Imaging contrast in SMM: the mechanical approaches

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Abstract. Scanning Microdeformation Microscopy (SMM) is a dynamic force microscopy technique, which uses a microcantilever vibrating at its resonance frequency (range of some kHz up to several 10 kHz) while scanning a sample surface. It uses a microcantilever associated with a tip of radius in the 1-20 microns range (contact radius is typically about 1 µm). The absolute amplitude and phase of the cantilever vibration as well as the frequency shifts of the cantilever resonance frequencies are measured. Due to the size of the tip, the SMM works on mesoscopic scale, allowing to investigate a large subsurface volume (typically about 10 times the contact radius between the tip and the sample). This paper is devoted to the contrast analysis in the SMM for the investigation of subsurface defects. More precisely we analyse different mechanical approaches to explain the contrast induced by the defects in the subsurface volume. The approach used for each case depends on the materials and geometry (size, depth, elastic properties). Experimental results performed on calibrated samples are presented, allowing us to give a clear analysis of the contrast sources.

1. Introduction
Near-field microscopes have been developed in order to analyze and to quantize the local elastic or viscoelastic properties at the surface of solids [1-4]. The Atomic Force Microscope (AFM) and derived instruments are useful tools to sense the stiffness of the surface when the vibrating tip contacts with the sample. In the case of defective samples, it has been proved that the close subsurface also contributes to the detected signal surface, thus the microscope is not limited to the surface investigation. But on the nanoscale, many atomic contributions act on the response of the system (sensitivity to Van der Waals forces, capillarity effects, adhesion,...) and the estimation of the local elastic constants is usually inaccurate. Moreover the unknown geometry of the tip and sample surface on the nanoscale limits the ability of accurate modeling. This explains that the quantization of local elastic properties remains a challenge. Thus, we have focused on a larger scale which enables to really quantize the sample properties. Our technique, called SMM [5,6], operates on the so-called “mesoscopic scale”: the contact radius is typically in the 100nm-1µm range for stiff materials. This scale yields a better knowledge of the geometry, and negligible atomic forces. Moreover, it enables an increased subsurface contrast because the depth of investigation is proportional to the contact radius. With AFM tips, the typical contact radius is in the range of 10 nm which limits the depth of investigation to below 100 nm. The aim of this paper is to give a phenomenological analysis of the subsurface investigation, based on solid mechanics: quasi-static approach which may be considered to be sufficient to explain the sensitivity of the SMM to subsurface defects. We present a heuristic approaches showing the effect of defective stiff or soft materials. Different behaviors are expected depending on the local elastic constants. Experimental results obtained with specifically machined test
samples confirm the results coming from the mechanical analysis. Finally, origins of the contrast in the images of microscopes based on contact vibrating tips are discussed.

2. Presentation of the SMM
The SMM physically is a large scale resonant AFM operating in contact mode. The scheme of the set-up is given in figure 1. The head of the microscope is composed of a piezoelectric transducer, a cantilever and a tip (made of diamond or sapphire); this hybrid device can be considered as an electromechanical resonator. The sample is scanned by a 3-axis translation unit, the vertical axis being used to adjust the value of the static contact force. The microscope can operate either in transmission or in reflection mode. In transmission mode, the tip is kept in vibrations with the excitation generator and the transmitted stress is detected with a second piezoelectric transducer on the back side of the sample. In reflection mode, a high sensitivity heterodyne interferometer is used to detect the amplitude and phase of the vibrating cantilever. A double-phase lock-in amplifier extracts amplitude and phase of the displacement above the tip. Moreover, the resonant frequency of the head can be detected by replacing the generator with an oscillator made of the cantilever, the piezoelectric actuator and a specific amplifier. The microscope shown can either be used to image the local elastic constants or to characterize close subsurface defects. For a known contact force (easily estimated with the knowledge of the contact position and the vertical motion of the cantilever), the sample stiffness can be estimated. In the presented study, the reflection mode has been used and different samples with subsurface defects have been investigated.

3. Phenomenological approach to explain the contrast of images
The SMM enables to detect the presence of subsurface defects. From a static point of view, the subsurface defect modifies the local stiffness of the structure, and consequently the resonant frequency of the SMM head. In the case of tip-object contact, we should consider two effects:

a) change in the contact stiffness related to the local elastic modulus of materials (the tip-surface interaction is equivalent to a spring which depends on the elastic constants).

b) variation of the structural elasticity, i.e. effect of the bending of the “surface membrane” caused by large subsurface defects. In this case, the observation depth is given by the global geometry of the sample and defect. The apparent stiffness may decrease when $E_{\text{defect}} < E_{\text{sample}}$ (E is the Young’s modulus); it is the case for subsurface holes. Conversely, it may increase if the stiffness of the defect is higher than the one of the sample: $E_{\text{defect}} > E_{\text{sample}}$.

In this section, we study the last case, corresponding to the effects of subsurface defects visible in the obtained images. Some interpretations have been given [7-10], but the interpretation of the results remains a challenge. We will first consider the softer case (the equivalent spring decreases because the
subsurface defect has a lower Young’s modulus. A representation of a simple square hole is given in figure 2.a. This defect can be approximated with a clamped beam shown in figure 2.b.

![Figure 2.a. Simple subsurface defect](image1)

![Figure 2.b. Equivalent beam](image2)

The modeling of the groove (defect under surface) with a clamped beam enables to simply estimate the stiffness of the structure. The spring modeling the equivalent beam of figure 2.b. can be expressed as (at the middle of the beam):

$$k_{elas} = 16EI\left(\frac{z}{L}\right)^3$$

where $l$ is the width of the beam, $z$ the thickness, $L$ the length and $E$ the Young’s modulus. The vertical spring $k_i$, equivalent to the tip-sample interaction may be expressed from the hertzian theory of contact[11]. Because the main effect comes from the variation of the vertical spring, we may assume that the frequency shift originates in the modification of the equivalent spring $k_{eq}$:

$$k_{eq} = \frac{k_i k_{elas}}{k_i + k_{elas}}$$

We have plotted the theoretical stiffness $k_{eq}$ versus the defect depth $z$ for the following values of parameters: $L = 60$ µm, $l = 30$µm, spring of the cantilever $k_c = 166$ N/m, tip radius $R = 20$ µm and applied force $F = 8$ mN. Result is given in figure 3.a. By using the classical theory of the vibrating cantilever[12,13], we have computed the variation of the resonant frequency for different values of the defect depth $z$. In this model, the variation of the value of the horizontal spring has been neglected. Figure 3.b shows the high sensitivity of the technique to specific subsurface holes (a rectangular shaped groove). The limit investigation depth is ~50 µm in the described experimental conditions.

![Figure 3.a. Value of equivalent spring $k_{eq}$ versus defect depth $z$](image3)

![Figure 3.b. Theoretical frequency shift versus defect depth $z$](image4)

Another method has been suggested to model the sample with a very stiff inclusion or defect. In this case, the beam equivalence is no longer valid and a different model should be used. Let us consider a stiff substrate and a thin soft layer above the substrate. With the help of the Gao’s functions

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[14], used to estimate the mechanical constants of composites materials, we propose an interpolation between the mechanical constants of the defect and those of the sample to determine modified effective constants:

$$E_{\text{int}} = \chi E_{\text{defect}} - (1 - \chi)E_{\text{sample}} \quad \text{and} \quad \nu_{\text{int}} = \chi \nu_{\text{defect}} - (1 - \chi)\nu_{\text{sample}} \quad \text{with} \quad \chi = \frac{z}{z_{\text{max}}} \quad (3)$$

where $\nu$ is the Poisson’s ratio, the index int meaning interpolated. The index “sample” represents the substrate and the index “defect” the layer over the substrate. The parameter $\chi$ is defined between 0 and $z_{\text{max}}$ (maximum observation depth which could be estimated experimentally). The evolution of the value of $E_{\text{int}}$ is given in figure 4 in the case of a resin (SU8) layer having a constant stiffness above a silicon wafer. The value of $z_{\text{max}}$ has been experimentally fixed to 60 µm.

![Figure 4. Interpolation of the Young modulus in the case of a resin layer onto a silicon wafer](image)

4. Experimental results and limits of the approach

Experimental results corresponding to holes inside sample has already been presented [8, 9]. The experimental exponent of the $z$ variable is a little bit smaller than the theoretical one, but experimental results are consistent with the heuristic theory from both mechanical and acoustic point of view. In order to determine the observation depth of the method, we have done series of experiments with different subsurface silicon gratings. The test samples had a quasi-plane surface (optical quality polishing). The tip was pressed onto this surface so that no surface effect was present in the images.

![Figure 5.a. Cross-section of the sample composed of a machined silicon wafer covered with resin layer; units are µm](image)  

![Figure 5.b. Amplitude image; size: 100*100 µm Operating frequency ~ 32 kHz](image)

Concerning the case of stiffer subsurface defect presented in this paper, we have realized different samples made of silicon grating buried under a polymer as shows in figure 5.a. The value of Young’s modulus of SU8 resin is about 4 GPa, very low compare to the Young’s modulus of silicon (169 GPa with the used crystalline orientation). The silicon grating was buried under 15 microns thickness of
SU8 resin. Figure 5.b. shows the amplitude image obtained by scanning the sample surface with a sapphire tip, as described above. The stress measured by the tip is obviously higher in the presence of a buried defect (thinner resin layer). The vibrating sensor (resonating head of the SMM) in contact with the sample senses a stiffer surface and the resonance frequency is up-shifted. Thus, amplitude image seems brighter in this area, as expected from the model. The contrast is high enough in the case of 15 µm thickness layer, as pointed out in figure 5.b. Different samples with variable layer thickness have been realized in order to check the previously shown model based on Gao’s functions. This phenomenological approach gives, with a good approximation, the image contrast as long as the layer is thin enough; because the resin surface is usually stiffer than the volume, optical observations of the surfaces after scanning have shown that the technique is non destructive. In the case of the presented sample, the theoretical frequency shift was estimated to be 2575Hz. The experimental value was 1850 Hz, close from the theoretical one in this multi-parameter experiment.

5. Conclusion
We have developed heuristic models based on the use of equivalent springs taking of account the contribution of a defect on the tip-object contact and evaluated the variations of vibration frequency of the cantilever. The subsurface investigation of defect using mechanical effects leads us to propose simplified analytical models giving a qualitative approach and estimating the contrast of the images. Two limit cases have been considered (subsurface defect very soft or very stiff compared to the surface layer). In the first case (softer defect), the main observed effect results from a bending of the layer between the tip and the defect. When the defect is very stiff, the proposed model is based on the Gao’s functions (interpolation) and the structure has been approximated with a gradient of elastic parameter. Globally, the mechanical proposed approaches give qualitative results that may be used to interpret the contrast observed in the images and also to estimate the main features of the investigated samples. Experimental results have demonstrated the validity of the models in specific cases.

We may note that the used tips (contact radius is typically about 1 micron) enable to detect large subsurface defects at a depth above 30 microns (the maximum investigation depth is usually in the 50-60 µm range). The accuracy of the technique for stiffness measurement is related to the knowledge of the different parameters (geometry, knowledge of the tip and cantilever constants, and applied force).

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