Real-time *in situ* epitaxial film thickness monitoring and control using an emission Fourier transform infrared spectrometer

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An emission Fourier transform infrared (E/FT-IR) technique for epifilm thickness measurement will be reported. The E/FT-IR technique takes advantage of the heated wafer as the source of IR radiation. It is a noncontact, nondestructive, real-time, and *in situ* epifilm thickness monitoring tool that we demonstrated to be useful for observing real-time growth rates and incubation times. In addition, this method is especially applicable to precise end point control when critical film thicknesses are required. Moreover, we have identified some limitations to this method, and found the operable temperature ranges. Furthermore, we have demonstrated, for the first time, a closed-loop feedback control of epitaxial silicon film thickness in a multichamber single-wafer chemical vapor deposition reactor.

I. INTRODUCTION

The epitaxial film thickness is a critical parameter that must be accurately measured and controlled. For example, it is necessary to ensure that outdiffusion and autodoping during device processing do not consume the entire epitaxial film. In addition, several bipolar transistor device parameters, such as breakdown voltage, junction capacitance, transistor gain, and high frequency performance, depend on the epilayer thickness. There are many thin film thickness measurement techniques.[1,2] However, they are either destructive or not applicable in the case of a silicon epifilm on a silicon substrate. The commonly used technique for epifilm thickness measurement is the reflectance measurement using an external IR source, and is widely used in industry.[3–5] But it is only used for *ex situ* measurement, that is, the measurement is done outside the growth or etching chambers. And it is difficult to be adapted for *in situ* measurement in its present form. The E/FT-IR technique, on the other hand, takes advantage of a heated wafer as the infrared source. By analyzing the IR emission, we have successfully obtained epitaxial silicon film thickness measurements on heavily doped silicon substrates.[6]

II. EXPERIMENTS

A Bio-Rad FT-IR (model FTS-40) spectrometer equipped with a room temperature deuterated triglycine sulfate (DTGS) detector was used to collect the E/FT-IR spectra (Fig. 1). To test the feasibility of using E/FT-IR for film thickness measurement, experiments were conducted using several different setups. It was initially tested using a furnace[6]; subsequently, it was tested *in situ* in a multichamber single wafer CVD reactor.[7] A description of the experimental setups is omitted here since they were reported elsewhere.[6,7] Although each of those E/FT-IR experiments

were conducted for different purposes, the principle of operation is still the same. The procedure of the FT-IR spectroscopy is straightforward: we start with a light source $I(k)$, passing through the interferometer, and the output signal $i(x)$, as detected by the detector, is the inverse Fourier transform of the source $I(k)$.

III. RESULTS AND DISCUSSION

A. Epifilm thickness measurements by E/FT-IR

A lightly doped film with refractive index n_1 and a thickness d on a heavily doped substrate with refractive index n_2 is selected for the thickness measurement. For simplicity, we will neglect the multiple reflections in the film. In the emission mode the wafer is kept at an elevated temperature (T),

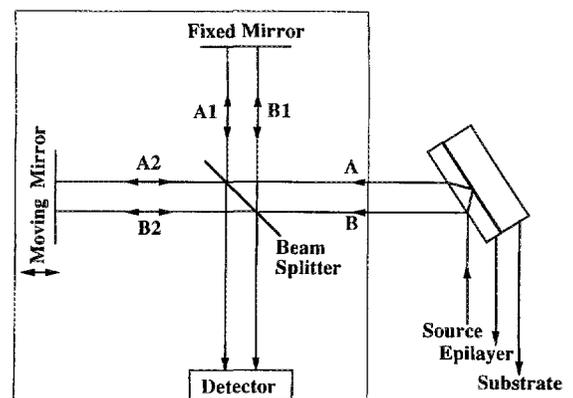**The Scanning Michelson Interferometer**

FIG. 1. Schematic of a scanning Michelson interferometer used to perform both the reflection and emission FT-IR studies.
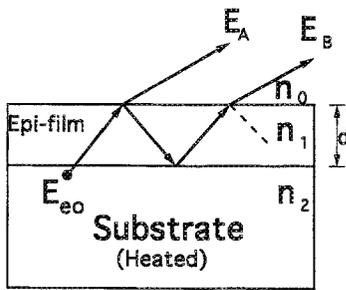
FIG. 2. Infrared emission from a substrate and an epilayer for film thickness measurements.

IR radiation is emitted by the heavily doped substrate (E_{eo}), and it travels through the film and arrives at the surface. At the film–ambient interface, part of the beam is emitted as beam E_A, while the remainder is reflected towards the substrate. The reflected beam traverses the film and is once again reflected by the film–substrate interface, then emerges from the film as beam E_B. A ray tracing diagram for the emission mode is shown in Fig. 2. Using the Fresnel reflection and transmission coefficients, light entering the Michelson interferometer (E_e) can be decomposed as

$$E_e(k) = T_{2,1}T_{1,0}e^{-0.5\alpha\Delta - j0.5k\Delta}E_{eo}$$
$$+ T_{2,1}R_{1,0}R_{1,2}T_{1,0}e^{-1.5\alpha\Delta - j1.5k\Delta}E_{eo}, \qquad (1)$$

where α is the absorption coefficient; $\Delta = 2dn_1 \cos \Theta$; Θ is the IR emission angle at the film–substrate interface; $R_{l,m}$ is the Fresnel reflection coefficient for normal incidence from substance l (with refractive n_l) to substance m (with refrac-

tive n_m); and $T_{l,m}$ is the corresponding Fresnel coefficient for transmission. We can calculate the IR emission intensity $I_e(k)$ using Eq. (1), which yields

$$I_e(k) = T_{2,1}^2 T_{1,0}^2 e^{-\alpha\Delta}I_{eo} + T_{2,1}^2 R_{1,0}^2 R_{1,2}^2 T_{1,0}^2 e^{-3\alpha\Delta}I_{eo}$$
$$- T_{2,1}^2 R_{1,0}R_{1,2}T_{0,1}^2 e^{-2\alpha\Delta}I_{eo} \cos k\Delta. \qquad (2)$$

The interferogram (IG) measured by the FT-IR spectrometer is the inverse Fourier transform of $I_e(k)$. A typical measured interferogram is shown in Fig. 3. By measuring the distance between the two side bursts, and assuming a constant refractive index for Si (e.g., $n=3.42$), the film thickness can be calculated from

$$d = \frac{\Delta}{2n \cos \Theta}. \qquad (3)$$

Therefore, in order to obtain the film thickness, the most important factor is to be able to identify the side bursts clearly. The side bursts result from the inverse Fourier transform of the last term in Eq. (2). Its magnitude determines the intensity of the side burst, and the phase determines the position of the side burst. In order to be able to identify the side bursts, the magnitude must be greater than the background noise. This means that $R_{1,2}$ must not be zero, hence the refractive index of the film and substrate must be different; $\alpha\Delta$ must be small, hence the absorption in the film must be small; lastly, the source intensity must be high enough to overcome noise. As temperature increases the first two conditions may not be satisfied, and the FT-IR thickness measurement fails; this determines the upper temperature limit. The third condition defines the lower temperature limit; we found the lower temperature limit to be about 50–100 °C for all of our samples. We observed that the upper temperature
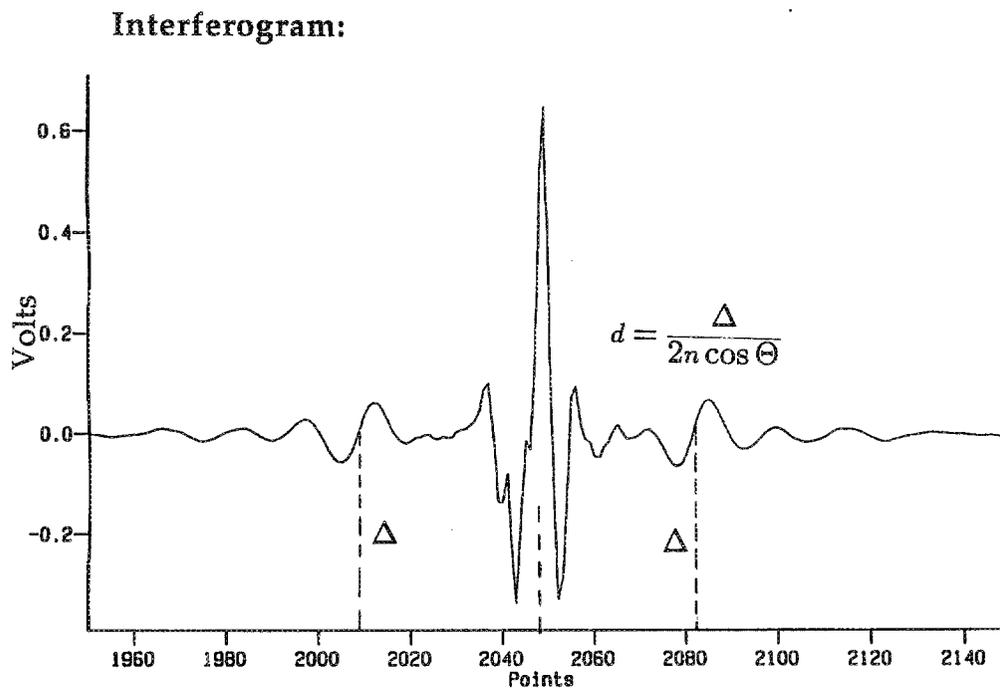
Interferogram:

FIG. 3. A typical measured FT-IR interferogram of a lightly doped epilayer on a heavily doped substrate.

limit depends on the substrate doping concentration. The intrinsic carrier concentration in the epifilm increases as the wafer temperature increases, and the upper temperature limit is reached when the carrier concentration in the film is about an order of magnitude below the extrinsic dopant concentration of the substrate. For a substrate with 6×10^{19} cm^{-3} dopants, the upper temperature limit is about 850 °C and the intrinsic carrier concentration in the undoped epifilm is about 4×10^{18} cm^{-3} at this temperature.

B. Real-time, *in situ* epifilm thickness monitoring and control using E/FT-IR

Although FT-IR spectroscopy using an external IR source is commonly and widely used in the semiconductor industry,[8] it is only used for *ex situ* measurements (i.e., the measurement is performed outside the process chambers) and it is difficult to adapt for *in situ* measurements. Compared to infrared reflection absorption spectroscopy, the infrared emission spectroscopy does not need an external light source; therefore it is easier to implement, and it is relatively less sensitive to surface roughness and flatness. Moreover, it is robust against vibration, rotation, and misalignment of the wafer. Thus, it is well suited for *in situ* monitoring of rotating wafers in production equipment. Consequently, emission FT-IR spectroscopy has significant potential as a real-time *in situ* process monitor.

The real-time film thickness measurement was demonstrated by depositing a film on a substrate with an existing epifilm. By monitoring the shift in the side bursts as a function of time, the relative change in film thickness can be found. The E/FT-IR technique has been applied to measure film thickness *in situ* and in real-time during the deposition processes. A film was deposited on a 3 μm epiwafer (substrate is n-type doped with resistivity equal to 0.001–0.002 Ω cm; the existing epilayer is also n-type with resistivity equal to about 0.5 Ω cm). The growing film thickness is the total measured film thickness minus the initial epifilm thickness. Figure 4 shows the real-time film thickness measurements during a thermal chemical vapor deposition (CVD) process at a temperature of 650 °C. The wafer is cleaned simply by dipping it in HF (10:1) solution without being rinsed before it was introduced into the CVD reactor. Deposition was carried out at a pressure of 2 mTorr by introducing 40 standard cubic centimeter per minute at (sccm) of pure silane. We have observed the transient behavior of this process. The slope of the curve gives us the growth rates, and we see that the initial growth rate is slower. Although the reasons for this change in growth rate is unknown at present, we believe it may be influenced by the initial substrate surface condition. In addition, the real-time *in situ* monitor can be used for end point detection and control. For example, if a 0.27 μm epilayer is desired, the deposition process can be terminated when the E/FT-IR gives a 0.27 μm epifilm thickness reading. For an even more precise end point control, the real-time film thickness measurements were used to extrapolate a real-time growth rate; therefore process end point was predicted in real time. The final thickness measurement was confirmed by *ex situ* FT-IR and high resolution cross-sectional transmission electron microscopy (HRXTEM)

which gave measured film thicknesses of 0.26 and 0.3 μm, respectively. This technique is particularly suited for real-time process monitoring, as it allows repeatable, fast, and accurate (0.01 μm precision) measurement of film thickness. Thickness information was fed back in real time to control film thickness precisely.

The closed-loop control was implemented on the Labview (National Instruments) platform using a Macintosh computer (Quardra 900 model with a Motorola 68040 microprocessor). Real-time thickness data were extracted from the Bio-Rad Digilab's computer (SPC-3200 workstation) through a standard RS-232 communication port. Two control algorithms were used: (1) first past the post (FPP) method; (2) linear forecasting. Mathematical models and other control algorithms, such as time series analysis and neural networks, were evaluated and described in detail in Ref. 9. The FPP algorithm was the most obvious approach for controlling the film thickness. The FPP algorithm monitors the thickness continuously. Once a measurement was on or exceeded the target, the process was terminated automatically. The termination step was carried out using a digital input/output (I/O) board (Lab-NB board from National Instruments) to turn off all process gases, mass flow controllers, and temperature controller. The E/FT-IR with 0.01 μm precision resulted in a control limit of 0.005 μm using the FPP method. The linear forecasting algorithm assumes that the growth rate of silicon follows a linear path. The growth rate was determined and continuously updated by taking the real-time measured film thickness data and performing a linear regression on them. The process end time was estimated and updated from the growth rate in real-time. When the growth rate was truly linear, the method reduced the effect of E/FT-IR measurement noise and improved the control limit to 0.003 μm. Currently, we are interfacing an analog I/O board to the mass flow controllers and temperature controller. When this is done, we will be able to perform real-time dynamic control
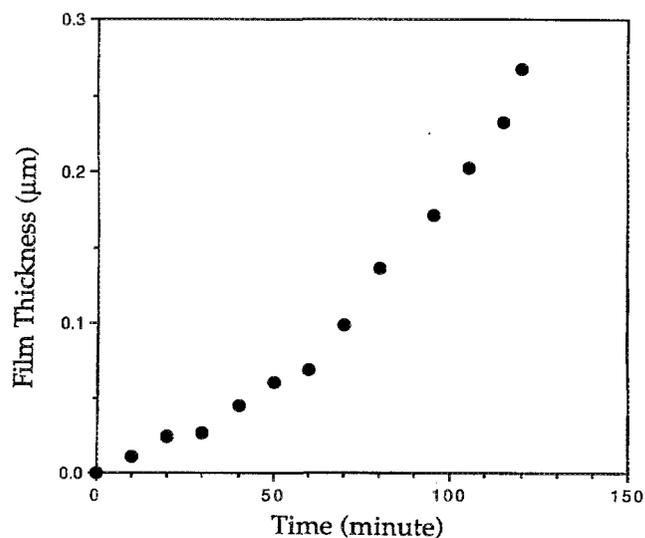

FIG. 4. Real-time *in situ* film thickness monitoring during a CVD process at 650 °C.
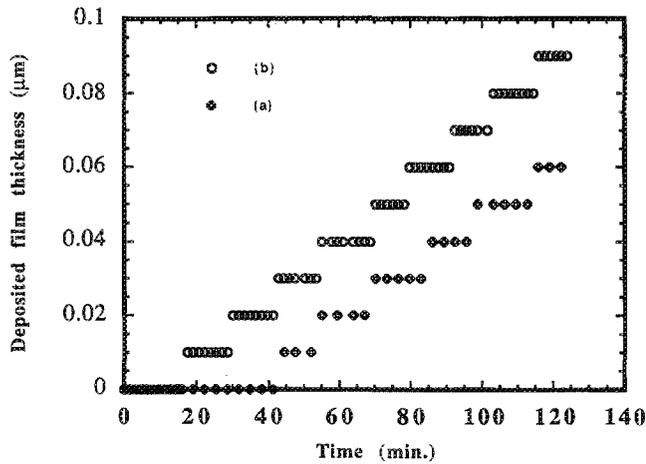
FIG. 5. Real-time *in situ* monitoring of growth incubation time and growth rate during a CVD process.

FIG. 7. Real-time *in situ* film thickness monitoring during a CVD process after hydrogen plasma predeposition cleaning.

over process temperature, gas flow rate, and gas composition.

In addition to real-time *in situ* monitoring and control of epifilm thickness, we have observed growth rate change and incubation time resulting from various predeposition wafer cleaning conditions. Figures 5 and 7 showed the real-time thickness monitoring results of three depositions that were performed under the same deposition conditions (as indicated in Fig. 7). Notice that multiple data points (shown in Figs. 5 and 7) clearly demonstrated the repeatability of the
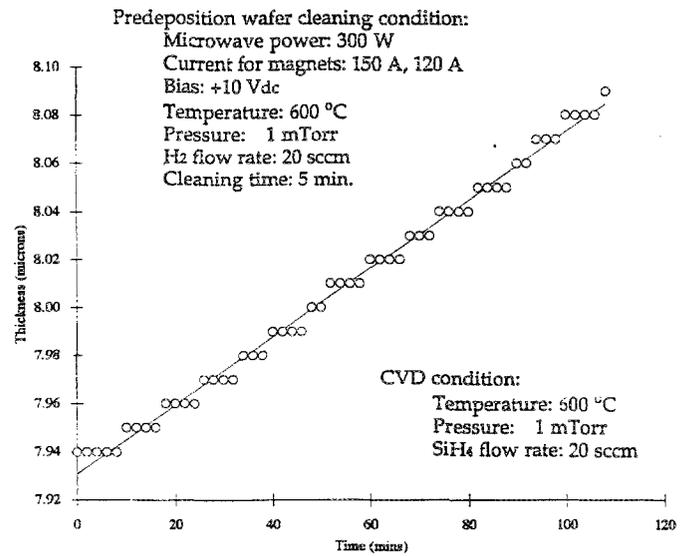
real-time E/FT-IR measurements. Figure 5(a) showed an incubation time over 30 min resulting from a deposition on a native oxide covered silicon surface. Subsequent cross-sectional transmission electron microscopy (XTEM) analysis [Fig. 6(a)] confirmed that the deposited film was polycrystalline silicon. Figure 5(b) did not show an incubation time; however, the growth rate was slower compared to that in Fig. 7. The corresponding XTEM picture [Fig. 6(b)] showed that the film grown with slower growth rate [Fig. 5(b)] was a defective epifilm. Figure 7 showed a defect free epifilm that was deposited after ECR hydrogen plasma cleaning. The deposition condition and the optimized plasma cleaning condition were specified in Fig. 7. The corresponding XTEM picture (Fig. 8) shows that this film was defect free and the epifilm–substrate interface was almost invisible. By real-time monitoring of incubation times and deposition rates, we were able to obtain qualitative information about the effectiveness of the predeposition wafer cleaning process and to assess the quality of the depositing film in real time.
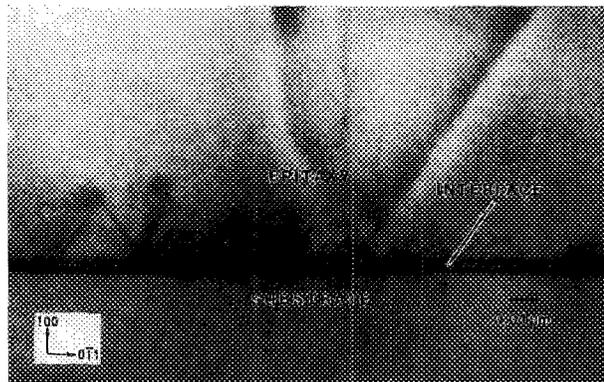
FIG. 6. Cross-sectional TEM picture of (a) a polysilicon film and (b) a defective epifilm.
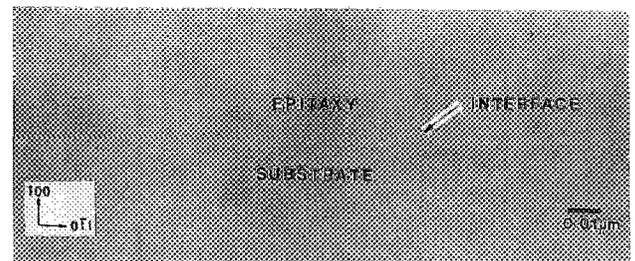

FIG. 8. Cross-sectional TEM picture of a defect free epifilm.

C. Conclusion

We have reported an emission FT-IR technique for epifilm thickness measurement. We have demonstrated that the E/FT-IR technique can be used as a noncontact, nondestructive *in situ*, real-time film thickness monitoring tool. Moreover, we have identified some limitations to this method, and found the operable temperature ranges. We attribute the lower temperature limit to low emission intensity, and upper temperature limit to the change in refractive index and increase in absorption due to increasing intrinsic carrier concentration in the epilayer. Finally, an application of E/FT-IR for real-time *in situ* monitoring of epitaxial silicon film thickness is demonstrated. From the real-time thickness measurements, growth rates can be obtained in real time and the end point epifilm thickness can be controlled precisely.

ACKNOWLEDGMENTS

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