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Infrared spectroscopic ellipsometry study of sulfur-doped $In_{0.53}Ga_{0.47}As$ ultra-shallow junctions

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Sulfur mono-layer doped $In_{0.53}Ga_{0.47}As$ films were investigated by infrared spectroscopic ellipsometry. The complex dielectric function of doped layers shows free carrier response which can be described by a single Drude oscillator. Electrical resistivities, carrier relaxation times, and active carrier depths are obtained for the shallow n-$In_{0.53}Ga_{0.47}As$ films. Our results indicate that sub-10 nm sulfur-doped layers with active carrier concentration as high as $1.7 \times 10^{19}\,cm^{-3}$ were achieved. Sheet resistances estimated from infrared spectroscopic ellipsometry are in good agreement with those obtained by electrical methods. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4882917]

InGaAs has high electron mobility and is a promising channel material to replace silicon in future n-channel metal-oxide-semiconductor field-effect transistors (MOSFETs).[1,2] Sub-10 nm junctions will be required in the source and drain (S/D) regions and extensions of nanoscale InGaAs channel FETs. A damage-free and uniform doping process that provides high doping concentration levels is desired. Also, three-dimensional (3D) structures such as fin-type field-effect transistors (finFETs) and nanowires would require a conformal doping technique to form the S/D or extension regions. These requirements may not be achieved with the conventional ion implantation technique. Silicon, which is commonly used to dope InGaAs, could lead to either n- or p-type doping, due to its amphoteric nature in InGaAs. This can be avoided by using sulfur as the dopant. It is known that a sulfur-containing solution such as ammonium sulfide $(NH_4)_2S_x$ provides effective surface passivation of III–V materials such as GaAs and InGaAs.[3–5] Chemisorbed sulfur atoms resulting from $(NH_4)_2S_x$ passivation can be used as a source of sulfur to dope GaAs and InAs n-type.[6,7] This technique is attractive due to its simplicity and suitability for commercial applications. Recently, n-InGaAs ultra-shallow junctions formed using sulfur monolayer doping (SMLD) technique were reported.[8–10] A prototype n-channel MOSFET using these shallow junctions in the S/D regions was demonstrated.[10]

Electrical techniques, such as four-point-probe (4PP) measurement, transmission line measurement (TLM), and Hall effect measurements, are routinely used for extracting sheet resistance R_s and active carrier concentration N of doped layers in semiconductors. However, sub-10 nm shallow junctions can be mechanically damaged by probe tips during 4PP measurement, leading to erroneous results.[11] Furthermore, TLM and Hall measurements in a doped layer could be inaccurate if the underlying layers in the heterostructures are conducting resulting in poor current confinement in the doped region of interest. Similarly, it should be pointed out that defining the carrier junction depth for ultra-shallow junctions in InGaAs using the chemical profile obtained from secondary ion mass spectrometry (SIMS) may not be very accurate. The contactless and non-destructive nature of infrared spectroscopic ellipsometry offers an alternative to overcome these issues. Infrared ellipsometry has been used for the electrical and physical characterization of complex layered semiconductor structures.[12] The presence of active carriers in the material modifies its optical response in the infrared range. The infrared dielectric function of a doped layer is dominated by free carrier absorption from which parameters such as electrical resistivity ρ, carrier relaxation time τ, and N can be determined using a classical Drude model.[13] The thickness of the doped layer t obtained from ellipsometry could be related to the junction depth. R_s is calculated from ρ and t of the doped layer. Additionally, knowledge of the effective mass of the band involved in conduction may allow us to extract the doping profile.[13,14] Infrared ellipsometry was applied to characterize sub-100 nm Si junctions formed by excimer laser annealing on bulk Si and Si-on-insulator.[15] R_s values for junction depths down to ~30 nm were reported.

In this Letter, we report the optical characterization of n-$In_{0.53}Ga_{0.47}As$ layers formed using SMLD process, by infrared spectroscopic ellipsometry from 0.05 to 0.65 eV. Electrical parameters of the doped layers were obtained from modeling the dielectric response with the Drude-like free carrier response. The results reveal that SMLD technique can dope the InGaAs with doping concentration in the order of $1.5 \times 10^{19}-1.7 \times 10^{19}\,cm^{-3}$. Our study shows that infrared spectroscopic ellipsometry may be used to characterize heavily doped ultra-thin $In_{0.53}Ga_{0.47}As$ films.

The starting substrates consisted of a p-type $In_{0.53}Ga_{0.47}As$ film with a doping level of $2 \times 10^{16}\,cm^{-3}$ epitaxially grown on heavily doped p-type InP substrates. The surface of the samples was cleaned with a mixture of hydrochloric acid (HCl) and H_2O in the ratio 1:3 to remove the native oxide prior to SMLD. A solution of 20% $(NH_4)_2S_x$ in H_2O was used for SMLD.[10] P-$In_{0.53}Ga_{0.47}As$ films were

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treated with the $(NH_4)_2S_x$ solution for 30 min at room temperature and capped with 20 nm SiO_2. In another set of samples, phosphorus pentasulfide (P_2S_5) was added to the $(NH_4)_2S_x$ solution[16,17] for the SMLD process step. This was followed by rapid thermal annealing of the samples from 550–600 °C to form n-$In_{0.53}Ga_{0.47}As$ films. The SiO_2 capping layer was then stripped using an HF solution. The doping concentration and the junction depth were tuned by varying the annealing temperature and the duration of annealing. Atomic force microscopy (AFM) showed that the root mean square (RMS) roughness of the InGaAs surface was between 0.3–0.8 nm after the SMLD process. The chemical profiles of the SMLD samples annealed at various temperatures were obtained using SIMS. Fig. 1 shows sulfur profiles for the SMLD samples along with an n-type reference $In_{0.53}Ga_{0.47}As$ film with a Si dopant concentration of 1.1×10^{19} cm^{-3}. A sharp sulfur profile is observed in the SMLD samples within the first 10 nm from the surface of the film. The profile for samples annealed at 600 °C have a longer tail extending up to ∼85 nm for S concentration above 1×10^{18} cm^{-3}, whereas it is ∼30 nm for the two SMLD samples annealed at 550 °C. This suggests that the samples annealed at 550 °C may exhibit shallower carrier junction depth compared to those annealed at 600 °C.

Spectroscopic ellipsometry measurements were carried out at room temperature using an infrared variable angle spectroscopic ellipsometer (IR-VASE) manufactured by J. A. Woollam Co. The ellipsometric angles Ψ and Δ were acquired at an angle of incidence of 65° from 0.05 to 0.65 eV. Initially, *in situ* Si doped n-type $In_{0.53}Ga_{0.47}As$ films with various thicknesses were characterized. The samples were modeled as a four-layer system containing an InP substrate, p-$In_{0.53}Ga_{0.47}As$ film, the n-$In_{0.53}Ga_{0.47}As$ film and a surface layer. The surface layer was modeled as a thin film consisting of 50% n-$In_{0.53}Ga_{0.47}As$ and 50% voids in the Bruggeman approximation.[18] The InP substrate and the lightly doped p-$In_{0.53}Ga_{0.47}As$ film used in growth experiments were characterized separately and the dielectric function obtained was used in tabulated form in the model. The complex dielectric function of n-$In_{0.53}Ga_{0.47}As$ film was described using an optical dispersion model given by[19]

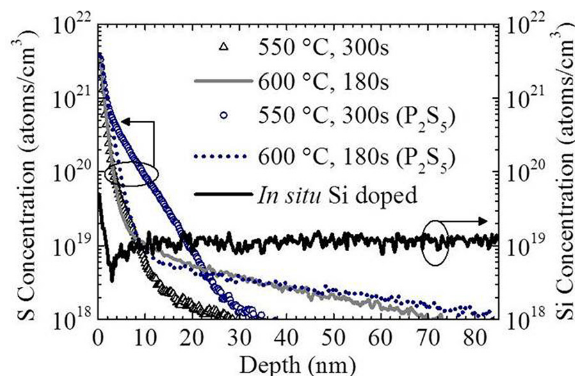

FIG. 1. SIMS profiles of SMLD samples annealed at various temperatures and time intervals. SIMS profile of an *in situ* Si doped reference n-$In_{0.53}Ga_{0.47}As$ film with a doping concentration of 1.1×10^{19} cm^{-3} is also shown. The SMLD samples annealed at 550 °C (dotted curves) appear to have a shallower junction depth compared to the samples annealed at 600 °C (solid curves) for S concentration above 1×10^{18} cm^{-3}.

$$\varepsilon(E) = C + \frac{-\hbar^2}{\varepsilon_0 \rho(\tau E^2 + i\hbar E)} + \frac{A_{pole}}{(E_{pole}^2 - E^2)}, \qquad (1)$$

where C is a fitting parameter that is independent of energy. The second term is related to the Drude lineshape that describes the free electron behavior in the conduction band. ρ and τ are related to the plasmon energy $E_P = \hbar\sqrt{\frac{1}{\varepsilon_{opt}\varepsilon_0\rho\tau}} = \hbar\sqrt{\frac{Ne^2}{\varepsilon_{opt}\varepsilon_0 m^*}}$, where $\varepsilon_{opt} = C + \frac{A_{pole}}{E_{pole}^2}$. The third term in Eq. (1) accounts for the dispersion caused by absorption outside the measured spectral range. $A_{\rm pole}$ is the magnitude of the pole and $E_{\rm pole}$ is the pole energy. The thicknesses of p-$In_{0.53}Ga_{0.47}As$ and n-$In_{0.53}Ga_{0.47}As$ and all the adjustable parameters in the model were fitted by a least-square procedure which follows the Levenberg-Marquardt algorithm.[20] Since the infrared wavelengths are not sensitive to the surface, the thickness of the surface layer was kept fixed at $\sim 2\sqrt{2}$ times the RMS value from AFM measurements.[21] We found that neglecting the surface layer from the model introduces an error of up to 20% in thickness and resistivity values of ∼3–4 nm shallow junctions. As a result, up to 6% error in R_s can be observed.

The model dielectric function of n-$In_{0.53}Ga_{0.47}As$ films is shown in Fig. 2. The SMLD $In_{0.53}Ga_{0.47}As$ samples show curves similar to the 35 nm thick *in situ* Si doped film indicating that their optical response is also dominated by the free carrier response. We verified using a point-by-point fit[19] that the model dielectric function is a realistic representation of the true dielectric function of n-$In_{0.53}Ga_{0.47}As$. The imaginary part of pseudo-dielectric function $\langle\varepsilon_2\rangle$ is shown in the inset of Fig. 2(b). Although the signal from p-$In_{0.53}Ga_{0.47}As$ film dominates the pseudo-dielectric function, we can still distinguish the ultra-thin SMLD sample from the starting substrate in the energy range below 0.2 eV where the free carrier response is observed. The pseudo-dielectric function corresponding to SMLD samples shifts upward as a thicker S-doped layer is formed on the starting substrate.

The parameters obtained from our fits are summarized in Table I. Fig. 3 shows the ρ, τ, and t of the heavily doped

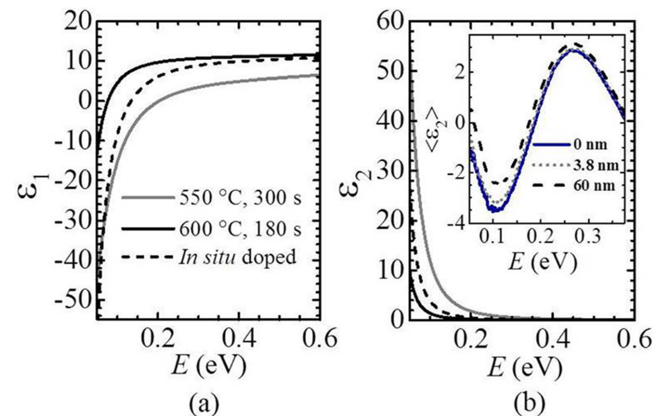

FIG. 2. (a) Real and (b) imaginary parts of the complex dielectric function of n-$In_{0.53}Ga_{0.47}As$ and an *in situ* Si doped reference n-$In_{0.53}Ga_{0.47}As$ film. The legend applies to both plots. The inset in (b) shows the imaginary part of pseudo-dielectric function of SMLD samples along with the starting substrate. The 3.8 nm SMLD sample has an active concentration of 1.7×10^{19} cm^{-3}, whereas the 60 nm SMLD sample has an active concentration of 3.8×10^{18} cm^{-3}.

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TABLE I. Electrical and physical parameters for SMLD and *in situ* Si doped n-$In_{0.53}Ga_{0.47}As$ samples. N and μ are obtained by assuming an effective electron mass of 0.074 m_e.

Sample number	Type	Annealing condition	ρ (mΩ-cm)	τ (fs)	t (nm)	N (10^{19} cm^{-3})	μ (cm^2 V^{-1} s^{-1})
1	*In situ* Si doped		0.729	33.2	35.2	1.1	790
2	*In situ* Si doped		0.533	43.9	45.0	1.1	1044
3	*In situ* Si doped		0.448	52.3	56.6	1.1	1242
4	*In situ* Si doped		0.375	62.3	86.6	1.1	1481
5	*In situ* Si doped		0.320	72.4	106.7	1.1	1720
6	$(NH_4)_2S_x$	550 °C, 300 s	1.46	11.9	2.7	1.5	282
7	$(NH_4)_2S_x$	600 °C, 180 s	2.17	32.4	56.6	0.37	770
8	$P_2S_5/(NH_4)_2S_x$	550 °C, 300 s	1.46	10.3	3.8	1.7	245
9	$P_2S_5/(NH_4)_2S_x$	600 °C, 180 s	2.17	33.1	60	0.38	787

n-$In_{0.53}Ga_{0.47}As$ films after SMLD. N and mobility μ are calculated from ρ and τ by using an effective conduction mass $m^* = 0.074\, m_e$.[22] N and τ are plotted as a function of t in Fig. 4. ρ for the SMLD samples is higher than the *in situ* Si doped samples. The samples annealed at 600 °C have larger t values compared to those annealed at 550 °C which is consistent with the SIMS trend. Also, the comparison of chemical profiles for the two SMLD samples annealed at 550 °C suggests that the one which was formed with P_2S_5 may possess larger active carrier depth. This appears to be corroborated by the measured t values. We estimated the active carrier dose from ellipsometry and the total sulfur dose from SIMS. The SMLD samples formed with $P_2S_5/(NH_4)_2S_x$ appear to show higher sulfur dose than the samples formed with $(NH_4)_2S_x$.[23] The activation efficiency at 600 °C (~5.7%) is about 3 times higher than the activation efficiency at 550 °C (~1.7%). We note that the activation efficiency achieved in our experiments is comparable to that achieved in SMLD InGaAs annealed at 700 °C for 30 s.[8]

τ for both SMLD and *in situ* Si doped samples appear to be strongly correlated to t. As thickness reduces, τ for the SMLD samples decreases. The values extracted are similar to the reference *in situ* samples with comparable thicknesses. Note that N is nearly constant at $\sim 1.1 \times 10^{19}$ cm^{-3} as expected for an *in situ* Si doped sample which was thinned down to 35 nm from 107 nm. Our sub-10 nm n-$In_{0.53}Ga_{0.47}As$ junctions also possess similar active carrier concentration of $\sim 1.5 \times 10^{19} - 1.7 \times 10^{19}$ cm^{-3}. The N and t obtained are similar to that reported by Kort *et al.*[24] Furthermore, the corresponding τ values appear to fall on the line extrapolated from the thicker reference samples.

R_s obtained from electrical measurements using the micro-four-point-probe (μ4PP) setup and TLM structures are compared with that obtained from infrared ellipsometry measurements in Fig. 5(a). It can be observed that the R_s of SMLD samples increases as t decreases despite the fact that N is higher in the ultra-shallow doped films. R_s of sub-10 nm doped films is ~3–5 kΩ/□. These are higher than the target set by ITRS. The trend of R_s decreasing with thickness is also observed in the *in situ* Si doped reference samples and can be expressed as

$$R_s \sim 8 + (2.4 \times 10^5) t^{-2}. \qquad (2)$$

This is shown in Fig. 5(b). Equation (2) predicts R_s values in kΩ/□ for sub-10 nm thick *in situ* Si doped n-$In_{0.53}Ga_{0.47}As$ films containing an active carrier concentration of 1.1×10^{19} cm^{-3}. The t^{-2} dependence of R_s in the SMLD samples is similar to that found in thin metallic films.[25] We note that the trend in R_s observed in our SMLD samples is identical to that found in sub-10 nm doped Si layers.[26]

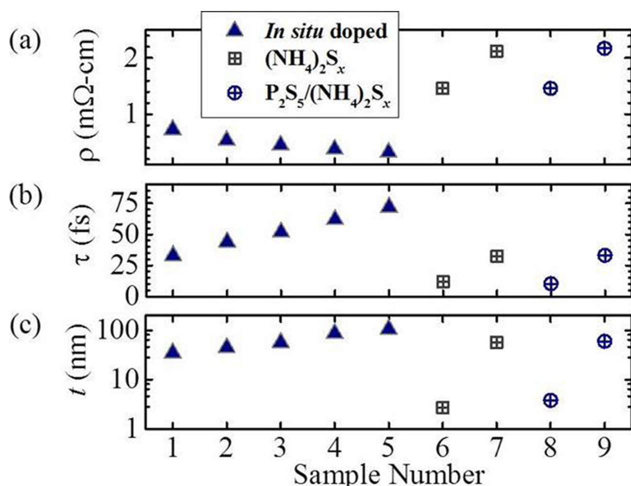

FIG. 3. Electrical and physical parameters of n-$In_{0.53}Ga_{0.47}As$: (a) resistivity, (b) relaxation time, and (c) film thickness of SMLD and *in situ* Si doped samples. The legend applies to all plots.

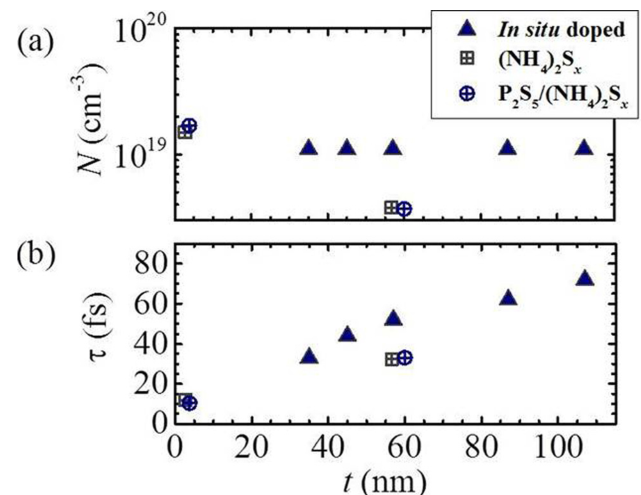

FIG. 4. Thickness dependence of (a) active carrier concentration N and (b) relaxation time τ for SMLD and *in situ* Si doped n-$In_{0.53}Ga_{0.47}As$ samples.

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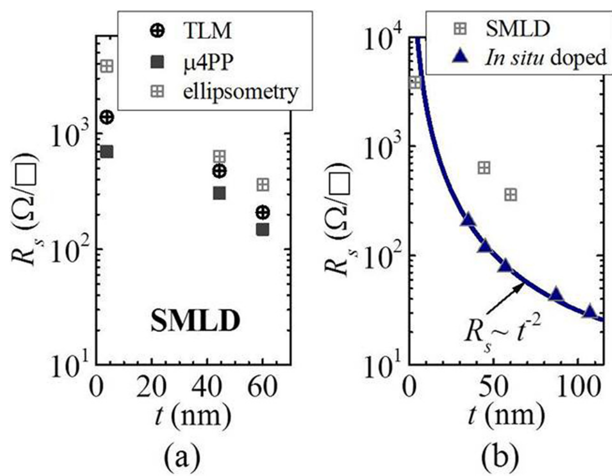

FIG. 5. (a) Comparison of sheet resistance R_s obtained by electrical and optical methods for SMLD $In_{0.53}Ga_{0.47}As$. (b) Thickness dependence of R_s obtained from ellipsometry for SMLD and *in situ* doped n-$In_{0.53}Ga_{0.47}As$ films. The solid curve is a fit with Eq. (2) to R_s values for *in situ* Si doped reference samples.

Equation (2) captures the essence of the Fuchs model to explain the dependence of R_s on t.[26] Our results indicate that N higher than $1.7 \times 10^{19}\,cm^{-3}$ in the ultra-thin SMLD samples would be needed to accomplish R_s below 1 kΩ/□ to meet the ITRS target. We are working towards optimizing the SMLD process and annealing conditions to enhance the active carrier concentration in our samples.

Of the two electrical methods, TLM shows better agreement with the ellipsometry values, whereas the μ4PP method seems to underestimate R_s values with respect to both TLM and ellipsometry. The agreement between the electrical and optical methods deteriorates as the doped regions get shallower with the μ4PP method underestimating R_s for ultra-shallow films with respect to TLM and ellipsometry. The discrepancy between ellipsometry and TLM results may be reduced by introducing active dopant profiles in the ellipsometry model. A systematic study is ongoing to detect the true active carrier concentration profile in our SMLD samples. The R_s values obtained by infrared ellipsometry with uniform doping profile are in good agreement with those reported by Loh *et al.*[9] We propose that the relevant parameters required in evaluating electrical properties of sub-10 junctions can also be investigated by infrared ellipsometry in a non-contact and non-destructive manner.

In conclusion, SMLD $In_{0.53}Ga_{0.47}As$ was studied by infrared spectroscopic ellipsometry. The optical response of n-$In_{0.53}Ga_{0.47}As$ films can be described with a Drude-like free carrier response from which electrical resistivity and carrier relaxation time are obtained. The results confirm that the monolayer doping technique results in ultra-shallow junctions ~3–4 nm with doping level as high as $1.7 \times 10^{19}\,cm^{-3}$. Infrared spectroscopic ellipsometry could be an alternative for simultaneous characterization of electrical and physical properties of heavily doped ultra-thin films for future technology nodes.

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