Structural Investigation by Atomic Force Microscopy

Dana Maria Copolovici,[*] Cecilia Sîrghie

Institute of Technical and Natural Sciences Research-Development-Innovation of "Aurel Vlaicu" University , Elena Drăgoi St., No. 2-4, Cod 310330, Arad, Romania

* To whom correspondence should be addressed: *D.-M. Copolovici, E-mail: danaban76@gmail.com*

Abstract

Nanotechnology is an emerging field of research that has been widely applied in different fundamental science and engineering areas. An example of a nano-based device is the atomic force microscope, which is a widely used surface scanning apparatus capable of reconstructing at a nanometric scale resolution the 3D morphology of a wide variety of samples. Therefore, due to its versatility, sensitivity and unique capability to reveal the nanoscale structure of the samples, atomic force microscopy (AFM) produced, in the last years, a vast increase of reports of its use to determine the topography, electric properties, nanomechanics and even nanomanipulations of various samples in the fields of materials science, chemistry, physics, biology, microbiology, medicine, engineering, food products, forensic, etc.

Introduction

The development of new nanomaterials with a vast variety of applications in our day-life leaded to the need of use of new techniques for the structural and physico-chemical characterization. One of the advanced methods of investigation is the atomic force microscopy (AFM), which uses a microscope that was obtained after the extended of the research in the area of scanning tunneling microscopes for investigation of electrically non-conductive materials, such as proteins, DNA, etc. Binnig and Quate reported in 1986 the first invented AFM which used a very small probe-tip at the end of a cantilever (Binnig et al., 1986) and in 1989 was available the first commercial AFM. The general components of AFM are the following:

- laser: for an excellent spatial resolution and a high resolution over the photodiodes detector;

- photodiodes: for high sensitivity and detection in two dimensions (measures the deflection of the cantilever);
- feedback loop (controls *z*-sample position);
- cantilever (spring which deflects as probe tip scans the sample surface);
- probe tip (senses surface properties and causes the deflection of the cantilever);
- piezoelectric scanner (positions the sample (x, y, z) with high accuracy);
- computer (controls the system and performs the data acquisition, display and analysis).

AFM can image the surface topography with high magnifications, up to 1.000.000x, comparable with electronic microscopes, and in three dimensions, z-direction being usually higher than the horizontal x, y-plane.

Performing a data search in Web of Knowledge we found approximately 104.200 publications that reported research that used information obtained by using AFM, from which 85.600 were scientific articles from diverse areas of fundamental research and applied technologies such as: materials science 34.000; films (thin films, alloys) 25.600; chemistry 30.000; polymer science 7.700; composites 3.500; 2.600 cells and their functionality, dentins 157, etc. AFM imaging is a common technique (Barth et al., 2011; Ando, 2012) used for determination of carbon nanotubes (Baer et al., 2010; Rao et al., 2013; Tessmer et al., 2013), composites (Wang et al., 2013; Zhang et al., 2013), wood pulp and paper properties (Maximova et al., 2001; Koljonen et al., 2004; Maximova et al., 2004; Chhabra et al., 2005; Hou et al., 2006; Knutson et al., 2007; Ahola et al., 2008; Deng et al., 2008; Fatehi and Xiao, 2008; Wan et al., 2010; Wang et al., 2010; Gilli et al., 2012; Leitner et al., 2013; Miao and Hamad, 2013), biological samples and their mechanisms (Hoffmann and Dougan, 2012; Kalle and Strappe, 2012; Dufrene et al., 2013; Han et al., 2013; Miron-Mendoza et al., 2013; Singh, 2013) chemistry (Barth, et al., 2011; An et al., 2012), etc. For example AFM methods were use for imaging and measurements of DNA related research (Kalle and Strappe, 2012). A current challenge in the life was to reveal and to understand how biological systems change their structural, biophysical and chemical properties to adjust functionality. Addressing this issue has been severely hampered by the lack of methods capable of imaging biosystems at high resolution while simultaneously mapping their multiple properties. The recent developments in force-distance (FD) curve–based atomic force microscopy (AFM) enabled researchers to combine (sub) molecular imaging with quantitative mapping of physical, chemical and biological interactions.

Material and methods

An AFM device, namely NTEGRA Probe NanoLaboratory (NT-MDT, Moscow, Russia), equipped with an M Plan Apo 100x magnification objective that has the numerical aperture of 0.70 (Mitutoyo, Kawasaki, Japan) and a RPC-TVPCI camera which helps to locate the sample position were used. Software Nova_1644 for manipulating and analyzing the recorded data was employed. For storing the optical information a CCD camera was utilized. The samples were added to two-sided tape on sapphire support and the measurements were carried out under ambient conditions (temperature: 22 ± 1 ºC, relative humidity: 40 ± 10). Noncontact ‘Golden’ silicon cantilevers (NSG30 from NT-MDT, Moscow, Russia) with a resonance frequency of 320 ± 80 kHz, were used. All samples were measured in semicontact mode (“tapping” mode) to determine the topography images. Different surface areas of the samples have been investigated, as are mentioned in the figures.

Results and discussion

Atomic force microscope equipped with confocal Raman spectroscope is currently used in Institute of Technical and Natural Sciences Research-Development-Innovation of “Aurel Vlaicu” University from Arad, Romania (Figure 1). We performed imaging and measurements for a wide variety of samples such as composites (Popa et al., 2013), plant bast fibers (Figure 2 a, b), paper sheets (Figure 2 c, d), plants.

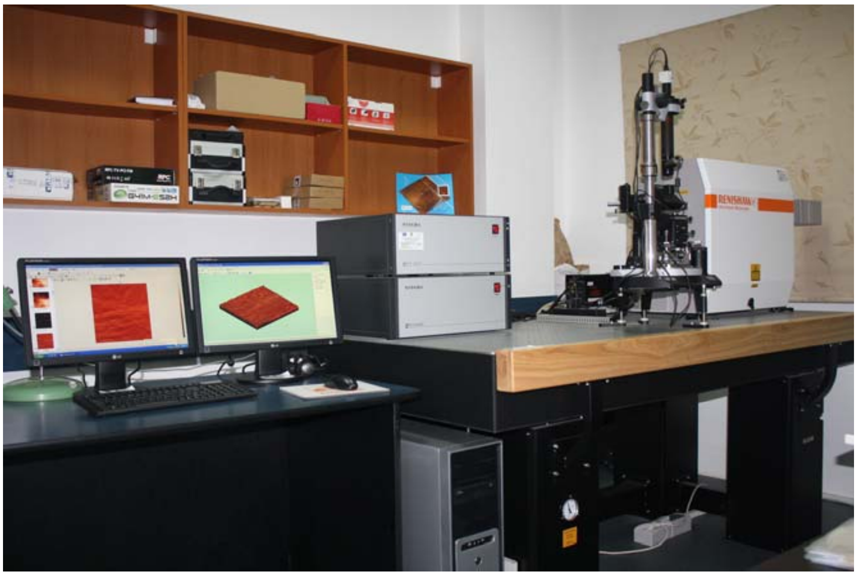

Figure 1. Atomic Force Microscope coupled with confocal Raman spectroscope used at ICDISTN of “Aurel Vlaicu” University.

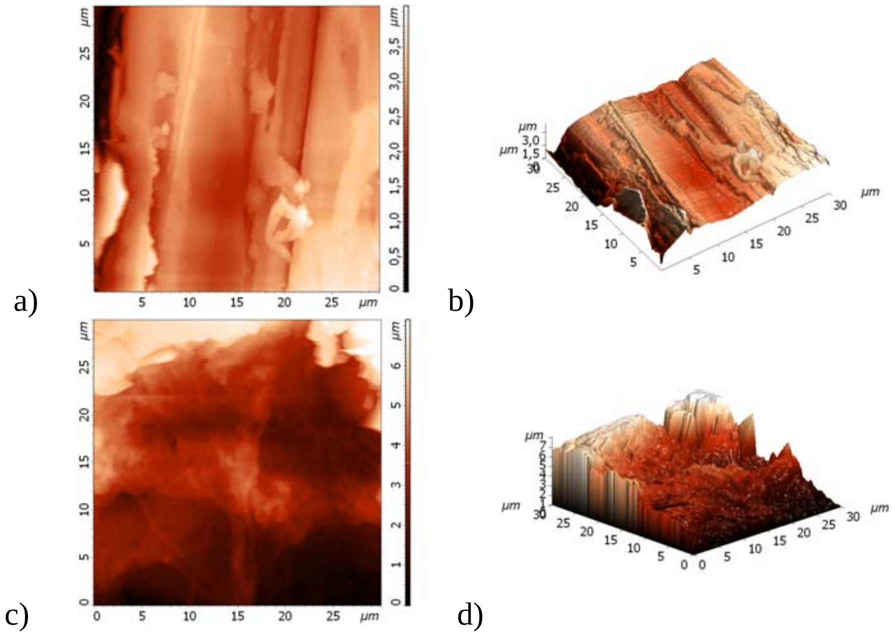

Figure 2. AFM images of the following samples:flax fiber a) 2D, b) 3D, paper sheet c) 2D, d) 3D.

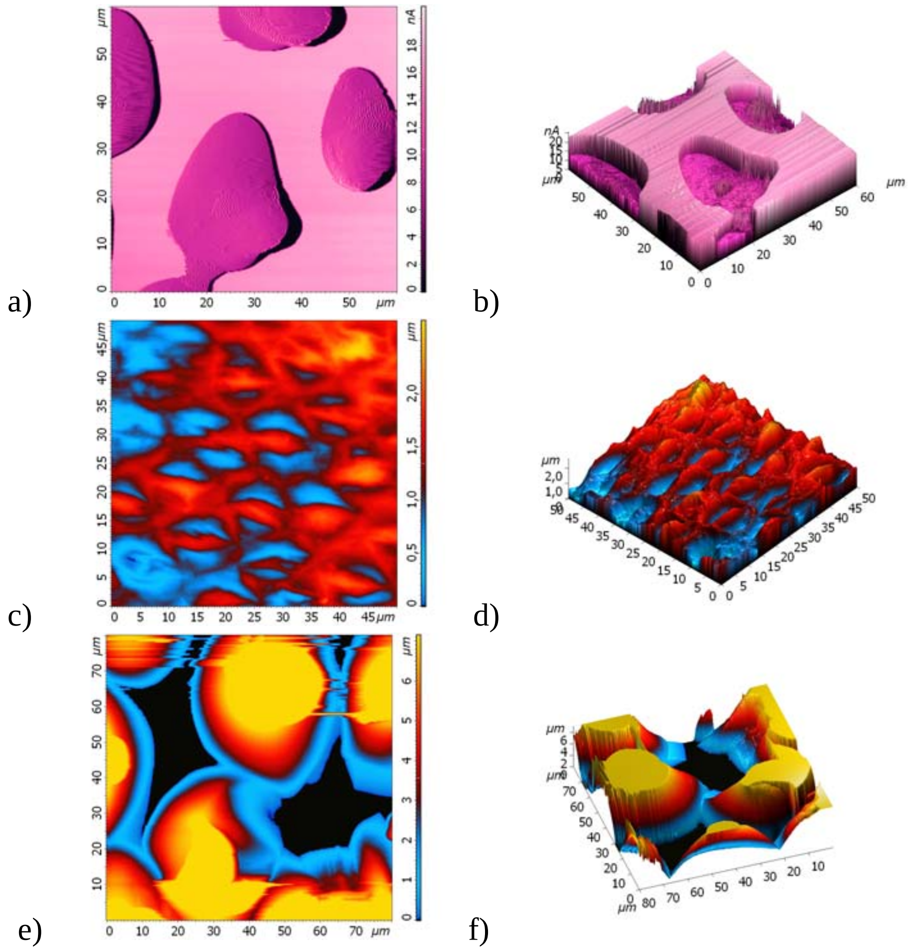

Figure 3. AFM images of petals of flowers: purple petal of *Hibiscus syriacus*: a) 2D, b) 3D; red petal of an *Hawaiian Hibiscus*: c) 2D, d) 3D; and protuberances from the edge of a petal of *Phalaenopsis amabilis* (Moth Orchid): e) 2D, f) 3D.

In Figure 3 are exhibited AFM images performed in semicontact topography (tapping) mode for Hibiscus petals and Moth Orchid petals. The AFM images unveiled the different morphologies of the petals measured.

Acknowledgements

This work was supported by project co-funded by European Union through European Regional Development Fund Structural Operational Program “Increasing of Economic Competitiveness” Priority axis 2, operation 2.1.2. ID project 679, cod SMIS CNSR 12638: “Bast plants – Renewable Strategic Resources for European Economy”. - „BASTEURES”. No. 210/2010 POS-CCE.

References

Ahola, S., Osterberg, M., Laine, J., 2008. Cellulose nanofibrils-adsorption with poly(amideamine) epichlorohydrin studied by QCM-D and application as a paper strength additive. Cellulose 15, 303-314.

An, B.-K., Gierschner, J., Park, S.Y., 2012. pi-Conjugated Cyanostilbene Derivatives: A Unique Self-Assembly Motif for Molecular Nanostructures with Enhanced Emission and Transport. Accounts of Chemical Research 45, 544-554.

Ando, T., 2012. High-speed atomic force microscopy coming of age. Nanotechnology 23.

Baer, D.R., Gaspar, D.J., Nachimuthu, P., Techane, S.D., Castner, D.G., 2010. Application of surface chemical analysis tools for characterization of nanoparticles. Analytical and Bioanalytical Chemistry 396, 983-1002.

Barth, C., Foster, A.S., Henry, C.R., Shluger, A.L., 2011. Recent Trends in Surface Characterization and Chemistry with High-Resolution Scanning Force Methods. Advanced Materials 23, 477-501.

Binnig, G., Quate, C.F., Gerber, C., 1986. Atomic Force Microscope. Physical Review Letters 56, 930-933.

Chhabra, N., Spelt, J., Yip, C.M., Kortschot, M.T., 2005. An investigation of pulp fibre surfaces by atomic force microscopy. Journal of Pulp and Paper Science 31, 52-56.

Deng, Y., He, B., Qian, L., 2008. Study on Mechanical Pulp's Fiber Surface Properties Using AFM and XPS, Proceedings of International Conference on Pulping, Papermaking and Biotechnology 2008: Icppb '08, Vol 2, pp. 43-46.

Dufrene, Y.F., Martinez-Martin, D., Medalsy, I., Alsteens, D., Mueller, D.J., 2013. Multiparametric imaging of biological systems by force-distance curve-based AFM. Nature Methods 10, 847-854.

Fatehi, P., Xiao, H., 2008. The influence of charge density and molecular weight of cationic poly (vinyl alcohol) on paper properties. Nordic Pulp & Paper Research Journal 23, 285-291.

Gilli, E., Schmied, F., Diebald, S., Horvath, A.T., Teichert, C., Schennach, R., 2012. Analysis of lignin precipitates on ozone treated kraft pulp by FTIR and AFM. Cellulose 19, 249-256.

Han, H.-M., Bouchet-Marquis, C., Huebinger, J., Grabenbauer, M., 2013. Golgi apparatus analyzed by cryo-electron microscopy. Histochemistry and Cell Biology 140, 369-381.

Hoffmann, T., Dougan, L., 2012. Single molecule force spectroscopy using polyproteins. Chemical Society Reviews 41, 4781-4796.

Hou, Q.X., Chai, X.S., Yang, R., Elder, T., Ragauskas, A.J., 2006. Characterization of lignocellulosic-poly(lactic acid) reinforced composites. Journal of Applied Polymer Science 99, 1346-1349.

Kalle, W., Strappe, P., 2012. Atomic force microscopy on chromosomes, chromatin and DNA: A review. Micron 43, 1224-1231.

Knutson, K., Pu, Y., Elder, T., Buschle-Diller, G.B., Yang, R., Thomson, C., Kim, D.H., Dang, Z., Ragauskas, A.J., 2007. Effect of photolysis on 17th/18th century paper. Holzforschung 61, 131-137.

Koljonen, K., Osterberg, M., Kleen, M., Fuhrmann, A., Stenius, P., 2004. Precipitation of lignin and extractives on kraft pulp: effect on surface chemistry, surface morphology and paper strength. Cellulose 11, 209-224.

Leitner, J., Zuckerstaetter, G., Schmied, F., Kandelbauer, A., 2013. Modifications in the bulk and the surface of unbleached lignocellulosic fibers induced by heat treatment without water removal: effects on tensile properties of unrefined kraft pulp. European Journal of Wood and Wood Products 71, 101-110.

Maximova, N., Osterberg, M., Koljonen, K., Stenius, P., 2001. Lignin adsorption on cellulose fibre surfaces: Effect on surface chemistry, surface morphology and paper strength. Cellulose 8, 113-125.

Maximova, N., Stenius, P., Salmi, J., 2004. Lignin uptake by cellulose fibres from aqueous solutions. Nordic Pulp & Paper Research Journal 19, 135-145.

Miao, C., Hamad, W.Y., 2013. Cellulose reinforced polymer composites and nanocomposites: a critical review. Cellulose 20, 2221-2262.

Miron-Mendoza, M., Koppaka, V., Zhou, C., Petroll, W.M., 2013. Techniques for assessing 3-D cell-matrix mechanical interactions in vitro and in vivo. Experimental Cell Research 319, 2470-2480.

Popa, M.I., Pernevan, S., Sirghie, C., Spiridon, I., Chambre, D., Copolovici, D.M., Popa, N., 2013. Mechanical Properties and Weathering Behavior of Polypropylene-Hemp Shives Composites. Journal of Chemistry.

Rao, C.N.R., Subrahmanyam, K.S., Matte, H.S.S.R., Maitra, U., Moses, K., Govindaraj, A., 2013. Graphene: Synthesis, Functionalization and Properties. International Journal of Modern Physics B 25, 4107-4143.

Singh, A.V., 2013. Biotechnological applications of supersonic cluster beam-deposited nanostructured thin films: Bottom-up engineering to optimize cell-protein-surface interactions. Journal of Biomedical Materials Research Part A 101, 2994-3008.

Tessmer, I., Kaur, P., Lin, J., Wang, H., 2013. Investigating bioconjugation by atomic force microscopy. Journal of Nanobiotechnology 11.

Wan, J., Wang, Y., Xiao, Q., 2010. Effects of hemicellulose removal on cellulose fiber structure and recycling characteristics of eucalyptus pulp. Bioresource Technology 101, 4577-4583.

Wang, B., Guan, D., Gao, Z., Wang, J., Li, Z., Yang, W., Liu, L., 2013. Preparation of graphene nanosheets/SnO2 composites by pre-reduction followed by in-situ reduction and their electrochemical performances. Materials Chemistry and Physics 141, 1-8.

Wang, B., He, B., Li, J., 2010. Study on Lignin Coverage of Masson Pine Fiber. Bioresources 5, 1799-1810.

Zhang, S., Yu, A., Song, X., Liu, X., 2013. Synthesis and characterization of waterborne UV-curable polyurethane nanocomposites based on the macromonomer surface modification of colloidal silica. Progress in Organic Coatings 76, 1032-1039.