Zsófia Kóczán

University of Sopron, Paper research institute,

APPLICABILITY OF AFM IN PAPER RESEARCH

ABSTRACT

Nowadays, there is a growing demand for paper to be used ever in different areas of industry/ To solve this, paper needs to be endowed with different properties that will make it more applicable in each area. In the paper industry there are a lots of materials and technologies which can increase the advantageous properties of paper, such as fillers, sizing agents, or smoothing. All these properties can be observed with machines which have been developed to test paper. In this study, we deviated from the general equipment which used for paper examinations. We used atomic force microscopy (AFM) to examine surface of paper in the nanometer size range.

INTRODUCTION

Atomic Force Microscopy

The AFM is a very-high-resolution type of microscope, with a final resolution equal to a fraction of a nanometer. The mechanical probe “touches” the surface of the sample, to gather information. The AFM was invented in 1982 by IBM scientists. The first AFM which was available commercially was introduced in 1989. **[1]**

The AFM has three, main functions: force measurement, topographic imaging, and material manipulation. **[1]**

In the case of force measurement, the AFM can be used to measure the forces between the probe and the sample. Using force spectroscopy, we can examine the mechanical properties of the material, like the Young's modulus, which measures stiffness.

For imaging purposes, it can create a high-resolution three-dimensional image of the surface (topography) from the forces exerted by the surface on the probe. **[1]**

The forces between tip and sample can be used to change the properties of the sample with a controlled way. For example, it can use atomic manipulation, scanning probe lithography and the cells local stimulation. **[1]**

© Kóczán Zs.

The major difference between AFM and other microscopy technologies is that AFM does not use lenses or electron beams. Therefore, it is not limited in spatial resolution due to diffraction or aberrations. It is not necessary to prepare the space for guiding the electron beam (by creating a vacuum) or marking the sample. **[1]**

Paper research

AFM has already been used for various studies in the field of paper research. In one research, thermal aging studies were made on the cellulose insulation paper of a power transformer using AFM. **[2]** In another study examined pulp. In this case they examined the surface properties of spruce kraft pulp cooked for different times as well as the bleached pulps. **[3]** In another research, the AFM was used to examine the cellulosic fibres from bleached Eucalyptus globulus kraft pulp. **[4]**

Overall objectives

In this study we explored the use of AFM in Paper investigation which is a key element of paper research studies focusing on the components and the geometry of the surface.

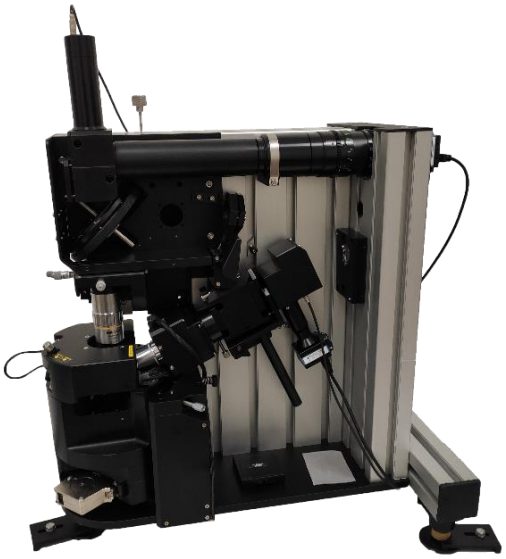
MATERIALS AND METHODS

We used an AFM produced by Horiba France SAS to examine paper samples It included an AFM head and the AFM Smart SPM.

Fig 1

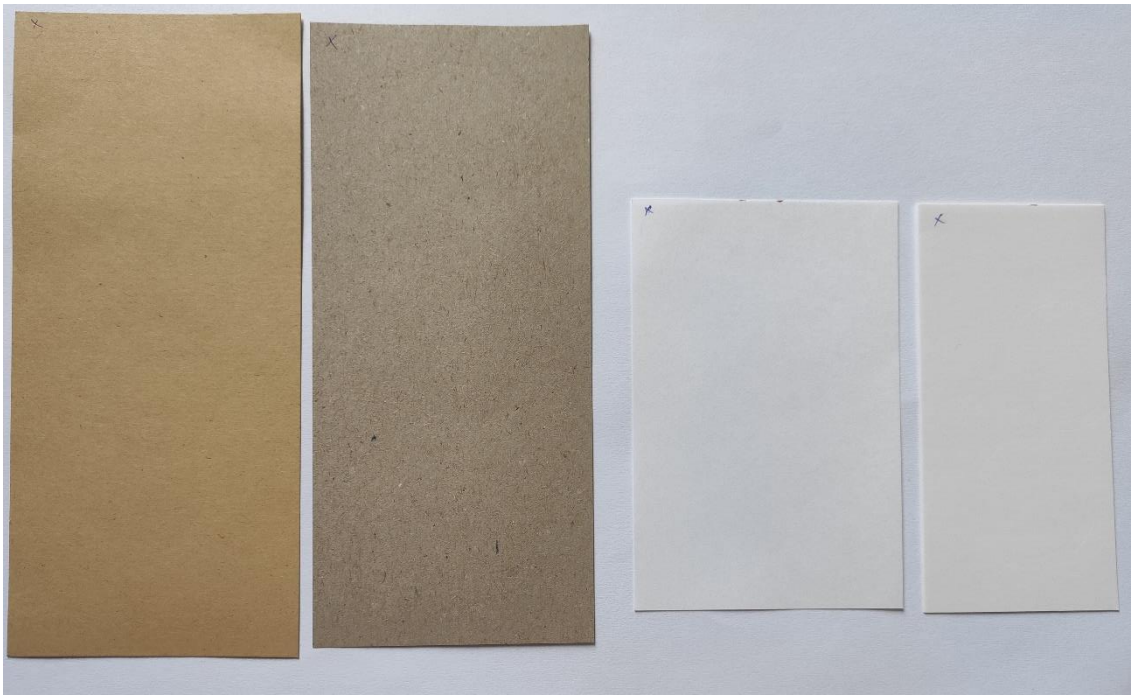
Atomic Force Microscopy by Horiba.

© Kóczán Zs.



For AFM imaging we used aluminum tips. Our tips were made by MikroMasch.

In this study we examined four different types of paper. The paper samples are shown in Fig. 2. The samples are shown from left to right. First is a brown paper with a 150 g/m^2 grammage, which seems to be made from secondary fiber material. The next type is a 90 g/m^2 paper made from recycled materials., making its color greyish. Third type is a 120 g/m^2 smoothed paper. which is very soft paper to the touch. The fourth type is a thicker paper, with 230 g/m^2 grammage. This is a coated paper because of the surface is very shiny. For each sample, the test was performed on a 5 x 5 μm surface.



© Kóczán Zs.

Fig. 2

The types of the examined paper samples.

RESULTS

The examination

Fig. 3 on the far left shows the height of the brown paper sample height, while the picture on the right shows the phase of the sample. The surface is not too smooth. The picture shows that there are a lot of different components. The related parts that are deeper in the dark places, while higher in lighter parts. If we compare the height of the picture with the phase image, we also see these related parts. We assumed that these are portions of the fiber. Between the fibers there are different materials. These can be fillers and starch particles.

The phase image depends on the interaction between the tip of the probe and surface of the paper sample. It depends on the properties of the surface, like adhesive or stiffness properties. **[5]**

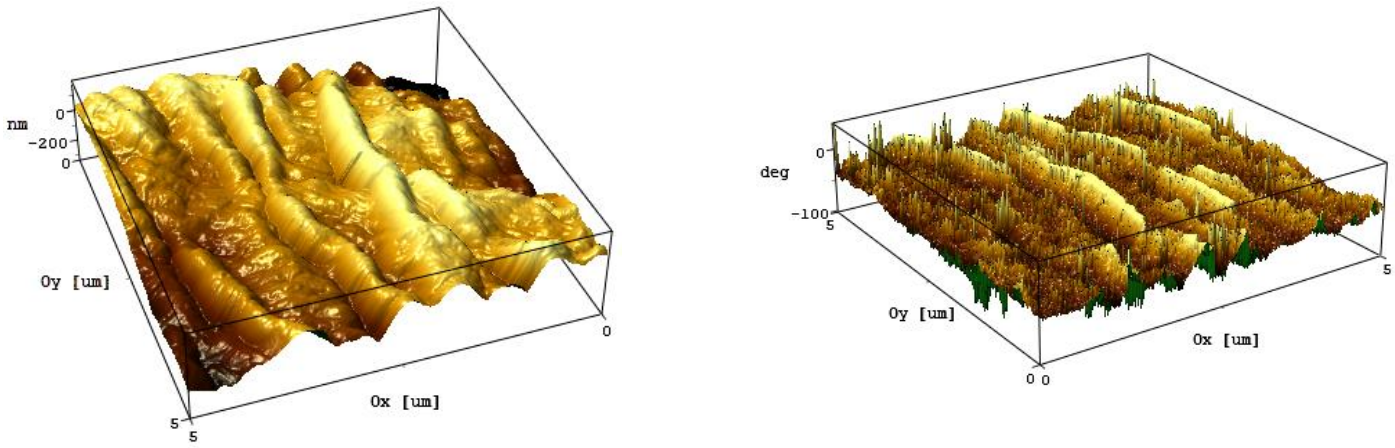


Fig. 3

Picture of the height of the first sample (left) and its phase (right).

The Fig. 4 shows the second, gray sample's height and phase. We can see this sample has rougher surface than the previous sample. using a scale of values ranging from -200 to 200 nm. On the picture of phase, we can observe large contiguous areas. These may indicate fillers which form an even part of the paper. Other areas where we see fluctuations may be starch granules and other additives of the paper.

© Kóczán Zs.

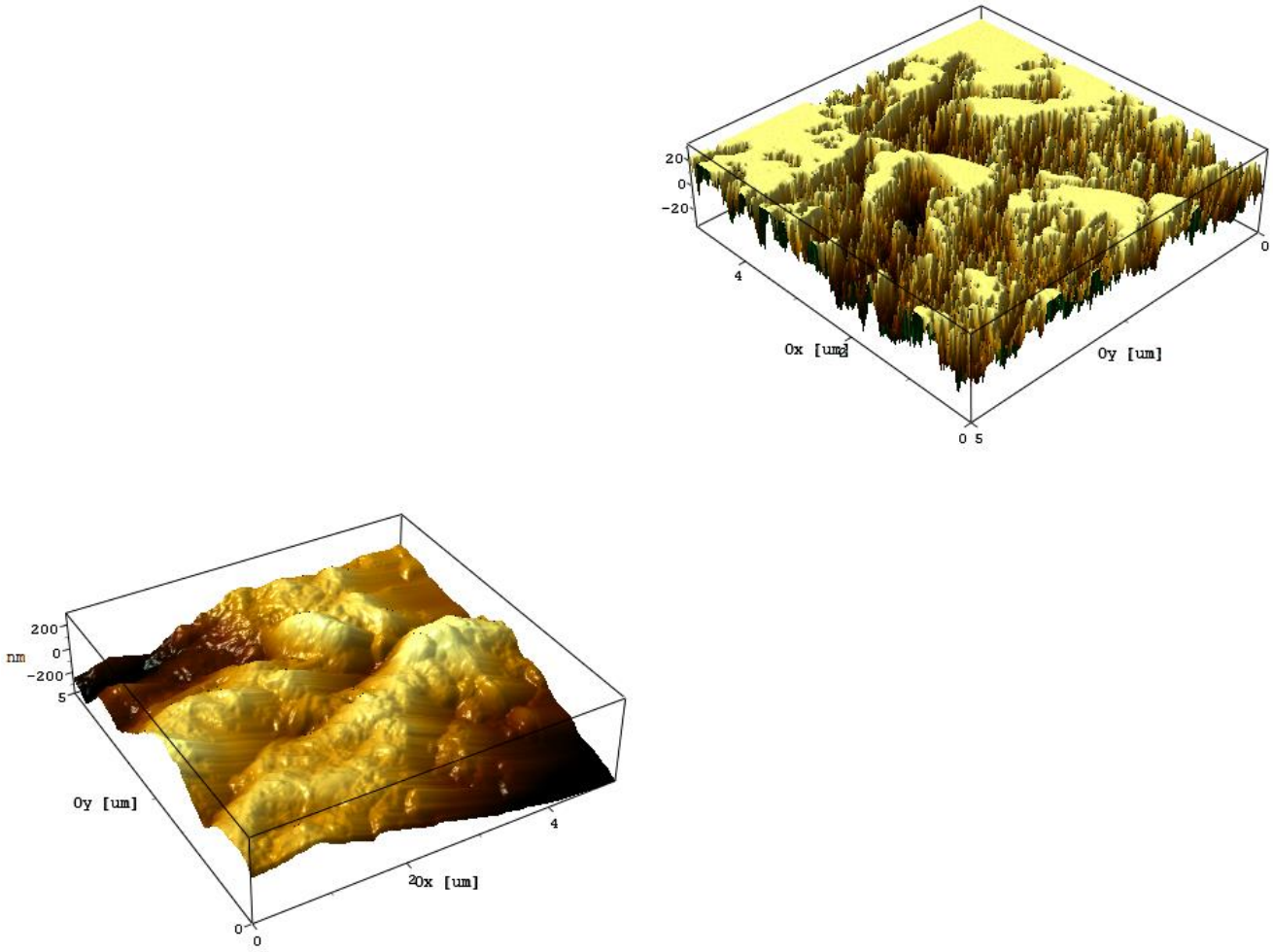


Fig. 4

The picture of the second sample height (left) and phase (right).

© Kóczán Zs.

The next sample is a white smoothed paper. If we touch this paper, we feel it is very soft, but in the picture, we see that at the nanometres size it has a varied surface. In the picture of phase, we see a contiguous area, which can be a fiber or fillers, starch grains, and sizing agents.

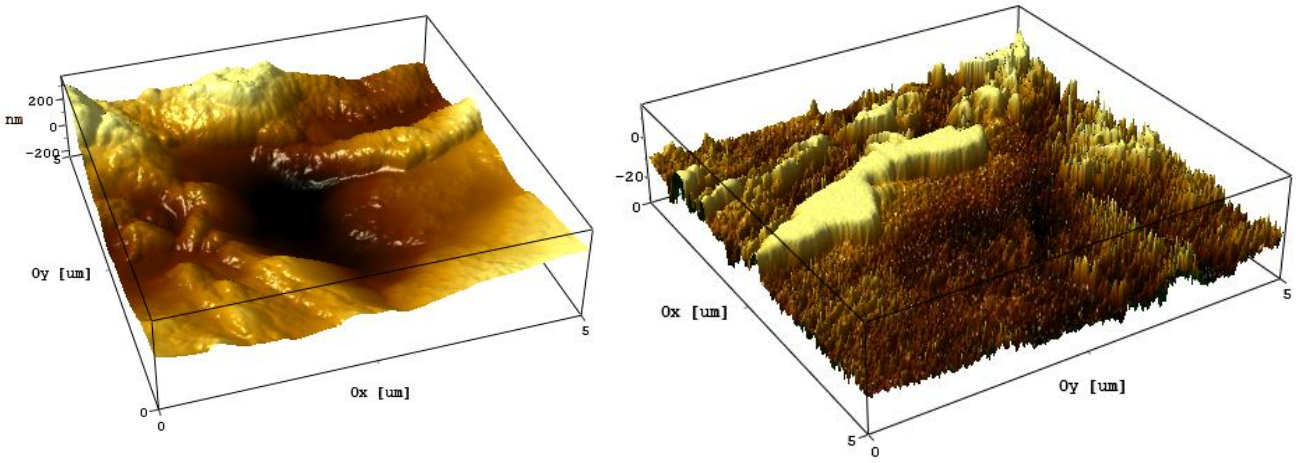
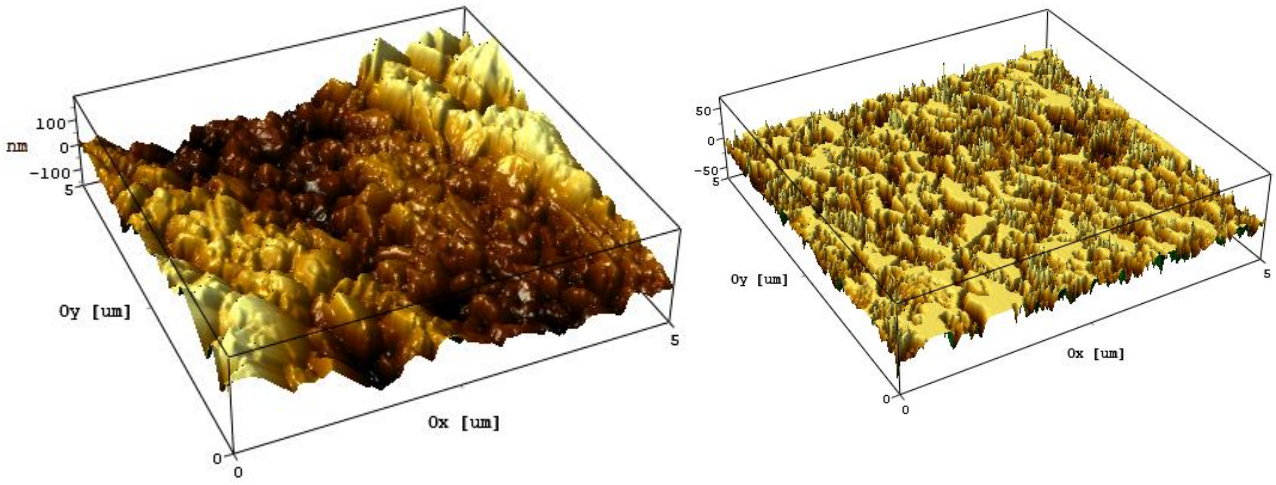


Fig. 5

The picture of the third sample height (left) and phase (right).

The fourth sample shows its height and phase. the height pictures shows many more mounds than in the previous samples. There is a sizing agent on the surface of the paper. On closer inspection we can see that the coating cannot create a perfectly smooth surface. The surface irregularities of the uncoated paper remain to some extent after coating. In the picture of phase, we can notice that the sizing agent did not create a completely connected surface. The numerous yellow cohesive parts are the sizing material, and the other areas between them show the uncoated or less coated parts of the paper.

© Kóczán Zs.



CONCLUSION

The AFM can be used for research on the surface characteristics and the materials of paper. In addition, AFM may be suitable for examining the various coatings which are created on paper. Furthermore, AFM could be used to examine paper fibers. The fibers can be analyzed in the pulping phase, and the results can be used to draw conclusions about the properties of the paper that will be made.

Fig. 6

The picture of the fourth sample height (left) and phase (right).

ACKNOWLEDGEMENT

The work was carried out as part of the "Sustainable Raw Material Management Thematic Network – RING 2017", EFOP-3.6.2-16-2017-00010 project in the framework of the Széchenyi 2020 Program. The realization of this project is supported by the European Union, co-financed by the European Social Fund.

REFERENCES

1. https://en.wikipedia.org/wiki/Atomic_force_microscopy
2. Liao Ruijin, Tang Chao, Yang Lijun and Chen Huanchao „Thermal Aging Studies on Cellulose Insulation Paper of Power Transformer Using AFM", 2006.

© Kóczán Zs.

3. Johanna Gustafsson, Laura Ciovica, Jouko Peltonen „ The ultrastructure of spruce kraft pulps studied by atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS)”, 2003.
4. A. T. Paiva, S. M. Sequeira, D. V. Evtuguin, A. L. Kholkin, and I. Portugal „Nanoscale Structure of Cellulosic Materials: Challenges and Opportunities for AFM”, 2007.
5. https://www.icspicorp.com/blog/phase-images

© Kóczán Zs.