown in Table 1.

Table 1 — Testing methods of chemical compositions of PVD multi-layer hard coatings

Testing method	Surface area		Cross-sectional area		Maps and line scans
	Metal elements	B, C, N and O elements	Metal elements	B, C, N and O elements	
EDS	Recommended (excluding Li and Be)	Recommended (excluding B and C)	Recommended (monolayer thickness more than 100 nm)	Recommended (excluding B and C)	Recommended
EPMA	Recommended	Recommended	Recommended	Recommended	Recommended
XPS	Recommended	Recommended	Recommended (only by etching)	Recommended (only by etching)	Recommended (destructive)
AES	Recommended	Recommended	Preferably recommended	Preferably recommended	Recommended
SIMS	Preferably recommended	Preferably recommended	Preferably recommended	Preferably recommended	Recommended (destructive)
XRF	Recommended	—	—	—	—
GDOES	Preferably recommended	Preferably recommended	Preferably recommended (only by etching)	Preferably recommended (only by etching)	Recommended (destructive)

5.2 Testing of layer structure

Different structures of PVD hard coatings observed by electron microscope, including columnar crystal, equiaxed crystal and amorphous, lead to different grain or crystallite types, boundary energy and texture, which influence their hardness, internal stress, toughness and adhesion. Therefore, structure testing is essential for coating evaluation.

PVD multi-layer hard coatings can be defined in two classes. The first class comprises several different layers consecutively, including the adhesive layer, transition layer, hard core layer and/or surface adaptive layer for lubrication, hydrophobicity, electroconductivity, etc, as shown in Figure 1 a). The other class comprises two kinds of layers, in which every two adjacent layers constitute a unit and the thickness is called the "modulation period" ($\Lambda = \lambda_A + \lambda_B$; λ_A and λ_B are the thickness of the A layer and B layer, respectively). It is called "nano-layered coating" when Λ is less than 100 nm or "super-lattice coating", as shown in Figure 1 b).

Methods such as SIMS, scanning electron microscope (SEM) and transmission electron microscope (TEM) are able to detect and confirm the layer structure of coatings. Detailed information about analysing the layer structure by TEM is given in Annex A.

a) **Several different layers** b) **Periodic structure**

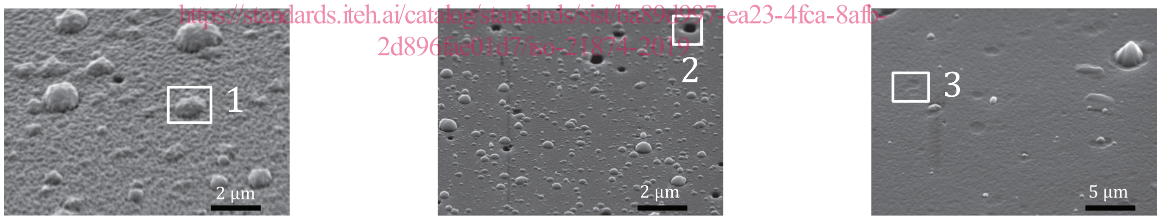
Key

1	adhesive layer	4	modulation layer A
2	transition layer	5	A layer
3	hard core layer	6	B layer

Figure 1 — Typical cross-section structures of PVD multi-layer hard coatings

5.3 Testing of surface deficiency

Droplets often occur in PVD hard coating processes. In addition, defects such as pinholes and shallow craters can form on the coating surface for some PVD techniques. Figure 2 shows the droplets, pinholes and shallow craters for arc ion plated coatings. The droplets are mainly from unreacted metal particles. The pinholes are from the shrinking of grain or crystallites during the coating growth. The shallow craters are from a spallation of the droplets.



a) b) c)

Key

1 droplet

2 pinhole

3 shallow crater

Figure 2 — Droplets and defects on the surface of arc ion plated coatings

These droplets and defects affect the mechanical properties, such as hardness, friction and wear of hard coatings, further influencing their service performance. Therefore, it is necessary to calculate the deficiency rate on the coating surface.

Surface deficiency analysis can be applied to evaluate the surface quality of coatings. Deficiency rate is defined as the percentage of the area composed of droplets, pinholes and shallow craters divided by the total observing area. A lower deficiency rate means better surface quality. A maximum value of 10 % for the deficiency rate should be an acceptable performance of polished coatings.

