Multiple-Angle Incident Ellipsometry Measurement on Low Pressure Chemical Vapor Deposited Amorphous Silicon and Polysilicon

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ABSTRACT

Multiple-angle incident (MAI) ellipsometry is used to study the optical properties of both amorphous silicon and poly-Si after different heat-treatments. At first, the error sensitivity of MAI ellipsometry has been studied and the optimum conditions to achieve the best precision are obtained. The optical constants and thickness of amorphous silicon deposited by LPCVD at 550°C are changed significantly after sample annealing. The optical constants of the amorphous silicon reached the values of single-crystalline silicon after annealing for both the high and low temperature treatments. A thickness shrinking phenomenon is observed when amorphous silicon is transformed to the polycrystalline form. This phenomenon is confirmed by cross-sectional TEM photography. The thickness shrinking ratio is 3.5 to 6.0%.

Polycrystalline silicon (poly-Si) films have been employed in various integrated circuit applications.[1-3] They are used as the gate electrode[4] and the high value load resistor[5] in metal oxide semiconductor field effect transistor (MOSFET) applications. Also, they are used as a diffusion source to form an emitter in very large scale integrated (VLSI) bipolar transistors.[6,7] In addition, they are used in nonvolatile electrically erasable programmable memory devices.[8] Polysilicon films are used in many other semiconductor devices. For example, they are used in charge-coupled devices (CCD) as gate electrodes,[9] and in solar cells as the substrate material.[10] Due to their vast application in integrated circuits, their electrical properties have been studied extensively.[1] The optical properties of poly-Si are somewhat different from those of single-crystalline silicon[11,12] due to the polycrystalline structure. They are dependent on the preparation conditions of the poly-Si film. For example, the refractive index of a poly-Si film varies distinctly for deposition temperatures above and below 580°C.[1]

Ellipsometry has been used for measuring the optical properties and the thickness of a poly-Si film.[13-20] There were two factors which made the measurements difficult (*i*) the ellipsometric period for poly-Si is small, only 850 Å at 632.8 nm. Therefore, the thickness of poly-Si must be known within this order and (*ii*) more than two sets of the measured data (Δ, ψ) for poly-Si must be obtained to calculate the real part, *n*, the imaginary part, *k*, of the refractive index, and the thickness, *t* of the poly-Si. For a conventional ellipsometer with a single incident wavelength and a single incident angle for any material, it is impossible to calculate the optical constants and the thickness simultaneously with one set of measured data (Δ, ψ). Hence, different technologies have been reported to solve this problem. For example, MAI ellipsometry[19] had been proposed to provide more sets of (Δ, ψ) data. Besides, techniques of various thicknesses of sandwiched SiO_2 before deposition of poly-Si films[18] and a zeroth-layer model method by measuring on a beveled poly-Si surface[20] have been proposed.

In this work, the optical properties of poly-Si films, both deposited at low temperature (<580°C) in amorphous form and deposited at high temperature (>580°C) in polycrystalline form and then annealed at various temperatures were studied by using an MAI ellipsometer. After determining the optimum conditions for the measurements by analyzing the system sensitivity of the investigated structure, poly-Si/SiO_2/Si structures were investigated by MAI measurements.

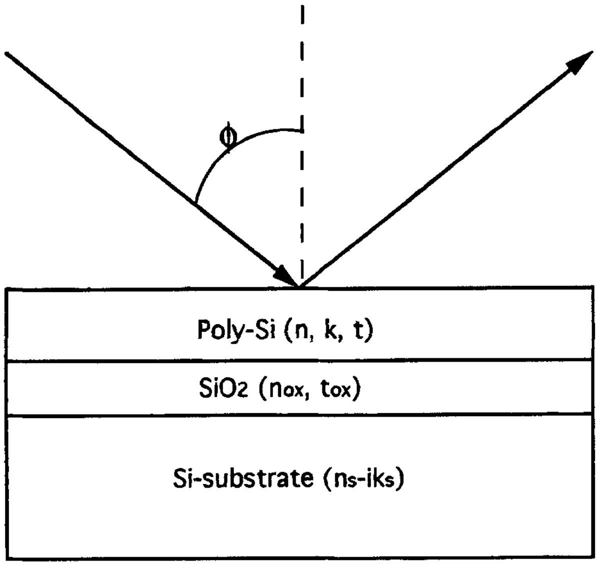

Fig. 1. The poly-Si/SiO_2/Si investigated or measured structure by MAI ellipsometry, where the unknown parameters are *n*, *k*, and *t*.

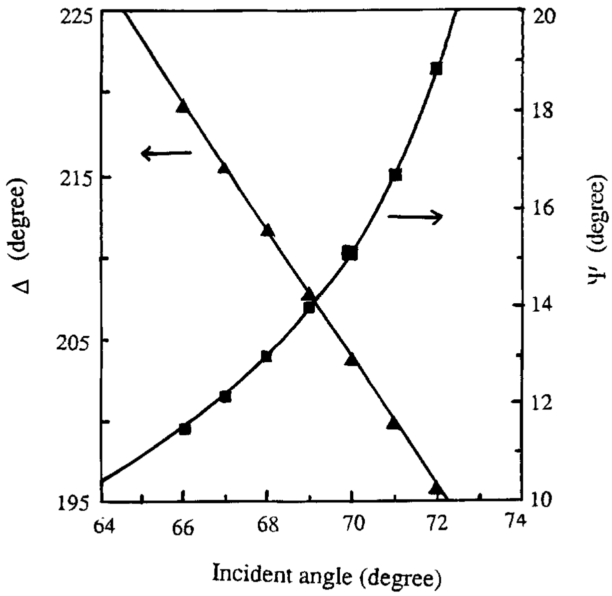

Fig. 2. The fitted (solid) line of the measured Δ and ψ values (solid marks) of the amorphous Si/SiO_2 (213 Å)/Si structure measured at various incident angles.

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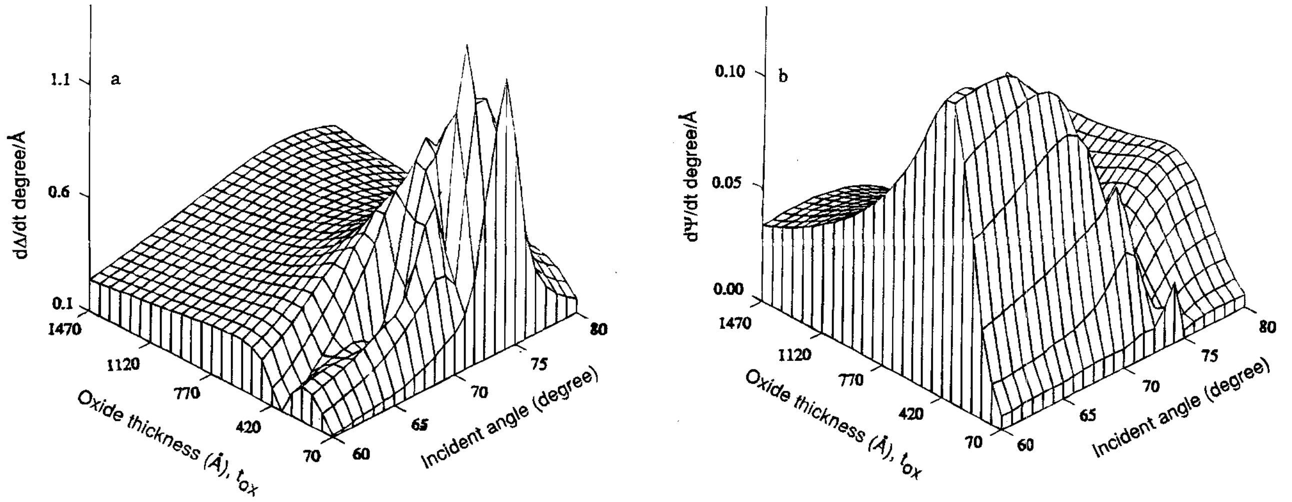

Fig. 3. (a, left) The sensitivity $d\Delta/dt$ vs. both the incident angle and the buried oxide thicknesses for the poly-Si (1000 Å)/SiO_2/Si structure. (b, right) The sensitivity $d\psi/dt$ vs. both the incident angle and buried oxide thicknesses for the poly-Si (1000 Å)/SiO_2/Si structure.

Sensitivity Analysis of MAI Ellipsometry

The sample structure investigated is shown in Fig. 1, where a poly-Si film was deposited on a thin SiO_2 film grown on an Si substrate. The light beam of the MAI ellipsometer is incident on the surface of the sample at an incident angle, ϕ, that varies from 60 to 80°. Using the sets (Δ, ψ) of the measured values at different incident angles, the corresponding values of the calculated complex refractive index, n-ik and the thickness, t, of the poly-Si film were chosen to fit curves as shown in Fig. 2. Due to both the inevitable instrument and measurement errors,[20] the fitting process usually embedded some unavoidable errors. Among the measurement errors, the thickness, t_{ox}, of the buried SiO_2 layer, affects the error significantly. It is desirable to choose an optimum t_{ox} thickness at the most sensitive range of incident angles and $d\Delta/dt$ to minimize the error.

Figure 3a shows a 3D $d\Delta/dt$ plot vs. t_{ox} and ϕ for the poly-Si film thickness of 1000 Å. To achieve a high precision during measurements, it is desirable to obtain the highest $d\Delta/dt$ value since a small error in t value results in a large deviation in Δ reading. Hence, in Fig. 3a, the thickness of the buried oxide must be chosen in the range of 200 to 300 Å, and the angle of incidence ϕ should be chosen in the range of 66 to 72°, since at this region, the sensitivity is the largest. A similar theoretical plot for $d\psi/dt$ is shown in Fig. 3b. The plot shows that $d\psi/dt$ is the largest at t_{ox} = 350 Å, and the incident angle range, ϕ, from 62 to 72°. However, the magnitude of $d\psi/dt$ is about one order of magnitude smaller than that of $d\Delta/dt$. Hence, in the MAI ellipsometry, the precision of the parameter Δ is more critical than that of the parameter ψ for reducing the fitting error. In conclusion, the optimum conditions for achieving the best precision for this MAI measurement are that the thickness of the buried SiO_2 must be chosen in the range of 200 to 300 Å, and the incident angle in the range of 66 to 72°.

Table I. Error simulations of MAI ellipsometry for *n, k,* and *t* of poly-Si film.

Deviation	n (error)	k (error)	t (error)
dn_s (−0.008)	3.985 (0.37%)	0.101 (1%)	1005 (0.5%)
dn_s (+0.008)	3.982 (0.45%)	0.102 (2%)	1007 (0.7%)
dk_s (−0.008)	3.984 (0.40%)	0.100 (0%)	1006 (0.6%)
dk_s (+0.008)	3.986 (0.35%)	0.103 (3%)	1005 (0.5%)
dn_{ox} (−0.01)	3.981 (0.47%)	0.105 (5%)	1007 (0.7%)
dn_{ox} (+0.01)	3.987 (0.32%)	0.098 (2%)	1005 (0.5%)
dt_{ox} (−5 Å)	3.982 (0.45%)	0.095 (5%)	1007 (0.7%)
dt_{ox} (+5 Å)	3.991 (0.22%)	0.108 (8%)	1003 (0.3%)
dt_{nox} (+10 Å)	3.986 (0.35%)	0.096 (6%)	1008 (0.8%)
dt_{nox} (+20 Å)	3.983 (0.42%)	0.090 (10%)	1012 (1.2%)

Corresponding to the different deviations in (i) optical constants of the substrate (n_s, k_s); (ii) optical constants of the SiO_2 (n_{ox}); (iii) thickness of SiO_2 (t_{ox}); (iv) thickness of native oxide (t_{nox}) for the poly-Si/SiO_2/Si-substructure estimated on the assumption that: (i) complex refractive index of poly-Si is 4.00 − i0.10; (ii) thickness of poly-Si film is 1000 Å; (iii) thickness of SiO_2 is 250 Å; (iv) refractive index of SiO_2 is 1.46; (v) thickness of native oxide is 10 and 20 Å.

To investigate the errors produced by small deviations in n_s, k_s, n_{ox}, t_{ox}, and t_{nox}, which are the real part, the imaginary part of the complex refractive index of the substrate, re-

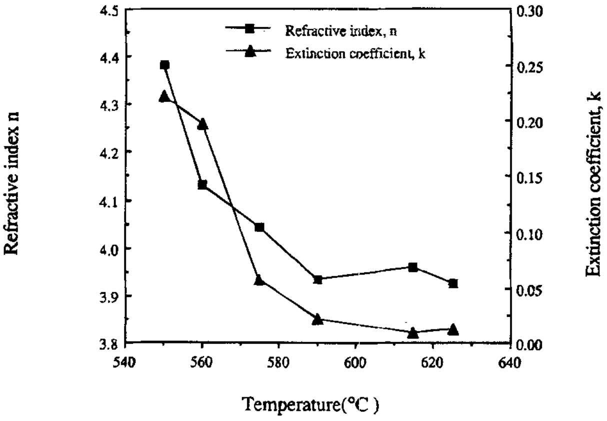

Fig. 4. The measured real and imaginary parts of the complex refractive index for the LPCVD poly-Si deposited at temperatures from 550 to 625°C.

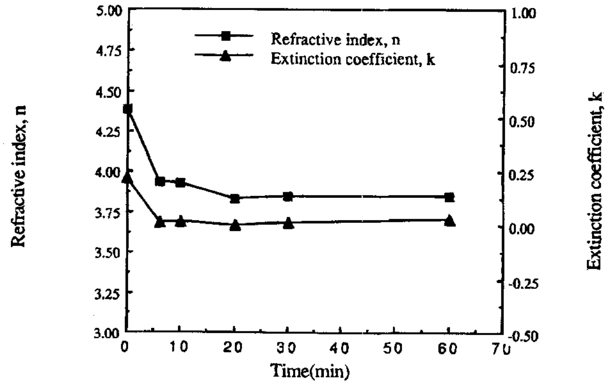

Fig. 5. The measured real and imaginary parts of the complex refractive index for the amorphous silicon annealed at 1000°C in an N_2 ambient for 6, 10, 20, 30, and 60 min.

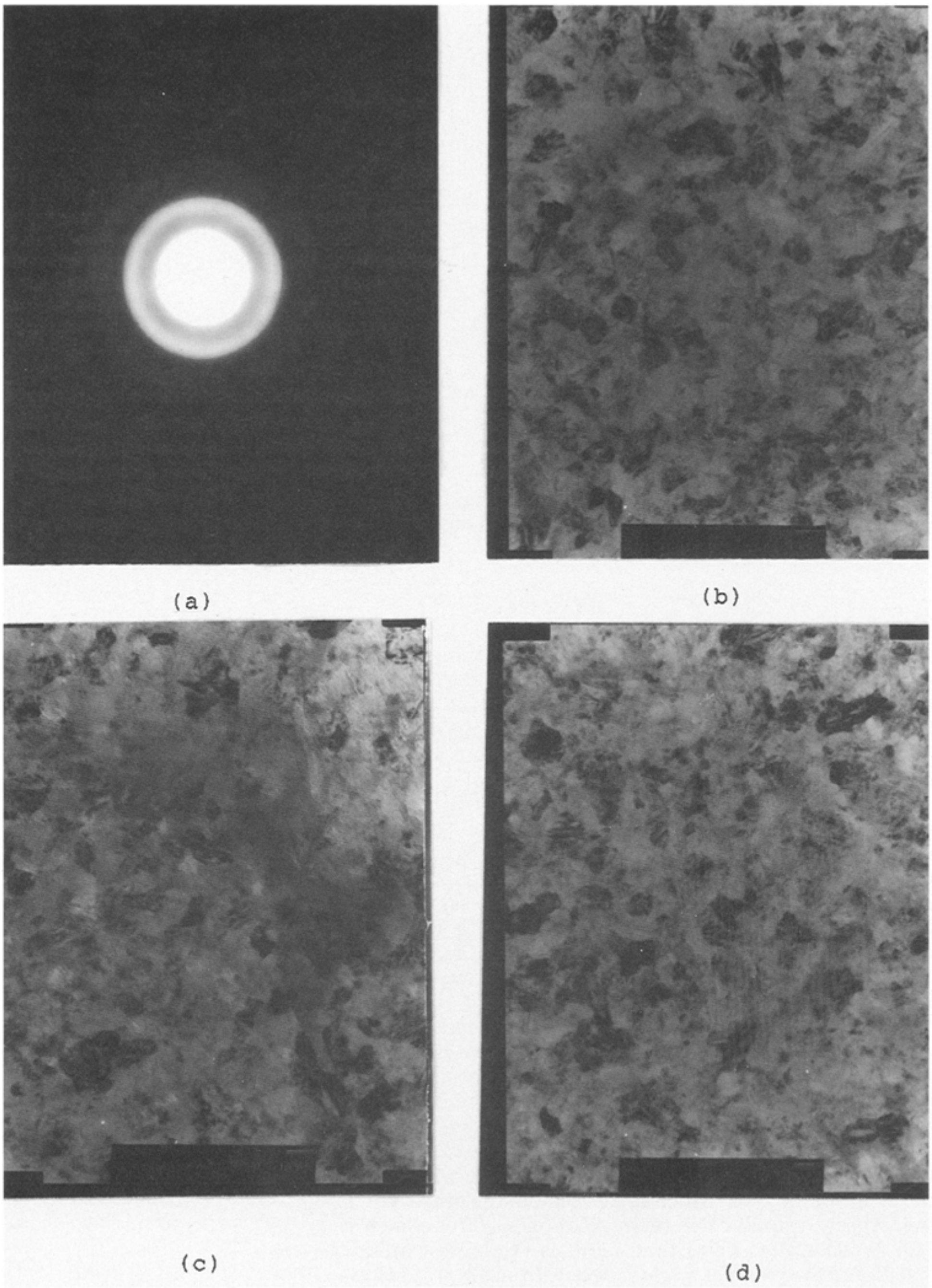

Fig. 6. (a) Electron diffraction pattern of the LPCVD poly-Si film deposited at 550°C; (b) TEM photograph of the amorphous silicon annealed at 1000°C for 6 min; (c) TEM photograph of the amorphous silicon annealed at 1000°C for 20 min; and (d) TEM photograph of the amorphous silicon annealed at 1000°C for 60 min.

spectively, the refractive index and the thickness of the buried SiO_2, and native oxide on poly-Si, respectively, a numerical simulation was carried out. The results are shown in Table I. In this simulation, it was assumed that t_{ox} = 250 Å, n_{ox} = 1.46, n_s = 3.858, k_s = 0.018, t_{nox} = 10 and 20 Å, and the complex refractive index and the thickness of the poly-Si film were 4.00 − i0.10 and 1000 Å, respectively. Seven sets of theoretical (Δ, ψ) values were taken as input for incident angles from 66 to 72°. In this table, if there is a −0.008 deviation in the assumed value of n_s = 3.858, the calculated errors of n, k, and t values are 0.37, 1, and 0.5%, respectively. The deviation in the table which causes a large error is dt_{ox}. An error of t_{nox} = 20 Å made a 10% error in the calculated k values. Nevertheless, the errors caused by these deviations are acceptable if the refractive index and the thickness of the buried oxide are measured carefully and precisely before the MAI ellipsometry is applied.

Experiments and Results

Sample preparations.—With the results of the above theoretical analysis, the following experiments were accomplished. Many poly-Si/SiO_2/Si samples were prepared and annealed at different temperatures. Their optical constants and thicknesses were measured by MAI ellipsometry.

Sample preparation conditions.—Silicon wafers were first cleaned and oxidized in dry O_2 at 1000°C for 15 min. The thickness and the refractive index of the thin oxide layer were measured by ellipsometry. They were 213 Å and 1.46, respectively. Then a poly-Si film was deposited by low pressure chemical vapor deposition (LPCVD) at different temperatures from 550 to 625°C. The thickness of the deposited poly-Si was controlled in the range of approximately 1000 Å for each deposition temperature. The MAI ellipsometry was applied with varying incident angle from 66 to 72°. The wavelength of the incident beam was 6328 Å.

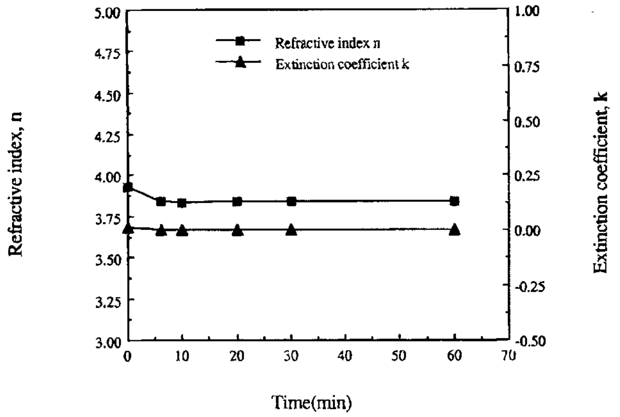

Fig. 7. The measured real and imaginary parts of the complex refractive index of a poly-Si film annealed at 1000°C in an N_2 ambient for 6, 10, 20, 30, and 60 min.

Before measurement, the samples were dipped with H_2O: HF = 50:1 to remove the native oxide.

Complex refractive indexes of the poly-Si deposited at different temperatures.—Figure 4 shows the measured complex refractive indexes of the LPCVD poly-Si films deposited at 550 and 625°C, where it can be seen that both n and k decreased with the increase in deposition temperature. Also, the n and k values nearly plateau when the deposition temperature reached 590°C. It was reported that poly-Si films deposited at temperatures <590°C were more like amorphous films and poly-Si films deposited at temperatures ≥590°C, were more like a polycrystalline structure.[21] In this study, the refractive indexes of the former condition were higher than that of single-crystalline Si value. For the latter condition, the complex refractive indexes reach that of single-crystalline Si at 6328 Å, 3.858 − i0.018.

High temperature annealing of amorphous silicon.—In a practical device fabrication process, poly-Si films are usually deposited at a low temperature (T_d < 590°C) in an amorphous phase and then annealed at a high temperature to crystalline phase with a large grain size.[22] The refractive indexes of poly-Si films deposited at low temperature (<590°C), and then annealed at high temperature were measured by MAI ellipsometry. Figure 5 shows the refractive indexes of the amorphous silicon deposited at 550°C and then annealed at 1000°C in N_2 for 6, 10, 20, 30, and 60 min. In this figure we see that both n and k values dropped quickly after annealing the film for 6 min, and they reach nearly steady state of constant values of 3.86 and 0.020, respectively, after annealing 20 min. It was expected that for 6 min annealing at 1000°C the film had transformed from an amorphous state into a crystalline one. Figure 6 shows transmission electron microscopy (TEM) photographs of the investigated film after it was annealed at 1000°C for 0, 6, 20, and 60 min, respectively. Figure 6a shows an electron diffraction pattern for the film deposited at 550°C indicating an amorphous structure but from Fig. 6b-d we see that the film was polycrystalline and also the grain size did not change as the film transformed to another polycrystalline phase after 6 min annealing. For comparison, the refractive indexes of poly-Si film deposited at 625°C and annealed at 1000°C for 6, 10, 20, 30, and 60 min also were measured by MAI ellipsometry and are shown in Fig. 7. The refractive indexes of the as-deposited samples were lower than that of the amorphous film shown in Fig. 5, and they essentially always rested at the same steady-state values of the poly-Si film.

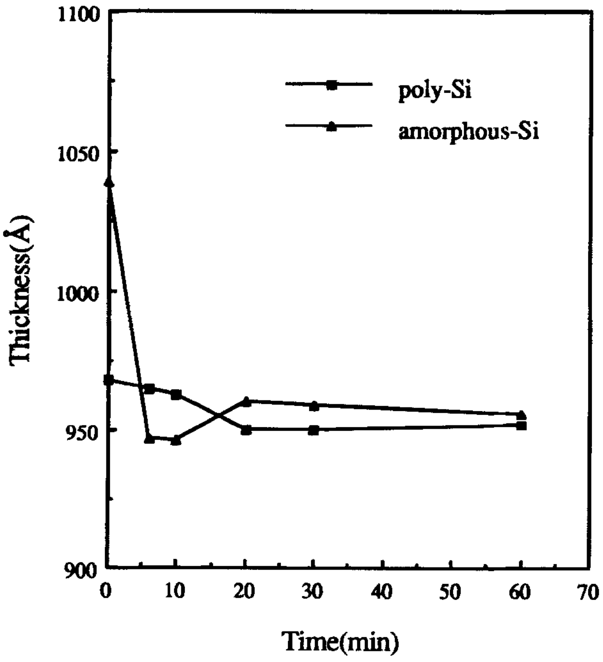

Fig. 8. Thickness variations of both the LPCVD amorphous silicon film and the poly-Si film annealed at 1000°C for 6, 10, 20, 30, and 60 min.

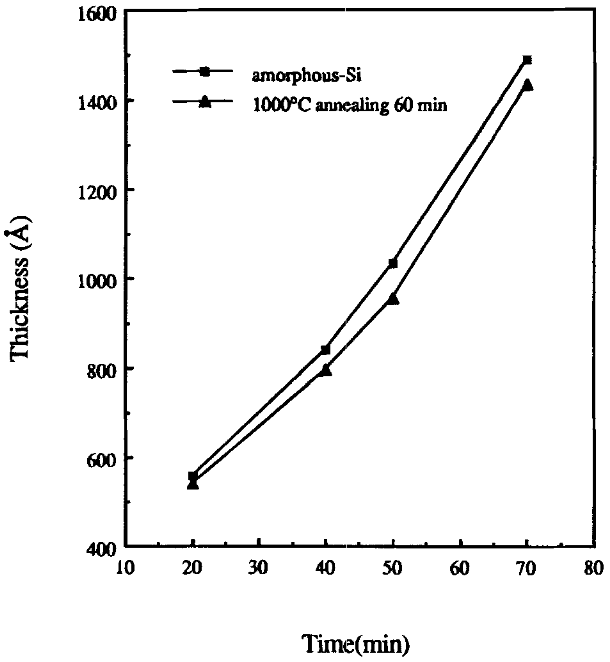

Fig. 9. Thickness variation of the different thicknesses amorphous silicon films deposited with different time after annealed at 1000°C for 60 min.

Figure 8 shows thicknesses of both the amorphous film and the as-deposited poly-Si film as they were annealed at 1000°C *vs.* time. For the amorphous film, the thickness decreased drastically at the initial 6 and 10 min annealings and then increased to reach a steady-state value of 956 Å. While, for the as-deposited poly-Si film, the thickness gradually decreased from 970 to 950 Å during the annealing process. Figure 9 shows another set of data of a group of amorphous films of different thicknesses with different deposition times after they were subjected to 1000°C annealing for 60 min. The average shrinkage on the thicknesses of these films was 3.5 to 6.0%.

Some samples were prepared for TEM photography to verify the thickness shrinkage. A cross-sectional view of the amorphous silicon/SiO_2/Si structure by LPCVD at 550°C is shown in Fig. 10a. The thickness of the amorphous silicon was estimated at 1120 ± 7 Å. Then the sample was annealed in an N_2 ambient at 1000°C for 60 min and shown in Fig. 10b. The thickness was estimated as 1066 ± 7 Å. The

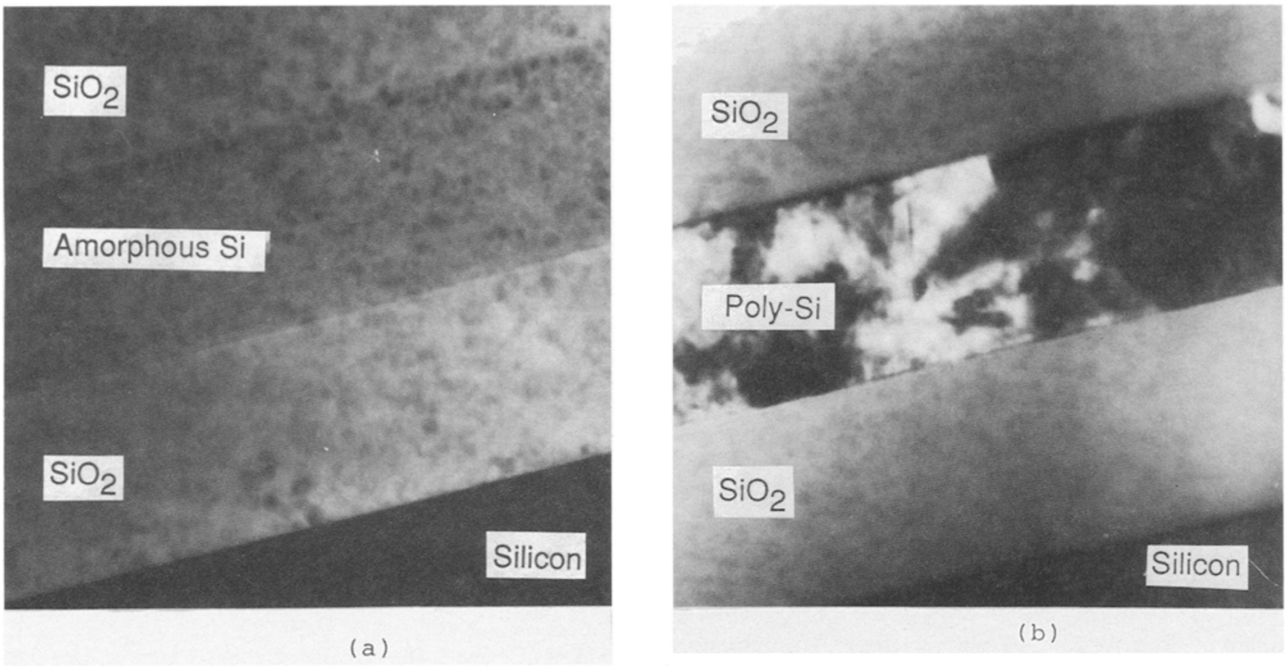

Fig. 10. (a) TEM photography of SiO_2/amorphous silicon/SiO_2/Si structure (300 K) and (b) TEM photography of SiO_2/poly-Si/SiO_2/Si structure (300 K).

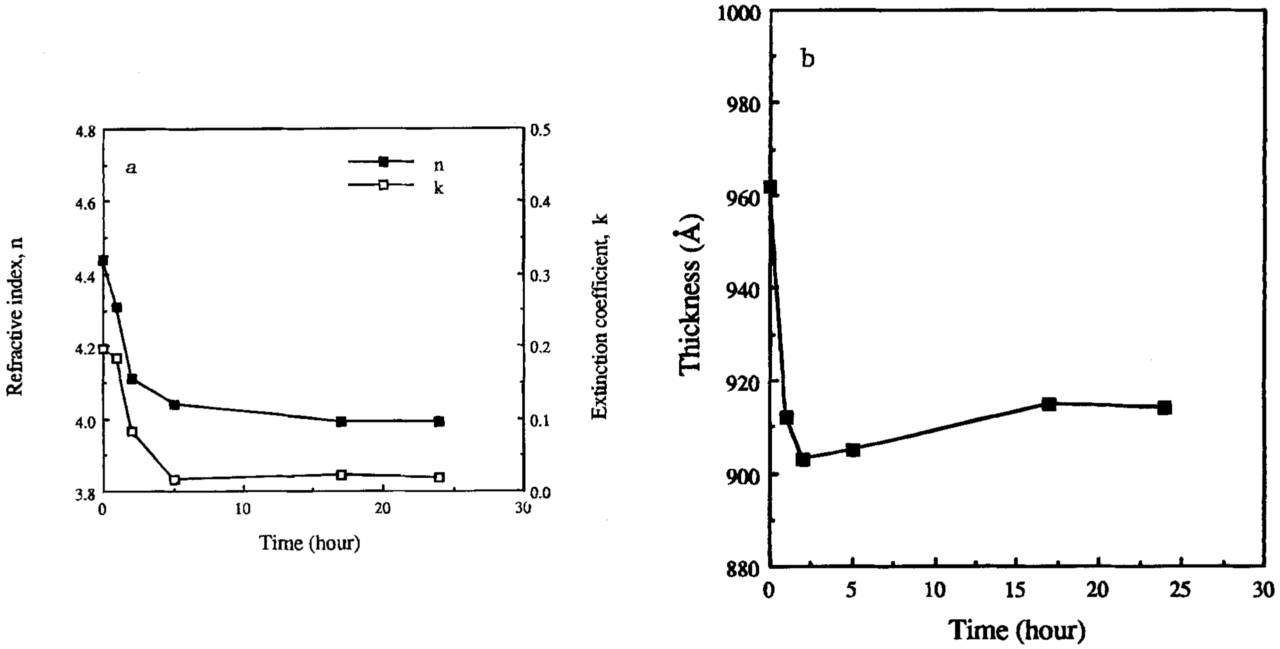

Fig. 11. (a) The optical constants *n* and *k* of the amorphous silicon film after annealing at 600°C for 0, 1, 2, 5, 17, and 24 h; (b) the thickness variation of the amorphous silicon film after annealing at 600°C for 0, 1, 2, 5, 17, and 24 h.

shrinking ratio of thickness was 4.8%. This was consistent with that determined by ellipsometry.

Low temperature annealing of amorphous silicon.—Poly-Si films may be obtained by annealing amorphous silicon at a temperature <600°C for a long time.[23] The strain and microstructure of these poly-Si films are better quality than those of films annealed at high temperatures. It is interesting to measure the complex refractive index and the thickness changes of these films under long-time low temperature annealing. Figure 11a and b shows the measured refractive index and thickness changes, respectively, of an amorphous film after annealing in N_2 at 600°C for 24 h. In Fig. 11a the refractive index of the amorphous film reached a steady-state value after annealing the film for 5 h. While, Fig. 11b exhibits an initial thickness shrinkage of the amorphous Si for annealing time up to 2 h, analogous to that of Fig. 8, and also the subsequent increase in thickness. The shrinkage ratio was also approximately 5.0%.

Conclusion

A MAI ellipsometer was used to study the optical properties of amorphous silicon and poly-Si after different heat-treatments. Initially, the error sensitivity of the MAI ellipsometry was studied to determine the optimum conditions for achieving the best precision of the MAI ellipsometry measurements. The optical properties of the amorphous silicon changed significantly when it was transformed to the polycrystalline form. Due to the high precision of the MAI ellipsometry, a thickness shrinking phenomenon was observed clearly during the measurement. This phenomenon was confirmed by TEM photography. The shrinkage ratio of thickness was 3.5 to 6.0%.

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Determination of Generation Lifetime in Trap-Rich and Layered Semiconductors by Metal-Oxide-Semiconductor Measurements

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ABSTRACT

The dispersion of metal-oxide semiconductor capacitance, $C(\omega)$, or of the normalized conductance, $G(\omega)/\omega$, is measured at a fixed inversion bias. A theory has been developed which relates the frequency f^* at which the step of the capacitance or the maximum of $G(\omega)/\omega$ occurs to the minority carrier generation lifetime. The technique can be applied to trap-rich and nonhomogeneously doped semiconductor material. We have used it for the special case of trap-rich n^-/n^+ amorphous silicon (a-Si). The resulting generation lifetime turns out to be $3.5 \cdot 10^{-13}$ s.

The minority carrier lifetime, $\tau_{n,p}$, of a semiconductor substrate is an essential parameter for the operation of a device made out of this substrate. Therefore its value is measured on silicon starting wafers on a routine basis. As a rule a metal oxide semiconductor (MOS) structure is formed on top of the substrate so that the generation lifetime τ_G, a value closely connected to the minority carrier lifetime, can be determined. The most common measurement procedure has been given by Zerbst,[1] which later appeared in many variants.[2] This procedure is subject to some restrictions which are of minor importance for the usual wafer applications. The technique fails, however, when, *e.g.*, the semiconductor layer is smaller than the equilibrium space-charge width in depletion or the transient of the pulsed C-V curve is faster than the capacitance recorder. In addition, for trap-rich semiconductors or layers containing sharp doping profiles no evaluation procedure exists. Recently we were led to some cases where we felt that a lifetime determination would be desirable and that these restrictions should be overcome. A typical example is nominally undoped (n^-) amorphous silicon whose surface has been implanted with phosphorus so that a shallow n^+ top layer is formed.

There have been a few attempts to deal with the above mentioned problems. Ferretti *et al.*[3] have extended the Zerbst technique to the cases of locally varying doping (and lifetime) profiles. Their work, however, is based on the as-

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