Applications of Atomic Force Microscopy in Textiles

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ABSTRACT
Potential applications of atomic force microscopy (AFM) in textiles are explained. For this purpose samples were carefully selected from both natural and synthetic fibers. Cotton, wool, conventional polyethylene terepthalate (PET), antibacterial PET, and antistatic PET were investigated by means of 3D topography imaging, phase imaging, and calculation of their Rq values. The distribution of the additives in the cross sections of antibacterial PET and antistatic PET were analyzed. Moreover, differences between inner and outer cross section of trilobal PET was observed by force spectroscopy. The results are discussed considering the fiber properties. It is concluded that AFM is a powerful tool to investigate different properties of textile fibers, and it gives valuable information.

Keywords: Atomic force microscopy, textile fibers, surface properties.

INTRODUCTION
Atomic force microscopy (AFM) is a surface analytical tool developed during the mid-1980s by Binnig, Quate, and Gerber as a daughter technique to scanning tunneling microscopy [1,2]. AFM was developed as an alternative for imaging either conducting or nonconducting surfaces. Therefore it allows investigating textile fibers without any coating and at ambient conditions.

In AFM, the tip is attached to a flexible cantilever and is brought in contact with the surface (*Figure 1*). The force between the tip and the surface is detected by sensing the cantilever deflection. A topographic image of the surface is obtained by plotting the deflection as a function of the *x-y* position. In a more common mode of operation, a feedback loop is used to maintain a constant deflection, while the topographic information is obtained from the cantilever vertical displacement [3].

Besides 3D imaging, AFM can be also used to determine the characteristics of a surface quantitatively. Thus, different surfaces can be compared quantitatively rather than qualitatively in a straight forward fashion. The simplest and most common method used for the observation of surface topography is called the root mean square roughness calculation (R_q). R_q values can be easily calculated using software supplied with the AFM instrument [2,4].

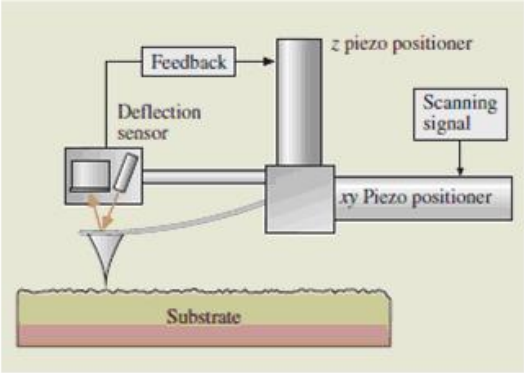


FIGURE 1. AFM sy stem [3].

Phase imaging is a powerful extension of tapping mode AFM that provides nanometer-scale information about surface structure often not revealed by other scanning probe microscopy techniques. By mapping the phase of the cantilever oscillation during the tapping mode scan, phase imaging goes beyond simple topographical mapping to detect variations in composition, adhesion, friction, viscoelasticity, and perhaps other properties. Applications include identification of contaminants, mapping of different components in composite materials, and differentiating regions of high and low surface adhesion or hardness. Phase imaging promises to play an important role in the ongoing study of material properties at the nanometer scale [5].

Another major application of AFM is force spectroscopy, the direct measurement of tip-sample interaction forces as a function of the gap between

the tip and sample (the result of this measurement is called a force-distance curve). For this method, the AFM tip is extended towards and retracted from the surface as the deflection of the cantilever is monitored as a function of piezoelectric displacement [6]. Force curves, provide valuable information on local material properties such as elasticity, hardness, Hamaker constant, adhesion and surface charge densities [7].

In this study AFM was used to investigate different properties of textile fibers by means of 3D imaging, phase imaging, calculating R_q values, and force spectroscopy. The samples were carefully selected to introduce the performance of AFM. The results are explained considering the fiber structures.

MATERIAL AND METHOD

In this study cotton, wool, conventional polyethylene terepthalate (PET), antibacterial PET, antistatic PET, and trilobal PET fibers were used. Different characteristics of the fibers were investigated by using different modes of AFM. 3D topography imaging; phase imaging and calculation of Rq value were performed for all the samples except trilobal PET. Investigating the distribution of the additives in fiber cross section was performed only for antibacterial PET and antistatic PET fibers. Force spectroscopy was used in order to observe the differences between inner and the outer parts of the trilobal PET fiber. The materials and the methods used in this study are summarized in *Table I*.

TABLE I. Materials and the methods used in the study.

Material	Method
Cotton	3D topography imaging, phase imaging, calculation of Rq value
Wool	
PET fiber, fully drawn	
Antibacterial PET fiber, fully drawn, contains silver ions	3D topography imaging, phase imaging, calculation of Rq value, investigating the distribution of the additives in fiber cross section
Antistatic PET, fully drawn, contains carbon black particles	
Trilobal PET fiber, fully drawn	Force spectroscopy

The AFM studies were performed on a Veeco Dimension 3100 atomic force microscope, in the Laboratories of Max-Planck Institute for Polymer Research (Mainz, Germany). The topography imaging of the surface of the fibers were carried out in tapping mode AFM. In these studies phase imaging of the fibers were also performed. For roughness calculations, the raw images were flattened (second order) and then the R_q values of the samples were calculated using the software (version v720) supplied with the atomic force microscope. Furthermore PET, antibacterial PET, and antistatic PET fibers were embedded in epoxy and had smooth cuts by microtome. The distribution of particles in fiber cross section was analyzed by contact mode AFM. The force spectroscopy studies were carried out with standard force-volume mode of AFM.

RESULTS AND DISCUSSION

AFM images of the samples are given in *Figure 2 and Figure 3*.

Cotton and wool fibers were analyzed with two different scan sizes in order to get more detailed information about their surfaces. Characteristic surface features were observed for all the samples. For cotton fibers the fibrillar structure and for wool the scales on the surface were recorded. PET showed a smooth surface with few impurities. On the other hand antistatic PET and antibacterial PET gave rougher surfaces due to the additives inside their structures.

R_q values of the fibers are given in *Table II*. R_q values are dependent on scan size; therefore they were calculated only for AFM images with the same scan size (2 μm × 2 μm). It was observed that wool gave the highest R_q value, while cotton gave the lowest. On the other hand PET, antibacterial PET, and antistatic PET gave similar R_q values. These results may be seen incompatible with the AFM images; however R_q is calculated from height variations from a mean surface level [4], which is the main reason for this difference. Moreover in most of the AFM images the scale cannot keep constant to get more detailed images. Therefore in determining the surface characteristics of the samples it is thought to be very useful to use R_q values, in addition to AFM images.

TABLE II. R_q values of the fibers.

Material	R_q (nm)
Cotton	7.02
Wool	22.7
PET	11.6
Antibacterial PET	11.7
Antistatic PET	12.0

AFM images of the cross sections of antibacterial PET and antistatic PET are given in *Figure 4*. In the cross section of antibacterial PET, the silver particles were not recognized. However in the cross section of antistatic PET the carbon black particles were clearly

seen. This shows that in antibacterial PET, the silver particles tend to aggregate on the surface of the fiber. On the other hand in antistatic PET, the carbon black particles spread homogeneously across the cross section of the fiber. Recently there is an increasing demand to fibers with additives. To understand the performance of these fibers it is very beneficial to know the distribution of the additives inside of the fiber. AFM can be easily used in such investigations.

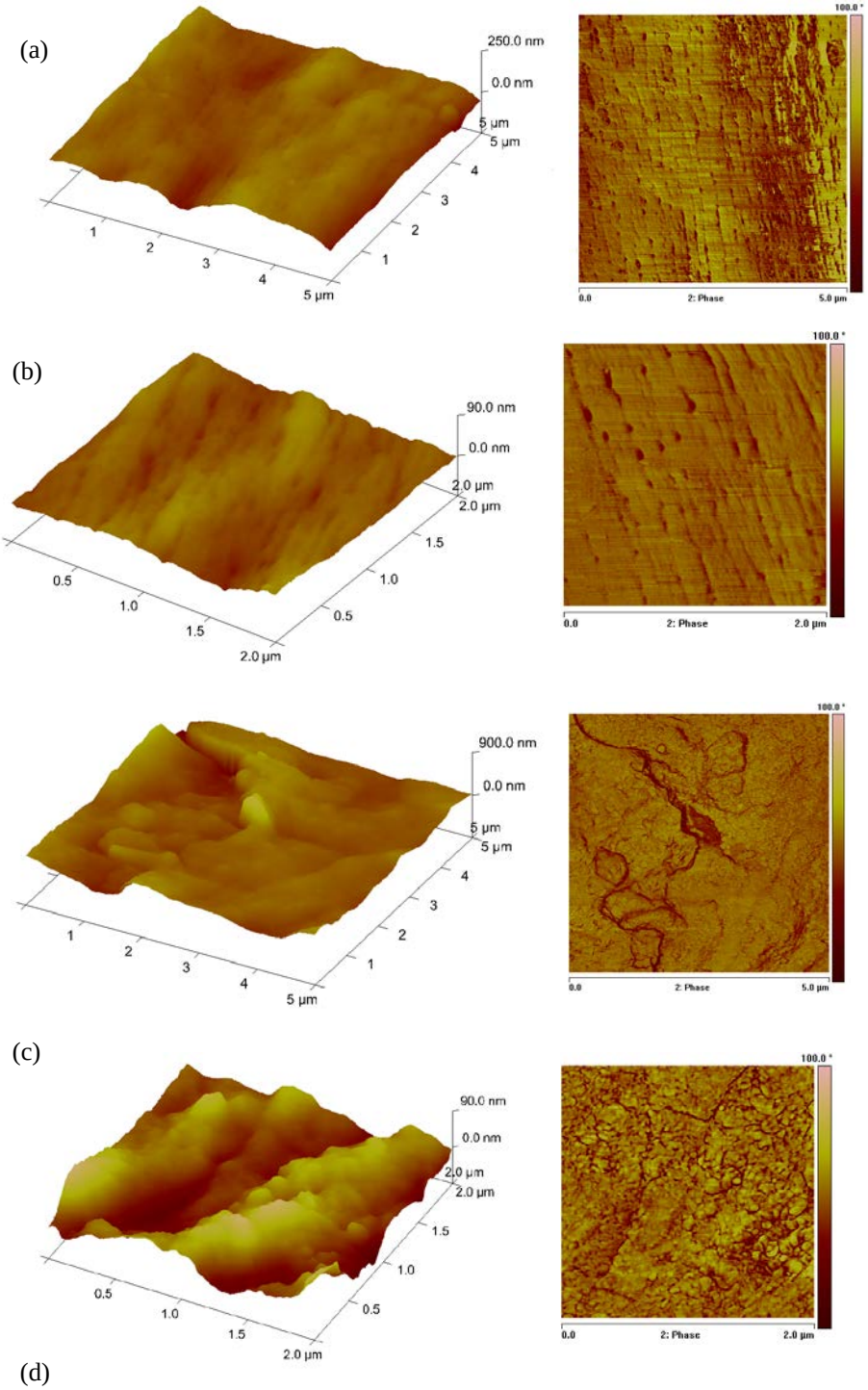


FIGURE 2. AFM images of the samples: a)Cotton topography and phase (5 µm × 5 µm), b) Cotton topography and phase (2 µm × 2 µm), c)Wool topography and phase (5 µm × 5 µm) d)Wool topography and phase (2 µm × 2 µm).

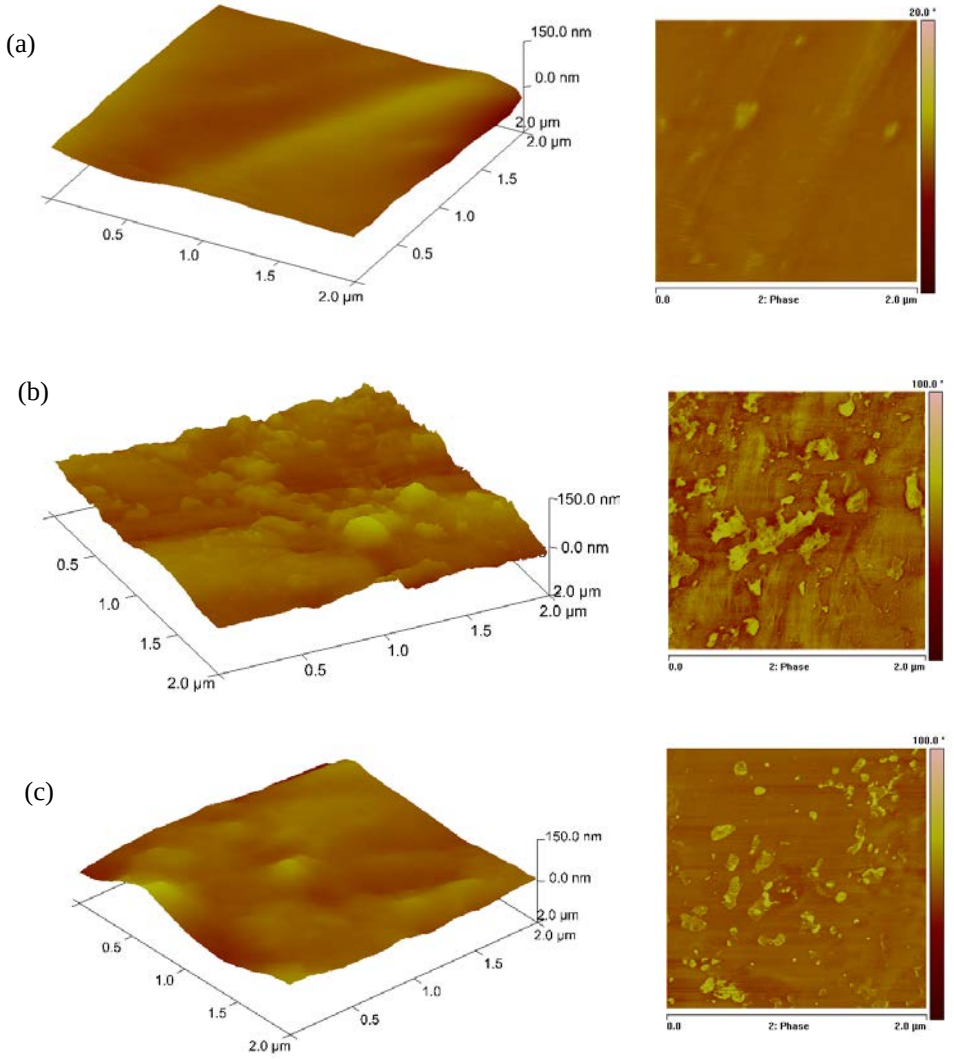
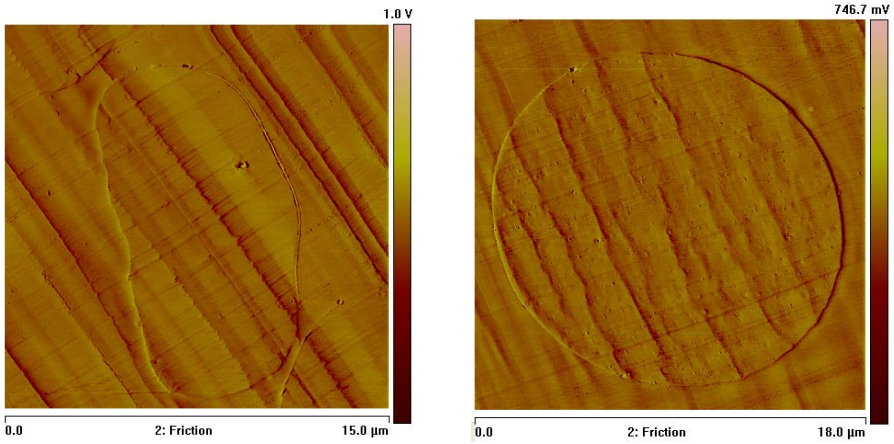


FIGURE 3. AFM images of the samples: a) PET, b)Antistatic PET, c) Antibacterial PET.



(a) (b)

FIGURE 4. AFM images of the cross sections of the fibers: a)Antibacterial PET friction, b)Antistatic PET friction.

Figure 5 shows the force-distance curves obtained from the cross section of the trilobal PET. The curves were taken from two different places: near the surface and near the center. The curves indicate no pronounced difference between the rim and the center of the cross section. Trilobal PET is produced by the melt spinning process. During melt spinning inner and outer parts of the fibers may show different solidification behaviors, resulting a non-homogeneous structure across the fiber. Detecting homogeneity of the fiber cross section is very beneficial, since it directly affects mechanical behavior of the fibers. AFM can be used to analyze the differences in the structure across the fiber cross section effectively.

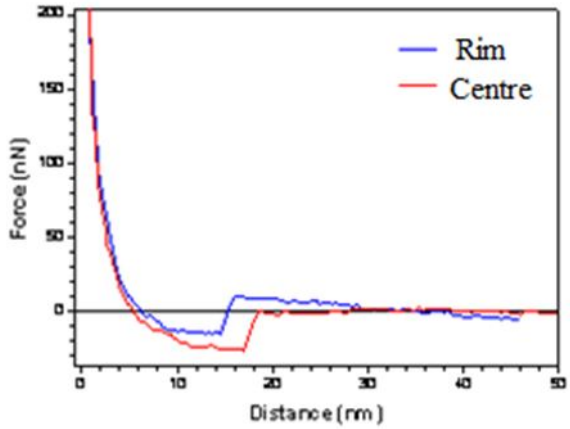


FIGURE 5. Force-distance curve of trilobal PET (Taken from 2 different places on the fiber cross-section. Near the surface and near the centre).

CONCLUSION

AFM gives 3D topography images of the fibers with high resolution. Calculation of R_q values allows determining the surface characteristics of textile fibers quantitatively. The distribution of particles in fibers with additives can be easily analyzed by AFM. Moreover the differences between inner and the outer part of fibers can be investigated by force curves.

It is concluded that AFM is a very powerful tool to investigate different properties of textile fibers, and it gives valuable information.

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