NT-MDT
Molecular Devices and Tools for NanoTechnology

SOLVER

SCANNING PROBE MICROSCOPE

Atomic Force Acoustic Microscopy (AFAM)

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PREFACE

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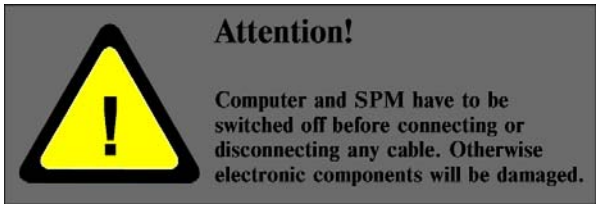

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Atomic Force Acoustic Microscopy

1 Introduction

1. Atomic Force Acoustic microscopy is a new SPM measuring mode developed at the Fraunhofer Institute of Nondestructive Testing, Saarbruecken, Germany [1, 2]. Other research groups worked on similar approaches and the reader may be check the references of the papers cited. This technique, licensed to NT-MDT, allows the measuring of qualitative and quantitative local elastic properties of different materials.
2. The basic idea is to excite the cantilever of an atomic force microscope into flexural vibrations when the tip is in contact with the sample (Fig. 1). The frequency of the eigenmodes of the cantilever depends, amongst other parameters, on the stiffness of the tip-sample contact and on the contact radius, which in turn are both a function of the Young's modulus of the sample and the tip, the tip radius, the load exerted by the tip, and the geometry of the surface. Such a technique allows one to determine the Young's modulus from the contact stiffness with a resolution of a few tens of nanometers.

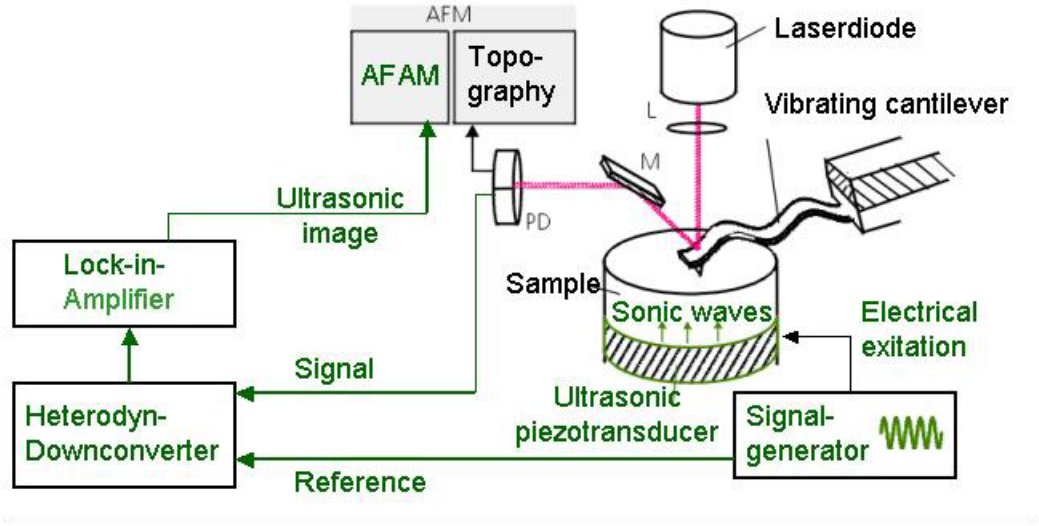

Fig. 1 Block-scheme of AFAM set-up.

In our AFAM setup the sample is coupled to a piezoelectric transducer with a center frequency of 2.5 MHz (Parametrics A106S or A103S). It emits longitudinal acoustic waves into the sample which cause out-of-plane vibrations of the sample surface. The surface vibrations are transmitted into the cantilever via the sensor tip. The cantilever vibrations are measured by a 4-sectioned photo-diode and evaluated by a lock-in amplifier. This setup can be used either to acquire cantilever vibration spectra or to take acoustic images. The latter are maps of cantilever amplitudes on a fixed excitation frequency near the resonance. The contact-mode topography image is acquired simultaneously with the acoustic

one. The frequency range employed covers the flexural modes of the cantilever from 10 kHz up to 1,5 MHz.

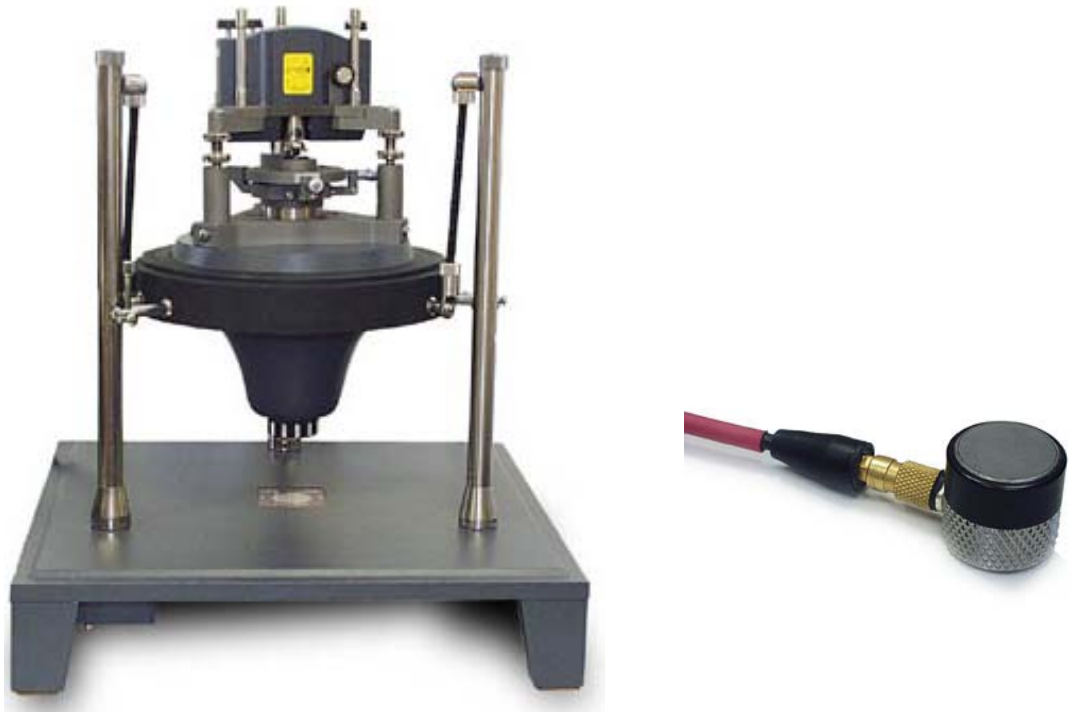

Fig. 2 P47H AFAM setup(left); ultrasonic transducer (right).

2 Theory

Like macroscopic beams, AFM cantilevers can vibrate in different types of acoustical modes, for example flexural, torsional or extensional modes. The equation of motion for flexural vibrations delivers an infinite set of vibration modes of n-th order with the wave numbers $k_n = 2\pi/\lambda_n$, {n = 1, 2, …}, the frequencies f_n, and the acoustic wavelength λ_n. The resonance frequencies for flexural vibrations are related to the wave numbers:

$$\frac{k_n^2}{f_n} = \sqrt{4\pi^2 \frac{12\rho}{b^2 E}} = c_B^2 ,$$

or $k_n = c_B \sqrt{f_n}$, where c_B is the characteristic cantilever constant. The constant c_B contains the density ρ of the cantilever material, the Young's modulus E of the beam and its thickness b.

When the sensor tip of the AFM is brought into contact with the sample surface, vertical and lateral elastic forces as well as adhesion and friction forces act between the sensor tip and the sample surface. The tip-sample forces are nonlinear with distance. They change the boundary conditions at the end of

the cantilever where the tip is located. The numerical solution of corresponding equations shows that, if the tip and the sample vibration amplitudes are kept sufficiently small, the resonance curves become symmetric and the tip-sample forces can be approximated by linear vertical and lateral spring dashpot systems Fig. 2). The linear equation of motion can be solved analytically, yielding the resonance frequencies of the system as well as the vibration amplitudes along the cantilever depending on the contact stiffness k*.

When lateral stiffness as well as lateral and vertical damping are neglected, the vertical contact forces can be estimated using the Hertzian model for a spherical indenter with radius R contacting a flat surface. The radius of the contact area according to the Hertz model is

$$a = \sqrt[3]{3F_N R / 4E^*},$$
$$\frac{1}{E^*} = \frac{(1-\nu_t^2)}{E_t} + \frac{(1-\nu_s^2)}{E_s}$$

where E^* is the reduced Young's modulus and ν_t , ν_s and E_t, E_s are the Poisson numbers and the Young's module of the tip and the surface, respectively. F_N is the normal force acting on the sensor tip. From the contact force the contact stiffness k* can be calculated:

$$k^* = \sqrt[3]{6E^{*2}RF_N}\ .$$

When cantilevers with spring constants around 1 N/m are used, the adhesion forces are usually larger than the forces applied by the cantilever. In order to control the static forces F_N, it is recommended to use cantilevers with spring constants 10-40 N/m, allowing to apply static loads of up to 2 μ N.

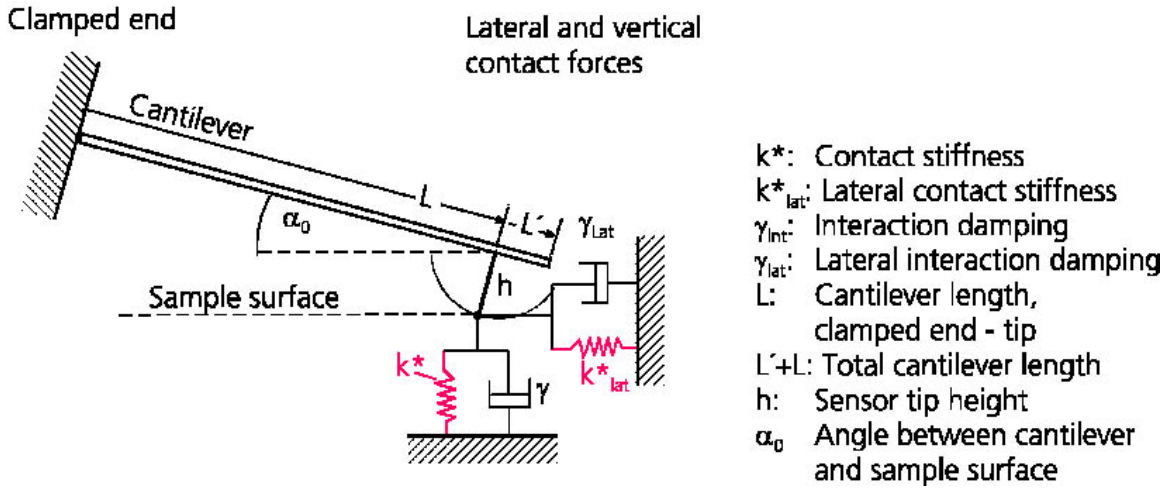

Fig. 3 Elastic beam model for the AFM cantilever.

3 Measuring Procedures

3.1 Mounting of a sample on AFAM ultrasonic transducer

Before mounting the sample make sure that surface of a side that's supposed to be attached to transducer is plane and smooth enough. That means the surface just should not contain any large steps or imperfections that could prevent the sample from tight contact with transducer. For mounting the sample you should use some material having good acoustic coupling properties. One of the most cheap and easy to get material for a such purpose is ordinary liquid state honey.

So first you should place a little droplet of this honey on surface of ultrasonic transducer (See Fig. 4). After that spread it uniformly all over the part of the surface that is going to come into contact with the sample. Than place the sample on the transducer and under slight pressure make several short moves of the sample along the surface of the transducer in order to provide optimal spreading of honey and the best adjacency of surfaces.

After you have finished all the manipulations described above it is highly recommended to start any measuring procedures only after a lapse of some time about an hour in order to let the sample to stick to the surface of transducer in the best way and also to avoid a vertical drift which can complicate process of measuring or even make it impossible.

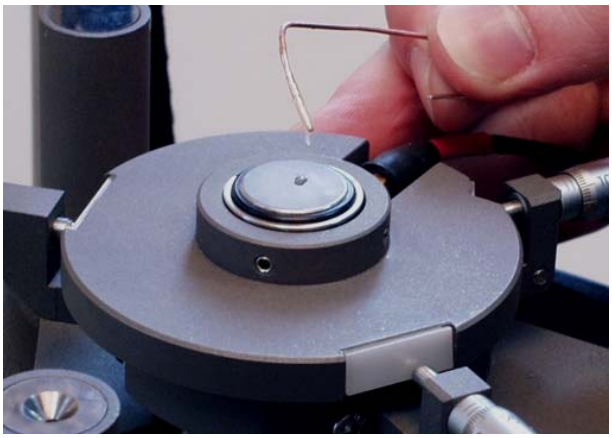

Fig. 4

Fig. 5

When you finish your measuring and need to remove the sample then in the case your sample is thin enough and not flexible, that is the most complicated case, you just should apply some lateral pressure to the sample (See Fig. 5). It will start gradual moving off then you can easily remove it. In order to facilitate sample removal, in the case of using of honey, slight warming up can also be recommended.

3.2 AFAM Imaging

AFAM imaging is the simplest acoustic imaging technique. With this technique one measures the amplitude of the cantilever flexural vibration near the contact resonance frequency simultaneously with the contact-mode topography imaging. To get the image one should perform the following sequence of steps:

1. Mount the sample on the transducer.
2. Apply modulation to the "Probe". Find and write down first two or three free flexural resonances of the cantilever (f_0, f_1, f_2).
3. Approach the sample in standard contact mode.
4. Apply modulation to "Ex6" connected to the piezoelectric transducer. Find the contact resonances of the cantilever. They should lie between free resonances ($f_0 < f_{0c} < f_1$; $f_1 < f_{1c} < f_2$; etc., see Fig. 6).
5. Adjust the amplitude of oscillations. The resonance peak should be symmetric. When it looks inclined or truncated that means that nonlinear effects [3, 4} contribute too much and you should decrease the driving voltage (Fig. 7).

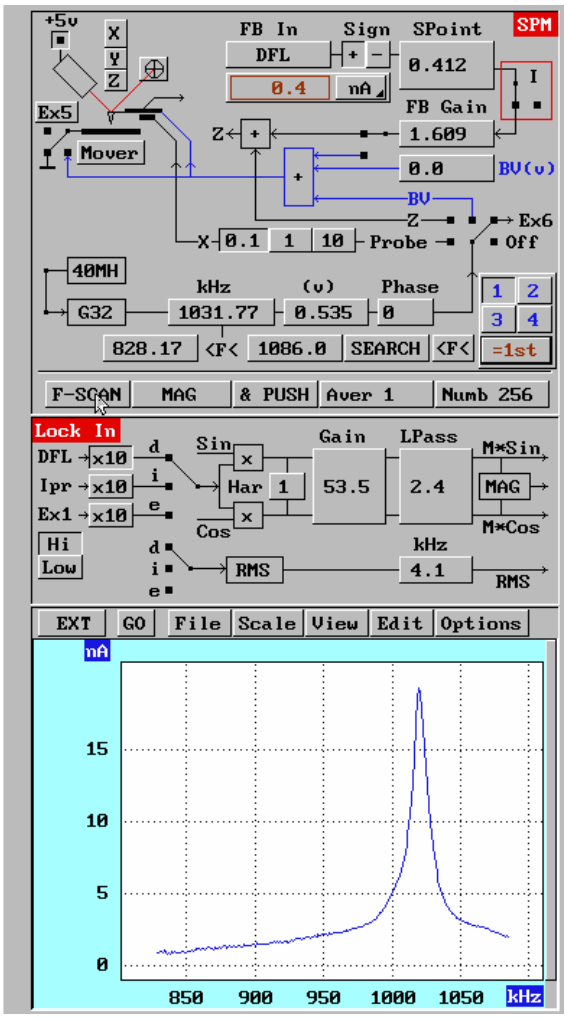

Fig. 6 Settings for AFAM measurements.

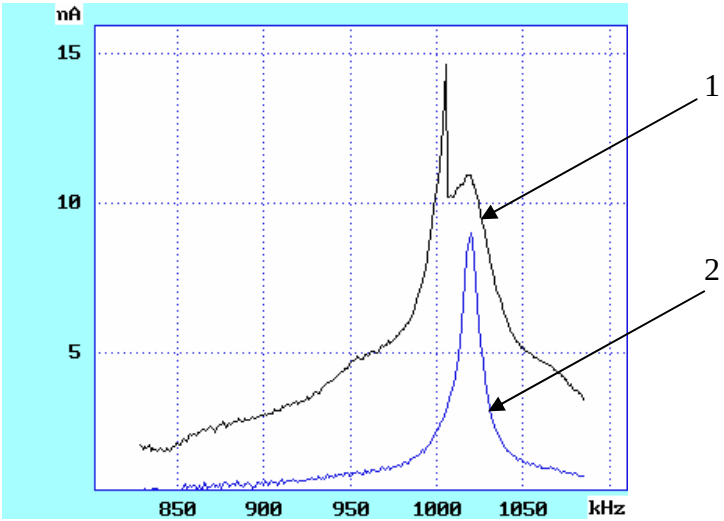

Fig. 7 Resonance curves for different excitation voltages. Graph 1 - Resonance curve distorted by nonlinear effects caused by high excitation voltage, here 1.0 V; graph 2 - Symmetric peak, obtained with lower excitation voltage, 0.25 V, suitable for measurements.

6. Take a force-distance curve and determine the load force. To obtain the load force one should determine the calibration coefficient between the DFL signal (in nA) and the applied force (in nN). An example of such a calculation is shown in Fig. 8. Here the DFL signal of 1 nA corresponds to a deflection of 27.8 nm, which in turn corresponds to the applied force $F = (27.8\ \text{nm}) \times k_c$, where k_c is the force constant of the cantilever shown in its specifications. In our case $k_c = 10$ N/m, so 1 nA of the DFL corresponds to 278 nN load force.

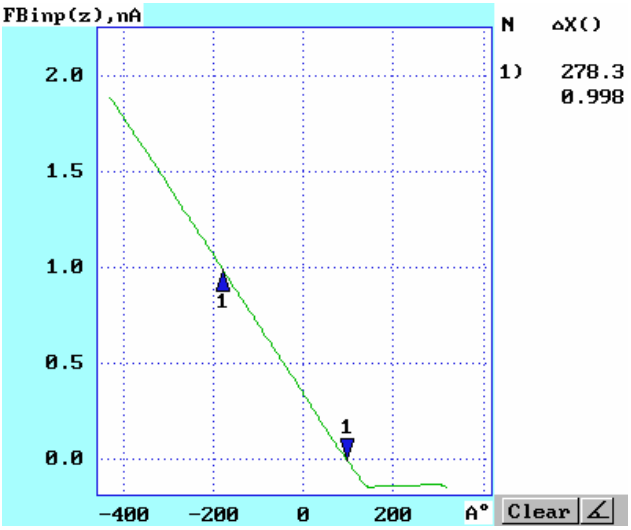

Fig. 8 Force distance curve.

7. Check whether the resonance peak shifts when you change the load. If it is not shifted decrease the load. The correct behavior is shown in Fig. 9. Here you can choose for example the force load corresponding to the graph 2.

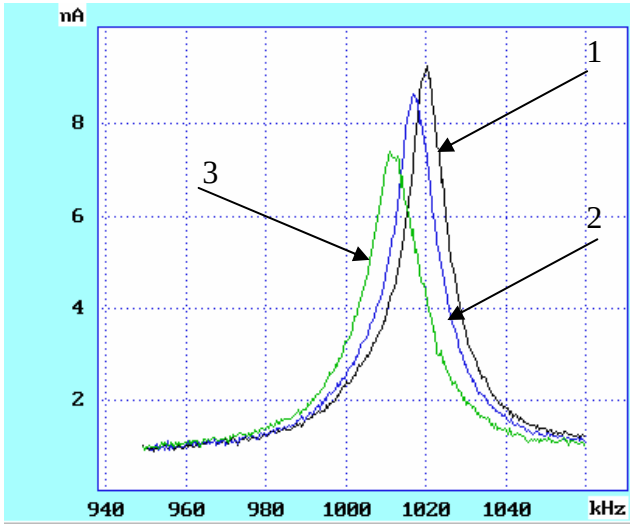

Fig. 9 Contact resonance curves measured with different applied loads: graph 3: F=50 nN; graph 2:– F=100 nN; graph 1: F=150 nN.

8. Set the driving frequency on the right slope of the peak.

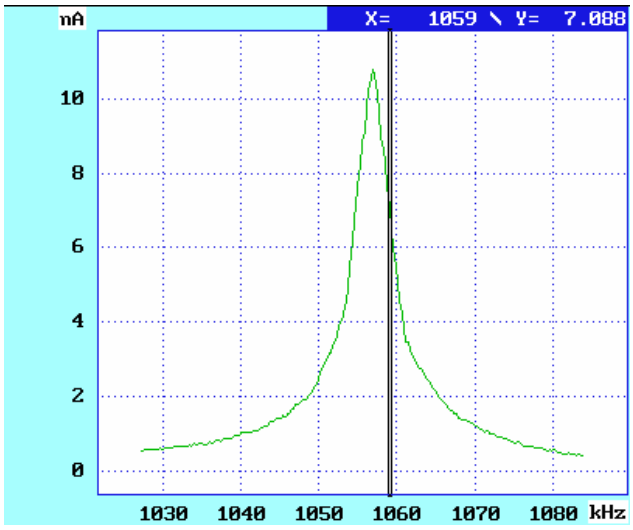

Fig. 10 Setting of the driving frequency for AFAM imaging.

9. Set measuring of “Height” signal in channel A and “MAG” signal in channel B

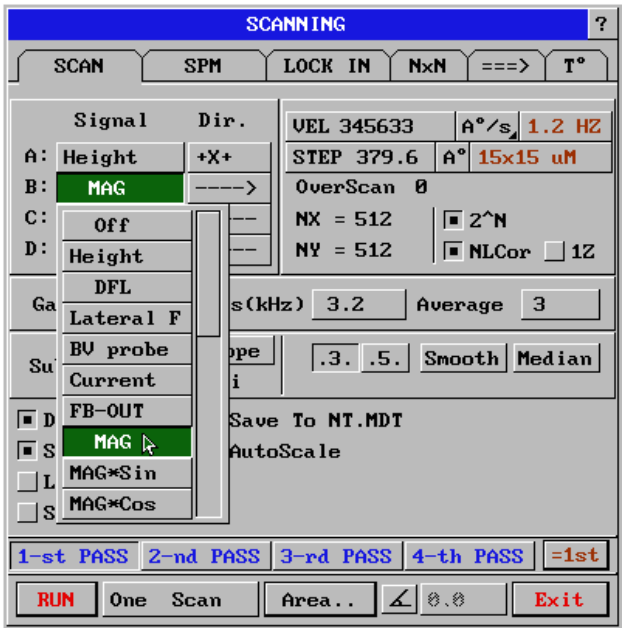

Fig. 11 Setting of the scanning options for AFAM imaging.

10. Execute scanning

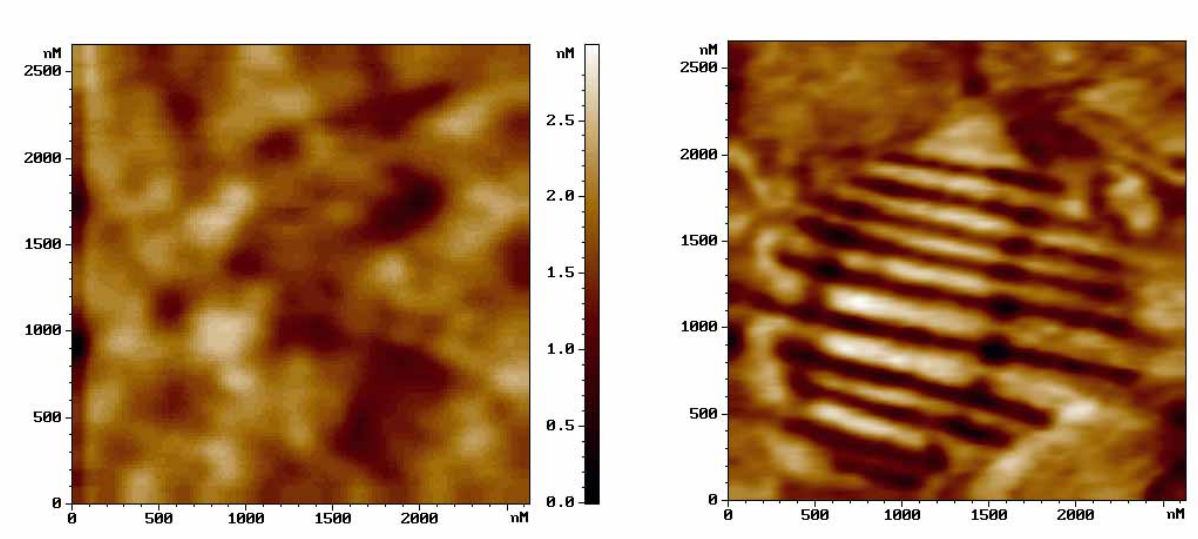

Fig. 12 Contact mode topography (left) and AFAM (right) images of stripe ferroelectric domains on a polished PZT sample of 1 mm thickness.

3.3 Contact-Resonance Spectroscopy Imaging.

The basic idea of contact-resonance spectroscopy (CRS) is the measurement of contact resonance properties (resonance frequency, Q-factor, peak amplitude, etc.) in a certain set of scan points /5/. Contrary to simple AFAM amplitude imaging that provides only contrast, CRS gives possibility to make quantitative calculations and to obtain local Young's modulus values at each point of the scan.

Our software allows one to measure the resonance frequency, the Q-factor and the peak amplitude simultaneously with the topography scanning, or to get a full resonance curve at each point of the scan and process this data afterwards. To make CRS one should perform the following steps:

1. Execute steps 1-8 for AFAM imaging (generally it is recommended to get first a good AFAM picture before CRS).
2. Set the frequency ranges and the number of points in the spectroscopy menu. The frequency range should be so wide that the peak position does not move out of the range along the scan, but not much wider than the peak width in order to keep good resolution. You can check the curve in different scan points to see how much the peak is shifted. The bigger the number of points is, the better is the accuracy of the measurements, but the time of measuring also grows. Usually 256 points are the optimal value.

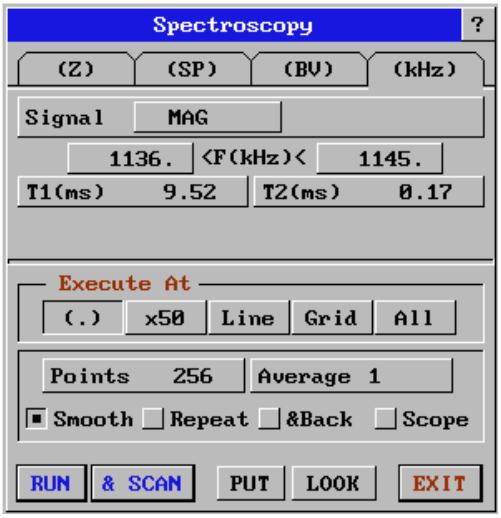

Fig. 13 Settings for contact- resonance spectroscopy.

3. Set measuring of the“Height” signal in channel A and the “Fr_kHz” signal in channel B. Simultaneously one can also measure the signals “Fr_Q”, the Q-factor of the resonance curve at each point and “Fr_Mag”, the peak amplitude of the resonance curve at each point.

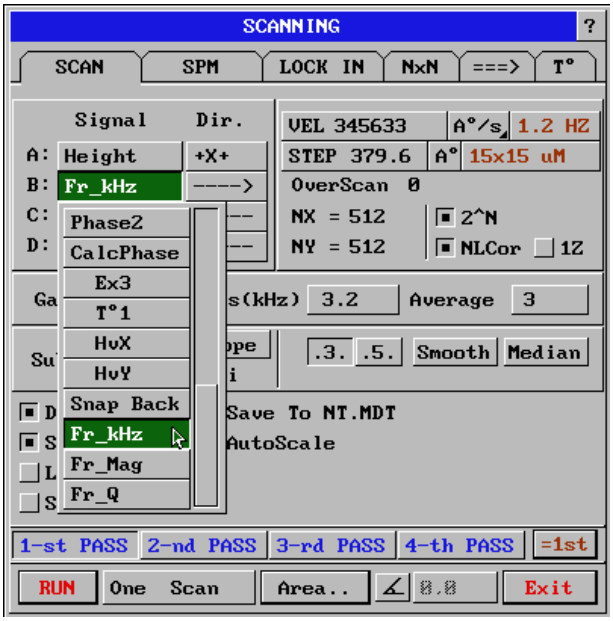

Fig. 14 Setting of the scanning options for CRS imaging.

4. Execute scanning. Note that such measurements take quite a lot of time, and it is therefore reasonable to use fewer points. Recommended are scans of 128 × 128 points.

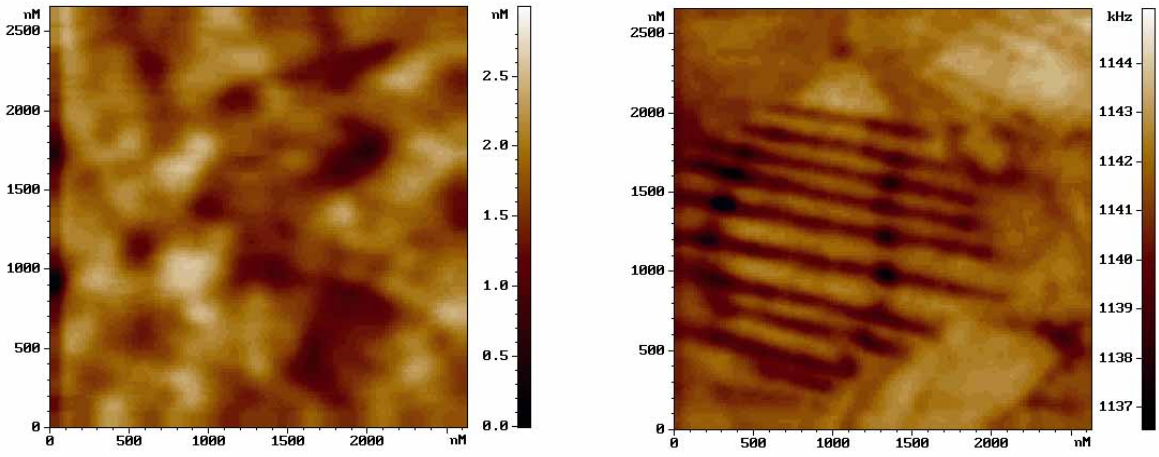

Fig. 15 Contact-mode topography (left) and CRS (resonance frequency) (right) images of stripe-like ferroelectric domains on a PZT sample.

References:

[1] U. Rabe, S. Amelio, E. Kester, V. Scherer, S. Hirsekorn, and W. Arnold, "Quantitative determination of contact stiffness using atomic force acoustic microscopy", Ultrasonics 38 (2000) 430-437.

[2] U. Rabe, S. Amelio, M. Kopycinska, S. Hirsekorn, M. Kempf, M Goeken, and W. Arnold, "Imaging and measurements of local mechanical material properties by atomic force acoustic microscopy", Surf. Interface Anal. 2002; **33**: 65-70.

[3] U. Rabe, E. Kester, and W. Arnold, „Probing Linear and Nonlinear Tip-Sample Interaction Forces by Atomic-Force Acoustic Microscopy», Surface and Interface Analysis 27, 386-391(1999)

[4] M. Muraoka and W. Arnold, "A Method to Evaluate Local Elasticity and Adhesion Energy Based on Nonlinear Response of AFM Cantilever Vibration", JSME International Journal, A44, 396-405, 2001

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