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Nanomechanical Characterization of SiO_x Film by Atomic Force Acoustic Microscopy

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Abstract: Atomic force acoustic microscopy (AFAM) has been developed in order to evaluate the mechanical properties of the material at the nanometer scale. The SiOx film on the silicon wafer was prepared successfully by plasma enhanced chemical vapor deposition (PECVD). In order to characterize the elastic property of the SiOx film , the images of cantilever amplitude were visualized when the sample was excited at 100kHz and 400kHz frequency. The acoustic amplitude images also were discussed at the different exciting amplitudes. The results showed that the acoustic amplitude images can provide the information about local elasticity of the materials.

Keywords: Atomic force acoustic microscopy; SiO_x film; acoustic amplitude image; local elasticity

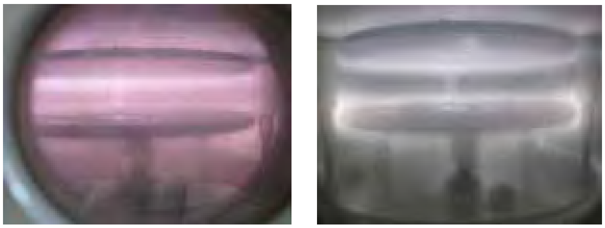
1 Introduction

With high barrier properties against oxygen or water vapour, SiO_x coatings are widely used as food and medical packaging barrier layer. SiO2 is also used as passive layer for micro-electron device. The plasma enhanced chemical vapour deposition (PECVD) has been utilized successfully to produce SiOx coatings on polymer substrates [1]. Atomic force microscopy can create a highly magnified three dimensional image of a surface[2].The trend towards nanometer dimensions in many emerging application demands new approaches to materials characters such as mechanical properties. Atomic force acoustic microscopy (AFAM) is a dynamical technique [3, 4, 5, 6], where the cantilever or the sample surface is vibrated at ultrasonic frequencies while a sample surface is scanned with the sensor tip contacting the sample. At a consequence, the amplitude of the cantilever vibration as well as the shift of the cantilever resonance frequencies contain information about local tip-sample contact stiffness and can be used as imaging quantities[7]. The AFAM technique has been demonstrated to be a powerful tool for the local investigation of the elastic prosperities of sample surface. Moreover, in the case of a thin film deposited over a substrate, AFAM measurements are less affected by mechanical properties of the substrate itself[8,9]. The AFAM images of the piezoelectric lead zirconate ceramic had been shown [5]. The local elastic properties and ferroelectric domain configuration of piezoelectric ceramics have been examined by atomic force acoustic microscopy [7]. The acoustic images of ferroelectric domain in PMN-PT were visualized by AFAM at low frequency below 10 kHz [10]. This wok represents a first attempt to using AFAM technique for SiOx film , obtained by plasma enhanced chemical vapour deposition (PECVD). We research the SiOx film on the silicon substrate by the AFAM, the acoustic response image and acoustic amplitude image were provided. The results showed the acoustic amplitude images can provide the information about local elasticity of the materials.

2 Experimental investigation

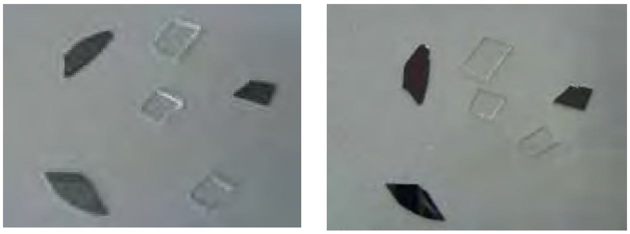
2.1 Material preparation

The single crystal silicon <111> wafers as the substrates were cleaned sequentially in an ultrasonic bath using ethanol, acetone and de-ionized water before they were mounted on the sample holder. The SiOx films were deposited by plasma enhanced chemical vapour deposition (PECVD) through glow discharge, shown in Fig.1. The background pressure is 4.1 Pa, oxygen flow rate was 21 sccm, monomer flow rate was 10.5 sccm, and input power (200W) was kept for 20 minutes during the deposition. Hexamethyldisiloxane (HMDSO) in mixture with oxygen (O_2) is used as deposition gases. Fig.1 (a) showed the discharge photography of Ar_2 at the 100W power, Fig.1(b) showed the discharge photography of mixture gas including oxygen, monomer and Ar_2 at the 200W power. The substrates were placed on the sample holder. Fig.2 showed the samples with and without the SiOx coatings.



(a) Ar_2 discharge at the 100W (b) Mixture gas discharge at 200W

Fig. 1 The photography of discharge different gases



(a) the sample without the SiOx coating (b) the sample without the SiOx coating

Fig. 2 The photography of the samples without (a) and with (b) the SiOx coating.

2.2 Experimental set-up

A modified commercial atomic force microscope (CSPM 5000, Ben Yuan, China) is used to image the sample surface and to control the static cantilever forces before the tip contacts the sample. An external frequency generator (AFG3021, Tektronix, USA) provides a stable sinusoidal excitation, which is applied to a piezoelectric transducer coupled to the back side of the sample. The transducer with a center frequency of 1MHz is used in thickness vibration. It emits longitudinal acoustic waves into the sample which cause out-of-plane vibration of the sample surface. These surface vibrations are transmitted into the cantilever via the sensor tip. The cantilever vibrations are measured by the optical beam-deflection sensor of the instrument, and the signal is fed to the lock-in amplifier (7280 DSP, Signal Recovery, USA) which also receives a reference signal at the excitation frequency. For imaging, a fixed excitation frequency is selected. While the sample surface is scanned, the amplitude of the cantilever is modulated by the lock-in amplifier and fed into an auxiliary channel of the AFM in order to image. Fig.3 and Fig 4 show the block diagram and photograph of the AFAM system

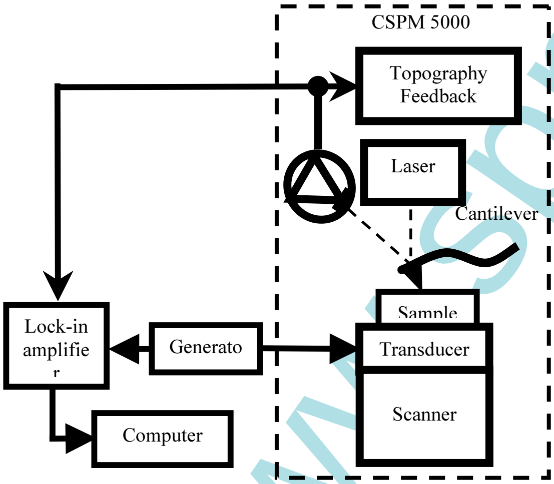


Fig. 3 Block diagram of AFAM system

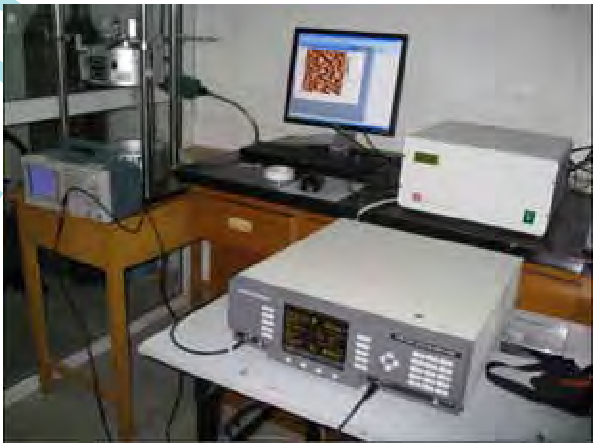


Fig. 4 Photograph of AFAM system

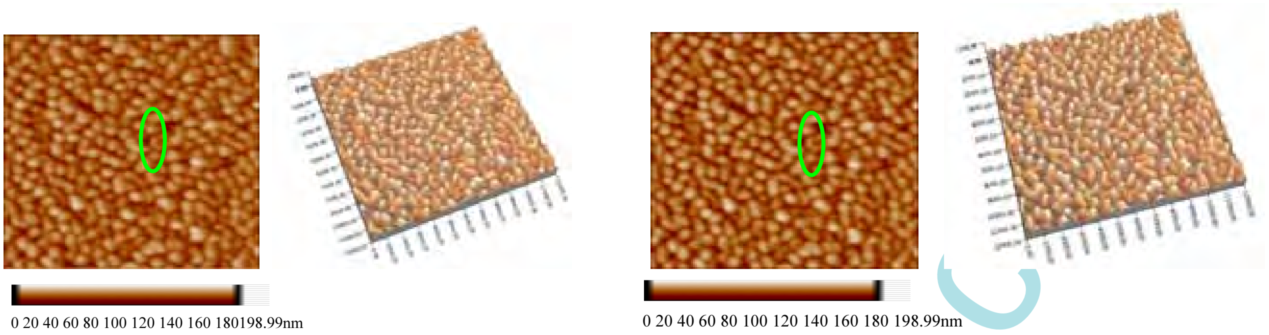
2.3 Signal obtaining

We can obtain the topography and acoustic respond images of same areas in contact mode using the same imaging channel of the AFM, but the latter was obtained when the sample was excited at ultrasonic frequency. In order to image the acoustic amplitude of the cantilever, we output the original up-down signal into the signal channel of lock-in amplifier and the excitation signal into the reference channel of lock-in amplifier, so the amplitude of the cantilever was modulated. When the sample was scanned, the acoustic amplitude images were obtained using the additional channel of AFM. In order to observe the acoustic amplitude of the cantilever affected by the excitation amplitude, we obtained the acoustic amplitude images with different excitation amplitudes. We also obtained the acoustic amplitude images at different excitation frequency in different areas of same samples.

3 Results and Analysis

3.1 Results

3.1.1 Acoustic response image

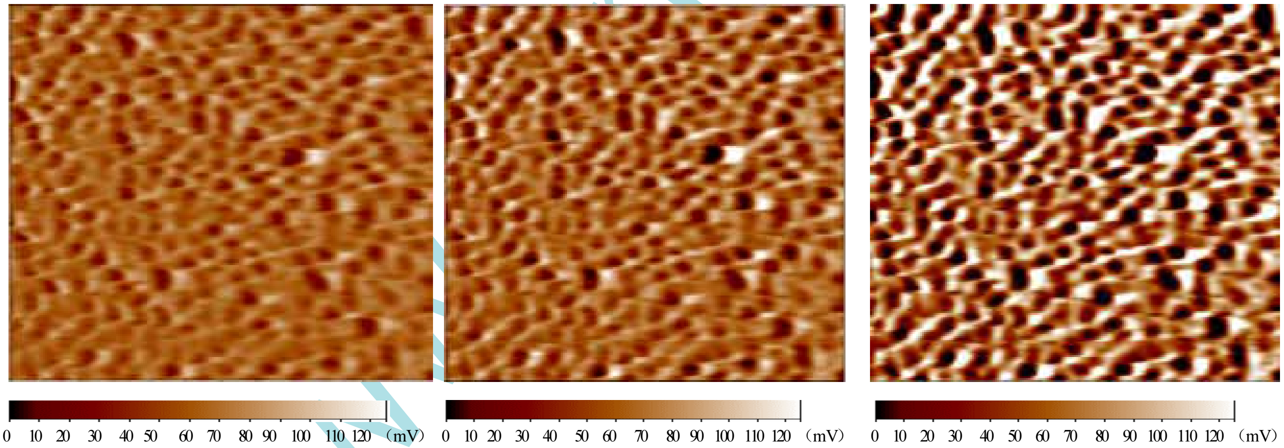


(a) topography image (b) acoustic response image

Fig. 5 The topography images (12μm×12μm) and acoustic response image of SiOx film on the silicon excited at 100kHz frequency

Fig.5 (a) is the topographic image of the SiOx in the static state, and presents the topography image of the crystal of the sample without any additional information. Fig.5 (b) is the acoustic response image of the acoustic signal at modulation frequency of 100 kHz and exhibits the inhomogeneous areas with remarkable bright and dark contrast. In comparison of the topography and the acoustic response image, it can be clearly seen that these microstructure feature in the acoustic image are almost the same as those appearing in the topographic image of SiOx and the contrast of the image is stronger, such as the in ellipse region. Therefore we can deduce that the acoustic response images reflect more clearly the real microstructure in SiOx film.

3.1.2 Acoustic amplitude images of sample excited by different amplitude signals

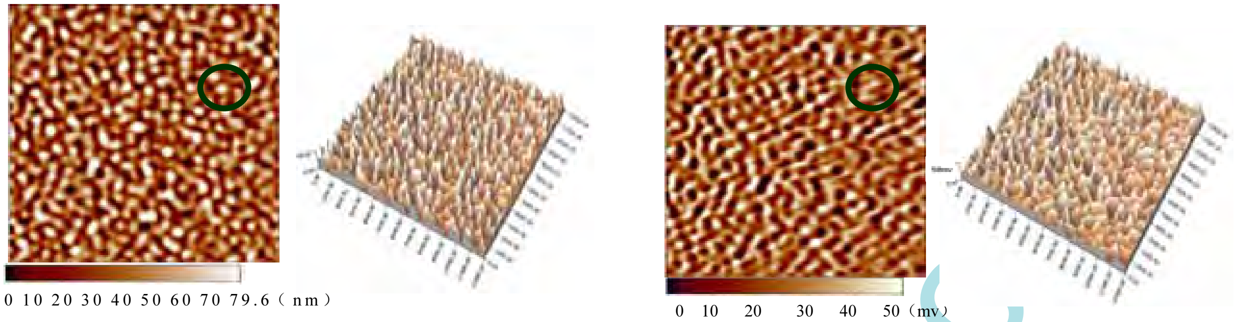


(a) A=1V (b) A=2V (c) A=4V

Fig. 6 The acoustic amplitude image of the tested sample with the different ultrasonic exciting amplitude (A) at the same frequency 100kHz.

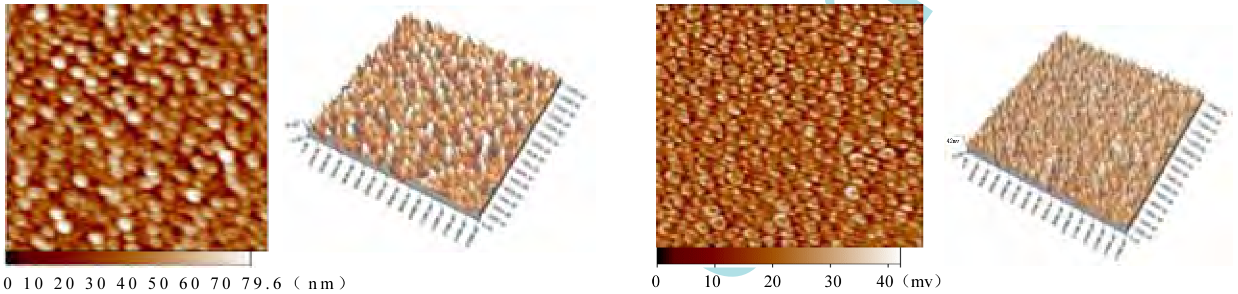
Fig.6 was the acoustic amplitude images while the sample was excited at the 100 kHz frequency and scanned with the sensor tip contacting the sample. The excitation amplitude was 1V, 2V, 4V. As the acoustic response images, the amplitude was bigger in brighter areas and smaller in darker areas. The measurement areas were same with the areas in Fig.5, so the acoustic response images of tested sample were same with Fig.5 (b). But we found that the contrast of brightness is inverse between the acoustic amplitude image and acoustic response image for same areas, such as Fig.5 (b) and Fig.6 (a). From Fig.6, we can find that the contrast of brightness increases with the excitation amplitude increasing, such as Fig.6 (a), (b) and (c).

3.1.3 Acoustic amplitude images of sample excited by different frequency signals



(a) Acoustic response images (b) Acoustic amplitude images

Fig. 7 Acoustic response (a) and acoustic amplitude (b) images of SiOx film excited at the 100 kHz frequency.



(a) Acoustic response images (b) Acoustic amplitude images

Fig. 8 Acoustic response (a) and acoustic amplitude (b) images of SiOx film excited at the 400 kHz frequency.

Fig.7 showed the acoustic response images and the acoustic amplitude images when the sample was excited at the 100 kHz frequency. The sample was prepared in the same condition with the sample of Fig.5 and Fig.6. The images of Fig.7 and Fig.8 showed different areas of same sample. In Fig.7, the sample was excited at the 100 kHz in one area. In Fig.8 the sample was excited at the 400 kHz in the other area. It was interesting that the contrast of the brightness was consistent between the acoustic amplitude image and acoustic response image, shown in Fig.8, but it was inverse, such as in Fig.7.

3.2 Analysis of the cantilever amplitude

In AFAM,the acoustic wave would transmit and result stress field in the sample when the piezoelectric transducer is vibrated under AC voltage. While in the case of acoustic amplitude imaging, the amplitude contrasts reflect the responses of SiOx film to the local elastic stress fields in the sample, which is closely relevant to the local contact stiffness between the tip and sample surface during scanning. Surfaces with higher elastic modulus generate contact resonance curves with increased center frequency and higher amplitude than soft surface [5, 9]. The vibration frequency spectra of the tip for different contact stiffness are shown schematically in Fig.9. The frequency corresponding to line (1) is below the frequency corresponding to the cross point of the two curves. The frequency corresponding to line (2) is above the frequency corresponding to the cross point of the two curves. From the Fig.6, we can find that when the excitation frequency (such (1) frequency in Fig.9) is below and nearer the contact resonance frequency of soft areas, the soft areas appear brighter (higher amplitude) in the acoustic amplitude image [5]. When the excitation frequency is above resonance frequency of the soft areas and near the stiff areas, the amplitude of the stiff areas is higher.

In AFM system, due to the feedback the repulsion in the higher areas of the topography is bigger, so the contact stiffness and contact resonance frequency is higher than the lower areas. So, when the excitation frequency is far from the contact resonance frequency of the brighter areas (equal to stiff surface) of the topography and near the contact resonance frequency of the darker areas (equal to the soft surface), the cantilever acoustic amplitude is lower in the stiff areas, the contrast in brightness between the acoustic response image and the acoustic amplitude image is inverse, such can be seen in Fig.7. When the excitation frequency is near the contact resonance frequency of the brighter areas (equal to stiff surface) of the topography, the contrast in brightness between the acoustic response image and the acoustic amplitude image is consistent, such can be seen in Fig.8.

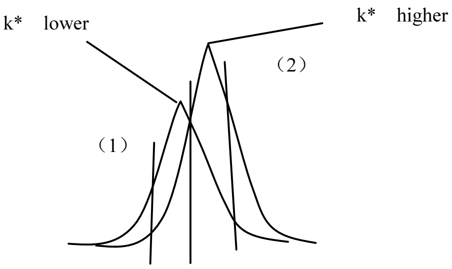


Fig. 9 Schematic spectra

4 Conclusions

We have shown that the contact resonances of AFAM cantilevers are a sensitive method for reflecting the elasticity of the materials. In this work, we think the regions with the higher (or lower) contact stiffness induced by the feedback and the topography as stiff regions (or soft regions). The soft surfaces have the low contact resonance frequency when the static force is same. When the excitation frequency is near the contact resonance frequency of the stiff surface, the cantilever amplitude of the stiff surface is higher. So the cantilever amplitude images can reflect the elasticity and subsurface information by changing the excitation frequency. At the same time, due to the feedback of the AFM, the contrast in brightness between the acoustic response image and acoustic amplitude image was consistent or inverse at the different excitation frequency. The results also showed that the contrast of acoustic amplitude between the soft and the stiff areas increased with the excitation amplitude increasing.

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