Nanocoating Surface Wear Assessment Using Methods of Non-Contact Scanning Probe Microscopy

Aleksandrs Filipovs[1], Natalija Filipova[2], [1-2]*Riga Technical University, Institute of Mechanical Engineering*

***Abstract* – Wear resistance of machine parts is the main exploitation index of the quality of the design, and application of nanocoatings provides effective resistance to wear and ensures long service life of machine parts. Since any surface loses its mechanical characteristics in the course of operation, it is expedient to control the surface in order to determine its state. This article describes the methods of measuring the state of nanocoatings, specifically wear, using non-contact methods, with the goal to already use a specific method for determining the durability of the investigated nanocoatings in the future.**

***Keywords* – Nanocoatings, wear, non-contact methods, microscopy.**

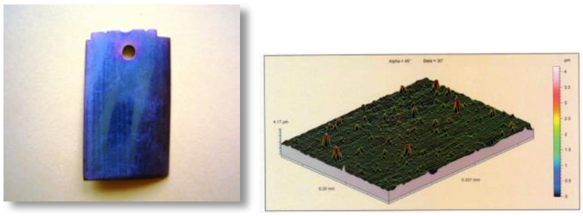
I. INTRODUCTION

In modern mechanical engineering there are very high requirements to the quality of machine parts. In this connection, product materials' characteristics such as wear resistance, heat resistance and hardness are complicated to ensure in the production of machinery parts, using all known metals and their alloys. That is why in the recent years very popular are combinations of elements of the Mendeleyev periodic table, made in the form of nanocoatings, which, thanks to their composition and structure, allow improving the detail surface quality by several times, thus matching the exploitation requirements of the structures.

The sphere of application of nanocoatings is extensive enough, it is automobile production, aircraft manufacturing, ship building and, of course, production of cutting tools for machining. The coatings are deposited on the surfaces of gas pumps, turbine engine blades, brake disks, tools; it means that the area of application is almost unlimited. But we must not forget that the nanocoating, in turn, will not be able to provide high mechanical properties throughout the entire time of operation, because the aggressive environment and the interaction with other coating surfaces will certainly influence the resistance of nanocoatings. In this regard, the methods of nanocoating control for identifying defects, especially wear, and ensuring the effective operation time of machine parts and tools will be explored.

II. INVESTIGATED COATING (TIAL)N

Within the scientific research project the analysis of the parameters of double nitride (TiAl)N compositional coating was carried out with the goal to improve the durability of machine parts and tools, which operate in aggressive environment. This coating has a cubic structure like TiN, but it has a smaller lattice period, and that influences its hardness. Coating (TiAl)N is stable at temperatures 710-830°C, while TiN coating begins to oxidize at 550°C. The reason is that the protective amorphous layer – Al_2O_3 – is formed, which prevents further oxidation. Consequently, the durability of the tool coated with (TiAl)N nanocoating is vastly superior than the life of the tool coated with TiN layer [1]. A sample with (TiAl)N nanocoating, which was deposited by ion-plasma spraying, is shown in Fig.1(a). Nearby, Fig. 2 (b) shows the topography of the coating, which reflects the nature of roughness.



a b

Fig.1. (TiAl)N nanocoating
a – investigated sample, b – surface topography

III. METHODS OF DETERMINATION THE WEAR OF THE NANOCOATINGS

Due to the fact that in the process of exploitation of machine parts the surface loses its mechanical properties, it is necessary to control or check the covered parts in order to identify the degree of wear and, respectively, the operating time of the nanocoating. Therefore, the aim of this paper is to analyse the methods of measuring the nanocoating wear in order to apply a specific method for determining the durability of the investigated nanocoatings in subsequent studies.

There are two methods of nanocoating analysis – the contact method, wherein the device sensor is brought in contact with the measurable object and the non-contact method, wherein the device sensor doesn't mechanically interact with the object of measurement. In this paper the second group of measurement methods will be described – the non-contact analysis of surface wear, because it allows avoiding the damage of the sample during the measurements.

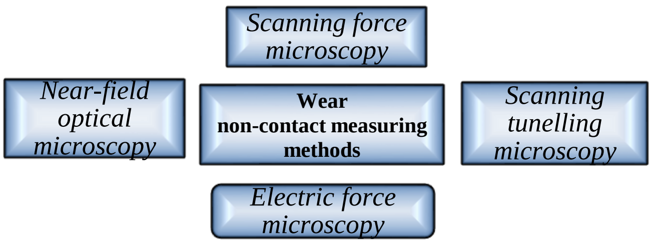


Fig.2. Non-contact methods for wear determining

Conventionally, methods of surface non-contact measurements, which include the determination of wear, can be divided into groups, represented in Figure 2.

The basis of all these methods is the principle of scanning probe microscopy –detection of the electrostatic, magnetic or electromagnetic interaction of the probe with the surface of the analysed object. Further in the article each of these methods will be considered in detail.

IV. Scanning Force Microscopy

The first method, the most commonly used one, is the method of scanning force microscopy, which in its turn is a subspecies of the atomic force microscopy, and its distinguishing feature is absence of repulsive forces, which makes it possible to use "soft materials" in research.

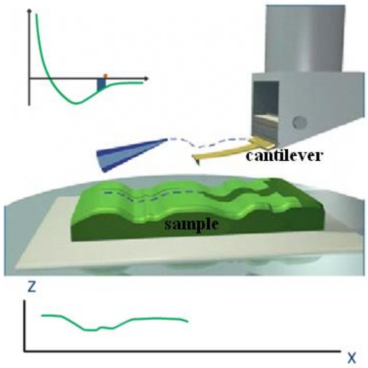


Fig.3. Scheme of the sample's measurement by non-contact scanning force microscopy (NC SFM) [2]

[2] NC SFM uses the principle of the definition of "amplitude modulation". The corresponding measuring scheme uses a variation of cantilever amplitude due to the interaction of the probe with the sample. Work using the method of NC SFM can be described in terms of the gradient-force model. In accordance with this model, in the limit of small amplitudes A while the cantilever approaches the sample the cantilever resonance frequency f_0 shifts by an amount df to its new value in accordance with the expression:

$$f_{eff} = f_0(1 - F'(z)/k_0)^{1/2} \tag{1}$$

where f_{eff} – the new value of the resonant frequency of the cantilever with a nominal value of stiffness k_o ;
$F'(z)$ – the gradient of the cantilever force interaction with the sample;
z – effective clearance of the tip-sample, in case of attraction value $df = f_{eff} - f_o$ (negative).

If the exciting frequency of the cantilever $f_{set} > f_o$, then the shift of the resonance frequency to lower values leads to a decrease of oscillation amplitude with the frequency f_{set} while approaching the sample. These changes in the amplitude A are used as an input signal in the feedback system. To obtain the scanned image using the method of NC SFM, first, it is necessary to choose a certain amplitude A_{set} as a setting, with $A_{set} < A(f_{set})$ when the cantilever is far from the sample surface. The feedback system brings the cantilever closer to the surface until its instantaneous amplitude A becomes equal to the amplitude of $Aset$ at the specified excitation frequency f_{set} of oscillation. Scanning the sample may start in the x-y plane with the retention by feedback system $A = A_{set}$ = constant for obtaining NC SFM image exactly at this point. The feedback leads the cantilever closer to the sample if A_{set} reduces at any point, and moves the cantilever from the sample if A_{set} increases. In general, as a consequence of the above model in the limit of small amplitudes A the scanned image can be seen as a relief of constant force gradient of tip-sample interaction.

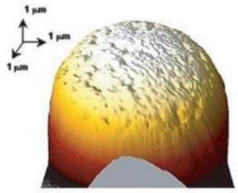


Fig.4. The surface model obtained by measurement by NC SFM [3]

V. Near-Field Optical Microscopy

[4] Traditional methods of obtaining optical images of the objects have significant limitations associated with the diffraction of light. One of the fundamental laws of optics is the existence of the so-called diffraction limit, which sets the minimum size *(R)* of the object, the image of which can be built by optical system using light with a wavelength λ:

$$R \approx \frac{\lambda}{2n} \tag{2}$$

where n – index of diffraction of the environment.

For the optical wavelength range the size is in the order of 200÷300 nm. In the near-field optical microscopy there are other principles of building the object image, which allow overcoming the difficulties associated with the diffraction of light, and which realize the spatial resolution of 10nm or better.

The basis of the device's working principles is the phenomenon of light transmission through subwavelength diaphragm (holes with the diameter much smaller than the wavelength of the incident radiation).

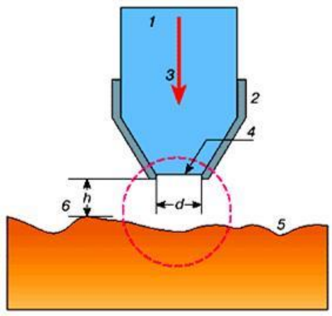


Fig.5.The scheme of near-field optical microscopy:
1 – a sharpened optical fiber; 2 – metal coating;
3 –radiation, extended through the probe; 4 –probe output aperture, $d<<\lambda$;
5 –the surface sample and the distance to the probe, $h<<\lambda$; circled - area of near-field contact [5]

From the physical point of view, near-field optical microscopy (NFOM) is based on the presence of quite identifiable traces of the interaction of light with a micro-object, located in the near field light, which is localized at distances much smaller than the wavelength. NFOM combines elements of conventional optics and scanning probe microscopy. A distinctive element of near-field devices is an optical probe (Fig.5.), which is usually represented as a sharpened optical probe, the external surface of which, except the vertex of cone, is covered with opaque metal layer.

The scanning probe collects the information from the sample surface with a resolution equal to the diameter of the aperture. For working with near-field optical microscope it is necessary to keep the probe over the surface at distances of the order of 10 nm or less. There are various solutions to this problem, but the most widely used is NFOM with the so-called "shear force" method of control of the distance between the probe and the sample.

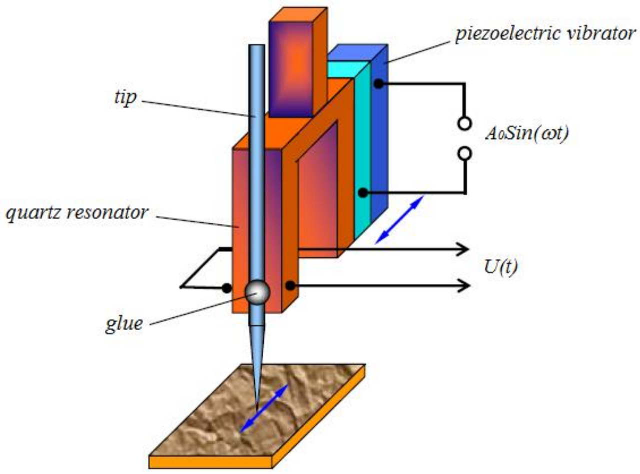


Fig.6.The scheme of „shear-force" sensor of distance probe-surface on the basis of tuning –fork-type quartz resonator [4]

The most commonly used are schemes of "share-force" control, using piezoelectric sensor based on tuning-fork-type quartz resonator (Fig.6). NFOM probe is attached to the quartz resonator by glue. Forced oscillations of a tuning fork, at a frequency close to the resonator frequency of the system "probe – quartz" resonator, are excited by an additional piezovibrator. The probe oscillates parallel to the sample surface. Measurements of the strength of the probe interaction with the surface are performed by detecting changes in the amplitude and phase of the bending oscillations at the excitation frequency. The theory of "shear force" control is quite complex, and here it is necessary to confine to the qualitative considerations. In the course of convergence of the probe and the sample, several effects are observed.

First, there is an additional dissipative probe's interaction with the surface due to the viscous friction forces (in the thin layer of air, which is adjacent to the surface, and in the thin layer of adsorbed molecules on the sample surface). This leads to the quality reduction of broadening of the system "probe-resonator" at the resonant frequency.

Second, the small distances of tip-surface increase changes in the oscillation modes in the system "probe-resonator". In a free state oscillation mode corresponds to oscillation of the rod with a free end, but during the approaching of the sample it is transformed into vibrations of a rod with a fixed end. This leads to an increase in the resonant frequency in the system "probe-resonator", it means to the shift of AFC (amplitude-frequency characteristics) to higher frequencies.

Changes in the amplitude and phase of the bending oscillations in the system "probe-resonator" are used as a feedback signal to control the distance "probe-surface" in the near field optical microscopes.

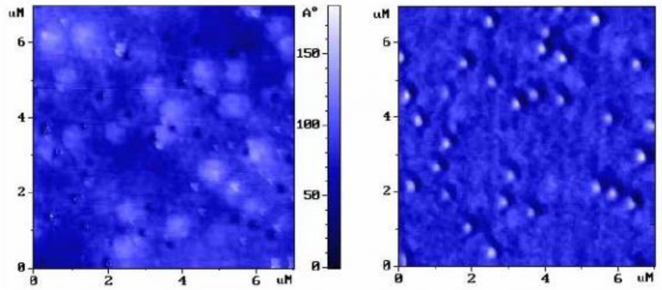


Fig.7. Surface topography image in "Shear force" mode [4]

VI. Scanning Tunnelling Microscopy

[4] Historically, scanning tunnelling microscope was the first in the family of scanning probe microscopes. The principle of STM is based on the phenomenon of electron tunnelling through a narrow potential barrier between the metal probe and a conductive sample in an external electric field. In STM the probe is applied to the sample surface at a distance of a few angstrems. A tunnel-transparent potential barrier is formed there, whose magnitude is mainly determined by values of the electrons' work function from the probe material P and from the sample S. The barrier can be considered as a rectangle with an effective height equal to the average work function of the material:

$$\varphi^{*} = \frac{1}{2}(\varphi_P + \varphi_S) \qquad (3)$$

As it is well known from quantum mechanics, the probability of electron tunnelling (transmission coefficient) through a one-dimensional rectangular barrier is determined by the following formula:

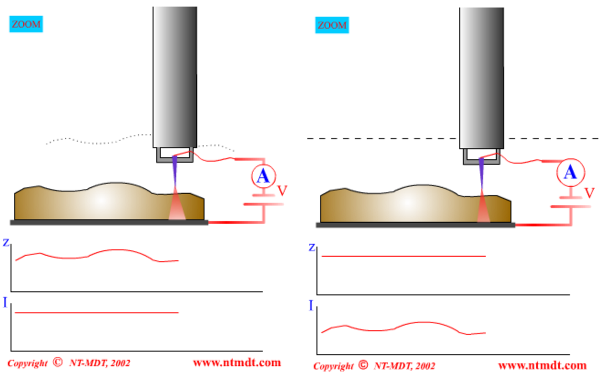
$$W = \frac{|A_t|^2}{|A_0|^2} \cong e^{-k\Delta Z} \qquad (4)$$

where A_0 – amplitude of the wave function of an electron moving to the barrier;

A – amplitude of the wave function of an electron passing through the barrier;

k – constant of the wave function in the region corresponding to a potential barrier;

ΔZ – the width of the barrier.



a b

Fig.8. Surface image formation by the constant tunnelling current method (a) and constant height method (b) [6]

Image of relief surface in STM is formed by two methods. Using the method of constant tunnelling current (Fig.8 (a), the probe is moved along the surface, carrying a raster scanning, thus, the change in voltage at *Z* – electrode of the piezoelectric element in the feedback (repeating with a great precision surface topography of the sample) is stored in the computer memory as a function $Z=f(x,y)$, and then reproduced by means of computer graphics.

In the study of atomically smooth surfaces it is often more efficient to obtain STM images of the surface by the method of constant height *Z=const*. In this case, the probe moves over the surface at a distance of a few angstroms, wherein changes in the tunnel current are detected as an STM image of the surface (Fig.8 (b)). The scanning is performed either when the OS is off, either at the speeds, which exceed the speed of OS reaction, so OS works off only smooth changes in surface topography. In this method very high scanning speeds and high frequency of getting the STM images are implemented, which allow monitoring the changes occurring on the surface almost in real time.

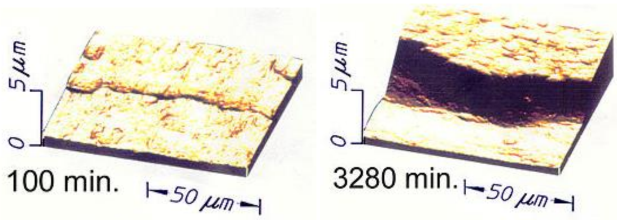


Fig.9. Sample surface, which is divided into two parts – the film-coated (Ta-C) and unprotected after 100 and 3,280 min abrasive machining [7]

[8] STM high spatial resolution is determined by exponential dependence of the tunnelling current on the distance to the surface. Resolution in a direction normal to the surface reaches a percentage of nanometer. Resolution in lateral directions depends on the quality of the probe and is determined mainly not by macroscopic curvature radius tip of the needle, but by its atomic structure. At the right preparation of the probe there is single protrusive atom or a small cluster of atoms with sizes much smaller than the typical radius of the curvature of the tip on probe's tip. Indeed, the tunnelling current flows between the surfaces on a distance equal to the value of the crystal lattice. Since the tunnelling current dependence on the distance is exponential, the current in this case flows mainly between the sample surface and the protruding atoms on the tip of the probe.

VII. Electric Power Microscopy

[4] In electric power microscopy electrical interaction between the probe and the sample is used to obtain the properties of the surface. Let there be submitted DC U_0 and AC $U_\sim = U_1 \cdot Sin(\omega t)$ between the tip and the sample. If a thin layer on the substrate is a semiconductor or dielectric, it may contain a surface charge, so that there is potential $\varphi(x, y)$ distribution on the sample surface. The voltage between the tip and the sample surface can be represented as:

$$U = U_0 + U_1 \sin(\omega t) - \phi(x, y) \tag{5}$$

The system „probe-sample" has a capacitance *C*, so that the energy of such system can be represented as follows:

$$E = \frac{CU^2}{2} \tag{6}$$

Then interaction of the electric force between the probe and the sample is:

$$\vec{F} = -grad(E) \tag{7}$$

And its *Z*-component can be represented as follows:

$$F_z = \frac{\partial E}{\partial z} = -\frac{1}{2} U^2 \frac{\partial C}{\partial z} \tag{8}$$

Since the value $\frac{\partial C}{\partial z}$ depends on the distance "tip-sample", the two-pass technique is used for the study of the dielectric properties of the sample . On the first pass cantilever oscillation was excited by pjezovibrator at a frequency close to the resonant frequency ω_0, and the AFM topography image is detected in the "semi-conductor" mode. Then the probe is removed from the surface to a distance z_0, AC (at a frequency $\omega=\omega_0$) voltage is supplied between the tip and the sample , and then the re-scan is performed (Fig.10).

On the second pass, the cantilever is driven into the oscillating notion at resonant frequency, wherein the cantilever is grounded or displaced. Capacitive force of tip-sample interaction (or rather its derivative) leads to a shift of the resonance frequency. Accordingly, the oscillating amplitude of the cantilever decreases, and the phase of its oscillation shifts. Both the amplitude and the phase of the oscillation may be measured and used to display the distribution of electric potential over the sample surface.

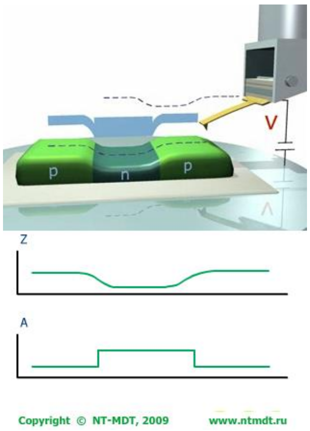


Fig.10. Surface scanning in the two-pass using the method of electric force microscopy [9]

Displays of the amplitude of phase deviations are determined by capacitive probe-sample force derivative of the tip sample capacity. As a result, non-contact EFM leads to higher resolution because the ratio of the parasitic capacitance of the probe cone and the flat part of the cantilever to the effective capacity of the tip of the probe is minimized.

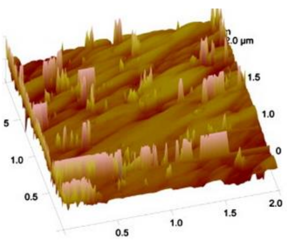


Fig.11. Topography of the surface, taken by electric force microscopy [10]

VIII. Conclusion

In conclusion, it should be noted that each of the aforementioned methods provides opportunity to make visual and quantitative detection of surface wear without damaging the sample. In these cases, specifically the change in topography of nanocoatings in the course of exploitation will determine the degree of wear. In turn, the choice of a particular non-contact method is determined by the requirements with regard to the quality of measurements and also the cost of measurement.

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Aleksandrs Filipovs, Assistant Professor of Riga Technical University, Doctor of Engineering Science (2003), International Welding Engineer-EWE (2000), MSB (bionics) (1994),BSME (mechanics) (1993).

Manager of RTU scientific research laboratory of welding process automation, address: Ezermalas Street 6, Riga, LV-1006; the Head of the Welding Department, Welding Engineer, Director of Inspection, Head of the Latvian Independent Testing Laboratory.

Membership in professional bodies: Councilor of Latvian Accreditation Committee, Councilor of Latvian Welding Association, Academic Councilor of "St. Petersburg Academy of Engineering Science". Contact data: Phone: +37129267763, E-mail: weld5@inbox.lv.

Natalija Filipova, a master student of Riga Technical University (the program "Production Engineering"), has a bachelor degree in machine and apparatus building and the qualification of a mechanical engineer (2012).

Work experience: welding engineer, expert of personnel certification centre, project manager (nanotechnologies, nanocoatings), the main job – research assistant, research areas: metrology, surface roughness, texture parameters. Address: Ezermalas Street 6, Riga, LV-1006. Phone: +37126347790, E-mail: natalija.filipova@rtu.lv.

Aleksandrs Fiļipovs, Natālija Fiļipova. Nanopārklājumu virsmu nodiluma noteikšana, izmantojot bezkontakta skenējošas zondes mikroskopijas metodes.
Sakarā ar to, ka nanopārklājumi kalpo par mašīnu detaļu un citu mašīnbūvniecības elementu aizsārgiznstrumentu, ir svarīgi kontrolēt to fizisko stāvokli, lai nodrošinātu efektīvu un drošu darba detaļu un konstrukciju darbību. Zem pārklājuma fiziskā stāvokļa saprot tā iespēju pretoties dilumam ekspluatācijas laikā, t.i. runa ir par detaļas kalpošanas laiku. Dotajā rakstā tiek izskatītas iespējamas nodiluma konstatēšanas metodes, pie tam norādītās metodes ir bezkontakta metodes, kas ļauj izvairīties no pārklājuma virsmas bojājumiem mērīšanas procesā. Nodiluma noteikšanas metožu pamatā ir skenējošā zondes mikroskopija, kura savukārt sadalās tādās plaši izmantojamas un pie nanopārklājumiem pielietojamām metodēm kā atomspēku, tuneļstrāvas, tuva lauka un elektrisko spēku mikroskopija. Darbā tika shematiski attēlota zondes mijiedarbība ar paraugu pie atšķirīgām bezkontakta mērīšanas metodēm un dažādu virsmu topogrāfijas mērījumu rezultāti. Kaut kādas konkrētas mērīšanas metodes izvēle ir nosacīta tiešā veidā ar mērījumu mērķiem, kā arī prasībām pret rezultātu precizitāti.

Александр Филипов, Наталия Филипова. Определение износа поверхностей нанопокрытий с помощью методов бесконтактной сканирующей зондовой микроскопии.
В связи с тем, что нанопокрытия являются защитным инструментом деталей машин и других элементов машиностроения, важно контролировать их физическое состояние для обеспечения эффективной и безопасной работы рабочих деталей и конструкций. Под физическим состоянием покрытия подразумевают его способность сопротивляться износу в течении эксплуатационного времени, т.е. речь идет о сроке службы детали. В данной статье рассмотрены возможные методы констатации износа нанопокрытий, при чем указанные методы являются бесконтактными, что дает возможность избежать повреждения поверхности покрытия в процессе измерений. В основе методов определения износа лежит сканирующая зондовая микроскопия, которая в свою очередь подразделяется на такие наиболее распространенные и применимые к нанопокрытиям методы как атомно-силовая, тунельная, ближнепольная и электросиловая микроскопия. В работе схематично отображено взаимодействие зонда с образцом при разных бесконтактных методах измерений и результаты измерений топографии различных поверхностей. Выбор какого-то конкретного метода измерений напрямую обусловлен целью измерений и требованиями к точности результатов.