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The study of harmonic imaging by AFM

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Abstract

Atomic Force Microscopy (AFM) is a powerful tool for the analysis of surface samples with accuracy of single atoms. The existing methods include surface roughness, porosity and hardness of the test portion of the sample. The article presents the preliminary studyof a new AFM method of surface analysis. The study indicates that there may be a correlation between intensity of a harmonic resonance frequency of the needle and the system response. The suggested correlation can characterize elasticity of the analyzed surface.

Keywords: atomic force microscopy, surface, harmonics.

Badania harmonicznych w obrazowaniu AFM

Streszczenie

Mikroskop sił atomowych (ang. Atomic Force Microscope – AFM) został wynaleziony w 1986 roku [1] jako alternatywa dla skaningowego mikroskopu tunelowego (ang. Scanning Tunneling Microscope – STM), którego nie można użyć do badań nad materiałami nieprzewodzącymi. AFM umożliwia pomiary materiałów zanurzonych w cieczach, co pozwala badać żywe preparaty biologiczne w warunkach zbliżonych do ich naturalnego środowiska [2]. W artykule przedstawiono zasadę pracy mikroskopu (rys. 1) oddziaływującego siłami van der Waalsa (opisanymi funkcją Lennarda – Jonesa) między ostrzem skanującym a próbką (1) (rys. 2) [3]. Opisano trzy podstawowe tryby pracy mikroskopu: kontaktowy, przerywany [4-6] oraz bezkontaktowy (2). Opierając się na dotychczasowych badaniach [7] wyznaczających różne właściwości materiału w zależności od ich budowy (rys. 3, rys. 4) przebadano próbkę warystora (rys. 5) pod kątem obecności i poziomu kolejnych harmonicznych pobudzającej częstotliwości rezonansowej w odpowiedzi układu. Przeprowadzone pomiary wskazują, że może istnieć związek między intensywnością kolejnych harmonicznych, a właściwościami badanej powierzchni.

Słowa kluczowe: mikroskop sił atomowych, powierzchnia, harmoniczne.

1. AFM surface examination technique

Atomic force microscope was invented in 1986 [1] as an alternative device to the scanning tunneling microscope (STM), which cannot be used for studies of non-conductive materials. The AFM, however, has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, and biological samples under conditions resembling their natural environment. Thanks to this property, AFM can be effectively used to study living cells [2]. Its additional advantage, in comparison with the STM microscope, is a non-destructivity – electric charges do not pass through the sample during the test.

The operating principles of AFM are very simple: the investigated material is placed on a piezoelectric tube, the sample is tested by a scanning needle tip positioned at the free end of the cantilever. Every surface depressions and bulges indicate changes of the cantilever deflection. Typically, the deflection is measured using a laser spot reflected from the top surface of the cantilever into an array of photodiodes (Fig. 1). This information is transmitted to a computer and the dedicated software generates a topography map and/or other properties of interest.

AFM allows the examination of twistings of the lever caused by the presence of forces parallel to the sample plane. Therefore, in addition to topographic measurements, the microscope can also provide information about mechanical properties of the sample, such as flexibility, adhesion force or friction.

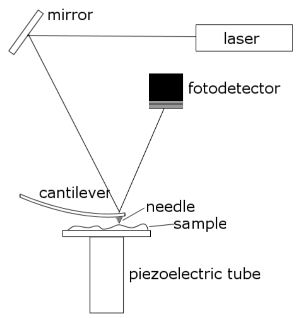


Fig. 1. Schematic illustration of AFM
Rys. 1. Schemat budowy AFM

Because of AFM's versatility, it has been applied to a large number of researches. The atomic force microscope has also gone through many modifications for specific application requirements. The most popular are three modes: contact, tapping and non-contact mode. Each mode responds to interatomic force between the probe tip and the surface. The forces are described by Lennard – Jones function [2]:

$$F = \frac{A_H R}{6\sigma^2}\left(\frac{\sigma^2}{s^2} - \frac{1}{30}\frac{\sigma^8}{s^8}\right), \qquad (1)$$

where: F –Lennard – Jones force, s – the distance between the particles, A_H – the Hamaker constant, R – tip radius, σ – finite distance at which the inter-particle potential is zero.

The force most commonly associated with atomic force microscopy is an interatomic force called the van der Waals force. Each mode is associated with different range of Lennard – Jones function (Fig. 2) [3]. Depending on the material, this function has different characteristic points (zero crossing – σ, and a minimum – ε), but its overall shape remains unchanged.

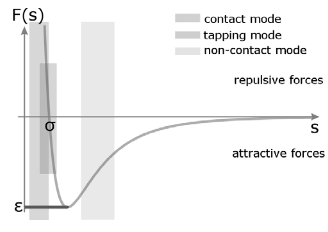


Fig. 2. F(s) Lennard-Jones potential function with marked AFM modes
Rys. 2. Funkcja F(s) Lennarda-Jonesa z zaznaczonymi trybami pracy AFM

In the contact region, the cantilever is held less than a few angstroms (10^{-10} m) from the sample surface, and the interatomic force between the cantilever and the sample is repulsive with a mean value of 10^{-9} N [2]. The AFM probe is scanned at a constant force between the probe and the sample surface to obtain a 3D topographical map. When the probe cantilever is deflected by topographical changes, the scanner adjusts the probe position to restore the original cantilever deflection. The scanner position information is used to create a topographical image.

Contact mode is used for hard material characterization. It is characterized by high-resolution images. Unfortunately, due to physical contact of the needle and the sample, the needle may be damaged.

In the tapping mode, the cantilever probe oscillates at or near its resonant frequency. However, the oscillation amplitude is greater than 10 nm, typically 100 to 200 nm. The oscillating probe scans at a height where it barely touches or “taps” the sample surface. The system monitors the probe position and vibrational amplitude to obtain topographical and other property information. When the needle touches the sample, the cantilever oscillates with high amplitude [4-6]. The advantage of tapping the surface improves lateral resolution for soft samples. Lateral forces such as drag, common in contact mode, are virtually eliminated.

The most important mode for the presented studies is non-contact mode. In the non-contact region, the cantilever is held on the order of tens to hundreds of angstroms from the sample surface. The cantilever oscillates at a frequency slightly above its resonant frequency where the oscillation amplitude is typically a few nanometers (<10 nm). The van der Waals forces decrease the cantilever resonance frequency. The decrease in resonant frequency combined with the feedback loop system maintains a constant oscillation amplitude or frequency by adjusting the average tip-to-sample distance. The lever oscillates above the sample with the resonant frequency and can be treated as a harmonic oscillator with the resonant frequency *f*:

$$f = \frac{1}{2\pi}\sqrt{\frac{c_{eff}}{m}}, \qquad (2)$$

where: c_{eff} – substitute spring constant, m – substitute mass of the cantilever with needle.

2. The study of harmonic intensities

The studies are intended to identify the characteristic points of the Lennard – Jones function for individual sample nanofragments. A varistor was used as the test material because previous studies [7] showed that the type and size of the varistor grains have a key influence on its properties, and thus the Lennard – Jones function. It was found that high quality varistors had a coarse texture (Fig. 3), while those of low quality - fine grained structure (Fig. 4).

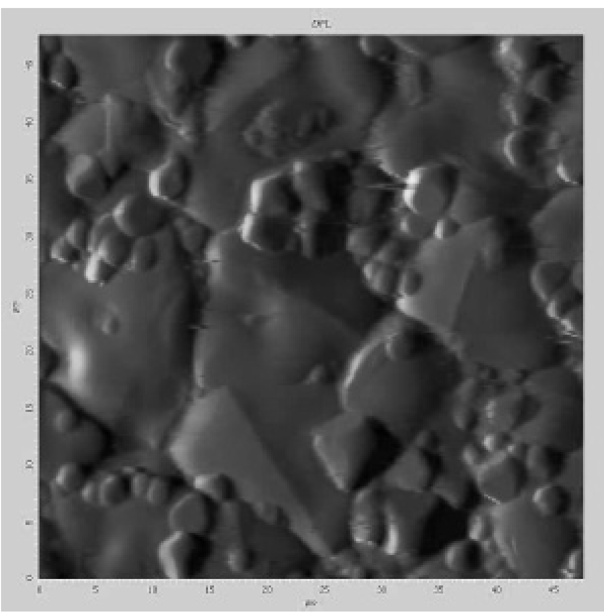


Fig. 3. The structure of a high quality varistor
Rys. 3. Struktura warystora wysokiej jakości

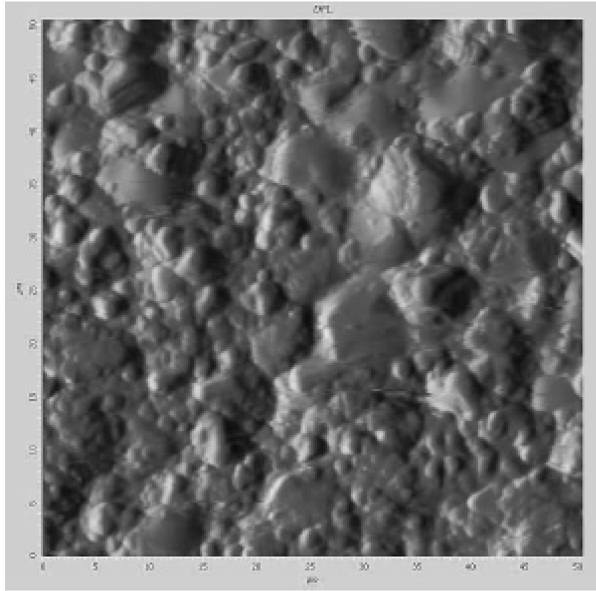


Fig. 4. The structure of a low quality varistor
Rys. 4. Struktura warystora niskiej jakości

The tests were carried out in non-contact mode. The cantilever with 17.97 kHz resonance frequency was used. The cantilever was stimulated by resonant frequency and during operating the sequences of 2 million samples were recorded. The sequences are a cantilever’s response on the spot “contact” with the sample. The tested sample’s fragment with 100 µm x 100 µm dimension is presented on Fig. 5.

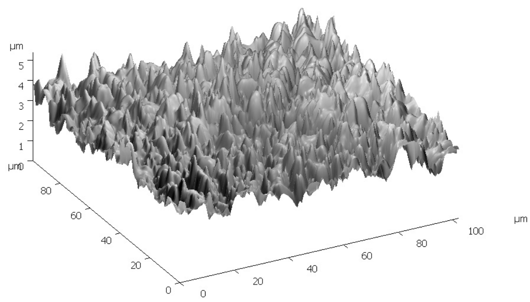


Fig. 5. Fragment of the sample
Rys. 5. Fragment badanej próbki

For analysis, six randomly selected measurements were used. Fig. 6 shows first five harmonics of the resonance frequency present in the system response.

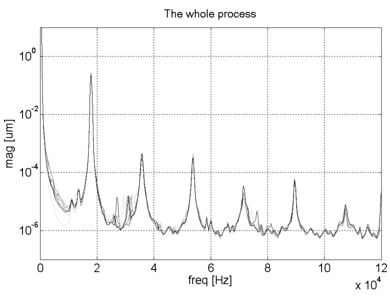


Fig. 6. First five harmonics of resonance frequency observed during five measurements
Rys. 6. Wykres pierwszych pięciu harmonicznych częstotliwości rezonansowej sześciu pomiarów

It is visible that the resonance frequency component is almost the same for all measurements. A significant difference is visible only for other harmonics (Fig. 7 – 10).

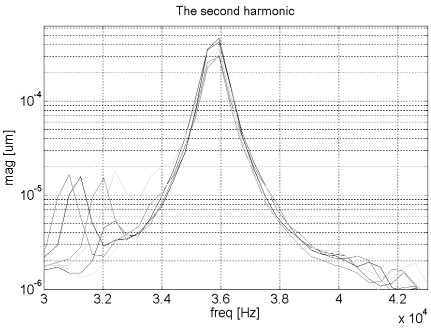


Fig. 7. The second harmonic
Rys. 7. Wykres drugiej harmonicznej

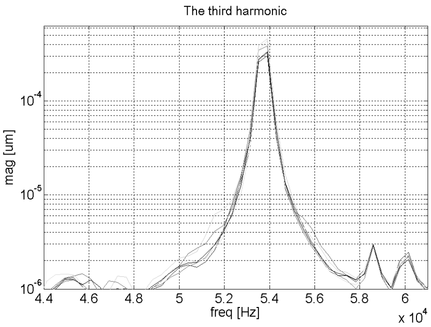


Fig. 8. The third harmonic
Rys. 8. Wykres trzeciej harmonicznej

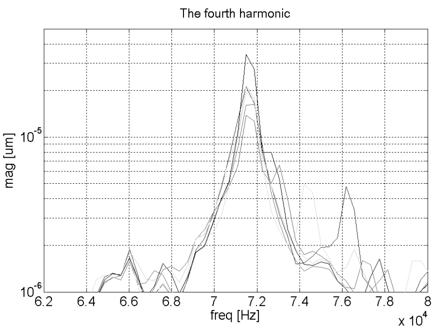
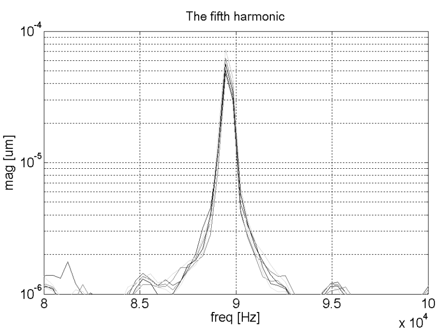


Fig. 9. The fourth harmonic
Rys. 9. Wykres czwartej harmonicznej



Rys. 10. The fifth harmonic
Fig. 10. Wykres piątej harmonicznej

Due to small differences between the level of zero and the first peak, the calculations were carried out for the second to the fifth peaks. The levels of successive harmonics are given in Tab. 1.

The relative differences in the harmonics intensities were calculated for the first measurement only. The results are presented in Tab. 2. As it can be noted the differences are significant.

Tab. 1. Harmonic levels
Tab. 1. Poziomy harmonicznych

Measurements number	Chart color	Harmonic level			
		second	third	fourth	fifth
1	niebieski	4,26E-004	3,06E-004	2,12E-005	5,05E-005
2	czerwony	3,06E-004	3,26E-004	1,38E-005	4,81E-005
3	zielony	3,65E-004	4,61E-004	2,16E-005	7,25E-005
4	różowy	2,97E-004	3,84E-004	1,62E-005	6,22E-005
5	cyjan	3,52E-004	4,36E-004	1,96E-005	7,18E-005
6	czarny	4,65E-004	3,33E-004	3,43E-005	5,61E-005

Tab. 2. The relative differences of the harmonic levels
Tab. 2. Względne różnice poziomów harmonicznych

Measurements number	Relative difference in harmonics intensities [%]			
	second	third	fourth	fifth
2	28,11	-6,64	34,87	4,79
3	14,35	-50,79	-1,79	-43,59
4	30,37	-25,56	23,85	-23,19
5	17,31	-42,77	7,54	-42,19
6	-9,16	-8,97	-61,64	-11,19

3. Conclusions

The presented studies show significant differences between intensities of the consecutive harmonics observed for AFM measurements of varistors. Thus, AFM measurements can detect nonlinearities within the probe oscillations that can be used to establish parameters of influence of the probe on the investigated material.

It is known that varistor grains have significant impact on their properties. Thus, association between the harmonics and the sample structure and thus determining the parameters of the Lennard – Jones function may be used as a new method of varistors properties testing.

Further studies are necessary to correlate the tested samples with measurements of harmonics. Such measurements need some modifications in the data registration process, which will be soon possible. The further work will deal with the detailed correlation of the observed harmonics with the Lennard – Jones function parameters.

4. References

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