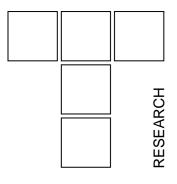
# Some Developments in Triboanalysis of Coated Machine Components



Surface engineering and coatings on surfaces of various auto- and machine components, bearings, piston rings, etc. have been since years proved to be a successful way of improving their performance [1-7].

However, quality of coated triboelements remains to be a subject of complex triboanalysis, using sophisticated methods of tribotesting, micro- and nanoindentation, micro- and nanoscratch, measurements of coating thickness and surface roughness, e.g. using non-contact confocal optical profilometry and related surface metrology techniques and software. Very often a physicist dealing with creating of novel deposition techniques and analyzing influences of plasma and high energy particles bombarding the solid surfaces on the properties of surface engineered components and a tribophysical background of their better performance. In tribology terms, plasma surface engineering and physical vapour deposition of coatings is playing a role of a control method for friction and wear, which has to be based on observation or measurement of output tribological parameters, characterizing a tribocouple and very exact selection of input parameters, aiming to provide the conditions, which would be maximally adequate for a concrete application of surface engineered components.

The goal of present paper is to create some guidelines to selection of an optimal set of analytical techniques and configuration for some specific equipment for triboanalysis of engineered surfaces and coatings, assisting research scientist to formulate clear tasks for an experimental investigation, to obtain the best possible instrumentation and finally to be able to carry out experimental research and collect the experimental data giving the answers on principle questions of engineering of tribological surfaces.

Keywords: surface engineering, triboanalysis, nano, micro indentation, scratch, roughness.

#### 1. SCHEMES OF TRIBOTESTING AND OPTIMAL CONFIGURATION OF TESTERS

Comprehensive testing of machines and equipment, is usually a multilevel and multiscale task [8-15], involving the field tests, bench tests, tests of specific units and components, whereas the last would be always subdivided into natural tests of real components and simplified elements, modeling a real tribology contact. On the other hand, versatile tribology systems in various industries are making a scientist, practically in all situations to keep in a mind some friction and wear fundamentals, including the laws of friction, describing physically, chemically and mechanically a real

Alexander A. Minewitsch TTZH Tribologie & Hochtechnologie GmbH, Germany E-mail: alexander.minewitsch@ttzh.com contact between moving solids [9, 13, 14, 16]. Despite some mutual effects in hard discs of computers, watches, orthopedical implants, autos, airplanes, machine tools, pumps, wind energy plants, etc., modeling of a specific contact situation, requires different approaches and surface analyses vary from nanometer to micrometer scale.

Bench tests we are presently focusing on can involve both model and real test of coated details in one and the same tester, having as a limitation some overall dimensions only. So, it would an applicability of mean maximum sizes of coated machine components like 150-250 mm, which actually covers a lot of applications, starting from nanotechnology and microtechnology and expanding to classical mechanical engineering on a macrolevel.

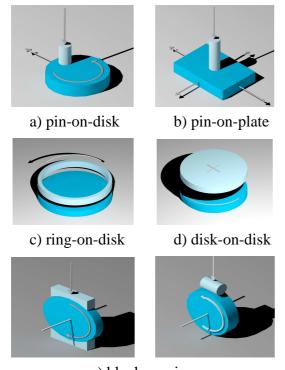
The initial questions for preparing a configuration of a tribotester are as follows:

- 1. How to select a model test, corresponding to a real situation?
- 2. What are the input parameters? Are they controllable with a sufficient accuracy?
- 3. Which parameters can be monitored? What is possible in situ (or in vivo) and ex situ?
- 4. How to create an affordable solution? Why a cheap solution could turn to become extremely expensive?
- 5. Where is a compromise between a universal testing machine and a special machine, performing only one specific function?

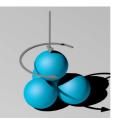
Really, there is always one more question in the minds "to test it yourself", i.e. to find out and to buy a tester for own use, or to involve a partner or a service company, specialized on tribometry and triboanalysis routinely. Both approaches have their advantages and disadvantages and final decision of this question, dependent on a lot of variables, is beyond the present paper.

## 2. REALISTIC SCHEMATIZATION

Realistic schematization of a tribosystem could involve several geometries like "pin-on-disk", "pinon-plate", "ball-on-disk", "ball-on-plate", "blockon-ring", "disk-on-disk", "parallel cylinders", "crossing cylinders", "4-ball" and some special ones, including various combinations of those mentioned above.



e) block-on-ring





f) 4-ball

g) crossing cylinders



h) parallel cylinders

Figure 1. Several typical schemes of tribological contact

Additional specific features are coming with the different sizes of the samples concerned, with their relative positioning, with their overlapping on the contact. Turning from geometry only to the materials aspects and choosing which of the samples (or maybe all available ones) have to be coated and which thickness of coatings (measured in micrometers or nanometers) of plasma modified, galvanic and different layers have to be taken into consideration bring more variables to our task.

## 3. KINEMATICS AND TYPES OF THE TRIBOSYSTEMS

Using a number of drives for providing the required relative movements of samples and counterbodies involve rotary, linear and reciprocating movements and combinations thereof.

Kinematically complicated tribotests can implement a combination of movements of both upper and lower specimens, this way providing an adequate modeling of a real situation in a configuration of a tribometer. For example, a rather simple linear motion could be used in a tribometer for wear and fretting tests, scratch-tester, microindenter and / or nanoindenter, which could be considered as some specific cases of tribometers, but also in the butterfly wear-track tests. Of course, depending on the required input parameters, each specific tribometer has to be equipped with a specific drive, having its individual portrait like a "Speed versus Torque Characteristic" and often a high precision resolution on a micrometer level. An example of a tribometer, implementing linear

reciprocating motion (having a possibility of fast changing of drives and this way providing its universality for testing under different of the aforementioned schemes, is shown on the Fig. 2).



Figure 2. Universal tribometer UMT

Some specific executions of these UMT / UNMT techniques have been described in the papers [17-18]. A variant of some tribotests with a more complicated kinematics have been developed in the paper [19] and obtained a name of a combinatorial approach in tribology. In case a physicist is concentrating on the "third body" for clarifying the influence of hard or soft coatings on performance of lubricating oils and greases some more specific testing techniques can be employed, as described in [20].

## 4. INPUT PARAMETERS

The most important parameters, characterizing a tribosystem are force (torque) and speed, which are fully predefining a choice of loading system and drive in a testing device. Of critical importance is a precision of measurements of these parameters, which is dependent on a concrete research project and requires a right selection of force and torque sensors, especially for a problem of distinguishing between several thin films, including those of nanometer thickness, determined by either their different compositions, or structures, or а combination of them both together with formation of some natural films, caused by the processes of friction and wear themselves. Load, measured in Newtons, milliNewtons, or microNewtons is actually considered to be a basic parameter only, while important related parameters are contact stress, impact force, oscillating load, etc. [9]. Parameters, related to sliding speed are average speed, rolling speed, spinning speed, impact speed, sliding/rolling ratio. Sliding distance (often given as a time of specific tests) can be in case of reciprocating tests characterized by amplitude

(connected with a precision of positioning) and frequency. Relevance of the model bench tests is usually a subject of thorough analyses. It always remains of primarily importance in adequate experiments for reproducing the wear mechanisms of the field tests. Another aspect is reproducing a temperature level and environmental situation of the test materials and coatings.

The latter question is technically implemented using elevated and high temperature chambers (incl. devices and sensors for precise temperature measurements) for the tests under elevated (up to 175°C) and high temperatures (depending on techniques used up to 1000°C or even 1600°C), including those, running in the protective gases; chillers and special cooling devices for negative and low temperatures; vacuum chambers for the tests in vacuum (and alternatively in special gases); humidity chambers for the tests under the controlled atmospheres, circulating oils, etc.

# **5. OUTPUT PARAMETERS**

Tribotests have since years demonstrated more complicated themselves to be much procedures, than only measuring friction and wear as performed in the mediaeval triboegineering. On the other side PC controlled input parameters being changed against time or sliding distance are causing the respective variations of the response in both open and closed engineering systems. So, the output parameters include measured forces and torques of friction, coefficient of friction COF, contact temperature, distribution of contact stresses and temperatures, changes of physical parameters of tribological surfaces and contacts. The latter aspect is connected with in situ (or in vivo) measurements of ECR (ESR) electrical contact resistance (electrical surface resistance) and remains an extremely sensitive method for catching a moment of breaking through of the deposited coatings and thin films. Supplementary is an AE acoustic emission analysis, bringing valuable semiquantitative information about origin of damages on the contact between a PVD coating and a counterbody, on the interface between a coating and a substrate in the body of coatings and in the deeper deformed substrate layers, modified with nanometer scale films with a primarily goal of improvement of contact conditions on the engineered surfaces of various components. One of additional aspects in analyzing of the obtained results is software for processing and interpretation of the output parameters and their presentation in

one of the forms, recognized by physicist, e.g. in the field of synergistic action of lubricating oils, modified with some more or less tribochemically aggressive additives and antifriction and anticorrosion PVD coatings, one of the popular interpretation of the results is a Stribeck curve (COF against ratio V/Fz – speed to normal load).

### 6. SOME CONCERNS ABOUT SELECTION OF A RELEVANT TEST

Relevant tribology test arranged in a bench top tribometer is a model, possibly repeating the prevailing wear mechanism of the real components and implementing the same type of contact, like point, linear or flat ones. This is not a must, that a complicated tribocontact, e.g. between a coated cutting edge, a metal chip and a machined workpiece has to be modeled using one scheme only. A detailed description of such practical situation can be done using several geometrical schemes and after collecting the results from several tribotests, e.g. "block-on-ring" plus "pinon-disk" (alternatively "block-on-ring" plus "pinon-plate"), depending on a specific cutting tool type and geometry, etc. [4].

As nominated by STLE members in 2005 [21], tribotesting belongs to the ten greatest events in tribology history, as it is this helped to make tribology from a technological endeavor to an interdisciplinary science.

# 7. SURFACE CHARACTERIZATION

Characterization of coated surface includes measurement and determination first of all of their geometrical mechanical and properties. Geometrical properties are analyzed using contact and non-contact profilometers and AFM - atomic force microscopes and devices. Hardness on the nano-, micro- and macro-levels and determination of elasticity modulus are done using the indentation techniques, which in the case of evaluation of thin films and coatings could be performed in static and in dynamic modes, assisting to eliminate any influence of substrate on the results of the measurements.

## 8. PROFILOMETRY

Traditionally measured 2D parameters, such as Ra, Rz, Rq, Rmax, etc. employing well known contact

techniques remain a complimentary to modern 3D surface topography analysis by means of chromatic confocal optical sensors and laser sensors. Submicrometer resolution in horizontal direction can go in this event down to 100 nm. Vertical range and respectively vertical resolution vary from 0,1 mm to 2,5 mm and from 2 nm to 40 nm for inductive (contact) sensors. Typical chromatic confocal (non-contact) sensors provide the resolution from 2 nm to 15 nm within larger vertical range from 460 µm to 3,7 mm. Laser noncontact sensors are giving a wider vertical range up to 10 mm, but worse resolution on a micrometer level. One of the popular models of optical profilometers NJ HP - 115 is demonstrated on the Fig. 3.



Figure 3. Optical profilometer NJ HP – 115

## 9. INDENTATION AND ELASTICITY MODULUS

Instrumented indentation is running according to ISO 14577 for measuring hardness, elasticity modulus, tensile and von Mises stresses, contact stiffness of various coatings and substrates. As described in [18], hardness tests as a part of tribotests and mechanical characterization involve a) wear tests for evaluation of coating friction and durability, b) scratch-hardness tests under constant load for scratch-resistance and microhardness measurement, c) scratch-adhesion tests under progressively increasing load for evaluation of the coating adhesion and scratch toughness properties, d) nano-indentation tests for coating nano-hardness and elastic modulus evaluation [18, 22].

### 10. MICRO-SCRATCH AND NANO-SCRATCH TESTS

Various physic-mechanical aspects of scratching have been described in [23], therefore we are

dwelling here on some semi-standardized procedures only. According to DIN V ENV 1071 part 3 (compare DIN Fachbericht 39, p. 223ff) scratch test has to run with a diamond (Rockwell C), free of contaminations, tip radius 0,2 mm, opening angle 120°. Presence of contaminants have to be checked after 10 tests using an optical microscope, impurities are to be cleaned by methanol and fine brush, or a diamond tip have to be changed. Loading from zero to 100 N with a speed 1,67 N/s. Speed of scratching is 0,167 mm/s, measuring time 60 s, scratching distance 10 mm, distance between scratching tracks is minimum three times of lateral elongation of recognizable layer damages (max. 1 mm).

Environmental requirement include anti-vibration table, room temperature  $22\pm5^{\circ}$ , air humidity below 80% (to avoid condensation on samples and diamonds). Critical load would be determined measuring an acoustic noise level by means of a sound converter (resonance frequency 200 kHz and minimum level of frequency ca. 30 kHz). Parallel with the acoustic measurements, analysis of damages is made by an optical microscope. If the layers and substrates cannot be identified using optical light, then access to some more methods, as SEM, phase contrast, etc. have to be provided. Detection of cohesion failures, i.e. formation of the cracks within deposited layers could require magnification x500.

## 11. TOWARDS COMPREHENSIVE TRIBOANALYSIS OF COATED MACHINE COMPONENTS

Comprehensive triboanalysis requires full scale tribotests plus a number of methods for chemical, physical and mechanical characterization of contacting coatings and films, aiming to distinguish specific tribophysical phenomena, serving to a longer life of coated and modified components. Full scale characterization involves multi-sensing tests, multilevel analysis and multi-resolution estimation of the triboparameters.

## REFERENCES

 K. Holmberg, A. Matthews: Coatings Tribology, Properties, Mechanisms, Techniques and Applications in Surface, Tribology and Interface Engineering, Second Edition, Vol. 56, Elsevier, Amsterdam, 2009, 560 p.

- [2] J. Takadoum: Materials and Surface Engineering in Tribology, 2008, Wiley, 226 p. First published in France in 2007 by Hermes Science/Lavoisier: «Matériaux et surfaces en tribologie».
- [3] *Tribology of Diamond-Like Carbon Films*, eds. Christophe Donnet, Ali Erdemir, Springer, 2008, 664 p.
- [4] A.A. Minewitsch: Wear of cemented carbide cutting inserts with multilayer Ti-based PVD coatings, Surf. & Coat. Technol., 1992, v. 53, No. 2, p. 161-170.
- [5] Hans Sjöström, A.A. Minewitsch: *DLC* coated bearings, In: "Modern Materials, Equipment and Technologies of hardening and recovery of machine elements", Novopolotsk, Polotsk State University, 1999, p. 26-27 (In Russian).
- [6] A.A. Minewitsch: Tribological Behaviour of Ti-B-Si-N CAE Coatings, 14th Int. Colloquium 13-15 January 2004 "Tribology and Lubrication Engineering", vol. I, ed.: W.J. Bartz, Ostfildern, Technische Akademie Esslingen p. 573-578.
- [7] A.A. Minewitsch: On Technological Heredity of Some PVD Coated Triboelements, Proc. 16<sup>th</sup> Int. Tribology Colloquium "Lubricants, Materials and Lubrication Engineering", January 15-17, 2008, ed. Wilfried J. Bartz, Technische Akademie Esslingen, paper 21.12.
- [8] Horst Czichos, Karl-Heinz Habig: Tribologie-Handbuch. Tribometrie, Tribomaterialien, Tribotechnik, Vieweg + Teubner Verlag, Wiesbaden, 2010, 757 S.
- [9] G.W. Stachowiak, A.W. Batchelor, G.B. Stachowiak: *Experimental Methods in Tribology*, Elsevier, Amsterdam, 2004, 354 p.
- [10] E.D. Braun: Methods and Devices for Tribotechnical Tests, In: Modern Tribology, ed. K.V. Frolov, Moscow, URSS, 2007, p. 367 – 414 (In Russian).
- [11]E. Santner, H.-J. Böhmer, L. Deters et al.: Reibung und Verschleiß von Werkstoffen, Bauteilen und Konstruktionen. Tribologische Optimierung und Schadenbekämpfung, Kontakt & Studium, Band 602, Renningen, expert verlag, S. 232, 2004.
- [12]B. Prakash: *Tribotesting*, In: Tribology in Engineering Design, B.V.A. Rao (Ed.), Institution of Engineers, India, p.152-165, 1993.

- [13] Fundamentals of Friction: Macroscopic and Microscopic Processes, eds. I.L. Singer and H.M. Pollock, Kluwer, Dordrecht, 1992, 621 p.
- [14] Nanotribology: Critical Assessment and Research Needs. Eds. S.M. Hsu and Z.C. Ying, Kluwer, Boston, 2003, 442 p.
- [15] S. Hogmark, S. Jacobson: *Hints and guidlines for tribotesting and evaluation*, Lubr. Eng., 1992, v. 48, No. 5, p. 401-409.
- [16] D. Tabor: *Tribology and physics*, J. Phys. D: Applied Phys., vol.25, No. 1A, p. A1-A2, 1992.
- [17] N. Gitis, A. Minewitsch: Integrierte SPM für Nano-Tribologische Charakterisierung von Oberflächen, Tribologie & Schmierungstechnik, GFT Tribologie-Fachtagung, 26.-28. Sep. 2005, Göttingen, 2005.
- [18] N. Gitis, A. Minewitsch: Tribomechanische Charakterisierung von Hartstoff-Beschichtungen im Nano-Mikrotester, GFT Tribologie-Fachtagung, 24.-26. Sep. 2007, Göttingen, Bd. II, S. 63/1-63/11, 2007.

- [19] M. Eglin, A. Rossi, N.D. Spencer: A Combinatorial Approach to Elucidating Tribological Mechanisms, Tribology Letters, 15, 3, pp. 193-198, 2003.
- [20] A.A. Minewitsch: Modern Equipment for Testing of Lubricants and Greases, Proc. of the VI Int. Conference "Lubricants Russia - 2010", November 10-11, 2010, Renaissance Moscow Hotel
- [21] Kathryn Carnes: The Ten Greates Events in Tribology History, Tribology & Lubrication Technology, June 2005, Vol. 61, No. 6, p. 38-47.
- [22] A.A. Minewitsch, S.N. Dub: *Mechanical properties of titanium nitride*, produced by magnetron sputtering, J. Mater. Technol. Tool., No. 2, p. 35, 1996.
- [23] *Scratching of Materials & Applications*, ed. Sujeet Sinha, Elsevier, Amsterdam, 2006, 319 p.