Thermal expansion of low-pressure chemical vapor deposition polysilicon films

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Polysilicon films were deposited using low-pressure chemical vapor deposition (LPCVD) onto oxidized silicon substrates, after which substrate curvature as a function of temperature was measured. The curvatures changed with temperature, implying that the thermal expansion of LPCVD polysilicon differs from that of the single crystal silicon substrate. Further, polysilicon films with tensile residual stresses displayed an increased thermal expansion, while polysilicon films with compressive residual stresses displayed a decreased thermal expansion. Following high temperature annealing, the residual stresses of the polysilicon films were reduced to near zero, and the thermal expansion of the polysilicon films matched that of the single crystal substrate. The apparent change in thermal expansion coefficient due to residual stress was much larger than predicted theoretically.

I. INTRODUCTION

The microstructure of low-pressure chemical vapor deposition (LPCVD) polysilicon films varies markedly with deposition conditions, particularly the deposition temperature. Films are amorphous at the lowest growth temperatures (lower than approximately 560 °C), display fine (approximately 0.1-μm diameter) ellipsoidally shaped grains at intermediate temperatures (approximately 560 °C to approximately 600 °C), and contain columnar (110)-textured grains with a thin fine-grained nucleation layer at the substrate interface at higher temperatures (>600 °C).[1,2] The fine-grained microstructure results from the homogeneous nucleation and growth of silicon crystallites within an as-deposited amorphous silicon film. In this "amorphous" regime, the deposition rate is just slightly greater than the crystallization rate. The columnar microstructure seen at the higher growth temperatures results from the formation of crystalline silicon films as-deposited, with growth being fastest in the ⟨110⟩ directions.

The residual stresses in these films are also sensitive to the deposition temperatures. The amorphous and the columnar films display compressive residual stresses, while the fine-grained films contain tensile stresses. The origin of the tensile stress in the fine-grained polysilicon arises from the slight volume decrease which accompanies the crystallization of the as-deposited amorphous material. The origins of the compressive stresses in the amorphous and columnar films are not entirely understood, but excess (interstitial) atoms incorporated during growth is one possible explanation. For polysilicon films deposited onto silicon substrates, thermal contributions to the residual stresses, i.e., thermal expansion mismatch, have not been previously considered, because the thermal expansions of the two materials—LPCVD polysilicon and single crystal silicon—have been assumed to be identical. We have now investigated the thermal expansion of LPCVD polysilicon films deposited on oxidized silicon substrates and found systematic differences that appear to be a function of the growth (or "intrinsic") stresses.

II. EXPERIMENTAL

A. Film deposition

For this investigation, five 100-mm-diameter (100)-oriented silicon wafers were used as substrates. The wafers were thermally oxidized at 1075 °C to grow 2.2-μm-thick SiO_2 layers on both sides of the wafers. Undoped polysilicon films were deposited on the substrates by LPCVD in a hot-walled horizontal tube furnace with an inner diameter of 225 mm, using SiH_4 at a flow rate of 100 sccm and a pressure of 300 mtorr

(39 Pa). The wafers were arranged vertically in the furnace, so polysilicon films were deposited equally on both sides of the substrates [see Fig. 1(a)]. For two of the wafers, the deposition temperature was 570 °C, and the deposition times were 210 and 307 min; the resulting film thicknesses were 790 and 1160 nm, respectively. These two wafers were both annealed *in situ* at 615 °C for 60 min to ensure complete crystallization, as the as-deposited films were partially amorphous. For two other wafers, the deposition temperature was 615 °C, and the deposition times were 100 and 156 min; the resulting film thicknesses were 750 and 1170 nm, respectively. For the fifth wafer, the deposition temperature alternated between 570 and 615 °C to create a 9-layer multilayer film.[1] The deposition started and ended at 570 °C, and the deposition times for the nine layers were 113.5, 78.7, 130, 40, 165.5, 59.3, 94.5, 54.5, and 136 min; the total thickness was 4210 nm. As described in Ref. 1, these deposition times were designed to produce a multilayer polysilicon film, termed a "MultiPoly" film, which displays a near-zero overall residual stress and near-zero overall residual stress gradient. The polysilicon film thicknesses were measured using a Nanometrics (Milpitas, CA) spectrophotometer.

B. Curvature measurements

After polysilicon film deposition, the curvature of each wafer was measured using a Frontier Scientific Measurements (San Jose, CA) model SMSi3800 room temperature stress measurement tool. This instrument scans a laser across the central 80 mm of the wafer, detects the
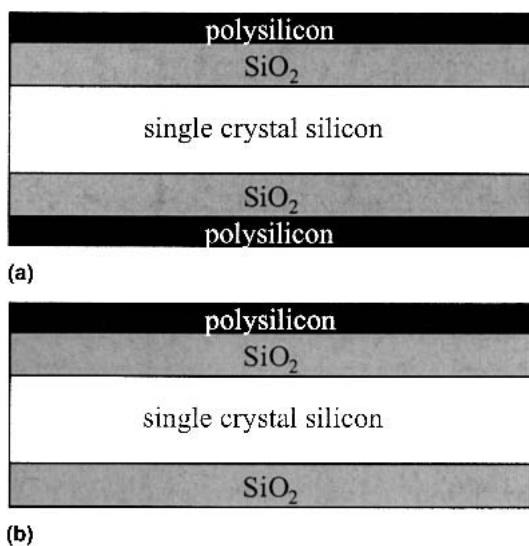

(a)

(b)

FIG. 1. Schematic cross-sections (not to scale) of the samples used in this investigation. (a) An oxidized silicon substrate with LPCVD polysilicon films on both sides. (b) The same substrate after one LPCVD polysilicon films has been removed by dry etching using a chlorine plasma.

position of the reflected laser signal, and thus allows the curvature of the wafer to be determined. Six scans across each substrate were made, with automatic turning of the wafer 30° between each scan. The data for all six scans were used to produce an average curvature.

Following this initial determination of the wafer curvature, the polysilicon film was removed from one side of each wafer using a dry chlorine plasma [see Fig. 1(b)]. The curvature of each wafer was measured again, and the residual stress σ of the polysilicon film was determined, using the Stoney equation[3]

$$\sigma = \frac{E}{(1 - \nu)} \frac{t_s^2}{6t_f} (K_f - K_i) \quad , \qquad (1)$$

where $E/(1 - \nu)$ is the biaxial modulus of the (100) silicon substrate (equal to 180.5 GPa[4]), t_s is the substrate thickness, t_f is the film thickness, K_f is the final curvature (after etching the film from one side), and K_i is the initial curvature. (The validity of using the Stoney equation in this analysis is addressed in the Appendix.)

The curvature of each substrate was measured as a function of temperature using a Frontier Scientific Measurements model 500TC high temperature stress measurement tool. Like the SMSi3800 room temperature model, this instrument also scans a laser across the central 80 mm of a wafer and monitors the reflected laser signal in order to determine curvature. The instrument operates in a flowing nitrogen ambient. During each run, the temperature of the wafer was increased from room temperature to 300 °C at a rate of 5 °C/min, and was then immediately cooled. The cooling rate began at 5 °C/min, but as the temperature neared 200 °C, the thermal mass of the system led to decreased cooling rates; cooling from 300 to 30 °C took approximately 100 min. Laser scans were performed approximately every 10 °C during both heating and cooling. Automatic substrate rotation is not possible in the 500TC instrument; therefore, two investigations were performed on each wafer, manually rotating the wafer 90° between the two. In one experiment, the scans were parallel to the major {110} flat present on production quality Si wafers, and in the other, the scans were perpendicular to the major flat. To examine the inherent uncertainties of the equipment, the curvature versus temperature measurements were also performed on an oxidized wafer which had 2.2-μm-thick SiO$_2$ layers on both sides but no polysilicon film.

C. Annealing experiments

The wafers containing the two thickest polysilicon films (the 1160-nm film deposited at 570 °C and the 1170 nm film deposited at 615 °C) were annealed at 1100 °C for 1 h in a flowing nitrogen ambient, following the curvature measurements described above. This high temperature anneal is known to drastically reduce

the residual stresses in polysilicon films.[1] After annealing, the curvatures of these two wafers were remeasured, both at room temperature and as a function of temperature to 300 °C.

III. RESULTS

A. Curvature measurements

The room-temperature curvature measurements and the calculated residual stresses for the five LPCVD polysilicon films are listed in Table I. The convention used is that a positive curvature indicates a concave shape (like a bowl) when the wafer is oriented with the film on top, and a negative curvature indicates a convex shape (like a dome) when the wafer is oriented with the film on top. A positive stress is tensile, and a negative stress is compressive. As described in Ref. 1, it is expected that the residual stresses of the polysilicon films are tensile when deposited at 570 °C and compressive when deposited at 615 °C. It was also determined in our earlier work that the magnitudes of the residual stresses varied with film thickness. The MultiPoly film was successfully designed to exhibit an overall residual stress that is much lower in magnitude than the stresses of its components.

The high temperature curvature measurements are shown in Fig. 2. Since bare silicon wafers do not necessarily exhibit axisymmetric curvatures (this is a purely empirical observation, based on measurements of dozens of silicon wafers), the linear curvatures measured on two successive 90° scans on a single wafer will not necessarily be identical, even though the thickness of the deposited films are very uniform across the entire wafer. This is seen in Fig. 2; the curvatures obtained for the scans parallel and perpendicular to the major flat are very similar in some cases [Figs. 2(b) and 2(d)] and dissimilar in other cases [Figs. 2(a), 2(c), 2(e), 2(f)].

The scans in Fig. 2(f) were taken from the bare oxidized wafer with no polysilicon. Inasmuch as the SiO_2 thickness was equal on both sides of the wafer, no change in curvature is expected with an increase in temperature, even though the thermal expansion of SiO_2 and Si differ

(approximately 0.5 and approximately 2.5 ppm/K, respectively). Therefore, any curvature changes that are detected must be due to variations in the instrument and its detection system. From Fig. 2(f), the inherent uncertainty of the curvature measurement is ±0.4 km^{-1}. In Figs. 2(a)–2(d), when the heating and cooling curves are compared, there appears to be a systematic error; the cooling curves always indicate a greater absolute value of curvature. However, this error is generally within the ±0.4 km^{-1} uncertainty. We have no obvious reason to suspect the validity of either the heating or cooling results, and therefore treat the heating and cooling data without bias. It is likely that the differences seen between the heating and cooling curves originate from instrumental error and do not represent anelastic behavior in the polysilicon films.

B. Thermal expansion determination from curvature measurements

Any change in curvature as a function of temperature can be interpreted in one of two ways: the thermal expansion of the polysilicon film differs from that of the substrate, or the biaxial modulus of the substrate changes with temperature. It is clear that the magnitudes of the measured curvatures in Figs. 2(a)–2(d) decrease with increasing temperature. This would occur if the substrates were becoming stiffer on heating, i.e., the biaxial modulus of silicon increases with increasing temperature. However, this is opposite to what is experimentally observed; silicon becomes less stiff as the temperature increases. Quantum atomistic simulations of silicon have shown that the bulk modulus of silicon decreases (but only by 0.6%) when the temperature increases from 25 to 300 °C[5]; therefore, this effect will be neglected. In the following analysis, the change in curvature is assumed to be caused by a difference in thermal expansion between the polysilicon films and the silicon substrate.

To use Eq. (1) to translate the curvature data in Fig. 2 into residual stress values, the initial curvatures (before etching away the polysilicon film from one side of each wafer) are required for each scan performed in the high temperature stress measurement tool. These linear curvatures were not measured, but can be deduced using the room temperature residual stresses listed in Table I. Using Eq. (1), the stress σ is given in Table I, and the final curvature K_f is the first (room temperature) result for each data set in Fig. 2; then the initial curvature K_i can be calculated for each scan. The same K_i is used for the entire scan, and the data in Fig. 2 are converted into residual stresses. The stress results for the five polysilicon films are plotted in Fig. 3. It should be noted that there are four data in Fig. 2(a) and one datum in Fig. 2(c) (all arrowed) that are considered outliers and are not included in Fig. 3.

TABLE I. Curvature and stress measurements for the five wafers with LPCVD polysilicon films.

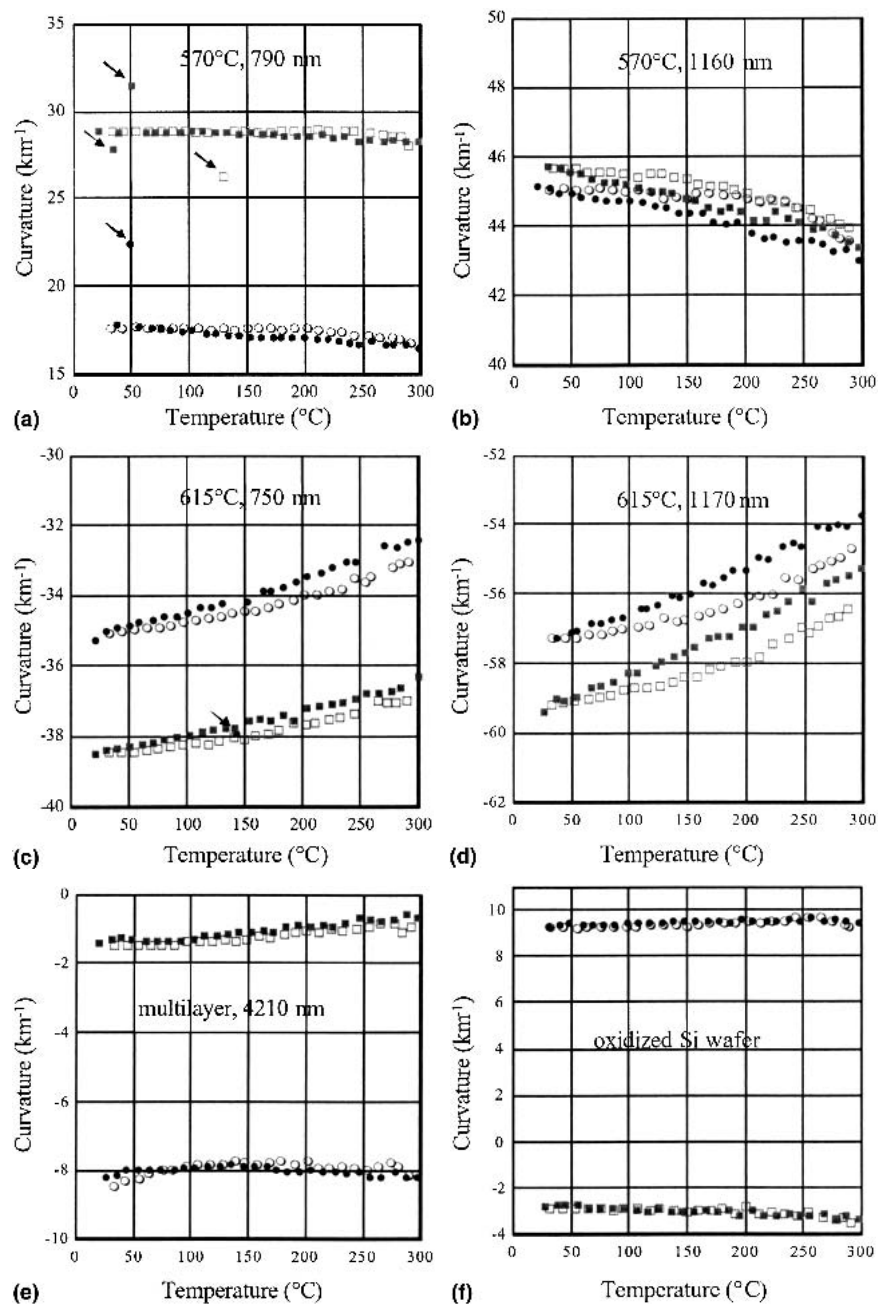| Deposition temperature (°C)	570	570	615	615	570/615
Film thickness (nm)	790	1160	750	1170	4210
Substrate thickness (mm)	0.515	0.506	0.540	0.501	0.524
Initial curvature (km^{-1})	−11.8	−0.5	1.7	−4.8	1.7
Final curvature (km^{-1})	19.8	33.9	−28.8	−44.2	−3.1
Film stress (MPa)	319	228	−357	−255	−9.6

FIG. 2. (a–e) Curvature versus temperature results for the five wafers with polysilicon films and (f) for the oxidized wafer with no polysilicon. Each plot is labeled with the polysilicon deposition temperature and film thickness. In each plot, the solid symbols indicate measurements taken during heating, and the open symbols indicate measurements taken during cooling. The squares are from scans parallel to the major flat of the wafer, and the circles are from scans perpendicular to the major flat. Note that the vertical scale is equal in magnitude for (b–e); it is doubled for (a) and increased by half for (f).

The calculation to obtain K_i just described is a convenient technique to normalize the two curvature scans (parallel and perpendicular to the major flat of the wafer) taken from each wafer. It also allows the curvature data to be expressed more conveniently as residual stresses, and to be plotted in a single graph in Fig. 3. However, it should be noted that in the calculations of the polysilicon

thermal expansion coefficients which follow, the K_i values do not play a role; it is only the slopes of the curvature plots in Fig. 2 that are important.

In Fig. 3, the stresses for the tensile polysilicon films deposited at 570 °C have slightly negative slopes, the stresses for the compressive polysilicon films deposited at 615 °C have slightly positive slopes, and the stresses

for the MultiPoly film are for all intents and purposes independent of temperature. As shown in Fig. 2, the stresses return to their original room temperature values after the complete heating-and-cooling cycle. Therefore, no permanent changes occur during the heating, and the stress variations must be due to a difference in thermal expansion between the polysilicon film and the single crystal silicon substrate. (Because the SiO_2 films exist symmetrically on both sides of the wafers, and because they are thin compared to the silicon substrates, they are ignored in this analysis. The validity of this assumption is
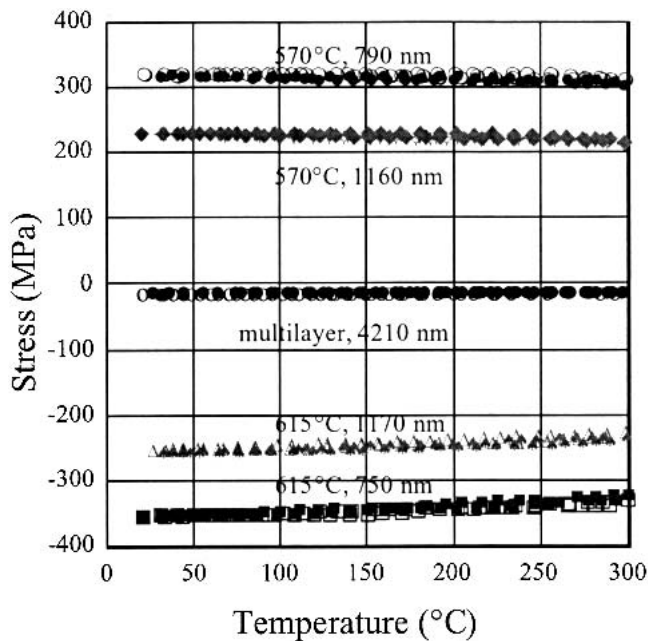

FIG. 3. Stress versus temperature data for the five polysilicon films. The open symbols are from scans parallel to the major flat of the wafer, and the solid symbols are from scans perpendicular to the major flat. The stresses are calculated using Eq. (1) and the data in Fig. 2. The initial curvatures are determined from the Stoney equation, assuming that the room temperature stresses were equal to the values listed in Table I.

addressed below.) Assuming a Young's modulus of polysilicon of 160 GPa (which is the generally accepted value for LPCVD polysilicon[6]), the slopes of the linear fits to the data in Fig. 3 can be translated into the differences ($\Delta\alpha$) between the linear coefficients of thermal expansion α of polysilicon and single crystal silicon. These are listed in Table II.

The scans from the tensile polysilicon films consistently display an average positive $\Delta\alpha$, the scans from the compressive polysilicon films consistently display an average negative $\Delta\alpha$, and the scans from the MultiPoly film consistently display a near zero $\Delta\alpha$ (Table II). The uncertainties in $\Delta\alpha$ for the five polysilicon films were calculated using the uncertainty in curvature of ± 0.4 km^{-1} described above. The values for $\Delta\alpha$ are plotted versus film stress in Fig. 4. It should be noted that the polysilicon films are subjected to biaxial stresses and not hydrostatic pressures. The equivalent hydrostatic pressures (equal to $\frac{2}{3}$ the biaxial stresses) are shown as the top abscissa on Fig. 4. The best-fit line has a slope of 1.3 ppm/K/GPa biaxial stress, or 1.9 ppm/K/GPa hydrostatic pressure. It should also be noted that α for single crystal silicon is not constant, but increases from 2.0 to 3.2 over the temperature range of interest.[7] However, the change in α due to an applied pressure will still be nearly constant across this temperature range.[5]

C. Annealing experiments

As expected, the residual stresses of the polysilicon films decreased considerably after the 1100 °C anneal. The stress of the film deposited at 570 °C dropped from 228 to 10 MPa, and the stress of the film deposited at 570 °C dropped from −255 to −5 MPa. The results for the high-temperature curvature measurements are shown in Fig. 5. Figures 5(a) and 5(b) can be directly compared to Figs. 2(b) and 2(d), respectively, which show the curvature measurements before annealing. It is readily

TABLE II. Difference in linear coefficient of thermal expansion between polysilicon and single crystal silicon ($\Delta\alpha$) determined from the data shown in Figs. 2 and 5. The uncertainties in the average values for each film were calculated from the uncertainty in curvature of ± 0.4 km^{-1}.

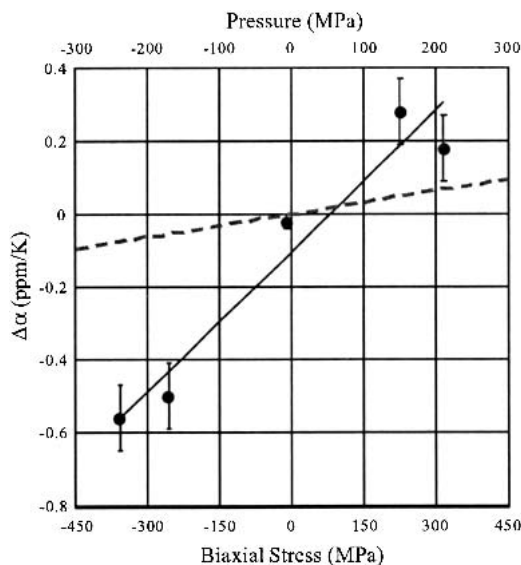| Deposition temperature (°C)	570				615				570/615	
Thickness (nm)	790		1160		750		1170		4210	
Orientation to wafer flat	//	⊥	//	⊥	//	⊥	//	⊥	//	⊥
$\Delta\alpha$ (ppm/K)	0.13	0.24	0.30	0.25	−0.49	−0.63	−0.52	−0.49	−0.03	0.00
	average = 0.18 ± 0.09		average = 0.28 ± 0.06		average = −0.56 ± 0.09		average = −0.50 ± 0.06		average = −0.02 ± 0.02	
					After annealing					
$\Delta\alpha$ (ppm/K)	0.02	−0.03					−0.07	−0.03		
	average = −0.01 ± 0.06						average = −0.05 ± 0.06			

FIG. 4. Results for the change in coefficient of thermal expansion for polysilicon. The solid line is a best fit to the data; the dashed line is a theoretical prediction.

apparent that both the curvature and the change in curvature as a function of temperature have decreased significantly for both wafers. From the data in Fig. 5, the $\Delta\alpha$ values were calculated to be -0.01 ± 0.06 ppm/K for the film deposited at 570 °C and -0.05 ± 0.06 ppm/K for the film deposited at 615 °C. These values are included in Table II.

D. Theoretical estimation of the thermal expansion

In the quasiharmonic approximation, and assuming a perfect crystal, the linear coefficient of thermal expansion α is given by[8]

$$\alpha = \gamma C_v / 3B \quad , \qquad (2)$$

where γ is the Grüneisen parameter [The Grüneisen parameter is a measure of the change in phonon frequency with a change in crystal volume.[8]], C_v is the specific heat, and B is the bulk modulus. Taking the derivative with respect to pressure P

$$\partial\alpha/\partial P = -\gamma C_v / 3B^2 \, \partial B/\partial P \quad . \qquad (3)$$

At 27 °C, γ for silicon is 0.452,[9] C_v for silicon is 1.7×10^6 J/K/m³ (equivalent to 1.7×10^6 Pa/K),[10] B is 100.75 GPa and $\partial B/\partial P$ is 4.72.[11] Therefore, $\partial\alpha/\partial P$ is 0.12 ppm/K/GPa. At 300 °C, γ is 0.55,[10] C_v is 1.9×10^6 Pa/K,[10] and $\partial\alpha/\partial P$ is 0.16 ppm/K/GPa. Accordingly, for the temperature range studied in this experiment, theory predicts the change in α due to changes

in residual stress to be approximately 0.14 ppm/K/GPa, which prediction is plotted as a dashed line in Fig. 4. The experimental data indicate a much higher change in α for the applied pressures associated with these residual stresses, 1.9 ppm/K/GPa.

Recently, atomistic simulations have been carried out to predict the pressure dependence of the thermodynamic properties of silicon. A quantum atomistic simulation using Stillinger–Weber potentials[5] predicts α of silicon at 27 °C to be 2.6 ppm/K under atmospheric conditions (zero applied pressure) and 1.8 ppm/K under an applied pressure of 10 GPa; this gives a $\Delta\alpha/\Delta P$ of 0.08 ppm/K/GPa. An *ab initio* study[10] predicts α of silicon at 27 °C to be 2.4 ppm/K under zero applied pressure and 1.9 ppm/K under an applied pressure of 2 GPa, for a $\Delta\alpha/\Delta P$ of 0.25 ppm/K/GPa. Both of these predictions are well below the results determined in this experiment, 1.9 ppm/K/GPa.

IV. DISCUSSION

The results indicate that for LPCVD polysilicon, residual stresses generate a much larger change in the coefficient of thermal expansion than would be expected from a theoretical analysis of single crystal silicon. The same result is seen for both tensile and compressive films. Further, upon high-temperature annealing, both tensile and compressive residual stresses reduce to near-zero values, and the coefficient of thermal expansion of the polysilicon becomes essentially equivalent to single crystal silicon.

The only obvious differences between LPCVD polysilicon and single crystal silicon are the presence of grain boundaries and presumably the incorporation of hydrogen in the polysilicon films; hydrogen incorporation is commonly found for LPCVD deposition of polysilicon. In our materials, the fine-grained polysilicon deposited at 570 °C has a grain size on the order of 300 nm, and each grain is internally twinned with a twin spacing on the order of 2 to 10 nm.[2] In the columnar polysilicon deposited at 615 °C, the column diameters are on the order of 700 nm, and this structure also contains a high density of twins, with an average spacing on the order of 3 nm, as measured parallel to the (110) plane.[12] Therefore, all the polysilicon films contain non-trivial amounts of "free volume" in the grain and column boundaries and in any incoherent twin boundaries.

Upon annealing to 1100 °C, the hydrogen will be released by the polysilicon films.[13] However, microstructural examination of the polysilicon films (unpublished data) after this annealing reveals only very modest reductions in twin boundary density and virtually no change in grain size or column diameter. This might seem to implicate hydrogen as the cause of the observed changes in thermal expansion behavior; however, since
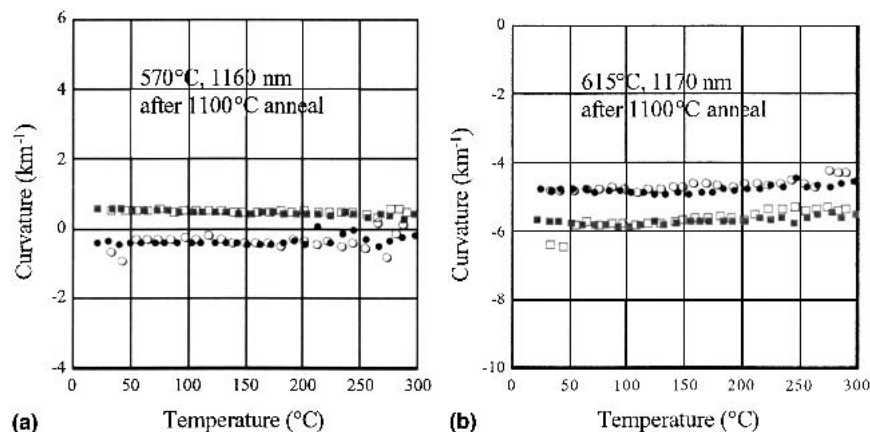
FIG. 5. Curvature versus temperature results for the two thicker polysilicon films after annealing at 1100 °C for 1 h. The symbols are the same as in Fig. 2. Figures 5(a) and 5(b) can be directly compared to Figs. 2(b) and 2(d), respectively. All vertical scales are equivalent.

hydrogen is present in both the tensile and compressive films, this cannot explain why the changes in thermal expansion are opposite for the two different stress states.

It must be recognized that thermal expansion coefficients reflect the anharmonicity of the interatomic potential—the short range repulsion interaction increases much more steeply with atomic spacing than does the relatively longer range attractive covalent interaction. The theoretical and atomistic analyses discussed in the previous section deal with perfect crystals and the response of the atomic bonds in those crystals to applied stresses. It is possible that the anharmonic response of those atoms associated with free volume, in the presence of tensile or compressive stresses, is different from atoms in perfect crystals or from those same "free volume" atoms in the absence of an applied stress. The net result is a thermal expansion coefficient α in polysilicon containing residual stresses that differs from α in single-crystal silicon and from α in polysilicon without residual stresses. It must be noted that stress–strain curves taken from similar LPCVD polysilicon films did not reveal any changes in Young's modulus up to 0.7% tensile strain (approximately 1 GPa stress).[14] However, it is questionable whether the anharmonic effects discussed here would be discernible in these measurements.

V. SUMMARY AND CONCLUSIONS

LPCVD polysilicon films with tensile, compressive or near-zero overall residual stresses were deposited onto oxidized silicon substrates. Substrate curvature versus temperature measurements were taken from room temperature to 300 °C, which indicated that the linear thermal expansion of polysilicon containing tensile stresses is greater than that of single crystal silicon, while the linear thermal expansion of polysilicon containing compressive stresses is smaller than that of single crystal silicon. On average, the change in α due to such residual

stresses is 1.9 ppm/K/GPa pressure. By contrast, theory predicts the change in α of single crystal silicon due to applied pressure to be 0.08 to 0.25 ppm/K/GPa in the temperature range of interest. The polysilicon films contain high densities of grain boundaries and twin boundaries. One possible explanation for the observed thermal expansion behavior is that the residual stresses cause changes to the anharmonicities of the interatomic potentials of those atoms associated with these boundaries.

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APPENDIX

The analyses performed in this paper use the Stoney equation [Eq. (1)] to calculate stresses in polysilicon films. The basic assumption of the Stoney equation is that the main substrate material, in this case single-crystal silicon, is so thick compared to any other layers that its mechanical properties dominate. In fact, the substrates used in this experiment are oxidized silicon, composed of approximately 500-μm-thick silicon wafers with 2.2-μm-thick SiO_2 layers on both sides. The calculation of the stresses listed in Table I assumed that the Young's modulus of the entire substrate was equal to that of single crystal silicon, even though the Young's modulus of SiO_2 is about half that of silicon. However, since the SiO_2 thickness is less than 1% of the silicon thickness, this assumption is reasonable.

The changes in curvature plotted in Fig. 2 were attributed to differences in thermal expansion between the polysilicon and the single crystal substrate. To determine the possible effects of the SiO_2 layers on the measured curvatures, a rigorous analysis employing laminate theory was performed on a model system, consisting of a 1-μm-thick polysilicon layer on a 500-μm-thick silicon wafer with 2.2-μm-thick SiO_2 layers on both sides. The α for both polysilicon and single crystal silicon was assumed to be 2.5 ppm/K, and α for SiO_2 was assumed to be 0.5 ppm/K. The stress of the SiO_2 was assumed to be zero at its growth temperature of 1075 °C, and the polysilicon deposition temperature was taken as 600 °C. For this model system, a temperature increase from 25 to 300 °C produced a curvature change of 0.04 km^{-1}. This is well within the error estimates of the curvature measurement, as discussed above and validates the use of the Stoney equation as a reasonable approximation to determine residual stresses from curvature data.