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Assessment of interface roughness during plasma etching through the use of real-time ellipsometry

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ABSTRACT

Real-time *in situ* ellipsometry was used to investigate the etching of SiO_2/silicon wafers with a high concentration of Cl_2. We monitored the temporal trajectory of the ellipsometric parameter Δ and then selected several points for *ex situ* study using atomic force microscopy (AFM). There was a clearly observable transition period in the trajectory near the endpoint of the SiO_2/Si interface. We studied the relationship between the ellipsometric parameter Δ and the same point in the AFM *ex situ* measurements. Three stages, thin film, the interface layer, and the substrate, were analyzed in this work.

1. Introduction

The manipulation and characterization of surface films has become a crucial issue in integrated circuits, micro-electro mechanical systems (MEMS) and photonic devices. Plasma etching is the standard etching technique used in the production of such devices. *In situ* monitoring of each process, for the determination of film thickness and surface roughness is essential for improving the physical and electrical properties of the devices. In current practice, one needs to establish "optimum" empirical values for the plasma etching process. This makes it necessary to use tools to monitor the film characteristics during processing. Generally, atomic force microscopy (AFM) and spectroscopic ellipsometry (SE) are used to measure the properties of thin films. AFM is a local measurement technique that scans the specimen surface with nanometer-scale resolution. However, several minutes are required for a typical scan, making it less suitable for in-line inspections. The ellipsometric technique on the other hand, which averages the optical response of a light spot, has poor lateral resolution. On the positive side, it is a fast measurement technique that is sensitive to the roughness of the surface. This non-destructive in-line inspection technique can be used to monitor the growth and etching of various materials.

Studies [1,2] comparing ellipsometric and AFM measurements of Si surface characteristics have been widely conducted in surface science research. They have mainly compared the surface roughness measured by AFM with the ellipsometric parameters measured by an ellipsometer. According to these studies, Fang et al. [3] has claimed that the parameter Ψ is strongly related to the incident angle and the real part of the refractive index of Si, and that it is insensitive to surface roughness and oxide thickness. Nevertheless, the ellipsometric parameter Δ is strongly affected by surface roughness and oxide thickness. Based on the results of these studies, one can say that Δ would be a favorable parameter for studying surface roughness and oxide thickness. Rolland et al. [4] compared results obtained using ellipsometry and AFM of the surface morphology of polysilicon films after different etching processes. From a comparison of the results of measurements of the ellipsometric parameter Δ and the roughness as observed by AFM, they concluded that the hemisphere geometric model had the best fit to these two parameters. However, they analyzed surface roughness only at a few specific etched points rather than monitoring the fabrication process. Irregularity and deformation of the surface were not taken into account during the etching process, and these conditions can affect the effective medium model (EMA) for surface roughness determination. Therefore, the behavior of the ellipsometric parameter Δ as a function of surface texture was not always predictable. Shamiryan et al. [5] focused on how the behavior of Δ is related to the surface morphology. They explored TiN to etching plasma and declared that the surface roughness can be easily characterized by Δ, that the change in Δ is proportional to the roughness if the roughness does not exceed 30 nm.

We constructed a real-time ellipsometric technique [6] for *in situ* monitoring of the etching process, focusing on the analysis

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of roughness at the interface between SiO_2 and the silicon wafer. The etching process was carried out in high-density inductively coupled Cl_2 plasma to form a rather rough surface for investigation. The ellipsometric parameter Δ was recorded and illustrated in real-time during etching of silicon oxide on silicon. The temporal trajectory of Δ showed behavior was not as expected. We observed a clear transition period near the endpoint of the SiO_2/Si transition. The surface textures in this transition period were etched in sections for *ex situ* study using AFM. We discuss the relation of the surface roughness with the ellipsometric parameter Δ during this transition period. The results indicate that not only the endpoint during etching but also the transformation in the surface morphology between silicon, silicon oxide and their interface can be examined. The different sections of the etching points are analyzed to build the basis for this method of nondestructive measurement.

2. Experimental details

A 30-nm-thick thermal oxide layer was deposited on a p-type Si (1 0 0) wafer at 900 °C through dry oxidation in an atmospheric pressure chemical vapor deposition chamber (APCVD). Etching was performed in an inductively coupled plasma (ICP) etching system in which both the ICP source and bias powers were operated at 13.56 MHz, with power levels of 800 W and 150 W under a pressure of 10 mTorr and a gas mixture of 95 sccm Cl_2/5 sccm Ar. In this study, a homemade photoelastic modulation (PEM) ellipsometer [6] was installed on the plasma etching chamber. Two windows were located on either side of the chamber to allow the laser light (532 nm, Thorlabs) to impinge on the sample surface. The area explored by the laser beam was approximately 2 mm × 2.5 mm at an incident angle of 60°. Ellipsometry is a surface diagnosis tool that measures the change in elliptic polarization of the light reflected from the surface. The change in polarization states is expressed in terms of two ellipsometric parameters Ψ and Δ, defined as follows:

$$\frac{r_p}{r_s} = \tan\Psi \exp^{i\Delta}, \tag{1}$$

where r_p and r_s represent the complex reflection coefficient of the parallel and perpendicular components of polarized light to the plane of incidence, respectively. Utilizing the measured ellipsometric parameters together with the optical model, one can deduce the film thickness and its complex refractive index. The advantages of using the PEM for ellipsometric measurement are that it provides a resonance frequency for synchronous detection, and that no parasitic error is caused by beam deviation during rotating polarizer/analyzer ellipsometry. In addition, during PEM ellipsometry, Δ can be deduced from the harmonic intensity ratios (1f/2f) of the modulated signal in order to measure the optical properties with minimum background noise in a way that is suitable for surface roughness and oxide thickness measurement. Before measurement, we calibrated the physical settings according to Refs. [7,8]. Calibration included the azimuth of the polarizer, the optical axis of the PEM, the incident angle, and the modulation amplitude of the PEM for obtaining accurate measurements. In addition to tracing the whole etching process, we also turned the ICP off at different points in order to examine the surface morphology at each position using an AFM (NS3a controller with D3100). A cantilever frequency of 300 kHz was used. To ensure that the form was identical for every sample, the tip, scan size, scan conditions and parameters of the measurement were the same. 256 × 256 pixel images were acquired at a scanning rate of 3 Hz.

3. Results and discussion

The ellipsometric parameter Δ measurements were displayed in real-time. Fig. 1 shows the temporal trajectory Δ for a 30 nm

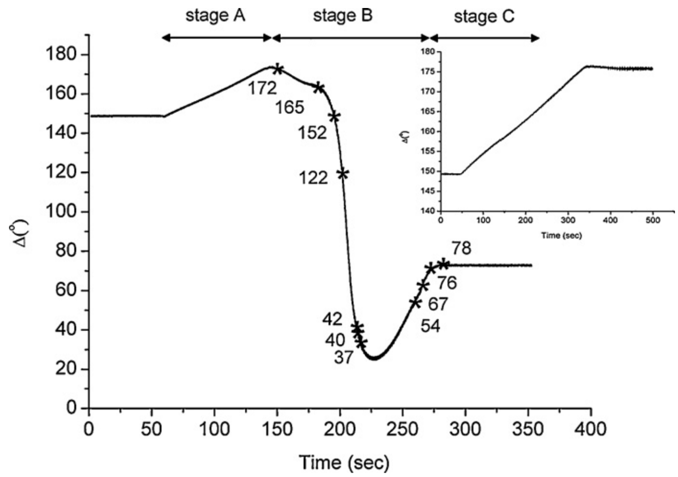

Fig. 1. Trajectory of the real-time ellipsometric parameter Δ measured by 532 nm laser diode during the etching of silicon oxide; sub-figure: same conditions but with low concentration of Cl_2 plasma during etching.

silicon oxide film on a Si substrate during the etching process. The transition of Δ near the endpoint of etching can be divided into the following three stages:

3.1. Blanket films

Initially, Δ increased steadily from 150° until reaching the highest point, 175°. The refractive indices of the silicon substrate and silicon oxide layer at 532 nm are 4.195–*i*0.0139 and 1.54, respectively (incident angle is 60°). One could calculate the Δ using the thin film model to obtain 148.7°. It can be seen in Fig. 1 that Δ started at 148°, which agrees with the theoretical value. When plasma ignition was on, Δ started to increase, indicating the removal of the oxide film from the silicon. As Δ approached 180°, the silicon oxide was completely etched off. The measured turning point was at 177.5°. The interface transition will be discussed in Section 3.2. From the Δ/time trajectory, we can conclude that the etching rate of SiO_2 is 0.375 nm/s.

3.2. Interface layer

After the etching reached the interface layer, there was a rapid decrease in Δ which started at a value of 175°, dropped to 35°, then

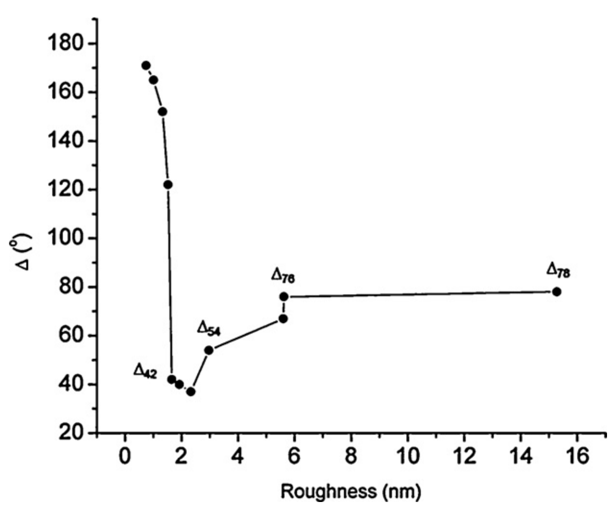

Fig. 2. Ellipsometric parameter Δ vs. RMS roughness as indicated by the height of the etched interface layer measured by AFM.

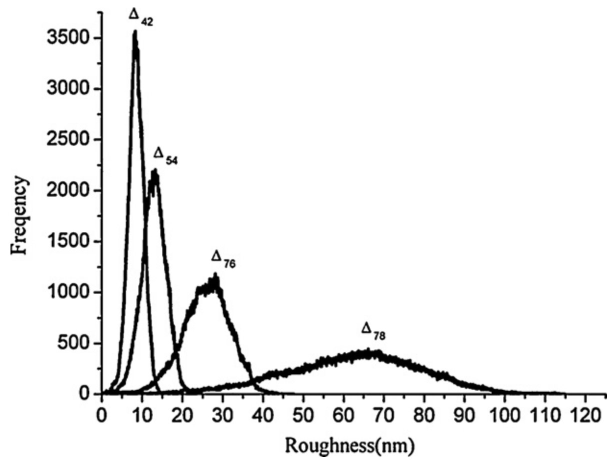

Fig. 3. Histograms of the surface height relative to the AFM topographies measured when Δ was at 42°, 54°, 76° and 78°.

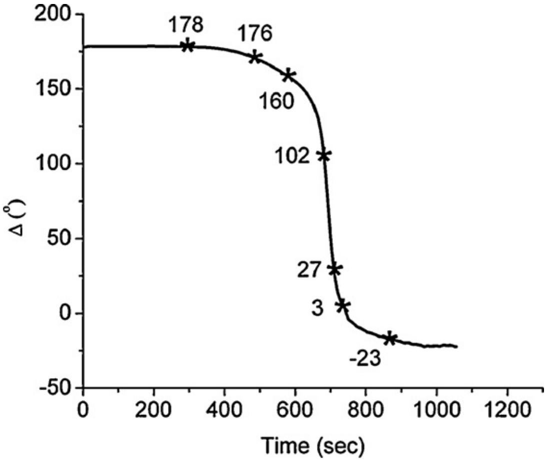

Fig. 5. Real-time ellipsometric traces at 532 nm during the etching of the silicon substrate.

rebounded back to a plateau (Δ = 76°). This phenomenon did not occur when the concentration of Cl_2 was low (see the sub-figure in Fig. 1). According to previous studies, the composition of this interface layer is 88% Si and 12% SiO_2 [4]. It is known that the etching rate during plasma etching of silicon oxide is far different from that of silicon during, especially with Cl_2. The roughness of the surface increased greatly with high concentrations of Cl_2 during the etching process. To observe the roughness, we etched several wafers under the same operating conditions. The initial conditions were the same but the ICP was turned off at different times at selected values of Δ_i (where i is the delta value in degrees) for further AFM *ex situ* analysis of surface textures. The relation of Δ_i to surface roughness is explored by AFM, as illustrated in Fig. 2. From this Δ/root-mean-square (RMS) roughness distribution diagram, one can separate the relation into two phases: (1) when the RMS roughness heights are less than 2 nm, Δ decreases linearly with respect to roughness height; and (2) when RMS roughness heights are between 2 and 6 nm, the relation is still close to linear, but Δ increases with respect to RMS roughness height. In the end, the Δ value is saturated up to 76° after the roughness exceeds 6 nm. We further analyzed the morphology near the turning points, such as the places where Δ switched from decreasing to increasing and the flattened point. We discuss four selected histograms of roughness height from the AFM topographies. The histogram of roughness heights gives us a diagram of the peak-to-valley height from the surface. Fig. 3 shows the height histograms at four points: when the lowest value of Δ reached is 42°, the roughness is narrow-banded at around 8.06 nm with 5 nm FWHM, and the surface topography looked like grass (Fig. 4(a)); then both of its roughness and Δ start to increase, but its highest frequency decreases and its band of roughness becomes broad. As can be seen in Fig. 4(b), the surface morphology transforms from grasslike (Δ = 42°) to pillarlike structures (Δ = 54°) and the value of Δ changes from decreasing to increasing. As Δ reaches the plateau (Δ = 76° and 78°), there is almost no variation in Δ, but its frequency of roughness is greatly reduced and its FWHM becomes very broad. These morphological changes can be observed in the AFM images shown in Fig. 4(c) and (d). In this interface region, the value of Δ cannot directly characterize the surface roughness, except for the linear relation when the roughness is less than 2 nm. However, one can also tell when it reaches the lowest value and starts to increase. This indicates that the morphology of the etched surface is gradually smoothed. The SiO_2/Si interface layer is often considered to be a uniform mixture of SiO_2, SiO_x and c-Si [9]. The

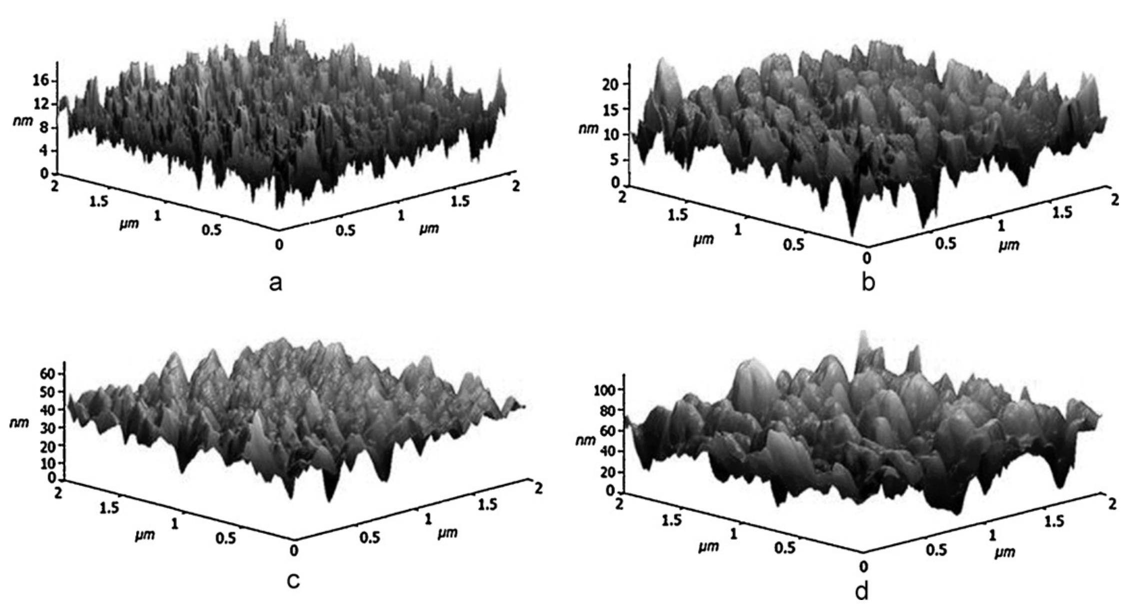

Fig. 4. Three-dimensional rendering of the AFM topography of the etched surface when Δ was at: (a) 42°, (b) 54°, (c) 76° and (d) 78°.

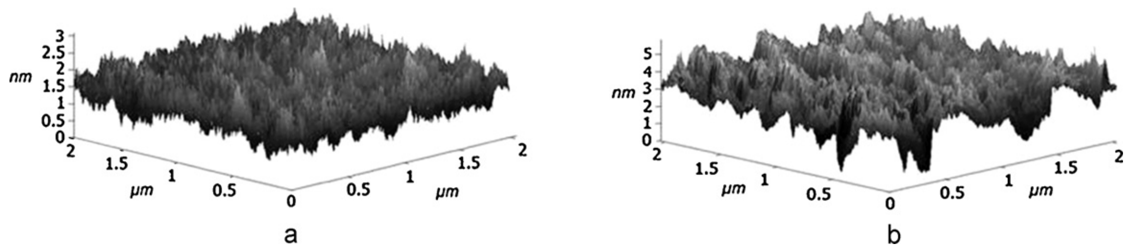

Fig. 6. AFM images of the etched bare silicon surface when Δ was at: (a) 178° and (b) 160°.

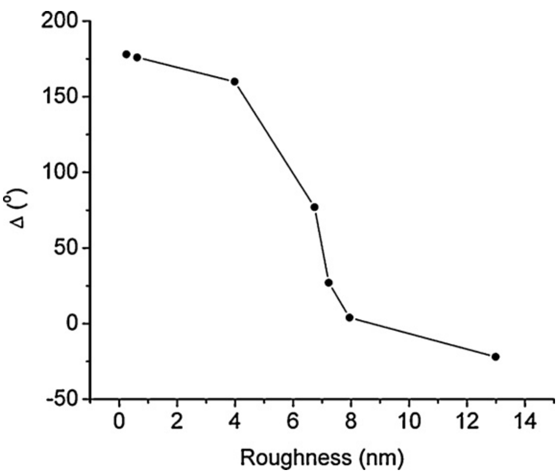

Fig. 7. Ellipsometric parameter Δ vs. RMS roughness height of etched silicon substrate measured by AFM.

difference in the plasma etching rate between silicon and silicon oxide (with chlorine content in the plasma) may be the main contributor to surface roughness [10]. The results obtained in this work agree with those obtained in previous studies [11,12] and confirm the relationship between the roughness as measured by AFM and the Δ as measured by ellipsometry at the interface.

3.3. Etching of silicon substrate

The etch rate drops upon reaching the substrate as shown in Fig. 1, when Δ reached 78°. The Δ value remained almost constant for a long time even when etching was still in process. To understand this phenomenon we etched a fresh bare silicon wafer. The etching rate of the bare silicon (measured by PEM ellipsometry) was very small, as shown in Fig. 5. During the etching process, the value of Δ remained constant for 6 min, then slowly decreased for another 6 min. After this, the value dropped sharply to zero. From AFM images, we also examined the surface texture with respect to the value of Δ for various processing intervals. We traced out the whole process and then picked up two positions for AFM images. By comparing AFM images taken in the first 6 min with that taken at the end of 12 min (see Fig. 6(a) and (b)), we can see that the surface was still smooth at the beginning then became rough. The association between surface roughness measured by AFM and the real-time ellipsometric parameter during the etching process is also shown in Fig. 7.

4. Conclusions

From real-time *in situ* ellipsometric measurements, we study the interface layer using the ellipsometric parameter Δ. A comparison of the roughness at selected points shows enhanced variation in Δ with a high percentage of Cl_2, which has never been studied using *in situ* ellipsometric measurement. We investigate the differences in etching rate for compound materials with different compositions. One can employ this real-time *in situ* ellipsometric technique to study the roughness effect during the etching process quantitatively, under different etching conditions, such as gas pressure and mixture.

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