Ultrasonic cure and temperature monitoring of photoresist during pre-exposure bake process

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ABSTRACT

A system of *in situ* ultrasonic sensors has been developed that can be used to monitor the photoresist prebake process. A high frequency phase measurement monitors the resist film properties while a lower frequency time of flight measurement monitors the corresponding wafer temperature. The high frequency measurement involves calculating the phase of an ultrasound signal as it is reflected from the silicon/photoresist interface. As the photoresist film changes in thickness and viscoelastic properties, the phase of the reflected signal will change. In this way, it is possible to follow how the photoresist film changes as it bakes; the solvent evaporates from the resist, decreasing the thickness and increasing the density. Results indicate that there is a phase minimum at a repeatable temperature, believed to be the softening or glass transition temperature (T_g). The lower frequency (200kHz) time of flight measurement employs PZT-5H piezoelectric transducers bonded to a quartz buffer rod. The transducer generates a Lamb wave in the wafer which is then detected at another location by an identical transducer. The time of flight of the Lamb wave through the wafer depends linearly on temperature. Using these two sensors, we can measure the wafer temperature and the photoresist properties during prebake; providing us with the information necessary for *in situ* process control.

Keywords: Photoresist, prebake, ultrasonic sensor, temperature, Lamb wave, time of flight, process monitoring, semiconductor manufacturing.

1. INTRODUCTION

In the past, the semiconductor manufacturing field has relied on empirical methods of process control to monitor lithographic processes. With the increased expense of the microelectronic fabrication process and the need for improved precision of feature size in DUV and eventually EUV lithography, there has been an increase in the use of *in situ* process monitoring techniques to control the lithographic process. In this research, we are addressing the issue of endpoint detection of photoresist pre-exposure bake. This softbake step is required to evaporate excess solvent from the photoresist as well as to relax the polymer chains into an ordered matrix.[1] Typical thickness changes for 1-2μm films range from 0.2-0.5μm, depending on how much of a delay there is between spin-coating and the bake. If, after the bake is completed, the solvent isn't fully evaporated or if the resist is over-baked, then the feature size of the resulting device or structure may not be as small as expected. It is also important that oven or hotplate be at a high enough temperature that the resist can reach and exceed its glass transition temperature (T_g); it is at this point that solvent and polymer chain diffusion is facilitated to the extent that significant evaporation can begin to occur. Several techniques are available to measure Tg but none of them has been applied *in situ* for endpoint detection of photoresist softbake[2-5]. There has been some research in endpoint detection of the prebake process [6], but the techniques do not provide information about the changing elastic properties of the resist. It is important to determine when the resist reaches the target thickness, but also to know whether or not it has reached glass transition during the bake.

Temperature uniformity is a continuous issue in semiconductor manufacturing and photoresist bake processes in particular. In order to obtain uniform critical dimension measurements across a wafer, it is necessary to have the same processing conditions across the wafer during the lithographic steps. At the Edward L. Ginzton Laboratory, several authors have applied ultrasonic technology to monitoring semiconductor processes. Lee, et al. [7-8] measured the change in Lamb wave velocity in silicon wafers during rapid thermal processing (RTP) to monitor temperature during processing. They found a linear relationship between the time of flight (TOF) through the wafer and wafer temperature. In related work, Degertekin, et al.[9-10] determined the effect of thin films on ultrasonic temperature measurements. The combination of these TOF wafer temperature measurements and a higher frequency cure monitoring sensor will allow monitoring of the changes in the photoresist film during prebake. Development and

0277-786X/98/$10.00

integration of these sensors as applied to the prebake process will facilitate controlling the prebake process *in situ.* This would provide an increase in process uniformity and throughput in a manufacturing environment.

2. MEASUREMENT PRINCIPLE

The two main issues that need to be considered for a given process are the uniformity across the wafer and the wafer to wafer repeatability of the process. In the prebake process, control of temperature uniformity as well as control of the changes in the resist film during the bake are desired in order to obtain a repeatable and reliable critical dimension at the end of the lithography sequence. For this reason, two measurements are presented here, a high frequency phase measurement to monitor the changes in the resist film during prebake and a lower frequency Lamb wave measurement to provide wafer temperature information. The integration of these two sensing mechanisms could eventually allow for feedback control of the baking process.

PHASE MEASUREMENT OF CURE STATE

The principle for this measurement was described previously.[11] The reflection coefficient for a plane wave incident from a medium on a layer was calculated using *Equation 1* from classical reflection theory.[12] In this equation, Z_1 is the acoustic impedance of silicon, Z_2 is the acoustic impedance of photoresist, Z_3 is the acoustic impedance of air, L is the photoresist thickness, and c_2 is the velocity of sound in photoresist. The phase of the reflection coefficient was predicted as a function of resist thickness and density using this equation. *Figure 1* illustrates the effect of a change in resist thickness from 2.6μm to 2.2μm as it solidifies. The change in phase expected is plotted as a function of measurement frequency to show the frequencies of highest sensitivity for this resist thickness. Optimum choice of frequency is necessary to get the maximum sensitivity to resist property changes.

$$R = \left[\frac{(1 - \frac{Z_1}{r_3})\cos k_2 L + j\frac{Z_2}{Z_3}\sin k_2 L}{(1 + \frac{Z_1}{Z_3})\cos k_2 L + j\frac{Z_2}{Z_3}\sin k_2 L} \right] \tag{1}$$

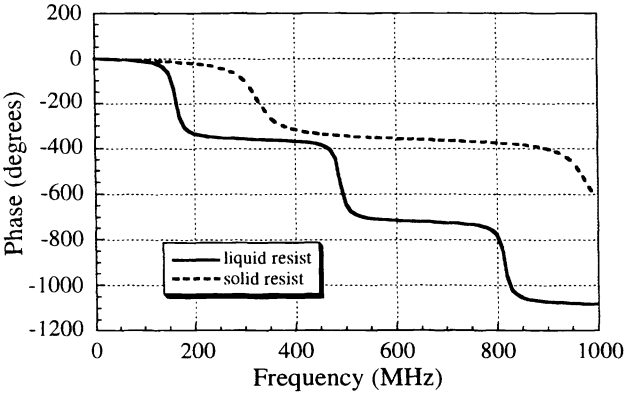

Figure 1: Reflection phase vs. frequency for liquid and solid photoresist (before and after pre-exposure bake, respectively).

There will also be a phase change in the reflected signal as the wafer changes temperature. During prebake, the wafer temperature typically increases from room temperature to 90 C. The relation from which the one-way phase change for a given temperature change can be calculated as given in *Equation 2.* It has been shown experimentally for silicon that this relationship is linear within the temperature range of the bake.[13] Here, d is the thickness of the silicon wafer, f is the frequency, v(T) is the bulk velocity in silicon at temperature T, T_0 is room temperature, 25C, and k_v is

the temperature sensitivity coefficient of the wave velocity. This relation was used to remove the effects of temperature on the measurements, with the calculated change multiplied by two to compensate for wave travel to and from the wafer surface.

$$\Delta\varphi(T) = 2\pi fd\left[\frac{1}{v(T_0)(1-k_v(T-T_0))} - \frac{1}{v(T_0)}\right] \tag{2}$$

LAMB WAVE TEMPERATURE MEASUREMENT

Another method for measuring changes in film thickness and density, as well as substrate temperature, is to measure the time required for a Lamb wave to propagate through the silicon wafer/resist structure. The time of flight (TOF) was used to quantify the time required for a Lamb wave to propagate through the wafer, from transmitter to receiver. A theory for Lamb wave propagation in a layered structure was developed previously in this laboratory.[14] Using this theory, the expected change in TOF can be calculated as the wafer heats and as the resist changes from a liquid to a solid during prebake. As with the phase measurement, the TOF is expected to change linearly as the wafer temperature increases during the bake. The effect of a thin (1-2μm) film of photoresist on this slope is negligible. *Figure 2* shows the theoretical change in TOF vs. temperature, at 200 kHz, as an 8" silicon wafer heats from room temperature at 25C to a typical prebake temperature of 90C. This change was calculated to be around 65ns for the change in temperature during prebake for a pathlength of 10 cm (plot shows normalized pathlength of 1cm). *Figure 3* illustrates the expected change in TOF vs. frequency, calculated by subtracting the TOF before prebake from the TOF after the resist has solidified; temperature effects were not included here. Again, a liquid resist thickness of 2.6μm and a solid thickness of 2.2μm were assumed. For a signal path of about 10cm, it can be seen that the change in TOF at 200kHz is about -3ns as the resist bakes. Similar results were obtained for calculations using resist thicknesses of 1μm. These changes are very small compared to the change in TOF due to wafer temperature changes; this would require the accuracy of the TOF measurement to be < 0.1ns in order to monitor the changes in resist thickness and density during cure. For this reason, the Lamb wave TOF measurement will be used only in developing a temperature sensor for prebake, while the more sensitive phase measurement will be applied to cure monitoring.

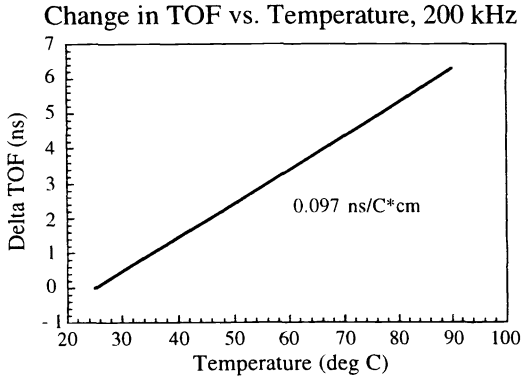

Figure 2: Delta TOF as a function of wafer temperature, calculated for a 200 kHz Lamb wave.

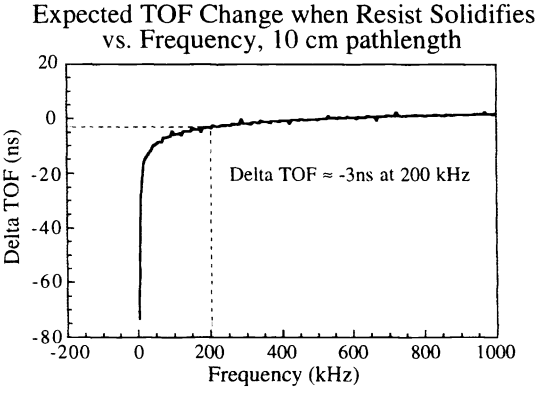

Figure 3: Delta TOF as a function of measurement frequency, calculated for a Lamb wave in photoresist changing from 2.6μm to 2.2μm (liquid to solid state).

3. EXPERIMENTAL SETUP

PHASE MEASUREMENT OF CURE STATE

In order to determine the changes in the photoresist film during the prebake process, we apply a high voltage pulse across a zinc oxide piezoelectric transducer that is resonant at 260 MHz. As shown in *Figure 4*, longitudinal waves excited by the transducer are coupled into the silicon wafer and its coatings by direct contact of the sapphire buffer rod to the wafer. These waves are reflected from the silicon/photoresist interface. The transducer serves as the receiver for the reflected signal which is continuously monitored during prebake. We digitize the signal using an HP 54520A

digital oscilloscope at 1G sample/sec. An anti-aliasing filter at 450 MHz was used. For each waveform obtained, the wafer temperature is measured using a thermocouple.

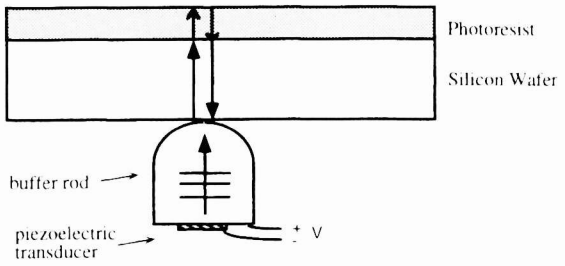

Figure 4: Illustration of sensor technology (not to scale).

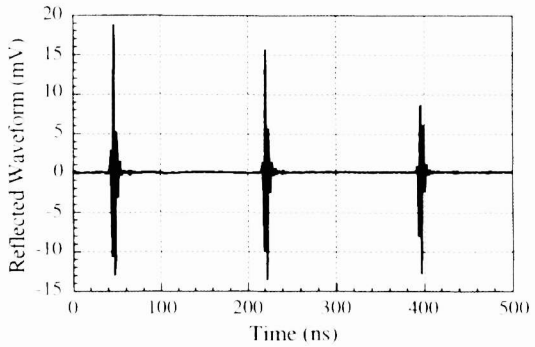

Figure 5: Sample reflected waveform used for phase calculation. The first three reflections from the silicon/photoresist interface are shown here.

A sample waveform of the first three reflection signals is shown in *Figure 5*. The waveform phase is determined by subtracting the phases of the first two reflection signals shown at 40ns and 220ns. Subtracting the phases of the two signals allows for the removal of unrelated systematic effects such as buffer rod temperature changes. The first set of waveforms obtained is used as a reference, thus the phase change is the parameter that is measured. Data is collected at a repetition rate of 3 seconds and the phase calculated using MATLAB® software.

LAMB WAVE TEMPERATURE MEASUREMENT

The method for TOF measurement was described previously by Lee, et al.[8] Briefly, Lamb waves are excited in a silicon wafer by a quartz pin piezoelectric transducer and detected by an identical transducer. The TOF is determined by the time difference between a chosen zero crossing in the echo waveform reflected from the pin/wafer interface and the received waveform that propagates through the wafer to the second transducer. A Stanford Research Systems SR620 Universal Time Interval Counter is used to measure the time difference between the two zero crossings. The setup shown in *Figure 6* was developed in order to incorporate the sensors into a hotplate design that could then be adapted to a manufacturing setting. It consists of a 2" thick aluminum plate into which the 3mm wide, 4.4cm long sensors are imbedded. The polished tips of the quartz sensors (radius of curvature about 100μm) extend above the plate by about 1mm so that the wafer can be placed on the sensors. These sensors replace the proximity heating pins of traditional bakeplates, keeping the wafer from coming into contact with the plate, but, in this case, also providing temperature sensing capability.

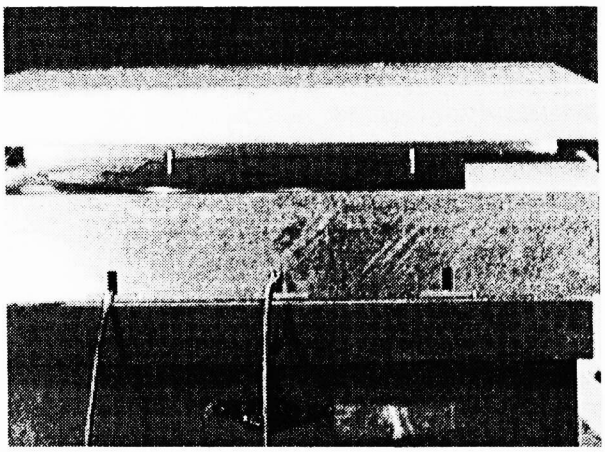

Figure 6: Hotplate design for Lamb wave temperature measurement setup.

4. EXPERIMENTAL RESULTS

PHASE MEASUREMENT OF CURE STATE

In this section, we present the results of phase measurement during photoresist removal and photoresist softbake. We first measured the overall change in phase when the resist was removed from the wafer and then the change in phase that resulted from the resist prebake process.

Phase Change During Photoresist Removal:

To evaluate the sensitivity of the phase measurement, the change in phase of the reflected signal was determined as a layer of photoresist was removed with acetone. The expected results for this change in resist thickness were calculated from theory, giving an expected phase change of about 50 degrees at the measurement frequency of 260 MHz.

For a typical resist removal experiment, an 8" silicon wafer was cleaned and spin-coated with a 2.4μm layer of Shipley 1813 resist, and the new coat was prebaked for 90 seconds on a hotplate at 90C. The wafer was placed on top of the transducer and two quartz support pins. The reflected ultrasound signal was monitored for about 30 seconds prior to removing the resist in order to obtain the measurement noise level; the noise is about 0.2 degrees peak to peak with a standard deviation less than 0.10 degrees. Acetone was then applied to the wafer to remove the resist layer, and the signal was monitored until the phase stabilized. For verification of repeatability, this experiment was also conducted at other points on the same wafer. *Figure 7* shows the reflected high frequency signal from the silicon/resist interface before and after the resist was removed from the wafer.

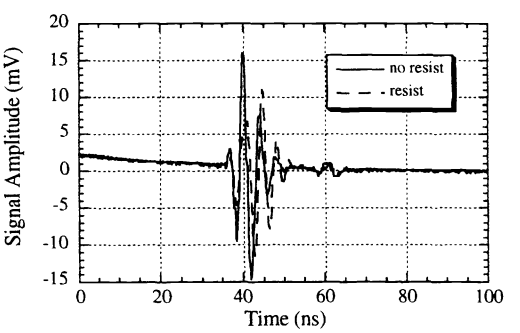

Figure 7: Reflected ultrasound signal before and after photoresist removal.

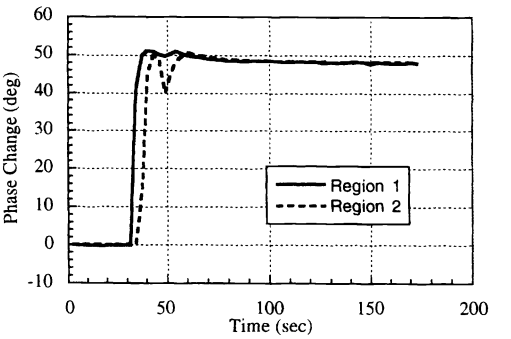

Figure 8: Experimental phase change during resist removal at two points on the wafer..

Figure 8 shows the measured phase change vs. time at 260 MHz. As indicated above, several seconds of data were taken prior to resist removal to obtain a noise figure, then the acetone was applied to the wafer. As seen in Figure 8, a 48 degree change in phase was measured when the resist was removed, agreeing well with the theoretically expected result of 50 degrees. The experiment was repeated on another part of the wafer with similar results plotted on the same graph in Figure 8. The spike in phase directly following removal in Region 2 is due to a second acetone application and the temperature effects associated with the subsequent evaporation of the solvent.

Phase Change During Resist Prebake:

Next, the phase change was measured during resist prebake to identify a significant and repeatable trend in the results that could be used to determine the state of cure at a given time. The expected change in phase during the bake was predicted to be a steady increase due to decreasing thickness and increasing density of the resist. The actual value of phase change would depend on the starting and ending point conditions of the film, including thickness, density, and velocity. This excludes the slowing effects of wafer heating, which are expected to be linear from Equation 2, as well as the changes in the elastic properties of the resist due to its softening. To measure the change in phase during prebake, first the temperature coefficient was found. A typical experiment involved cleaning an 8" wafer and measuring the increase in phase as the bare wafer was heated to prebake temperatures of about 90°C. In this way, the

slope of the linear relationship between phase and temperature was obtained. *Figure 9* shows the results of two runs on the same wafer, giving a k_v of about -28 x 10^{-6} /°C. This slope was later used to remove the temperature effects from the total phase change during a cure involving that wafer. *Figure 10* shows a sample calculation of the phase change due to temperature effects, with the upper plot representing data taken from a thermocouple attached to the wafer as the wafer was heated. The lower plot, then, is the phase change due to that temperature change, calculated using the slope in Figure 9.

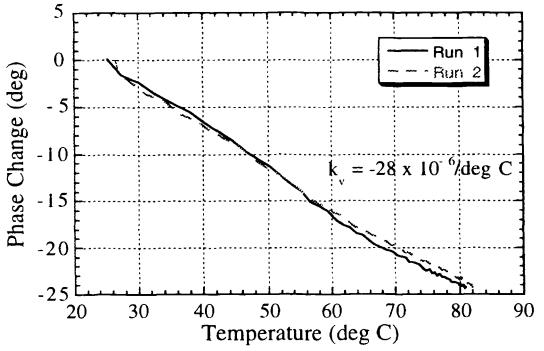

Figure 9: Phase Change vs. Temperature during heating of a bare 8" silicon wafer. This determines the slope of the linear relationship between phase and temperature so that temperature effects can be removed from the cure measurement.

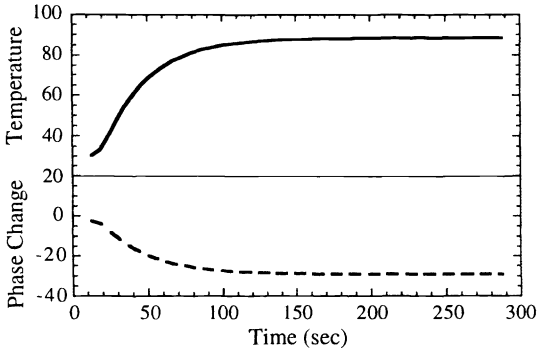

Figure 10: Sample calculation of the phase change resulting from temperature changes in a heating wafer. Temperature is in units of degrees Celsius and phase change is in degrees.

Once the temperature coefficient was determined, the wafer was coated with 1813 resist at a spin speed of about 3000 rpm. The coated wafer was immediately placed on the setup and a heating lamp was turned on to begin the prebake. The reflected waveforms and temperature data were obtained once every 3 seconds for about 200 seconds, well beyond the usual bake time of 90 seconds. The total phase change was calculated vs. time and temperature and the temperature effects subtracted using the slope determined in the first part of the experiment.

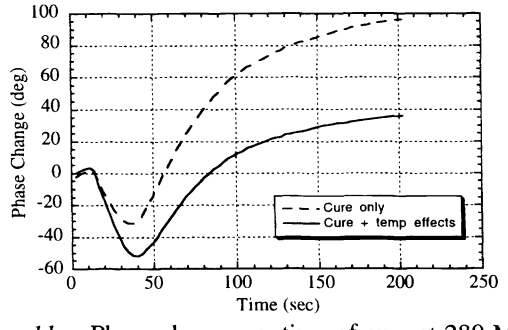

Figure 11: Phase change vs. time of cure at 280 MHz, with temperature effects (solid line) and after temperature effects have been inverted out (dashed line).

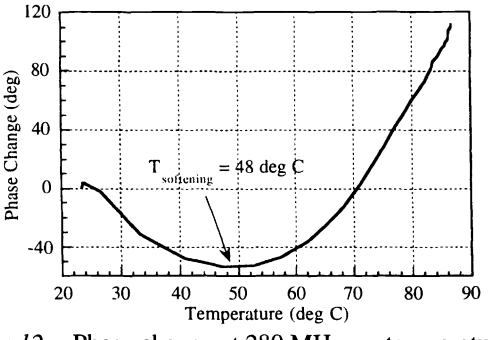

Figure 12: Phase change at 280 MHz vs. temperature during prebake.

The measured phase vs. time of cure is plotted for a typical run in *Figure 11*, both with and without the temperature effect. The phase is plotted at 280 MHz since the measurement sensitivity was highest at this point. There is an initial decrease in phase followed by a rapid increase which eventually levels off. It is believed that the initial decrease in phase is due to a the film softening during heating. Also, the rapid increase in phase that follows the minimum is likely due to the onset of solvent evaporation. This occurs after the resist has softened, allowing for the diffusion of

solvent molecules that facilitates evaporation. *Figure 12* shows the change in phase again, this time plotted against temperature. There is a repeatable phase minimum occurring at about 48 C. We believe that the temperature of this minimum represents the characteristic glass transition temperature of the resin/solvent mixture. This is important because it indicates the point at which significant evaporation and polymer relaxation occur and, therefore, the point at which significant prebaking begins. It also can provide us with a temperature calibration point that can be used in determining the temperature profile of a given prebake cycle. This would be useful in resist processing since the temperature uniformity is known to affect the final critical dimension (CD) of a feature that is being processed. For both Figures 11 and 12, the final thickness of the resist after prebake was 2μm.

LAMB WAVE TEMPERATURE MEASUREMENT

Results of Lamb wave measurements of temperature on bare wafers indicate that the measured slope is well-approximated by the theoretical calculations. A typical time of flight change vs. temperature plot is shown below in *Figure 13*. This plot shows the change in TOF as a function of the wafer temperature as it increased from 25 to 90C, a typical range of wafer temperature during prebake. The measurement pathlength was 9.5cm in this case, so the slope of the curve was divided by 9.5 to normalize it with repect to distance, resulting in a measured slope of 0.097ns/C*cm. This was in agreement with the temperature sensitivity calculated above and shown in Figure 2. The variation in slope from run to run was approximately 5%, probably due to thermal expansion of the apparatus holding the sensors in the hotplate. This variation would need to be improved before the measurement could be applied for *in situ* temperature measurement or the eventual goal of manufacturing feedback control.

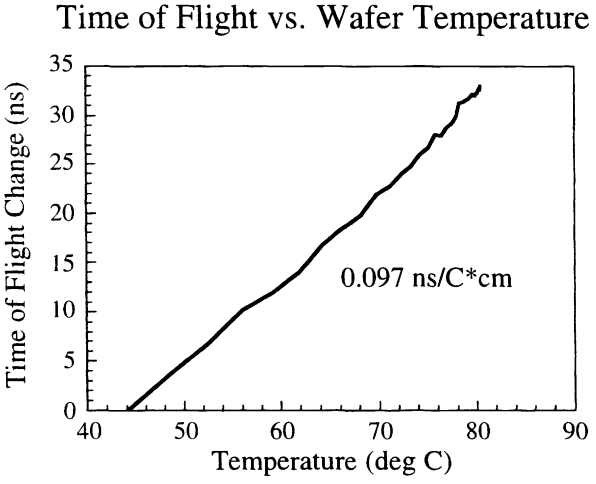

Figure 13: Results of TOF measurement of Temperature change during prebake.

5. CONCLUSIONS

An *in situ* ultrasonic method has been developed to measure the glass transition temperature of prebaking photoresist by monitoring the phase of a high frequency reflection from the silicon/resist interface. Since it is at the glass transition temperature that the resist softens and the diffusion and evaporation of the solvent molecules in the resist become significant, a measure of the glass transition temperature allows us to determine resist cure state. This method can be combined with a lower frequency Lamb wave temperature measurement to provide cure and temperature information simultaneously during the pre-exposure bake process.

6. ACKNOWLEDGEMENTS

This research was funded by the Defense Advanced Research Projects Agency of the Department of Defense and monitored by the Air Force Office of Scientific Research under Grant No. F49620-95-1-0525.

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