

**University of Ulm
Institute for Micro- and Nanomaterials**

**Lab
„Materials Science“
Summer Term 2007**

Group 9: Adelheid Grob & Sukhum Ruangchai

E: Atomic Force Microscopy
lab on April, 26th 2007

1 Questions

1.1 Advantages and Disadvantages of AFM vs. SFM/TEM and STM

1.1.1 AFM vs. SFM/TEM

- AFM provides extraordinary topographic contrast, direct height measurement and unobscured views of surface features (with AFM no coating is necessary)
- with AFM 3-dimensional images are possible
- AFM works without extensive sample preparation
- For AFM no vacuum is necessary.

1.1.2 AFM vs. STM

Disadvantage of STM	Advantage of STM
applicable only to conducting samples (AFM can be applied to conducting and non-conducting samples)	in some cases you'll get a better resolution
with AFM the writing voltage and the tip-to-substrate-spacing can be controlled independently	the force-distance dependence in AFM is more complex than the tunneling current-distance dependence in STM
AFM is more versatile	

1.2 Difference between Si- and SiN-Probes

Si-Probes are used for Tapping Mode AFM, they can be much stiffer than SiN, therefore they have a larger force constant and resonant frequency compared to SiN-Probes.

SiN-Probes are used for Contact Mode AFM, they have a lower resonant frequency. For CAFM cantilevers which are soft enough are required.

1.3 Forces in CAFM

In CAFM there are the following forces:

- shear forces
- normal forces between the tip and the sample, e.g. strong ionic forces or weak van der Waals forces. The magnitude of these can be estimated as 10^{-7} N for the ionic forces and 10^{-11} N for the van der Waals forces.
- meniscus forces of fluid contaminant layers, adsorbed water carbon and so on
- tribology forces

1.4 Resonance frequency ω'_0 in TAFM

The resonance frequency ω'_0 of a cantilever interacting with the surface sample can be calculated from its free air frequency ω_0 in the following way:

$$\frac{df}{dz} = m_{eff}^{1/2}(\omega_0^2 - \omega_0'^2)$$
$$\rightsquigarrow \omega_0' = \sqrt{\frac{\omega_0^2 - \frac{df}{dz}}{m_{eff}^{1/2}}}$$

with ω_0 being the resonant frequency in air, m_{eff} being the effective mass, depending on the mass distribution and geometry of the tip/cantilever assembly, $\frac{df}{dz}$ is the force gradient.

1.5 Controlled Variable in CAFM and TAFM

In CAFM the controlled variable is the deflection of the cantilever, which is kept constant. That means the scanner is moving up and down depending on the value of the instantaneous deflection, which is controlled optically. You have to take care, that the force on the material while scanning and approaching is not too big - otherwise the sample or the cantilever can be damaged.

In TAFM the controlled variable is the amplitude of the oscillating cantilever, which is kept constant. This is done by a feedback loop, which keeps a constant RMS of the oscillating signal, which is controlled by two photodiodes. During approaching the surface it can happen, that the cantilever gets stuck in the sample.

1.6 Mean Roughness

The Mean Roughness R_a is defined as

$$R_a = \frac{\sum_{i=1}^N |Z_i - Z_{CP}|}{N}$$

with Z_i being the current z-value, Z_{CP} the z-value of the center plate and N the number of samples.

So, the mean Roughness R_a can be the same for totally different surface profile, because it is a average.

Another method for characterization is the Root Mean Square Roughness (RMS Roughness), which is defined as

$$\mathbf{RMS} = \sqrt{\frac{\sum_{i=1}^N (Z_i - Z_{ave})^2}{N}}$$

with Z_{ave} being the average z-value of the given area.

2 Experiment

2.1 TAFM

First we operated in Tapping AFM Mode.

2.1.1 Fe-Ni-Alloy-Bridge

In this part of the lab we took a closer look at the roughness of a Fe-Ni-Alloy-Sample. The result is shown in Fig. 1.

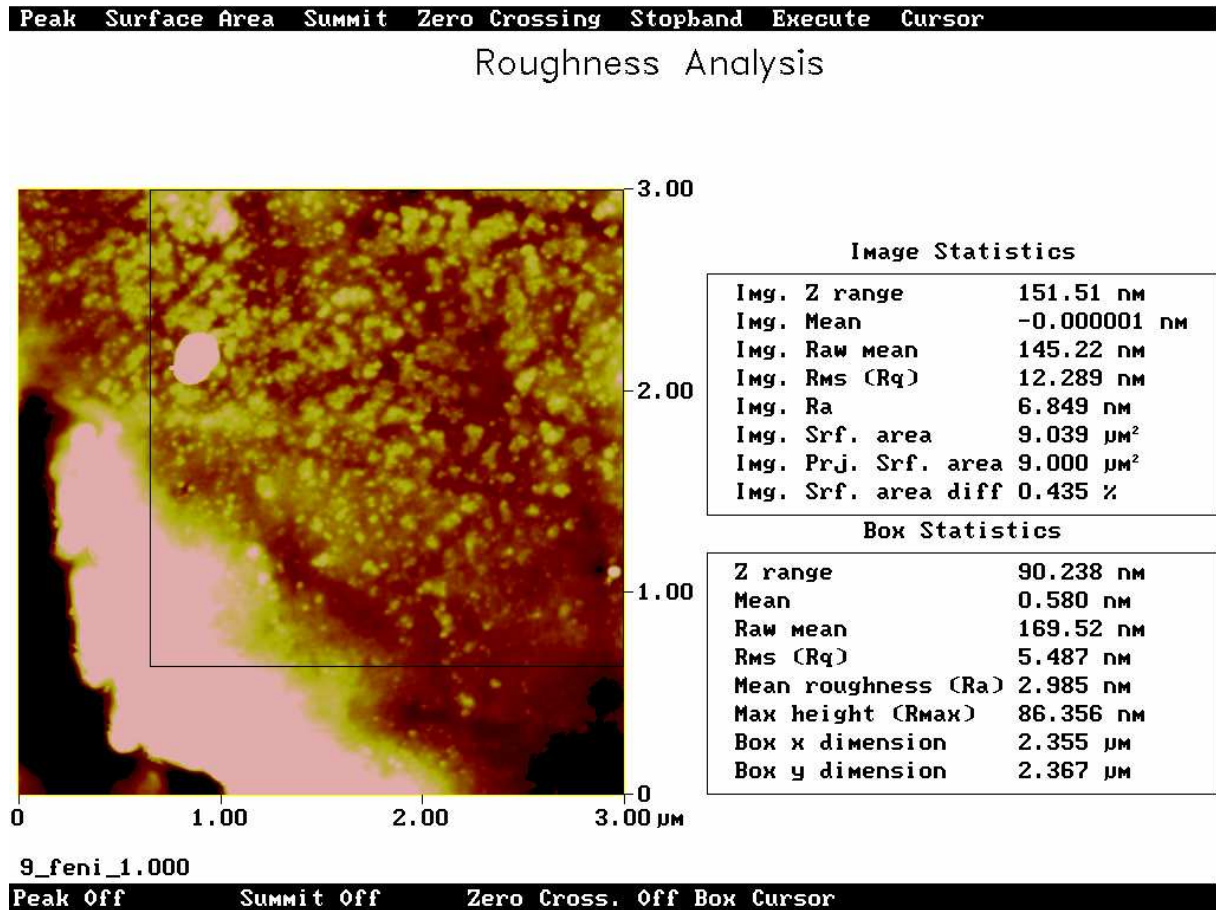


Fig. 1: Fe-Ni-Alloy

With the software we then calculated the Mean Roughness R_a and the Root Mean Square Roughness **RMS**. We got the following values:

$$R_a = 2.985\text{nm}$$

$$\text{RMS} = 5.487\text{nm}$$

Another (smaller) section (see Fig. 2) of the image gives as the following values:

$$R_a = 1.512\text{nm}$$

$$\text{RMS} = 1.945\text{nm}$$

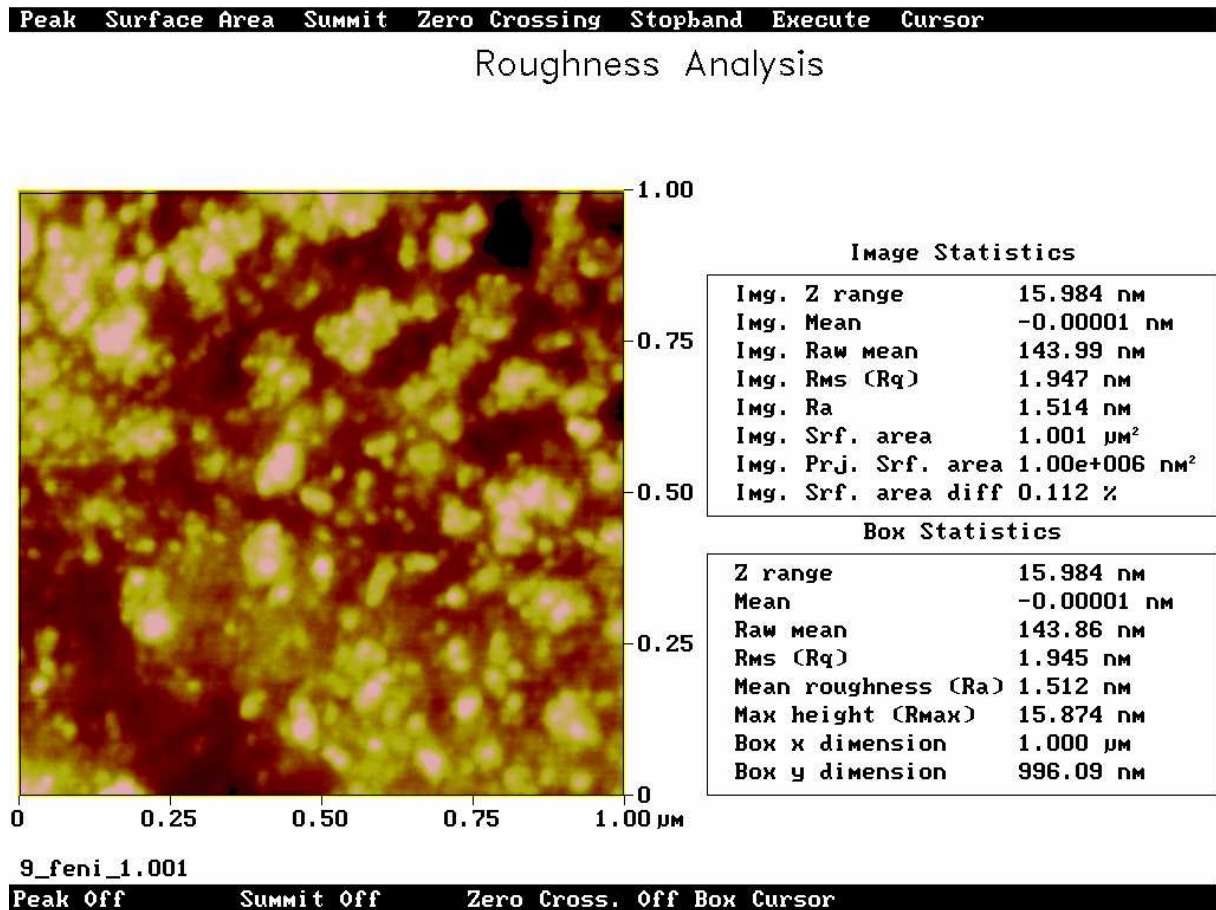


Fig. 2: smaller section of Fe-Ni-Alloy

The next thing we did, was to measure the grain angle, that means the angle between two grains on the surface. We selected two grains and measured with the software the angles of each grain to the surface as can be seen in Fig. 3.

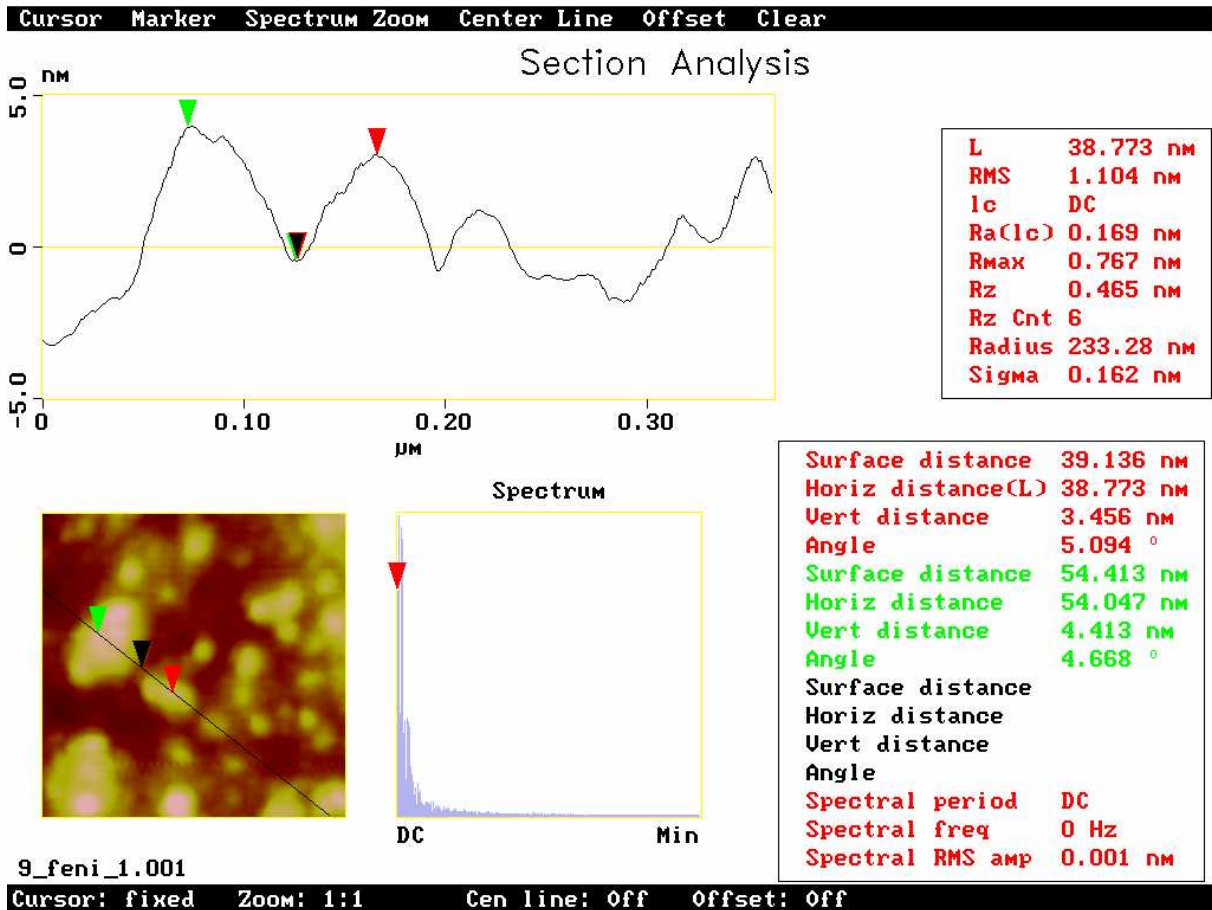


Fig. 3: Fe-Ni-Alloy, angle between two grains

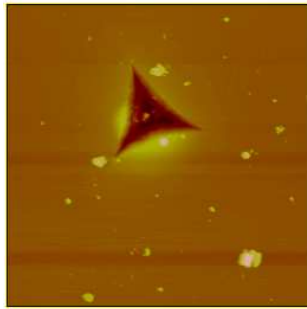
So we got as an resulting angle between the two grains:

$$180^\circ - 5.094^\circ - 4.668^\circ = 170,238^\circ$$

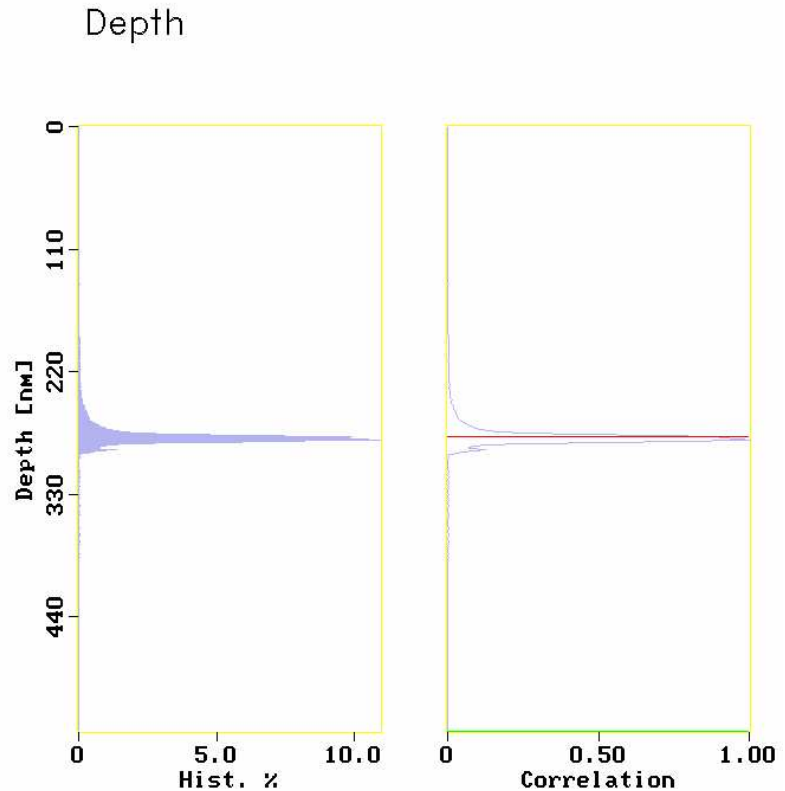
2.1.2 Si-Wafer with indents

Now we had a second sample out of silicon with a big scratch on it and 9 indents. Our goal was to focus on one of these indents and measure its depth. In Fig 4 you can see the indent and a curve which shows the distribution of heights of the sample. The most points in the sample are at a height of ca. 270 nm, that means at this point is the surface and at

the “lowest” point in the diagram is the minimum of the indent. So you can measure the distance between the surface and the lowest point and you’ll get the depth of the indent. In this case the depth is 264.20 nm. The value “Depth at Max” of 281.43 nm means, that most of the surface is at that height (whereby 0 nm as deepest point is the ground of the indent).



Peak to Peak	264.20 nm
Filter cutoff	0 nm
Total peaks	2
Depth at max	281.43 nm



9_inden1.000

File: default

Fig. 4: indents in Si-Sample

Then we inverted the measured image and took a 3D-Look at it, the result you can see in Figure 5. We suppose that the indent is not a perfect pyramid, because there is perhaps some dust in it.

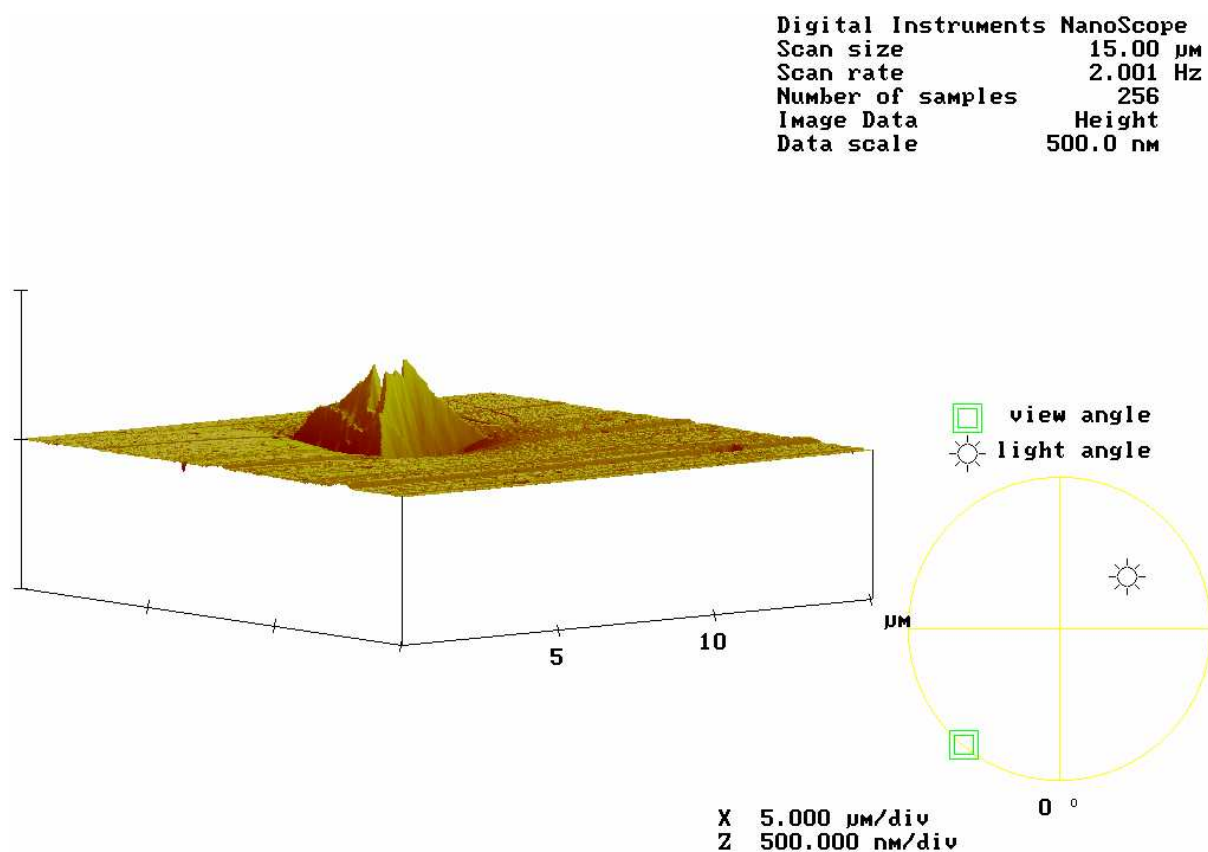


Fig. 5: indents in Si-Sample, 3D-Plot

2.2 CAFM

In the second part of the experiment we operated in Contact AFM-Mode.

2.2.1 Calibration Sample

First it was necessary to calibrate the AFM with a known calibration sample, so that the software knows, which forces are combined to which height. As you can see in Figure 6 the sample was a lattice with a surface distance of nearly $10\ \mu\text{m}$ and a height of the periodic elements of $0.20\ \mu\text{m}$. Because we were interested in it, we measured the height of a dust particle laying on the surface. Its height is $54.441\ \text{nm}$.

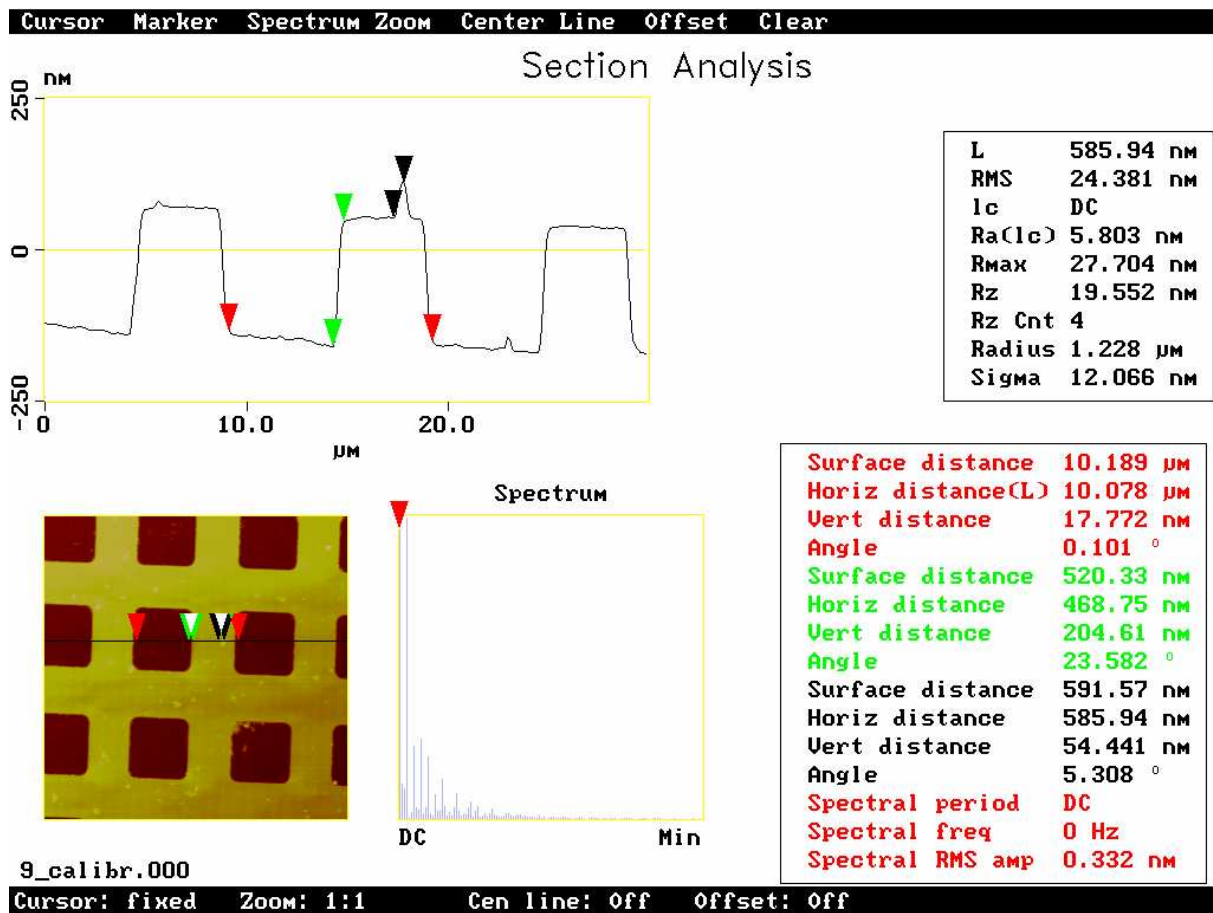


Fig. 6: Calibration sample

2.2.2 Chip for high frequent appliances

The last thing we did is to make a 3D-CAFM image of a chip with gold contacts used for high frequency appliances. You can see the result in Fig. 7

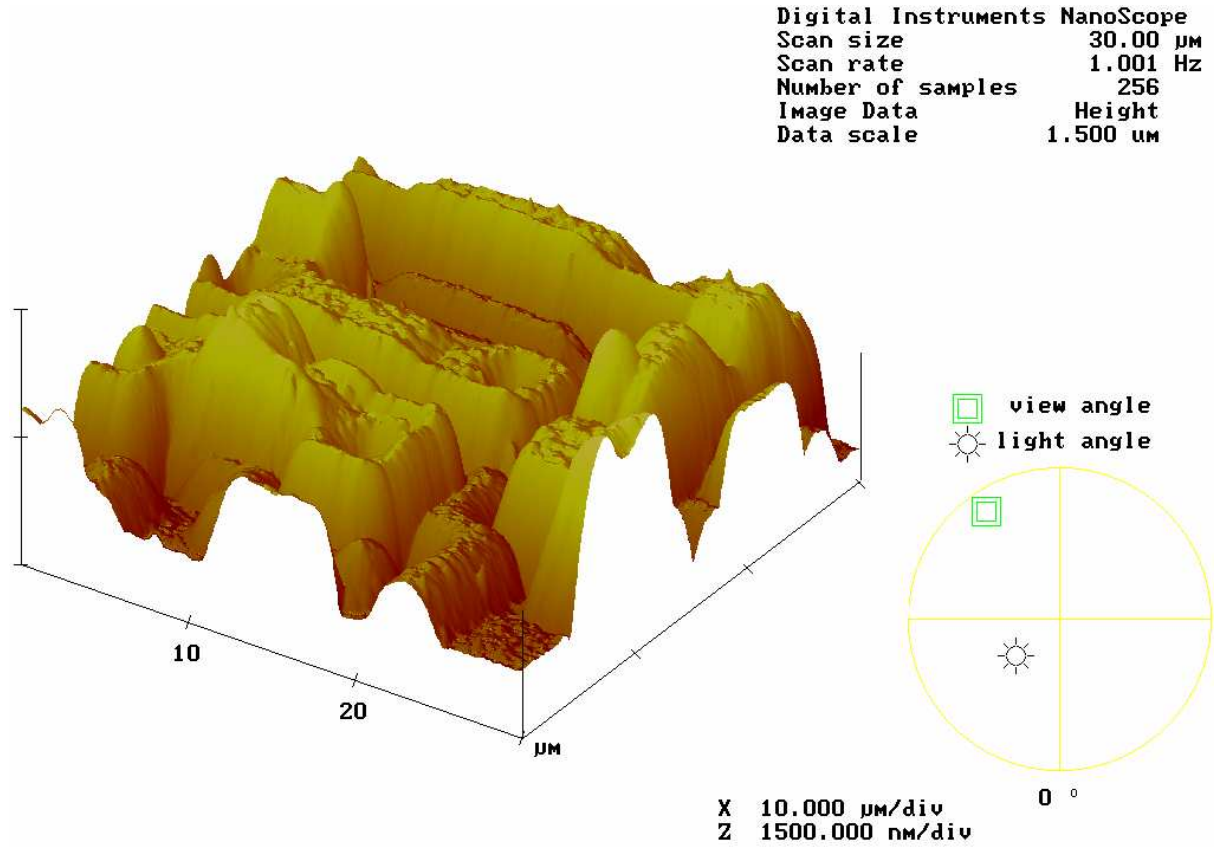


Fig. 7: 3D-Plot of a Chip