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Advanced techniques for nanotribological studies

Key words

Nanotribology, nanotribometry, atomic force microscope (AFM), nanoindentation.

Słowa kluczowe

Nanotribologia, nanotribometria, mikroskop sił atomowych (AFM), nanoindentacja.

Summary

The advanced techniques used in nanotribological studies are discussed. Various techniques such as Atomic Force Microscopy (AFM), Surface Force Apparatus (SFA), Nanoindentation technique, etc. are described and their suitability to study friction on a nano- scale are evaluated. Some examples of the use of these instruments for nanotribological studies are given.

1. Introduction

Nanotribology is very fascinating area of tribological science and technology. It can reveal the behaviour of nanotribological systems down to nearly the atomic level. The results are useful in understanding the behaviour of

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macrotribological systems as well. The nanotribological studies have both a fundamental character and the results are applied in the Micro Electro Mechanical Systems (MEMS), Hard Disk Drives (HDD) technologies as well as in nanotechnologies (e.g., in nanoimprint lithography).

Nanotribology started practically after the construction of first atomic force microscopes (AFM) in the 1980's [1-11]. The first experiments were carried out in the IBM laboratory in San Jose (USA) by M. Mate [2, 3]. He can be treated as the pioneer of Nanotribology followed by R. Kaneko in Japan and B. Bhushan in the USA [6, 8, 9]. Presently, dynamic progress can be observed, because many physicists and chemists have joined the research community of tribologists, the demands for the results of the nanotribological studies have been greatly increased.

2. Advanced techniques for nanotribological studies

The techniques used for nano- and macrotribological studies are completely different. The typical tribometers and rubbing pairs are not applicable in nanotribological research. The tribological parameters such as friction (friction coefficient) and wear rate are usually accompanied by the results of the adhesive (stiction) studies (pull-off force, stiction force, wetablility, surface energy) as well as by nanomechanical properties (nanohardness, elasticity modules on nano-scale – usually the properties of ultra-thin (with thickness below 1 μm) films are needed to understand the tribological data. In addition, such specific tests as nano-scratch is used but not for e.g. the estimation of the coating`s adhesion to the substrate but rather for rapid recognition of the tribological properties in particular of very thin films (e.g. 3-5 nm thick films applied as overcoat on magnetic layer in computer hard disks).

The fundamental instrument used in nanotribological studies is the atomic force microscope (AFM) [1, 6]. It is the device that enables the studies of friction and wear as well as estimate many other characteristics of the rubbing components on the atomic level. The principle of the instrument is shown in Fig. 1.

The rubbing pair is composed of the tip (usually with a very small radius = 5-100 nm) fixed to the cantilever (flat spring), which is in contact the sample material. The area of contact can be very small (e.g. with radius 0.5 nm). The applied load are usually on the level of nN-μN and sliding speeds in the range of 0.02-100 μm/s. The study of friction to obtain the friction map is a scan performed in the area below 50 μm x 50 μm. The additional surface topography (2D and 3D images), scratch test with the visualisation of its results as well as force-distance curve (to estimate pull-off force and to perform nano-indentation) can be done with the use of the same cantilever.

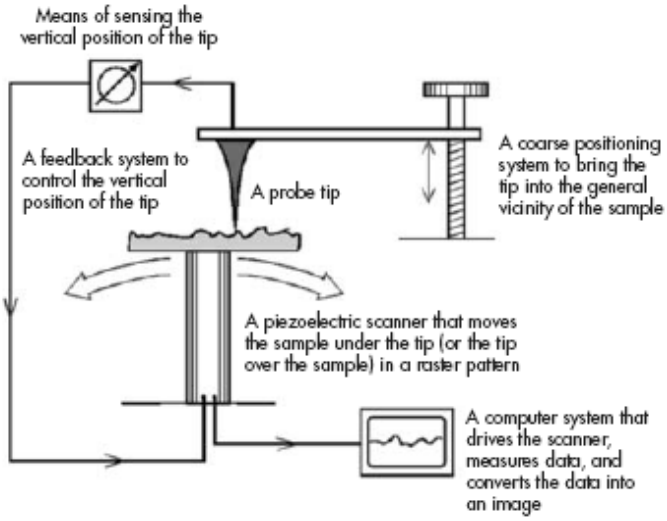

Fig. 1. Schematic of atomic force microscope (AFM)
Rys. 1. Schemat budowy mikroskopu sił atomowych (AFM)

It is evident that, with the use of the cantilever with very sharp tip, the friction map will be presenting the lateral force that results from the adhesive interactions (material properties) and the mechanical interactions that result from the roughness. However, in nanotribological studies, the surfaces of the tested samples are very smooth so the effect of the roughness can be very small.

The results of the studies with the use of a AFM enable one to compare the tribological, adhesive, and mechanical properties of often studied ultra-thin films. The results of the measurements are very useful for the evaluation of the quality of materials useful in the construction of, e.g. MEMS devices. This is particularly important to the surface topography as confronted with the friction map.

The nano-scratch and nano-wear tests enable one to compare the wear resistance of tested materials (in particular ultra-thin films) devoted to the construction of the dubbing components of micro/nanosystems. Such test forces to use special cantilevers (usually very stiff) with very hard (diamond) tip. At scratching, the maximum depth of the scratch scar is evaluated at the defined load, scratch speed, and mode of scratching (single scratch, multiplied scratch). The shear force is very often also measured. The nano-wear test is rather complex, time consuming, and expensive. At the first moment, the surface of the sample is scanned at a low load to obtain its image. Then, at a smaller scan area (e.g. 2 x 2 μm), the nano-machining line by line is realised to form a nano-crater in the tested material (film). By the comparison of two scans, one obtained before the nano-machining and the second one after the wearing process, the wear depth, and the shape of the nano-crater can be visualised. Different shapes

of the crater can be easily obtained and visualised (e.g. a nano-well with "nano-stairs").

The second device, which is very useful to study friction on nano-scale, is the surface force apparatus (SFA) invented by J. Israelachvili and D. Tabor [7]. It was invented to study the molecular force interactions (Van der Waals forces) of very flat (mica) surfaces, being in intimate contact but having relatively small curvatures (Fig. 2). Nowadays, the SFA is also applied to study interfacial lateral (friction) forces at the shearing ultra-thin (molecular) of lubricant both in static and dynamic conditions. The interferometry is usually applied to identify the film thickness.

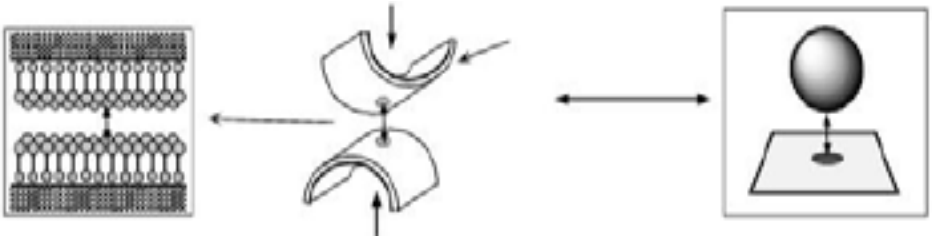

Fig. 2. Schematic of components (surfaces) used in studies of surface interactions and friction (shearing) with use of the surface force apparatus. Left-surfaces coated with a thin layer of liquid (lubricant) are used in centre–surface interactions, and the right model is used for theoretical studies of surface interactions

Rys. 2. Szkic elementów stosowanych w badaniach z użyciem aparatu do pomiaru sił powierzchniowych. Po lewej – badane powierzchnie pokryte cienką warstwą cieczy (smaru), w środku – oddziaływania powierzchni, po prawej – model do analizy teoretycznej oddziaływań powierzchni

In the studies of friction on the atomic level, sometimes it is useful to apply the quartz crystal microbalance [2, 6, 9]. The pioneer of these studies was J. Krim (USA). In this case, the oscillating quartz crystal is used as the heart of the instrument.

The Quartz Crystal Microbalance (QCM) is used to measure friction dissipation for adsorbed layers sliding over surfaces. The QCM consists of a thin single crystal of quartz with metal electrodes deposited on its top and bottom surfaces. A thin film of atoms or molecules adsorbed causes the resonance frequency of the crystal to shift to a lower frequency. The film slides over the surface and dissipates its kinetic energy via friction, which broadens the resonance. The QCM measurements of interfacial friction are limited to sliding systems with very low friction, such as adsorbed rare gases or small physisorbed molecules. Noble metals are used for electrodes to have lower bonding energies. The schematic of the QCM is presented in Fig. 3.

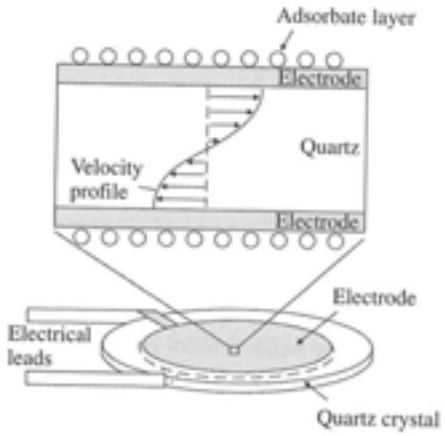

Fig. 3. Schematic of quartz crystal microbalance
Rys. 3. Schemat mikrowagi zbudowanej na drgającym krysztale kwarcu

For the practical studies of tribological properties of micro/nano-tribosystems, special microtribometers are constructed [12-16]. They are used to identify the friction/wear between rubbing components, e.g. in hard disk drives, MEMS/NEMS devices, etc. In the last case, special test structures integrated with actuators and sensors to applied loads and move the components as well as to measure friction are fabricated with the use of microelectronic silicon technology [17]. After use, the microtribometer can be discarded.

In nanotribology, nanomechanical and adhesive studies are also very important. Very effective is nano-indentation with the use of special nano-indenters [18]. This technique enables one to estimate nanohardness and elasticity modulus of very thin films. The adhesive studies are realised with the use of AFM via the realisation of force-distance curves (FDC). Such test enables in particular the estimation of the pull-off force needed to remove the tip (probe) from the contact with a sample. At well-known geometries of the contacting surface (tip–sample), this FDC procedure enables one to estimate interfacial surface energy. A very effective additional test is the wetablility test, which, using very small droplets, enable one to estimate surface energy on a very small area. The droplets can have very small diameters: 30-50 nm.

3. Applications of advanced techniques in nanotribology

The AFM found the most effective application in nanotribological studies. The family of Scanning Probe Microscopes (SPM) based on the sharp tip is very wide now. The application of only AFM creates very exciting possibilities that are schematically show in Fig. 4.

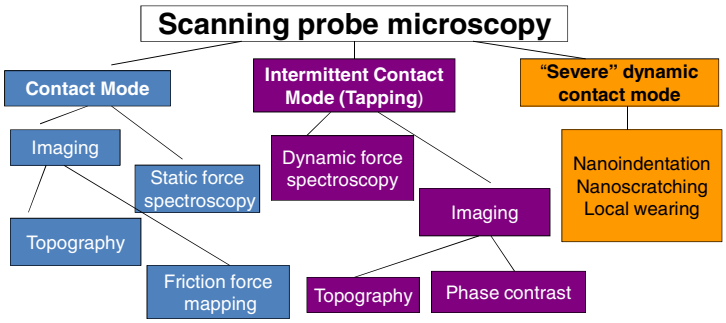

Fig. 4. Modes of operation of the scanning probe microscopy/atomic force microscope used for nanotribological/nanomechanical studies
Rys. 4. Mody pracy mikroskopu skaningowego z sondą/mikroskopu sił atomowych przy badaniach nanotribologicznych/nanomechanicznych

Contact mode is most common in the study of the surface topography and friction. Tapping mode is necessary to study the surface topography of polymeric (elastic) materials (Fig. 5).

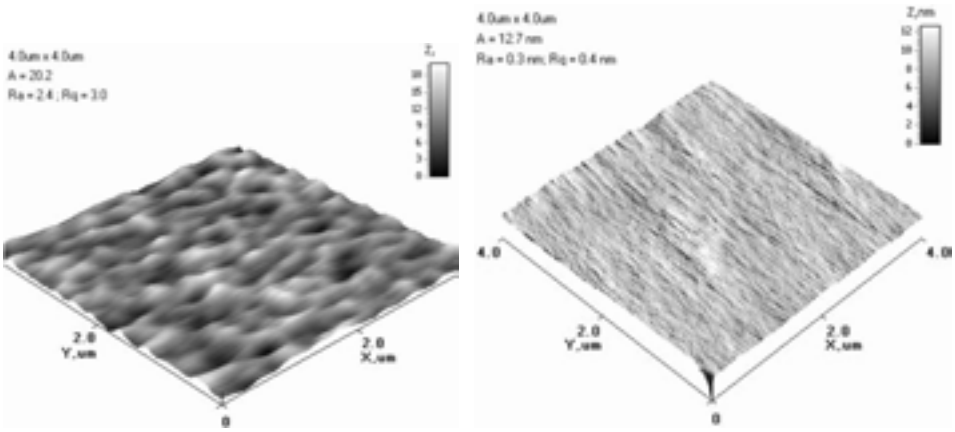

Fig. 5. Examples of surface topography of two polymeric resist materials used in nano-imprint lithography. AFM images obtained in tapping mode
Rys. 5. Przykłady topografii powierzchni dwóch polimerowych rezystów stosowanych w technice nanoimprintingu otrzymane przy pracy AFM w modzie tapping (oscylująca sonda)

In nanotribological studies with the use of a AFM, the friction loop (friction force vs. displacement at forward and backward movement of the sliding element) can be easy created (Fig. 6), which can be used to estimate static and friction coefficient of friction as well as preliminary displacement of the surface layers of the contacting material before transition to sliding can be identified.

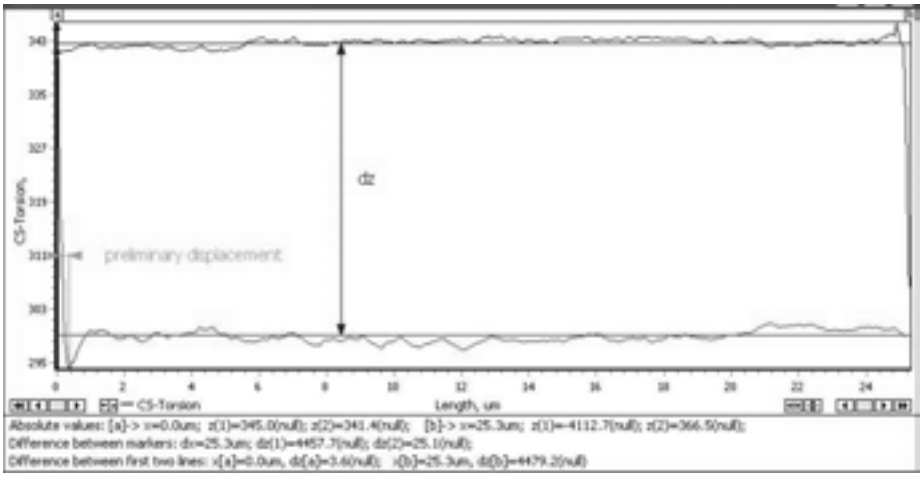

Fig. 6. Friction loop obtained with the use of AFM
Rys. 6. Pętla tarcia uzyskana przy użyciu AFM

The scratch test is relatively simple and cheap, and it can give some interesting results about the wear resistance of the tested material (ultra-thin films) (Fig.7). The depth of scratch scar can be used as criterion for preliminary estimation of wear resistance.

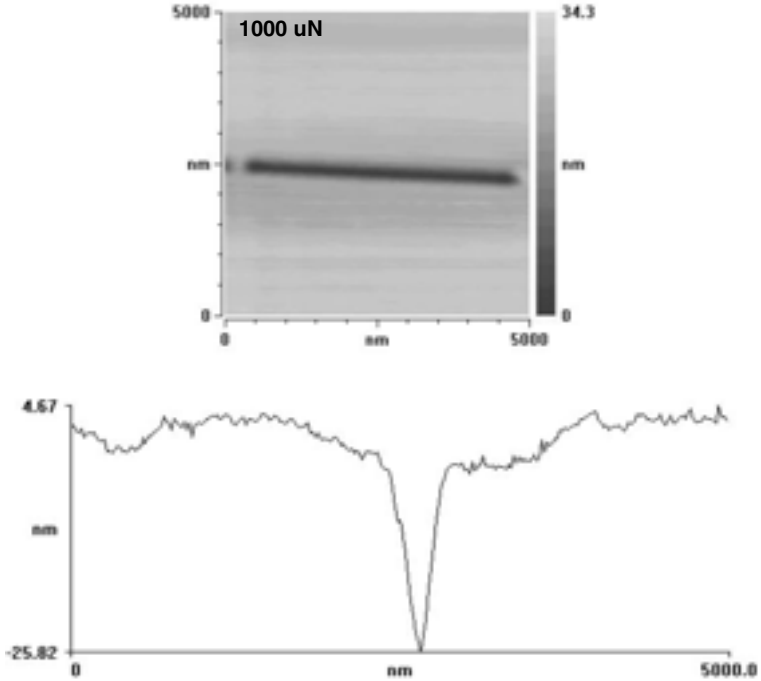

Fig. 7. Scratch scar and its cross-section obtained with a diamond tip on carbon coating
Rys. 7. Obraz śladu zarysowania igłą diamentową powłoki węglowej

The nano-wear test can be easily carried out with the use of a AFM and stiff cantilever with a diamond tip. The wear crater is visualised with the use of the same tip (Fig. 8).

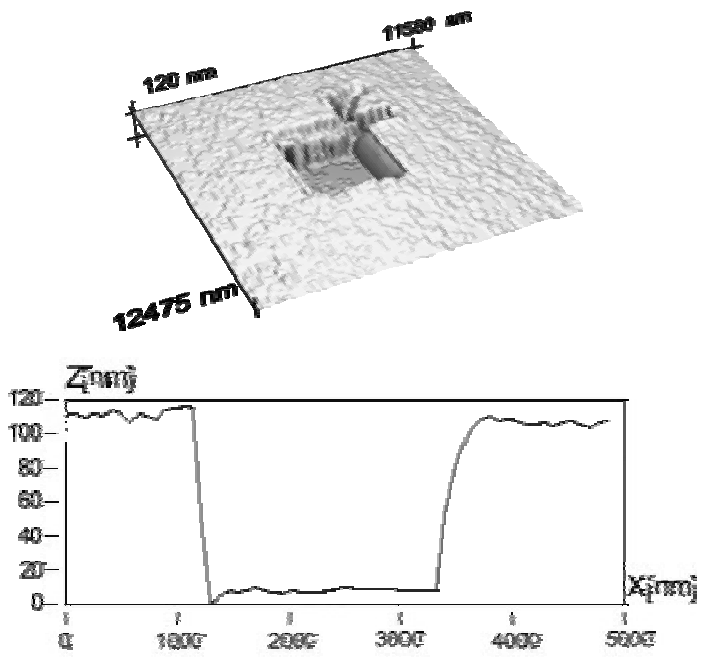

Fig. 8. Example of nano-wear scar and its cross-section
Rys. 8. Przykład krateru nanozużycia i jego przekrój

The nano-indentation enables one to easily identify nanohardness and the elasticity modulus of very thin films. The continuous load-distance characteristics (Fig. 9a) with the Pharr and Oliver formula is used for [16]. Also, a nice AFM image of very small indention can be obtained by scanning (Fig. 9 b).

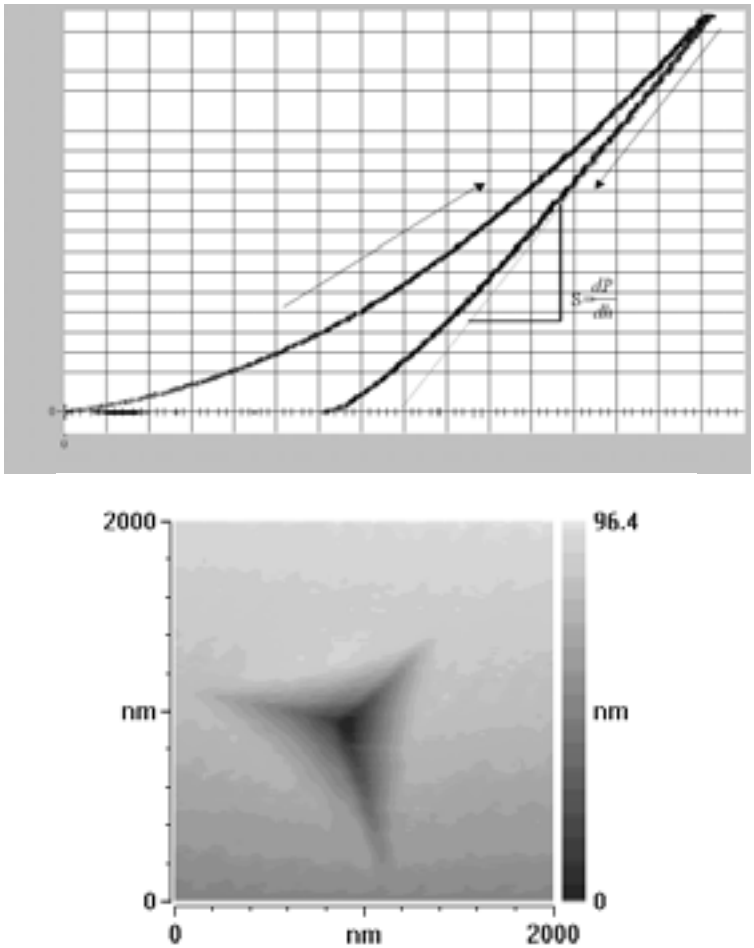

Fig. 9. Nanoindentation: a – Load vs. indentation depth during loading and unloading, b – AFM image of indent
Rys. 9. Nanoindentacja (nanowgłębnikowanie): a – siła vs. głębokość odcisku przy obciążaniu i odciążaniu, b – odcisk uzyskany z AFM

Summary

Nanotribology has become a very import and interesting area of advanced interdisciplinary studies. The results of nanotribological studies are very important for understanding the fundamentals of tribological phenomena, and they play a very important role in the progress of nanotechnology, in particular, in the area of MEMS/NEMS, magnetic recording, and nano-imprint lithography technologies.

The advanced techniques used in the studies of tribological problems on a nano-scale have enabled rapid grow of this area of science and technology. In particular, the inventions of the scanning probe microscopy techniques and the AFM have enabled much progress in the nanotribological studies.

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Zaawansowane techniki badań nanotribologicznych

S t r e s z c z e n i e

W artykule przedstawiono zaawansowane techniki badań nanotribologicznych. Opisano różne techniki, takie jak mikroskopia sił atomowych (AFM), aparat do pomiaru sił powierzchniowych (SFA), techniki nanoindentacji (nanowgłębnikowania) i oceniono ich przydatność do badania tarcia w nanoskali. Podano też przykłady wykorzystania niektórych technik do badań nanotribologicznych.