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Measurement of Physical Thickness of Native Oxide Using Accurately-Calibrated Atomic Force Microscope

Shintaro Aoyama, Yoshinori Nakagawa and Tadahiro Ohmi

Department of Electronics, Tohoku University, Sendai 980, Japan
PHON./FAX 022(263)9395

Physical thickness of various native oxide grown on Si surface had been studied with accurately-calibrated atomic force microscope. For this purpose the method to calibrate the height (Z-axis) in the AFM image had been established. Based on accurate calibration, native oxide thickness grown in the atmosphere and various wet chemical solution for wafer cleaning were measured. Layer by layer growth of native oxide in the atmosphere had been confirmed from first grow step. The thickness of native oxide grown in the wet chemical solutions were compared with the value of XPS evaluation and some differences were confirmed.

1. Introduction

In ULSI technology toward manufacturing of sub-half micron devices, it becomes important to control native oxide growth on the Si surface. Especially to obtain ideal Si-metal contacts and high quality epitaxial Si films at low temperature, suppression of the native oxide growth is absolutely needed ,i.e., native oxide free processing[(1),(2)]. On the other hand, the presence of native oxide(preoxide) right before thermal oxidation becomes one of the factor dominating the insulating performance of very thin gate oxide, because the thickness ratio of the preoxide to net thermal oxide relatively increases as the gate oxide is thinner[(3)(4)]. Thickness measurement method of thin oxide films such as the native oxide has been developed by using XPS evaluation and detailed growth mechanism of native oxides has been investigated based on XPS evaluation[(5)-(7)], where the native oxide thickness is evaluated under the assumption of native oxide having quality equivalent to the thermal oxide. The purpose of this paper is to present a possibility to measure physical thickness of the native oxides films on the Si surface by means of accurately calibrated atomic force microscope (AFM). For this purpose we establish a calibration method of height(Z-axis) in the AFM images[(8)]. Practical thickness of the native oxide grown on the p(100)Si surface in various ambiences was studied.

2. Experimental

In the calibration procedure of AFM, firstly the standard samples which have fine steps were prepared. The structure was fabricated on the surface of thermal oxide film on the Si substrate. The thickness of the oxide film was 1000Å. Patterned SiO_2 surfaces were etched by buffered hydrogen fluoride (BHF) which has an extremely low etching rate of around 1Å/min level to control the fine etching depth by changing etching time. The arrangement of 2μm×2μm square pattern with a space of 1μm as shown in Fig.3 was utilized as the etching pattern. The etching depth was obtained from the difference of the oxide thickness before and after etching process at same point to measure. The thickness was measured by ellipsometer(model AEP-100, Shimazu Corp.) which has minimum detection angle of 0.005 degrees, while in the measurement of 1000Å silicon dioxide film on silicon substrate, precision was σ=0.17Å. In the entire process to prepare the sample, the surface micro-roughness was taken into consideration in order to obtain accurate calibration at sub-nanometer or nanometer scale steps formed by wet etching. The average surface microroughness of less than 1Å was yielded on the oxide surface. Secondly the patterned oxide surfaces of prepared samples of various etching depth were observed by the AFM(model SFA300, SEIKO INSTRUMENTS INC). The AFM apparatus was separated from the external microvibrations by using the active damper[(9)] and the sound insulating box in order to get clear AFM image and measurement accuracy. To obtain AFM images from sample surfaces the area of 10μm square were scanned with a speed of 10μm/sec and 256 lines. By correlating the step height measured by the AFM with the etching depth measured by the ellipsometry, the sensitivity of the piezo actuator was determined.

In the procedure to obtain the native oxide thickness by AFM measurement, the step height of mask thermal oxide (A)(around 30Å) is measured at first as shown in Fig.1(a). Secondly the step height (B) is obtained after oxidation process as shown in Fig.1(b). (A−B) gives "expanded" native oxide thickness above original Si surface. After striping mask oxide and native oxide, the step height (C) is evaluated as shown in Fig.1(c) where the value of (C) indicates the recessed Si consumed in native oxide growth. In the case native oxide etching was accompanied with its growth such as (NH_4OH + H_2O_2)(APM) clean, the procedure shown in Fig.1(d) was followed and thickness was obtained from (B+C−A). In these procedure same pattern and same measurement condition as in the calibration procedure were employed.

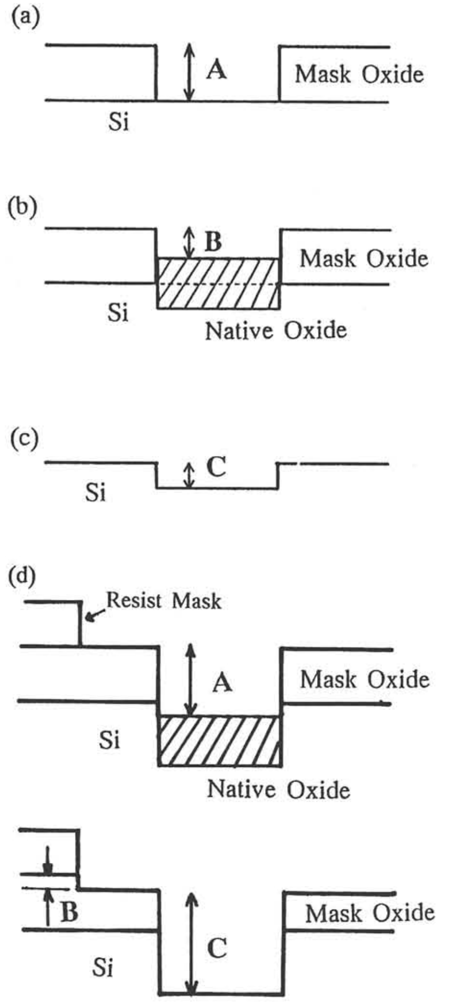

Fig.1 Procedure of measurement of native oxide thickness. (a) Before native oxidation. (b) After native oxide growth. (c)After strip oxide. (d)Measurement procedure in the case native oxide growth is accompanied by etching simultaneously.

3. Result and Discussion

Fig.2 shows the etching characteristic of the BHF used in the patterned etching, where the etching depths measured by the ellipsometry are plotted on the vertical axis and the etching time on the horizontal axis. It is seen from Fig.2 that the dependence of the etching depth is almost proportional to the etching time in sub-nanometer and nanometer region. The etching rate of 1.13Å/min was obtained from the slope of the fitted line. In Fig.3, the AFM image of the patterned surface of the SiO_2 film for various step heights were shown. Each step height was formed with the etching time shown in Fig.2. Clear step images corresponding to the etching depth were observed. In this picture, the Z−axes are re−illustrated in the scale after the calibration just discussed. The correlation of step height data obtained from AFM images with the etching depth measured by the ellipsometry is shown in Fig.4. In this figure, the step height is presented as output voltage of the piezo actuator which correspond to each step height. The linear relationship is observed in spite of well known second order hysteresis curve of piezo−electrics which was supposed to arise from a small displacement relative to the dimension of the piezo element. From the slope of the fitted line, the sensitivity of the piezo actuator can be obtained.

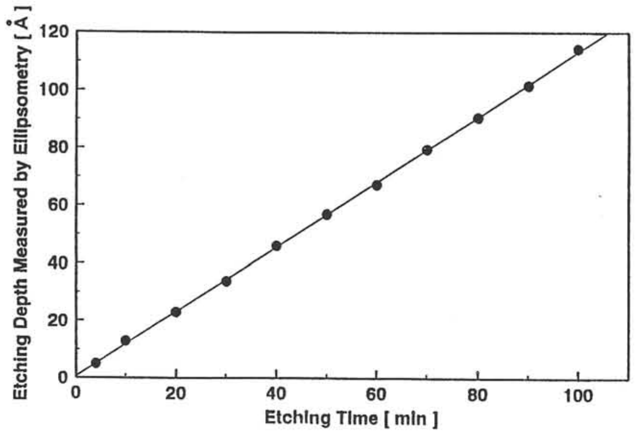

Fig.2 The etching characteristic of the thermal oxide by extremely low etching rate BHF.

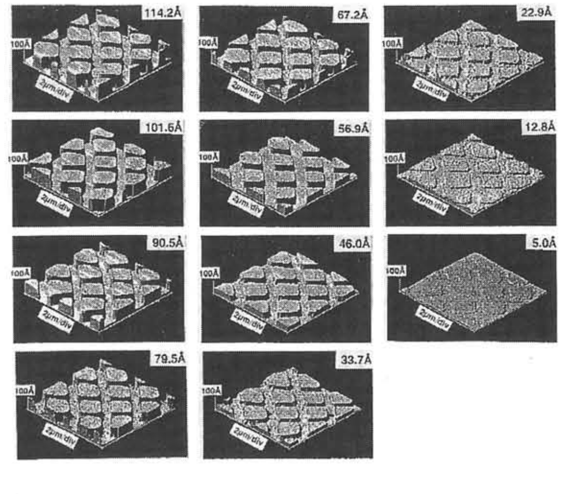

Fig.3 AFM images of standard samples of various etching depth.

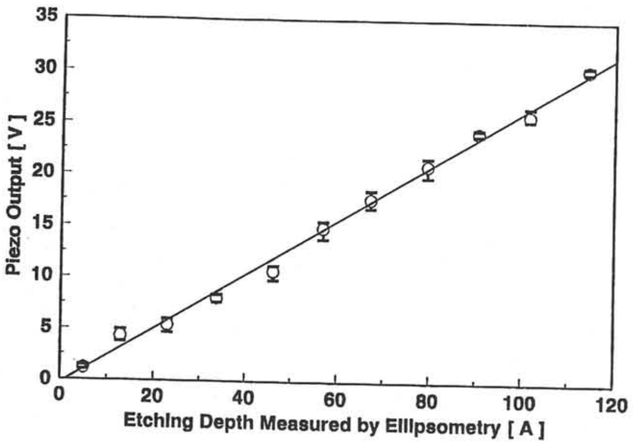

Fig.4 Correlation of PZT output in AFM images with etching depth measured by ellipsometry.

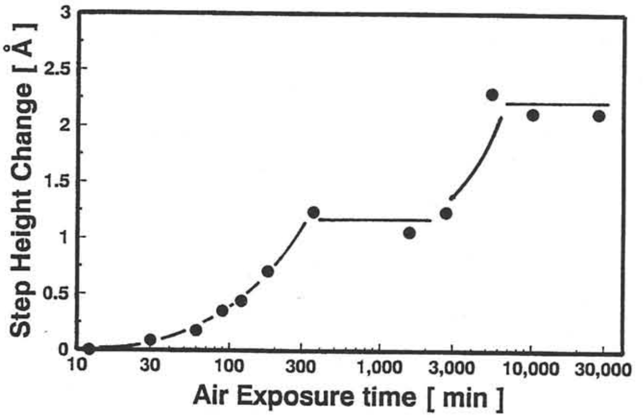

Fig.5 Change of step height(B) because of native oxide growth with air exposure.

	SPM	HPM	APM
AFM	13.2Å	6.0Å	10.1Å
Recessed Si	4.0Å	4.0Å	-
XPS	6.5Å	6.4Å	6.5Å
ΔE	3.91eV	3.92eV	3.86eV

SPM: $H_2SO_4 + H_2O_2$ mixture
HPM: $HCl + H_2O_2$ mixture
APM: $NH_4OH + H_2O_2$ mixture
ΔE: Binding energy shift of Si_{2p}

Table 1 Comparison of native oxide thicknesses under various wet chemical processes.

In Fig.5, change of (B) is plotted as a function of air exposure time. The change exhibits layer by layer growth of native oxide on Si surface. At time 300min first layer was grown up. The value of the step height change well coincide with half thickness of XPS evaluation discussed by M. Morita et.al.[6] The oxide film which has the density of like thermal oxide can be grown in the atmosphere. Table.1 shows various native oxide thickness measured by AFM compared with XPS evaluation. The differences of the thickness values suggest that the film quality of native oxide depend on the process. Native oxide grown in ($H_2SO_4 + H_2O_2$)(SPM) cleaning has less density than thermal oxide.

4. Conclusion

A calibration of the height in AFM image was studied. The structure fabrication on Si wafer was carried out by a process taking care of surface microroughness. The calibration was successfully performed on the scale of sub-nanometer by using silicon dioxide steps fabricated by the wet etching using advanced BHF having an extremely low etching rate. With this BHF etching possibility to fabricate the step structure of less than 1nm on thermal silicon dioxide on silicon substrate was confirmed.

Physical thickness of native oxide grown in the various ambiences had been measured. The native oxide film quality depends on the growth ambience.

3. Acknowledgement

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