ISSN 1318-0010
KZLTET 31(6)529(1997)

METHODS FOR THE VALIDATION OF ADVANCED THIN HARD PROTECTIVE COATINGS - AN EUROPEAN PROGRAM

METODA ZA VALIDACIJO NAPREDNIH TANKIH ZAŠČITNIH PLASTI - EVROPSKI PROGRAM

PETER MAYR, H. VETTERS, A. SCHULZ

Stiftung Institut für Werkstofftechnik, Bremen, Badgasteinerstr. 3 D, 28359 Bremen

Prejem rokopisa - received: 1997-10-01; sprejem za objavo - accepted for publication: 1997-10-21

Specific surface engineering technologies are widely used to improve decisively product characteristics whereas thin hard coatings are increasingly adopted in a broad range of industries. The quality assurance infrastructure for coated products is essential to determine their functional properties. For the reliable assessment of the intrinsic properties (coating thickness, hardness, chemical compositon, adhesion) as well as the tribological performance standardised test methods are required. International groups from research institutes and industries are working on this actual topic to develop comprehensive test methodologies including instrument calibration and reference samples. These will then be proposed to committees for standardisation for adoption as European standards.

Key words: thin hard coatings, characterisation methods, properties, standards

Specifične površinske inženirske tehnologije se široko uporabljajo za povečanje karakterističnih proizvodov in tanke trde prevleke se uporabljajo v širokem spektru te industrije v naraščajočem obsegu. Zagotovitev kakovsti infrastrukture za prekrite proizvode je bistvena za določitev njihovih funkcionalnih lastnosti. Za zanesljivo opredelitev specifičnih lastnosti (debelina, trdota, kemijska sestava in adhezija prekritja) in triboloških lastnosti so zahtevane standardne testne metode. Mednarodne skupine iz raziskovalnih institutov in iz industrije delajo na tej aktualni temi s ciljem, da razvijejo razumljive metodologije za testiranje vključno s kalibracijo instrumentov in referenčnimi etaloni. Te bodo kasneje predložene komitetom za sprejem kot evropski standardi.

Ključne besede: tanke trde prevleke, metoda karakterizacije, lastnosti, standardi

1 SURFACE ENGINEERING GAINING ADVANCED THIN HARD PROTECTIVE COATINGS

Specific surface engineering technologies are widely used to improve decisively product characteristics. Thin hard coatings are increasingly adopted in a broad range of industrial sectors as a means of achieving advanced products. The mechanical properties of these advanced coatings will determine the service life because elastic and plastic behaviour control fracture and adhesion of the coating. The use of wear resistant coatings can lead the retention of better tolerances through the lifetime of tools or machine parts. In all these cases mechanical reliability requires optimum matching of the different components to be "assembled", that can only be obtained by model calculations requiring precise knowledge of the characteristics and functional properties[1]. It is established that surface coatings and treatments can lead to considerable improvements in the performance of a wide range of components. Modern PVD and CVD hard coating techniques often allow for alternative, environmentally friendly solution to wear protection[2-4].

2 QUALITY ASSURANCE - A KEY OBJECTIVE

As reported[5,6], a broad selection of materials is being offered for ceramic coatings. When selecting a coating for certain application, the substrate, the interface and possible interlayer and the environment influence strongly the on service condition of the coated part. A wide variety of coating procedures leads to a wide range in functional properties, which can be classified by methods of test[7]. In order to allow industry to take full advantage of this rapidly developing area it is essential to establish a quality assurance infrastructure for coated products by standardised test methods. In view of fostering international collaboration between research organisations, standard bodies and industry on pre-standardisation research in advanced materials the "Versailles Project on Advanced Materials and Standards" (VAMAS) is fulfilling a unique role. Within several technical working areas (TWA) formal links with international standards organisations and increasing industrial involvement have been formed. In fact one activity on developing methods of test of thin hard coatings is now established[9]. The European Committee for Standardisation (CEN) has also established a technical committee on ceramic materials with a working group "Methods of test of ceramic coatings" (CEN TC 184/WG 5).

3 CHARACTERISATION OF PROPERTIES

Many methods exist for the measurement of properties of coatings **(table 1)**, but for thin hard coatings is a great paucity of standards. Improved test methods, therefore need to be developed or the existing procedure should be formalised and draft standards prepared. The test routine should lead to a subsequent process control during the production routine which includes preliminary quality control as well as quality control after the coating procedure. Their properties are defined as: hardness, residual strain, composition, morphology, microstructure, thickness of coating, elasticity, electrical conductivity, magnetic susceptibility, thermal conductivity, etc., and on service conditions are indicated by wear and corrosion resistance, fatigue and life time oriented behaviour, thermoshock stability, and other characteristics[8-10].

Table 1: Characterisation of reference samples

composition bulk materials	spectroscopy, wet chemical analysis
composition of coatings	X-ray fluorescence, EPMA, AES, GDOS, SIMS, SNMS
thickness of coatings	cap grinding, cross-sectioning, X-ray diffraction, step height measurement, NDT-test methods
roughness	3D profilometry
microstructure	optical microscopy, SEM, TEM, XRD phase-analysis
grain size and texture	XRD, quantitative metallography
coating internal stress	X-ray diffraction, flexural strain, Rayleigh wave analysis
density	weighting and volumina evaluation
hardness, hardness gradient	indentation method
adhesion of coatings	scratch testing, indentation method
modulus of elasticity	ultrasonics, acoustic microscopy

4 THE VALIDATION OF METHODS OF TEST

In practice, many of the qualitative tests are used and recommended, because they are suitable for several coating materials, simply to handle and commonly based on visual examination[13]. Because of limited applicability of the standard pull test a modified shear test is recommended for the test of adhesion of ceramic coatings[12]. In practice often accepted is the Rockwell indentation test[13] for the qualitative examination of thin hard coatings, where the remaining plastic deformation after indentation causes a "spalling off" of the layer, so an area of failure remains. But in spite of reference samples producing calibration data under reproducible measurement conditions this method of test is not valuable until now to be adapted as a standard.

4.1 Reference specimens for calibration

Calibration requires the production of an ensemble of well characterised specimens with hard coatings of perfectly controlled chemical composition, microstructure, and thickness uniformity, likely to become certified ultimately as reference specimens. The following methods are used to assess these properties.

4.2 Measurement of the coating thickness

For the measurement of the coating thickness the methods of stylus profilometry[14] and cap grinding[15] are employed. A further development of the cap grinding method allows the in-situ detection of the coating relief during abrasive extraction[16]. This method can also be modified for the determination of the coating resistance against abrasive wear under defined friction control.

4.3 Measurement of the chemical composition

Chemical analysis[23] was carried out from various layers of defined (N/Ti)-composition which has been realised by reactive magnetron sputtering with nitrogen where the N-concentration has been varied by trimming the gas flow[18]. The spectrochemical analysis of the layer led to a constancy of the composition of ±1% atomic%, which has been certified by reference labs[18].

4.4 Measurement of the coating hardness

It was found by round robin comparison that existing standards methods of hardness tests on hard coatings are rarely valuable for certification. Hardness measurement by load-indentation[18,19] on TiN coated test plates of tool steel showed under certain conditions, regarding the surface quality, chemical homogeneity, smooth interface morphology, hard substrate material, reproducible hardness values of 20 GPa by means of a test force between 250 mN (HU 0.25) and 1 N (HU 1) within an error width of less than ±2,5% of the value as determined above. These platelets, described in[18], could act as calibration samples for hardness measurement.

The surface morphology, respectively the surface roughness, plays an important role on accuracy and reproducibility. Doubtless, the calibration of the instruments, their stability and constancy during the test periods give a guarantee for exact results. Several procedures, especially for the nanohardness scale, have been developed and controlled through various round robin tests[19].

4.5 Adhesion of the coating

In the same way the influences on scratch test sensitivity and reproducibility of results are carried out. Obeying the whole system, stylus geometry and adhesive debris, surface quality, the existence of lubricants, humidity and temperature the function of influencing parameters is studied[22]. Then, using various coating/substrate composites of defined combination, scratch test failure modes are analyzed and various features catalogued. Improving the NDT techniques for in-situ control during process routines, ultrasonic test procedures are validated[20]. For thin hard films it will be realised by surface acoustic measurement techniques. The input of one specified research work is to enhance and calibrate the surface acoustic wave velocity measurement technique vis a vis reference coated substrate standards with the aim of developing a comprehensive test methodology for coating thickness. This measurement methodology will then be proposed to CEN for adoption as an European standard.

5 ASSESSMENT OF TRIBOLOGICAL PERFORMANCE

Engineering components often suffer from wear and friction resulting at best in energy inefficiencies and losses in performance, and in the worst cases catastrophic failure. There has been considerable recent interest in improving the tribological performance of components by the application of wear resistant coatings to the surface of the components[22].

For reliable assessment of the tribological performance of potential coating systems, robust and effective testing procedures are required. Testing of the cavitation erosion by ultrasonic affects in water can be estimated as a valuable method for standardisation[18].

Current friction and wear testing procedures are not sufficiently well defined to allow data comparisons, and evaluation of the significance of reported data is difficult. The work to be carried out in one prenormative research[22] involves the development of improved procedures for uniaxial sliding wear testing; reciprocating sliding wear testing and abrasive testing.

6 ASSESSMENT OF LIFE TIME OF COATED PARTS

Whatever the main technological function may be, in practice mechanical properties of the coating substrate compound will determine the life time with on service conditions because the interactive elastic and plastic behaviour of coating and substrate controls the fatigue endurance, fracturing conditions and the adhesion of the coating. At first, the aims of the research work conducted in one current program[22] are to develop and validate the methods of test to determine the elastic properties of the surface layer. In comparison to the tribological test procedures quantitative assessments based on scientific investigations due to structure and composition of reference samples are provided. These items will involve the development of reference materials and the conduction of round-robin intercomparisons.

7 REFERENCES

[1] D. S. Rickerby & A. Matthews: Advanced surface coatings, A handbook for surface engineering, Blackie, London, 1991

[2] S. Bull & D. S. Rickerby: Compositional, microstructure and morphological effects on the mechanical and tribological properties of chromium nitrogen films, Surface and Coatings Technology, 43/44 (1990) 732 -744

[3] E. Lugscheider, P. Jokiel: Particle reinforced coatings - processing and mechanical properties, Surface Engineering (ed. P. Mayr, Proc. Of the International Conf. On Surf. Eng. 1993, Bremen, FRG, DGM Informations GmbH Frankfurt/Oberursel ISBN 3-88355-189-9, 1993, 159-164

[4] K. Keller: Economical and ecological aspects as design criteria for coating processing, Surface Engineering (ed. P. Mayr, Proc. Of the International Conf. On Surface Engineering 1993, Bremen, FRG, DGM Informations GmbH Frankfurt/Oberursel ISBN 3-88355-189-9, 1993, 3-12

[5] D. S Rickerby et al.: The development of advanced erosion resistant coatings for gas turbine compressor applications, BRITE-EURAM Rep. BE-3339 Rep. (Brussels, 1994)

[6] H. Holleck: Binäre und Ternäre Carbid- und Nitridsysteme der Übergangsmetalle, Materialkundl, Techn. Reihe 6, Verl. Gebr. Borntraeger, Berlin, Stuttgart, 1984

[7] W. Gissler, H. Jehn: Advanced Techniques for Surface Engineering, Dordrecht, 1992

[8] S. Ronkainen et al.: Mechanics of Coatings (Ed. D. Dawson, C. M. Taylor, M. Godet) Elsevier Sci. Publ., 1990, 453-462

[9] S. R. J. Saunders: VAMAS (Versailles Project on Advanced Materials and Standards), Technical Working Area Project Initiation Form: Property Measurement of Thin Films and Coatings, Tokyo, Dec. 1992

[10] J. P. Celis et al: Corrosion of Electrodeposited, PVD and CVD Coated Metallic Materials, Surface Engineering (ed. P. Mayr, Proc. Of the International Conf. On Surface Engineering 1993, Bremen, FRG, DGM Informations GmbH Frankfurt/Oberursel ISBN 3-88355-189-9, 1993, 43-53

[11] European Envisaged Standard ENV 1071-5, Methods of Test of Ceramic Coatings: Determination of the Porosity, 1995

[12] ISO standard 2819, Metallic Coatings on Metallic Substrates - Electrodeposited and Chemically Deposited Coatings - Review of Methods for Testing Adhesion

[13] H. Jehn, G. Reiners: Charakterisierung dünner Schichten, DIN Fachbericht 39, Beuth Verlag GmbH., Berlin, Wien, Zürich, 1993, ISSN 0179-275X

[14] European Envisaged Standard CEN ENV 1071-1, Determination of the Coating Thickness; Step height measurement by means of a contact profilometer, 1992

[15] European Envisaged Standard CEN ENV 1071-2, Determination of the Coating Thickness; Cap grinding method, 1992

[16] H. Klümper-Westkamp: Prakt. Metallographie (German DGM, Spec. Vol. 26, 1994), Mikrotribometer zur Qualitätssicherung von Randschichten (Pat. DE-P 4434661.1) 433-442

[17] European Envisaged Standard CEN ENV 1071- 3: Characterization of adhesion: Scratch adhesion test, 1992

[18] A. Schulz, H.-R. Stock, H. Vetters and P. Mayr: Qualifizierung von Prüfverfahren und Standards für die Dünnschichttechnologie, Final Report Programme OSTEC 13N6242, (German Fed. Ministry of Education and Research BMBF, Feb. 1996)

[19] S. R. J. Saunders et al.: Hardness (Mechanical Property) Testing of Surfaces, NPL Report DMA (D) 198, BCR MAT CT 92 - 0027, 1996

[20] BRITE EURAM BE 4398: Non Contacting Surface Characterization of Ceramics and Coatings with Micro Acoustic Waves (C-MAW), Final Report R/CJG/1093/95, 1996

[21] K. Holmberg: Friction and Wear Mechanisms of Thin Surface Coatings on Metal Substrates, Surface Engineering (ed. P. Mayr, Proc. Of the International Conf. On Surf. Eng. 1993, Bremen, FRG, DGM Informations GmbH Frankfurt/Oberursel ISBN 3-88355-189-9, 1993, 13-22

[22] Development and Validation of Test Methods for Thin Hard Coatings (FASTE) EU- MS&T MAT1-CT 940045, Workprogramme, 1994 - 1997

[23] European Envisaged Standard CEN ENV 1071-4; Determination of the chemical composition, 1995