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Depth sensitivity of subsurface imaging using atomic force acoustic microscopy: FEA Study

Xu Yan 1, 3, Wei Xu 1, 3, Qian Cheng 1 and Zheng Xu 1, 3

1 Institute of Acoustics, Tongji University, Shanghai, 200092, People’s Republic of China
2 Department of Spine Surgery, Tongji Hospital, Tongji University School of Medicine, Shanghai, 200065, People’s Republic of China
3 These authors contributed equally to this work.

E-mail: gotoxvzheng@tongji.edu.cn

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Abstract

Atomic force acoustic microscopy makes it possible to image objects below the surface of a sample. Such subsurface imaging capabilities are of great interest in electronics, semiconductors, (bio) materials and manufacturing, polymers and microbiology. In this article, we report a numerical method to study the subsurface depth sensitivity. We calculated the depth sensitivity of atomic force microscopy and atomic force acoustic microscopy for subsurface objects of different heights and elasticity, as well as for different probe radii, applied forces and indentation depths. We also conducted experiments to validate the simulations. The results indicate that atomic force acoustic microscopy has higher subsurface depth sensitivity than atomic force microscopy.

1. Introduction

In recent decades, atomic force microscopy (AFM) has developed into a characterization tool to investigate new materials and devices on the nanoscale. For example, AFM is used to measure parameters such as elastic moduli [1–3], adhesion [4], viscoelasticity [5, 6], particle size and concentration [7], device integrity [8], material surface roughness [9, 10], and the topography of nanostructural materials [11–14]. In atomic force acoustic microscopy (AFAM), which is derived from AFM, either the tip or the sample is excited with an ultrasonic wave. This method can be used to image objects below the surface of a sample. Such subsurface imaging capabilities are of great interest in several fields, such as electronics, semiconductors, (bio)materials and manufacturing, polymers and microbiology [15].

Many methods have been proposed to improve the imaging resolution of both AFM and AFAM. For example, to improve the AFM image quality, the van der Waals force was eliminated by immersing the tip and sample in fluid [16]. In another study, single-walled carbon nanotubes were used as probes for high-resolution imaging [17, 18]. Moreover, Rabe and Arnold applied ultrasonic vibration at the bottom of the sample to detect the ultrasonic vibration amplitude with AFM (i.e. the basis of AFAM), achieving a depth sensitivity of about 100 nm [19]. To further increase the resolution of AFAM, Fukuma et al proposed the use of a fixed-frequency excitation signal to drive a cantilever and detect the change of the phase difference between the cantilever deflection and excitation signals [20].

To further increase the AFAM imaging quality, understanding of its mechanism is necessary. Schwarz et al introduced an analytical model for the calculation of the frequency shift using harmonic potentials, whereby they considered the cantilever approach process as a highly nonlinear problem [21]. Their results revealed a shift of the resonance frequency during approach of the cantilever to the surface. Melcher et al studied the physical origins of phase contrast in dynamic liquid-phase AFM. Their results indicated that the phase contrast derives primarily from a unique energy flow channel that opens up in liquids owing to the momentary excitation of higher eigenmodes [22]. Our group also investigated the interaction mechanism between the tip and sample in the contact mode of AFAM using the mass–spring model. We showed that good AFAM image contrast could be obtained when the the system is operated at resonance frequencies of the sample and the tip [23].
Cantrell investigated theoretically the phase contrast of AFAM. Their results indicate that dissipative force parameters dominate phase contrast at low drive frequencies, whereas conservative force parameters dominate phase contrast at high drive frequencies. However, the authors neglected the complex problem of surface oscillations in their calculations [24].

It is difficult to calculate the depth sensitivity of either AFM or AFAM theoretically, because most models have 2D axial symmetry, whereas most structures investigated when assessing depth sensitivity are non-asymmetric. The model that we previously used to study the mechanism of AFAM is based on the mass–spring model, in which the contact area is neglected. However, variation of the contact area influences the depth sensitivity of AFAM. Furthermore, a time-dependent approach should be included to calculate phase contrast in AFAM [25]. Based on the reasons outlined above, the depth sensitivity of AFM and AFAM has seldom been investigated. In particular, the depth sensitivity of AFAM subsurface imaging has never been studied.

In this paper, we propose a method to simulate the scanning process of AFM and AFAM as a first step to determining the subsurface depth sensitivity and the mechanism of imaging is discussed.

2. Numerical and experimental details

Numerical calculations were conducted using finite-element method software (COMSOL Multiphysics, COMSOL AB, Stockholm, Sweden). The geometry and boundary conditions are shown in figure 1 (a). For comparison, a photograph (taken with a digital microscope; Hirox, KH7700, Japan) of the probe with the cantilever is shown in figure 1 (b). The length of the cantilever and the height of the probe in the simulation (figure 1 (a)) is set to be the same as measured (figure 1 (b)). The tip of the probe is a semi-sphere with a nominal radius of 10 nm. The sample used in the simulation is a cube (20 (width) μm × 100 (length) μm × 10 (height) μm). The density, Young’s modulus and Poisson ratio of the sample are 1050 kg m⁻³, 1.5 × 10⁵ Pa and 0.47, respectively. The values are close to those of biological soft tissue [3]. An object is buried in the subsurface of the sample. The density and the Poisson ratio of the subsurface object are set to match those of the sample, whereas the Young’s modulus is different. A constant force is applied at the end of the cantilever to ensure contact between the probe and the sample. A contact pair is set on the surface of the probe and the surface of the sample. The contact is calculated using the penalty method. For simplicity, the Van der Waals force and friction are neglected. In the simulation, the maximum of the indentation depth is about 2 nm, which is 1/5000 of the sample height. The force from the substrate can be neglected according to our previous results [3].

The bottom of the sample is fixed in AFM mode (contact mode), but set to vibrate at a certain frequency in AFAM mode. In AFAM mode, the resonance frequency of the probe with the sample is used as the excitation frequency for the bottom vibration. Under these conditions, the vibration amplitude of the cantilever is at its maximum and the images have high contrast [23]. The vibration frequency at the bottom is selected as the lowest mode of the eigenfrequency (59 kHz) of the probe. The value of the eigenfrequency is the same to the resonance frequency when the sample is not loaded. In experiment, the Young’s modulus of biological sample is much smaller than that of the probe. Therefore, the difference of the eigenfrequency to the resonance frequency of the cantilever is neglected. Figure 1 (c) is the lowest eigenmode of the probe.

We increased the density of the mesh near the contact area in both the tip and sample. The model contains about 6000 quadrilateral elements and the degree of freedom is about 30 000. In AFAM, the time step is set to 1/
10 of the period of the vibrations at the bottom. When finished, the displacement along the $y$-axis is recorded (AFM) and the phase is acquired (AFAM). Then, the probe moves to the next point. The probe sweeps the surface every 0.2 nm, in a 20 nm region near the left boundary of the subsurface object along the $x$-axis. It takes about 6 h to finish one calculation in our workstation (DELL, E5-2650 v4, 512 G).

We used the depth sensitivity to evaluate the resolution of AFM and AFAM subsurface imaging. As shown in figure 1, the probe scans along $x$-axis on the surface of the sample. The scanning curve of the probe is shown in figure 2. In figure 2, the $x$-axis displacement indicates the probe space coordinate on the surface of the sample and the $y$-axis data represents the displacement of the probe for AFM and the phase change for AFAM. The subsurface object influences the scanning curve when the probe scans along the $x$-axis. This is because the existence of the subsurface object changes the strain map in the sample and further influences the displacement of the probe along $y$-axis. The depth sensitivity ($r_x$) is defined as the 25% to 75% interval between the maximum and minimum of the step function [26]:

$$y_1 = a + 0.25 \times (b - a)$$

$$y_2 = b - 0.25 \times (b - a)$$

where $a$ and $b$ are the minimum and maximum of the $y$-axis data.

The depth sensitivity is defined as

$$r_x = x_2 - x_1$$

where $x_1$ and $x_2$ are the locations of the space axis corresponding to $y_1$ and $y_2$, and $l$ is the length of the image (50 μm in our experiment). Thus, the decrease of $r_x$ indicates an increased subsurface depth sensitivity.

According to the definition of depth sensitivity as is shown in equation (3), the $r_x$ value indicates the sensitivity of the AFM and AFAM to the subsurface object. The depth sensitivity decreases by the increase of the $r_x$. In AFM measurement, the depth sensitivity is determined by the probe curvature radius. The indentation depth in AFM is about 2 nm. In AFAM measurement, the sample, the selected working frequency, the eigenfrequency of the cantilever and the curvature radius of the probe influence the depth sensitivity. Numerical results indicate the resolution of the AFAM is higher than that of AFM when the working frequency is the resonance frequency of probe-sample system in most of the cases. The depth sensitivity of AFAM decreases when the working frequency deviates from the resonance frequency of probe-sample system.

3. Results and discussion

We investigated the subsurface depth sensitivity of AFM and the results are shown in figure 3. The influence of the height of the subsurface object on the depth sensitivity is shown in figure 3(a). The elasticity of the subsurface object, probe radius and contact force are $6 \times 10^5$ Pa, 10 nm, $5 \times 10^{-12}$ N, respectively. The results indicate that the depth sensitivity increases with increasing height of the subsurface object. This is because the strain in the sample quickly decreases with increasing depth [3]. When the depth of the subsurface object is small, it indicates the surface of the subsurface object is close to the surface of the sample. Thus the probe is influenced...
greatly by the subsurface object, which further influences the scanning curve. Therefore, by the increase of the subsurface object height, the depth sensitivity also increases.

We investigated the influence of the Young’s modulus of the subsurface object on the depth sensitivity and the results are shown in figure 3(b). The height of the subsurface object, probe radius and contact force are 5 μm, 10 nm, 5 × 10^{-12} N, respectively. The depth sensitivity first decreases and then increases with increasing Young’s modulus. The depth sensitivity becomes zero when the Young’s modulus of the subsurface object reaches that of the sample. This suggests that the depth sensitivity is determined by the difference between the Young’s modulus of the subsurface object and that of the sample. The ratio of the stress to strain increases with increasing Young’s modulus of the subsurface object. When the Young’s modulus of the sample is greater than that of the subsurface object, for a given stress, the strain and y-axis displacement decrease with increasing Young’s modulus. By contrast, when the Young’s modulus of the subsurface object is greater than that of the sample, the strain and y-axis displacement also decrease with increasing Young’s modulus. Therefore, the depth sensitivity is determined by the difference between the Young’s modulus of the subsurface object and that of the sample.

We then investigated the influence of the probe radius on the depth sensitivity and the results are shown in figure 3(c). The height and elasticity of the subsurface object, and contact force are 5 μm, 6 × 10^5 Pa, 5 × 10^{-12} N, respectively. The resolution decreases with increasing radius of the probe tip. This is because the contact area increases as the probe radius is increased. For a given applied force to the cantilever, the indentation depth decreases with increasing probe tip radius. This decreases both the y-axis displacement and depth sensitivity.

Next, we investigated the influence of the applied downforce and the results are shown in figure 3(d). The height and elasticity of the subsurface object, and probe radius are 5 μm, 6 × 10^5 Pa, 10 nm, respectively. The depth sensitivity increases with increasing applied downforce, which also increases the stress and the strain difference in the sample. In other words, the indentation depth will affect the depth sensitivity. The depth sensitivity increases by the increase of the indentation depth.

Figure 3. Subsurface depth sensitivity of AFM as a function of (a) height, (b) elasticity of the subsurface object, (c) probe radius and (d) applied force.
We studied three points \((x = 45 \mu m, x = 50 \mu m, x = 55 \mu m)\) in the scanning process of the probe. Figure 4 is the strain map of the probe-sample system. In figure 4(a), the subsurface object is marked by the red dotted frame. The three scanning points shown in the figure 4(a) are marked by the red hollow circles. The strain maps at three points correspond to the figures 4(c)–(e), respectively. The strain maps of these three points are similar as is shown in figure. Since the size of the probe is far smaller than the sample, the difference between the \(y\) displacement and strain map in (c), (d) and (e) is difficult to be observed. Therefore, the \(y\) displacement of the probe was used to evaluate the difference of the strain maps of these points. Figure 4(b) is the \(y\) displacement curve of the probe. In figure 4(b), points \(c, d\) and \(e\) correspond to figures 4(c)–(e), respectively. In figure 4(b), the \(y\) displacement of the probe deviates from the amount of change in displacement at \(d\) point. With \(d\) point as the demarcation point, the influence of the probe mainly comes from the sample on the left side. The sample and the subsurface object have a major influence on the probe on the right side. In the \(y\) displacement curve of the probe, the effect of the subsurface object on the probe increase by the decrease of distance between the probe and the subsurface object. The change of \(y\) displacement of the probe represents the change in probe-sample system strain.

Furthermore, we numerically simulated the depth sensitivity of AFAM and the results are shown in figure 5. The depth sensitivity relates to the height of the subsurface object, the elasticity of the subsurface object and the radius of the probe tip. In figure 5(a), the elasticity of subsurface object, probe radius and maximum indentation depth are \(6 \times 10^5 \text{ Pa}, 10 \text{ nm}, 2 \text{ nm}\), respectively. In figure 5(b), the height of subsurface object, probe radius and maximum indentation depth are \(5 \mu m, 10 \text{ nm}, 2 \text{ nm}\), respectively. However, the subsurface depth sensitivity depends on the phase contrast: the detected phase is determined by the ultrasound propagation time in the material. Because the sound speed changes in the subsurface object when its elasticity differs from that of the sample, the phase contrast depends on the height of the object and the elasticity difference between it and the sample. Therefore, the subsurface depth sensitivity increases with increasing subsurface object height and with increasing difference between the elasticity of the subsurface object and the sample. The detected phase is a space-averaged signal of the contact area, and the contact area increases with increasing probe radius (figure 5(c)). In figure 5(c), the height and elasticity of subsurface object, and maximum indentation depth are \(5 \mu m, 6 \times 10^5 \text{ Pa}, 2 \text{ nm}\), respectively. Therefore, the depth sensitivity decreases with increasing probe radius. In addition, the relation between the indentation depth and the depth sensitivity is studied. In figure 5(d), the height and elasticity of subsurface object, and maximum indentation depth are \(5 \mu m, 6 \times 10^5 \text{ Pa}, 10 \text{ nm}\), respectively. It can be observed in figure 5(d) that the resolution is independent of the maximum indentation depth of AFAM in the range studied. In AFAM mode, to ensure contact between the probe tip and the sample, the maximum indentation depth was set to 2 nm. The change of the contact area was almost the same for all maximum indentation depths. Thus, the phase contrast is almost independent of the maximum indentation depth in the range studied.

![Figure 4](image-url)
The depth sensitivity of AFM and AFAM are compared in figures 6(a) and (b) for different heights and elasticities of the subsurface object, respectively. In both cases, the depth sensitivity of AFAM is better than of AFM under most conditions. The subsurface imaging with AFM is based on the feedback force: when the probe scans the sample where an object is buried in the subsurface, the feedback force changes owing to the elasticity of the object and the probe radius.
difference between the subsurface object and the sample. A change of the height or the elasticity of the subsurface object influences the depth sensitivity. By contrast, subsurface imaging with AFAM is based on the signal difference: the acoustic signal applied to the bottom of the sample propagates to the upper surface of the sample and is detected by the probe tip. Thus, for AFAM, a change of either the elasticity or height of the subsurface object affects the received signal. The change of the signal (i.e. AFAM) is more sensitive than the change of the force (i.e. AFM). We note that the phase contrast has its maximum around the peak of the resonance, where a slight change of the material properties results in great phase contrast. Moreover, in a real situation, the density and the Poisson ratio of the subsurface object will differ from those of the sample, which will further increase the phase contrast in AFAM.

Using the proposed frequency and method, we compared AFM and AFAM imaging for neural stem cell on a glass slide, as shown in figure 7. In the experiment, cantilever stiffness, contact force, tip diameter and resonance frequency are 15.7 GPa, 243 nN, 10 nm, 66.2 kHz, respectively. The contour of the nucleus is much clearer for the AFAM image than the AFM image. Furthermore, the cytoskeleton of the cell can be clearly observed in the AFAM image, whereas it can hardly be observed in the AFM image. These results are consistent with those of our numerical simulation since the AFM expresses the force difference and the AFAM expresses the phase difference as has been mentioned above.

Although AFAM has higher depth sensitivity than AFM in subsurface imaging, its phase data is more complex than that of AFM, because it includes information relating to the elasticity, height, density and the Poisson ratio of the subsurface object. Therefore, it is not clear what these parameters change in response to the bottom vibration. In future work, we will investigate a method of parametric inversion of the subsurface material using AFAM at different frequencies.

4. Conclusion

We numerically investigated the subsurface depth sensitivity of AFM and AFAM. Our results indicate that the depth sensitivity increases with increasing height of the subsurface object, increasing the difference between the elasticity of the sample and the subsurface object, and decreasing probe tip radius. The results also indicate that the depth sensitivity of AFAM is higher than that of AFM in most cases. By this numerical method, the lateral subsurface resolution can be estimated and it is instructive to improve the subsurface depth sensitivity. Furthermore, we will try to make standard samples and test a ‘flat’ surface with a buried object. In current stage, it is difficult to determine Young’s modulus using AFAM for an unknown subsurface object. The related works will be carried out in the near future.
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ORCID iDs

Zheng Xu @ https://orcid.org/0000-0002-2271-9089

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