In Situ Monitoring of Crystallinity and Temperature during Rapid Thermal Crystallization of Silicon on Glass

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ABSTRACT

A novel technique is presented for simultaneously measuring temperature and crystallinity in situ during the rapid thermal annealing of thin Si films on transparent substrates for active matrix liquid crystal display applications. This technique makes use of acoustic waves to monitor temperature by measuring changes in Lamb wave velocity with temperature. Since this technique is independent of emissivity, it enables accurate tracking of crystalline phase transitions along with temperature, based on changes in the optical absorption properties of the film. This provides a methodology for closed-loop control and end-point detection. The experiments on thin amorphous Si on fused silica demonstrate temperature repeatability of 2%. Also, the technique proved sensitive enough to detect the onset of nucleation, as evidenced by transmission electron microscopy.

Introduction

Thin film transistors (TFTs) find extensive applications in active matrix liquid crystal displays (AMLCDs). There is substantial interest in the development of a glass-compatible polycrystalline TFT technology. The ability to fabricate low temperature polysilicon TFTs will enable the incorporation of driver circuitry onto glass display substrates, improving performance at a reduced cost. Low-cost glasses have strain points of approximately 600°C. Therefore, to prevent glass warpage, it is necessary to minimize thermal budget and peak process temperatures. Rapid thermal annealing (RTA) to crystallize amorphous films to form the channel is a promising technique for the processing of polycrystalline TFTs. With RTA, it is possible to produce high-performance TFTs without sacrificing process throughput. There has been progress in recent years in the development of a glass-compatible RTA-based TFT technology.7 In situ end-point detection during RTA will allow the minimization of the net thermal budget. The ability to measure temperature accurately is critical to ensuring glass compatibility and enabling process control and modeling.

Temperature measurement is an important problem in RTA. Pyrometry is the most widely used technique for this purpose.3 The measurement principle of pyrometers is the temperature dependent radiation from the sample wafers. Since the radiation is strongly dependent on the emissivity of the radiating body, temperature measurement is affected by the process chamber and heating lamp geometry, the surface conditions, and the doping level of the wafer.4 Assuming these properties are known a priori and their variation is small, pyrometers can be calibrated for temperature measurement with some real-time correction algorithms.5 The properties of thin films present on the wafer surfaces are also shown to have a significant effect on emissivity.6 The material, thickness, and crystallization phase of these films all affect emissivity. During the RTA of thin films on transparent substrates, crystalline phase transitions take place, resulting in significant emissivity variations. Consequently, a method that is particularly insensitive to emissivity is required for accurate in situ temperature measurement in these kinds of processes.

An acoustic temperature sensor relying on the temperature dependence of elastic constants has also been used for in situ temperature monitoring in semiconductor processing.7 The sensor can be used in the whole 20 to 1000°C range whereas pyrometers require high temperatures (>400°C) for acceptable accuracy. The acoustic technique is virtually independent of optical parameters of the sample, enabling isolation of emissivity variations from the temperature measurements. Since emissivity variations due to phase transitions determine the heating profile, the acoustic sensor provides crystallinity information through accurate temperature measurements. The heating of the substrate is a strong function of surface film absorption coefficients, which change with crystallinity. Therefore, the ability to isolate surface conditions from temperature measurement enables the tracking of crystalline phase changes.

In this paper, the methodology for using the acoustic temperature sensor as a thin film crystallinity sensor is presented. A description of the experimental RTA system with an integrated acoustic sensor is provided and results obtained with Si films on fused silica substrates are presented.

Measurement Principles

Acoustic temperature measurement.—The elastic constants of common semiconductor processing materials have linear temperature sensitivity in the order of 10⁻⁴/°C, which is large enough to make accurate measurements.8 The acoustic temperature sensor uses A, mode Lamb waves in the 200 kHz to 1.5 MHz range to monitor the changes in elastic constants with temperature. To couple the Lamb waves to the substrate, the existing quartz support pins of an RTA system are modified by bonding a PZT-5H transducer at one end and shaping the other end in the form of a tip in order to have a reliable contact with the substrate as in Fig 1. The acoustic waves are generated by the PZT-5H transducer by applying an electrical pulse at its terminals. These waves are guided in the quartz rod. Part of the acoustic energy is coupled to the A, mode in the substrate at the rod-substrate contact and the rest is reflected to form an echo signal at the transducer terminals. The Lamb waves propagating in the substrate are detected at another contact point and converted to an electrical signal at the receiver. The time-of-flight (TOF) for the Lamb wave propagation is determined by measuring the time difference between particular zero crossings in the echo and the received signals. This enables isolation of velocity changes.

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![Fig. 1. Cross section of acoustic sensor system.](image-url)
in the quartz pins, determined from the echo signal, from velocity changes in the substrate. The Lamb wave velocity, and therefore the TOF, is determined by the elastic constants. By monitoring TOF, the temperature of the substrate can be monitored. Using a theoretical model to link TOF to the elastic constants and temperature coefficients, the average temperature along the measurement path can be estimated in situ with ±1°C accuracy.

Since the temperature measurement depends only on the variation in elastic properties, the measurement is independent of optical parameters including emissivity. Also, the acoustic energy is fully confined in the sample plate eliminating the interference from external sources. At a frequency of 250 kHz, the A0 mode has a wavelength of approximately 5 mm in a 0.5 mm thick fused silica substrate plate. This results in a uniform acoustic power flow along the thickness of the substrate. Since the deposited films are very thin (100 to 150 nm) compared to the substrate, the measured temperature is dominated by the bulk substrate temperature and the effect of variations in film thickness is negligible.

Crystallinity tracking.—The isolation of emissivity variations from temperature measurement provides a unique means of tracking crystallinity in situ. The optical absorption coefficient of thin amorphous Si films is higher than the absorption of an equivalent polycrystalline film. Hence, thin amorphous Si films are significantly more opaque than polycrystalline films of the same thickness. As a result of this, the absorption of light from the heating lamp by the film drops substantially during the amorphous-to-crystalline phase transition. This is shown in Fig. 2, which illustrates the thermodynamic cross section of the system, along with heating characteristics for bare and film-coated fused silica substrates. Clearly, the heating of the substrate occurs primarily through conduction from the surface film. This is shown in Fig. 2, which illustrates the thermodynamically cross section of the system, along with heating characteristics for bare and film-coated fused silica substrates. The acoustic sensor is isolated from the RTA chamber by a water-cooled stainless steel base plate. The ends of the quartz rods with the bonded piezoelectric transducer are isolated by O-rings to prevent contamination in the chamber. The cooling is necessary to protect the transducer from the high temperatures which cause degradation of the transducer material. The 3 mm diam acoustic sensor operates around 250 kHz with 40% fractional bandwidth. Three sensing pins are placed asymmetrically around the edge of the wafer to enable multipath measurements for tomography. The distance between the sensor locations used in this experiment was 9.2 cm, resulting in an approximately 40 μs travel time for the A0 mode Lamb waves. Unlike other solid materials, the velocity of Lamb waves increase with increasing temperature in fused silica, clearly visible in Fig. 4. The relation between the TOF and the temperature is linear in the range from room temperature to more than 700°C.

In order to infer temperature from the TOF data accurately in real time, the scaling factors should be calculated. For this purpose, the temperature coefficients for the substrates used in the experiments were obtained experi-

**Experimental Methodology**

The samples used for this experiment were prepared on fused silica substrates. 100 nm low temperature oxide (LTO) SiO2 was deposited by LPCVD on the substrates.

![Fig. 2. Thermodynamic properties of system, showing film-dependent absorption.](image1)

![Fig. 3. Cross section of Stanford Rapid Thermal Multiprocessor.](image2)
An acoustic sensor operating around 500 kHz was placed in an oven, where isothermal conditions can be obtained, and the temperature of a fused silica substrate was varied in the 20 to 75°C range while recording the TOF. Then, using a theoretical model, the coefficients were inverted from this TOF data. Calculated coefficients have an estimated 4% error due to temperature dependent behavior of the transducers and uncertainties in the oven temperature and distance measurements. With the parameters of the acoustic sensor in the RTA chamber, and using the measured elastic temperature coefficients, the TOF to temperature conversion factor was calculated to be $-3.11 \, \text{ns/°C}$.

The correlation between TOF and crystallinity was studied using TEM. The films were etched off the underlying substrate using HF and floated onto a Cu grid. These films were analyzed at different magnifications to determine relative crystallinity. Diffraction patterns were also obtained for comparison by using the TEM in selective area diffraction (SAD) mode.

**Results and Discussion**

The temperature vs. time plot for the recipe used is shown in Fig. 5. The peak temperature of the fused silica wafers is measured to be 588°C with a standard deviation of 12°C, determined over several runs, providing a repeatability of approximately 2%. This variation is due to variations in ambient chamber temperature, as well as sensor resolution limits and noise. Note that this temperature is averaged over the entire acoustic path length. Since the RTP lamp was designed for silicon substrates and not transparent substrates, uniformity is suboptimal and local temperatures vary substantially, as determined by tomography and visual inspection. Clearly, the temperature drops during the amorphous-polycrystalline transition. Films which were initially polycrystalline, on the other hand, do not exhibit this drop in temperature, and also do not heat up as much initially, verifying the higher absorbance of the amorphous films. Samples were withdrawn at points A, B, C, and D during the anneal cycle and studied using transmission electron microscopy (TEM).
TEM micrographs and diffraction patterns for samples annealed to the points indicated are shown in Fig. 6-9. The sample annealed to point "A" along the temperature-time curve is still completely amorphous, as apparent from Fig. 6. This is in agreement with substantial work done on crystallization which suggests that crystallization begins only after a specific incubation time.

The high sensitivity of the technique is apparent upon examination of the micrograph for samples annealed to point "B", shown in Fig. 7. A few small nuclei are present within the sample. The diffraction rings, which exhibit no significant tightening to the naked eye, do not reveal this. However, the temperature curve shows a drop almost immediately afterwards. Further into the anneal, at point "C," samples have large grains surrounded by patches of amorphous material, indicative of incomplete crystallization, shown in Fig. 8. Upon restabilization of the temperature, at point "D," the samples are fully crystallized and
have no large amorphous regions visible. Additional annealing does not increase grain size any further. The diffraction rings are well defined. This is shown in Fig. 9.

To further analyze the progress of crystallization, the intensification of the lines in the diffraction rings was studied using a normalized quantization image enhancement technique. This illustrates the crystallization progress clearly. This is shown in Fig. 10. The tightening of lines is clearly visible. The correspondence to temperature data is excellent, suggesting that tracking is reliable. Repeatability of the experiments was also verified using multiple anneals. Deviations were found to be minimal.

Our results indicate that the acoustic sensor is an excellent means of monitoring temperature and crystallinity in situ. The system has high sensitivity to phase changes, and tracks temperature, independent of surface film conditions. The sensitivity of the sensor to crystallinity can be used to provide input for a closed loop control system for multistep anneals, using a system similar to the one shown in Fig. 4. For example, the technique could be extended to provide an initial high temperature step to reduce the incubation time, followed by a low temperature quench upon nucleation, to suppress further nucleation and potentially result in the growth of extremely large grains. The temperature sensors have been extended to provide full-wafer tomography of temperature and crystallinity by using multiple sensors over different paths across the wafer. This could also be used for closed loop control to reduce nonuniformities across large area display substrates. Since the sensor measures substrate temperature independent of film temperature, it could be used to develop techniques to minimize warpage while maximizing film heating.

**Conclusions**

A novel technique for simultaneously monitoring temperature and crystallinity during the rapid thermal annealing of thin Si films on quartz/glass substrates has been presented. Acoustic sensors are used to measure the variation of sound velocity with temperature, enabling the extraction of the variation in elastic coefficients with temperature. From measured constants, it is possible to extract the temperature of the substrates used. This technique for measuring temperature is independent of surface emissivity. This enables tracking of film crystallinity based on variations in optical absorbance. The technique has been demonstrated to be highly repeatable and sensitive. Phase changes can be tracked from the onset of nucleation to full crystallization. This should result in optimized techniques for the production of high perfor-

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**Fig. 9. TEM and SAD for sample annealed to point "D": complete crystallization.**

**Fig. 10. Progressive tightening of diffraction rings.**
Crystal Growth in Silicon Chemical Vapor Deposition from Silane

The Role of Hydrogen

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ABSTRACT

Crystal growth mechanisms in chemical vapor deposition (CVD) of silane are studied in some detail. An atomic view reveals that the free energy change upon the formation of a small crystal on the (100) silicon surface in Si-CVD from SiH$_4$ is always negative, due to the presence of hydrogen. Therefore, nucleation and step growth are unnecessary, in contrast to crystal growth from melt. Epitaxial growth in this case takes place through iterative adsorption and desorption reactions on the periodic surface sites of the substrate. Slow desorption reactions result in vacancies and distorted bond angles which hinder epitaxial growth. Based on this model, the role of H desorption in low temperature Si epitaxy from SiH$_4$ is investigated. A thermodynamic analysis implies that H desorption is the limiting factor to low temperature epitaxy in the pressure range of 1 to 760 Torr, and the pressure dependence of minimum epitaxial temperature is derived as $T_{\text{ep}}(^\circ \text{C}) = 106 \log P_{\text{H}_2}(\text{Torr}) + 668$. Below 1 Torr, adsorption rate becomes the limiting factor to low temperature epitaxy.

Introduction

Crystal growth studies began with bulk crystal growth from melt, such as Czochralski growth. In those studies two contributions were usually considered in the energetics: surface energy and volume energy,

$$\Delta G = \Delta G_s + \Delta G_v, \quad [1]$$

where $\Delta G$ is the total free energy change upon the formation of a new crystal, $\Delta G_s$ is the volume energy change, and $\Delta G_v$ is the surface energy change. $\Delta G_s$ is always positive which leads to an energy barrier $\Delta G^*$ for small crystals and therefore only those crystals larger than a critical size $r^*$ are energetically stable (Fig. 1). This is the origin of the concepts of nucleation and critical nucleus. Subsequent growth takes place at the atomic step formed by the critical nucleus and the growing surface to ensure lower energy configurations for incoming atoms, the so-called step growth. The key point here is that each event, both microscopic and macroscopic, must proceed toward a lower energy configuration to be stable.

Studies on crystal growth in chemical vapor deposition (CVD) have adopted the above model, i.e., they assumed the same energy terms as in Eq. 1. As a natural result, an energy barrier again exists for small crystals, and critical nucleus is again necessary.

However, recent scanning tunneling microscopy (STM) observations in CVD of silane on (100) silicon surface provide no evidence of the presence of critical nucleus. The STM results are summarized here: (i) single adspecies SiH$_3$ and SiH$_2$ are observed on (100) — $\sqrt{2} \times 1$ Si and give a random distribution on the surface at room temperature, (ii) as temperature rises, the surface morphology shows a random distribution of clusters of all sizes, from single adspecies to big islands, and (iii) at even higher temperatures, clusters, from single adspecies to big islands, all disappear, and only miscut-induced terrace steps are observed.

These observations can be understood if the free energy change for small crystals is negative (Fig. 1), combined

REFERENCES