Chapter 1

Introductory Chapter: Why Atomic Force Microscopy (AFM) is One of the Leading Methods of Surface Morphology Research of all Engineering Material Groups

Tomasz Tański, Bogusław Ziębowicz,
Paweł Jarka and Marcin Staszuk

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1. Introduction

Forming part of the group of scanning probe microscopy, the atomic force microscopy (AFM) allows to imaging of surface topography of all groups of engineering materials with very high resolution, relative to all three axes. AFM enables of obtaining very large magnifications up to 10^8 and resolution of 0.1 nm in the x, y, and 0.01 nm axes in the z axis. Compared to classic scanning electron microscopy (SEM), it allows to imaging of dielectric material surfaces without the need for applying conductive layers and allows the determination of surface roughness parameters without the need for additional tests. Therefore, this method is still being developed and is recognized in many areas of science and technology. The special significance of AFM is emphasized in the research on the surface quality of functional materials, using the possibility of surface analysis at the nanometric level. The field of AFM applications, due to the enormous possibilities unavailable to other research techniques, is constantly expanded [1–4].

The idea of AFM is based on the use of interatomic short-range interactions whose intensity depends on the surface topography of the test sample subjected to the research. The analysis of the surface in terms of the shape and location of characteristic points most often carried out by means of AFM is in three modes [5–7].

It enables very accurate imaging of the surface topography of the static-contact mode (the microscopy of repulsive interactions), in which the distance between the atoms of the probe and atoms of the exanimated surface is in the range of 1 Å and the pressing force of the probe is in the range of 10-11 to 10-7 N. However, the limitation of the use of the mode in the case of materials with low hardness (thin organic layers) is the need to apply low-pressure forces by danger of damaging the sample [5].

In the dynamic-resonance mode (long-range attraction microscopy—van der Waals forces, magnetic forces, electrostatic forces), i.e., noncontact mode, the probe is located 1–10 nm from the sample surface. The mode provides lower resolution of images than in the contact mode; however, it can be used to analyze the surface of materials with very low hardness and easily deformable [6].

In intermittent-contact mode (resonant microscopy of repulsive interactions), the probe introduces oscillation at a frequency close to the resonant frequency (50–500 kHz) at a distance enabling the analysis of interactions of both long-range forces and short-range forces and the blade periodically comes into contact with the surface of the sample [7].

A common feature of all available modes is the method of image creation as a result of measuring the forces occurring between the microprobe and the surface under test. The microsphere is placed on a springy lever (beam) which is bent under the influence of interatomic forces. Deflection of the beam is processed by the photodetector for a current signal, which allows obtaining a surface image of the sample. Due to the specificity of surface imaging, AFM is effective for virtually all groups of engineering materials. This method has no limitations due to the mechanical, optical, or electrical properties of the materials [3–5, 8].

In the case of ceramic layers with high hardness, the use of AFM provides detailed information on the morphology of the surface layers obtained in PVD and CVD (chemical vapor deposition) processes produced as coatings, among others on ceramic tool materials. For example, the analysis of Ti (C, N) + Al_2O_3 + TiN coating surfaces allows to show a topography characteristic of the subsurface Al_2O_3 layer consisting of numerous polyhedra. In this case, the used atomic force microscopy enabled the imaging of the nanodimensional structures and the precise measurement of the height of the structural surface elements by profile analysis which is unachievable by the use of other surface observation techniques, e.g., scanning electron microscopy [9].

An extremely important element of a properly conducted surface topography survey is the analysis of results containing the appropriate interpretation of processes and phenomena occurring on the surface of the material being tested. AFM image analysis is carried out using programs dedicated to specific hardware applications used to analyze and measure basic parameters of the image surface, such as quantitative measurements of surface features in selected areas, statistical data such as geometric area, surface area, and the ratio of surface area to geometric surface.

The use of AFM is developed in particular in the fields of material engineering in which very precise measurement of surface quality is necessary due to both the most modern methods of obtaining surface structures and the specific use of obtained materials. For this reason, AFM is often used in the research of dental materials and, in particular, the layers of materials used in dentistry produced by the most modern methods providing very high-dimensional accuracy and surface quality. The ALD (atomic layer deposition) method plays an important role in dental prosthetics as one of the most modern coating techniques. ALD is a variant of the CVD (chemical vapor deposition) that allows the coating deposition of very accurately reflecting the topography of the substrate and ensures control of the thickness of the deposited coating with accuracy to the layer of atomic thickness, which allows coating complex-shaped surfaces [3].

In the case of layers of materials used in dentistry produced using the ALD method, the application of AFM for surface layer morphology tests is an important supplement to the surface quality tests carried out so far. It is extremely important to examine the roughness of the created layers, the low value of which makes it practically impossible to use a different test method. An important issue is also the ability to determine the quality of the layer made with very high magnification [4, 9].

The area of application of AFM is also surface investigations of materials whose physical properties (e.g., lack of electrical conductivity, low hardness) make it difficult to analyze with other methods, and determination of surface morphology is extremely important due to their potential application.

Atomic force microscope enables detailed analysis of topography of thin organic layers or composites with organic matrix and micro- and nanostructure fillers. The results of the analysis of topographic images obtained using this method allow to obtain detailed information on the state of the surface, damage on the nanoscale, and lumps of material deposited with possible agglomerates in the case of nanoparticle-strengthened composites. One of the basic advantages of AFM from the point of view of organic materials research is the possibility of imaging with a very large magnification surfaces of nonconductive materials. The complementary method of examining the surface of materials can be scanning electron microscopy (SEM) in the case of nonconductive organic materials forces coating their surface with thin conductive layers (gold, silver, and aluminum, among others) in order to perform the test [3, 4, 8, 9].

In addition, in case if thin layers with nanometric thickness (less than 100 nm) are from organic materials of mostly very low density, the embedding of the conductive layer can cause a significant decrease in the accuracy of imaging, e.g., in the form of fractures of additional conductive material. The necessity of depositing thin conductive layers on the surface of organic material also makes it impossible to carry out further research on a thin organic layer forming, for example, a heterojunction p-n due to a change in the optical properties of the system. In addition, thin layers of organic materials are easily degraded under the influence of the electron beam used in SEM. This makes the AFM an irreplaceable imaging method that provides an image of the surface of organic materials with the resolution capability of the order of a single atom. The low mechanical strength and, in particular, the low abrasion resistance, thin organic layers, and nanocomposites with an organic matrix make that in the research of this type of materials, the noncontact mode is most often used. In addition, in the case of composite layers with nanofillers, due to the high tendency to form agglomerates with sizes above 1 μm, the use of noncontact imaging avoids the risk of damage to the probe [3, 9].

An extremely important advantage of AFM is the ability to describe quantitative surface quality by determining roughness coefficients. The quantitative analysis of the surface topography of layers is carried out based on surface unevenness parameters. The basic parameters describing surface topographies are RMS (rough mean square) and Ra parameter. The RMS parameter is defined as the standard deviation from the mean value calculated from the area based on the point grid (characterized by the height of Zi) according to the formula 1:

$$RMS = \sqrt{\frac{1}{n} \cdot \sum_{i=1}^{n} \left(Z_i - \overline{Z}\right)^2} \quad (1)$$

where n is the number of points, Z_i is the height of each point for the z coordinate, and $\bar{Z}$ is the average value of the sample height for the z coordinate.

AFM also allows thickness measurement of thin organic layers, which is extremely convenient especially for materials with low hardness. Methods that use the determination of layer thickness based on measurement of optical properties require complex calculations or are relatively time-consuming. Using AFM, thickness measurement is simpler and faster. The measurement consists in removing the material of the layer up to the substrate material. Then, the measurement of surface topography at the boundary of the layer and the substrate is conducted. Through the linear analysis of the height change value in the topographic profile, it allows to specify the layer thickness. However, the accuracy of the test should depend on the total exposure of the substrate and the selection of a representative place thickness test. Therefore, especially in the case of layers with complex topography, it is necessary to perform measurements several times in order to determine the statistical average [9].

Author details

Tomasz Tański*, Bogusław Ziębowicz, Paweł Jarka and Marcin Staszuk

*Address all correspondence to: tomasz.tanski@polsl.pl

Division of Materials Processing Technology, Management, and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, Gliwice, Poland

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