Characterization of tellurium and silicon as n-type dopants for GaAsBi

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Abstract
Films of n-GaAs$_{1-x}$Bi$_x$ films were grown via molecular beam epitaxy using both Si and Te as dopant sources. Electron mobility was characterized by Hall effect measurements as a function of carrier concentration and Bi content for films with bismuth fractions of $x = 0.02$ and $x = 0.06$. While GaAsBi:Te shows lower majority carrier mobility than GaAsBi:Si at low Bi concentrations, the two become comparable as Bi content increases. Furthermore, it was observed that in the presence of bi-metallic Bi-Ga droplets on the film surface, films doped with Si display p-type behavior, likely due to Si preferentially occupying group-V sites. The use of Te as a dopant always resulted in n-type epilayers, making it a more reliable dopant choice for high Bi content films. Finally, ex situ annealing was studied as a method to improve majority carrier mobility in GaAs$_{0.98}$Bi$_{0.02}$:Te films, with a 10 min anneal at 350 °C resulting in a 30% improvement in electron mobility. Improvement of film quality was confirmed through spectroscopic ellipsometry examination of film optical properties. Annealing at higher temperatures resulted in electrical, optical, and structural degradation of the GaAsBi films.

Keywords: GaAsBi, molecular beam epitaxy, Hall measurements, ellipsometry

(Some figures may appear in colour only in the online journal)

1. Introduction
GaAs$_{1-x}$Bi$_x$ has garnered interest in recent years for use in infrared (IR) applications due to the dramatic effect of Bi incorporation on III–V band structure. The interaction between the Bi impurity state and the host valence band edge results in a significant band gap reduction [1]—up to 84 meV/%Bi for dilute concentrations [2]. Bi incorporation may also reduce the temperature dependence of the band gap [3] and cause significant spin-orbit splitting, which for sufficiently high concentrations can lead to reduced Auger recombination [4]. Together, these make GaAs$_{1-x}$Bi$_x$ well suited for use as the active layer for near- and mid-infrared optoelectronic devices, such as in a thermophotovoltaics [5] multijunction solar cells [6–8], LEDs [9–11], or in IR lasers [4, 12]. To optimize the design of these devices, the properties of doped materials must be characterized electrically and optically.

As the Bi impurity state in III–V alloys is thought to primarily affect the valence band [1], the electronic properties of p-type GaAsBi have been studied in great detail. Dopants such as carbon [13, 14] and beryllium (Be) [14–16] have been explored and their resulting impact on hole mobility has been measured. Additionally, previous work in the literature has shown that the p-type conductivity of unintentionally doped (UID) GaAs$_{1-x}$Bi$_x$ films increases with increasing Bi content due to the acceptor-like nature of Bi-Bi pairs or clusters that form in the films [17, 18]. While some groups have reported
The n-type behavior of GaAsBi has been studied primarily with silicon (Si) as the dopant source. Electron mobility of GaAsBi:Si has been shown to be stable up to \( x = 0.013 \), but it degrades rapidly at higher Bi fractions [22]. The measured donor concentration in GaAsBi decreases at higher Bi content due to increased compensation for the natural acceptor-like states in undoped GaAsBi [21, 23, 24]. Additionally, as silicon is a group-IV element, its behavior as an amphoteric dopant in the GaAs(Bi) system can lead to n-type or p-type behavior depending on the V/III flux ratio [25]. While GaAsBi:Si films that exhibited p-type behavior demonstrated electrical properties in line with GaAsBi:Be/C films, the necessity of near-stoichiometric growth conditions for high Bi-content GaAsBi films could lead to p-type or compensated doping behavior when attempting to grow Si doped n-type films for device purposes.

As an alternative to Si for n-type GaAsBi, we explored the use of a group-VI dopant, tellurium (Te). Initial work on gas source MBE has shown that Te can provide comparable electron mobilities as Si in GaAs films under conventional growth conditions: 580 °C, high V/III ratio [26]. However, the relatively high growth temperatures in GaAsBi growth can lead to undesirable GaTe microcomposites forming in the epilayers [27]. In the GaSb-based alloy system, group-IV n-type dopants are typically not used because the lower V/III ratio necessary for high quality films [28] can result in Si incorporating on the group-V site rather than the group-III site [29]. As the growth conditions of GaAsBi are more akin to GaSb than GaAs, low growth temperatures and near-stoichiometric V/III ratios [30], it is possible that Te is a more suitable n-type dopant for this material system.

In this paper, we report the room temperature mobility trends for GaAsBi doped with Te, Si, and Be as a function of both dopant incorporation and Bi fraction. The bismide compositions (band gaps) studied in this work were \( x = 0.02 \) (1.25 eV) and \( x = 0.06 \) (1.04 eV), which are suitable for subjunctions of a multijunction solar cell or for near-IR lasers. The mobility of GaAsBi:Te was found to be lower than GaAsBi:Si for low Bi compositions. At higher Bi fractions, the mobility of GaAsBi:Te and GaAsBi:Si were comparable. However, Te performed better as an n-type dopant over Si in the presence of Ga-Bi surface droplets, which induced amphoteric behavior of the Si dopant. The mobility and resistivity of GaAsBi_{0.02}:Te films was improved up to 30% by rapid thermal annealing at 350 °C for 10 min.

2. Experimental details

Samples were grown by molecular beam epitaxy (MBE) on a Veeco GENxplor system. The group-III elements and the Bi were supplied by a solid-source effusion cells and a valved cracker source was used to provide a flux of As\(_4\). The dopant sources were elemental Si, elemental Be, and GaTe. Beam equivalent pressure (BEP) measurements were taken before film growth using a retractable ion gauge calibrated to N\(_2\). The temperature of the substrate during growth was measured using band-edge thermometry with a kSA BandiT system. The surface was monitored using a Staib reflection high-energy electron diffraction (RHEED) system.

Epilayers were grown on 1/4 pieces of 2° epi-ready (001) oriented UID GaAs, loaded into the MBE chamber after a 4-hour load lock bake at 180 °C. The wafer was heated to 620 ± 5 °C under As\(_4\) overpressure to remove the native oxide before an UID buffer was grown at \( \sim 0.45 \) ML s\(^{-1}\). For GaAsBi samples grown on GaAs, the buffer layer was 500 nm of UID GaAs grown at 580 ± 5 °C with a (2 × 4) surface reconstruction. For samples grown on partially relaxed InGaAs underlayers, used to lattice match the GaAsBi alloy and prevent Bi rejection under high compressive strain [31], 100 nm of GaAs was first grown at 580 ± 5 °C before 500 nm of InGaAs was grown at 475 ± 5 °C with a (4 × 3) surface reconstruction. The Ga\(_{1-x}\)As composition was chosen to lattice match the capping GaAsBi layer. The relaxed growth rate for the ternary buffer layer ranged from 0.5–0.6 ML s\(^{-1}\) depending on the composition.

After buffer growth, the substrate was cooled to 260 ± 12 °C at 30 °C s\(^{-1}\) and the As\(_4\) flux was adjusted to achieve an As\(_4\)/Ga BEP ratio of \( \sim 12.5 \). Based on (2 × 4) to (4 × 2) RHEED transitions at conventional GaAs growth temperatures, this As\(_4\)/Ga BEP ratio was near the stoichiometric point for high-temperature GaAs growth. For GaAsBi films grown on InGaAs, the substrate thermocouple temperature corresponding to the desired BandiT temperature for film growth was determined before InGaAs film growth. This was done because the presence of a 500 nm InGaAs buffer layer was found to affect the temperature measured by BandiT and, thus, prevents accurate growth temperature determination. GaAsBi growth was initiated by opening the Ga and Bi shutters simultaneously. The layers were grown at \( \sim 0.45 \) ML s\(^{-1}\) based on GaAs RHEED intensity oscillation growth rate measurements. GaAsBi films exhibited a (1 × 3) surface reconstruction during droplet-free growth. For samples grown on GaAs buffers, the entire GaAsBi layer was doped. For samples grown on InGaAs buffer layers, 50 nm of UID GaAsBi was grown before the doped layer. The Bi fraction was tuned by adjusting the Bi flux and holding all other growth parameters constant: As\(_4)/Ga\) ratio, growth rate, substrate temperature. Doped GaAsBi_{0.02} films were grown to 500 nm and doped GaAsBi_{0.06} films were grown to 250 nm. These thickness limitations were chosen to avoid Bi-rejection and droplet formation.

After samples were removed from the MBE system, differential interference contrast optical microscopy (DIC/Nomarksi) was utilized to check for the presence/absence of Ga-Bi droplets on the sample surface. High resolution x-ray diffraction (HRXRD) was performed on a Bruker D8 and data was simulated using the Bruker Leptos software. Bi fraction was determined by simulations of collected 2θ-ω 004 line scans and 224 reciprocal space maps.
Vegard’s law was used to simulate the GaAsBi films in Leptos, assuming the GaBi lattice constant was 0.633 nm and the elastic constants were the same as GaAs at low Bi concentrations [2].

Optical properties were measured using a J.A. Woollam variable angle spectroscopic ellipsometer (VASE) and optical data was analyzed using the J.A. Woollam WVASE software. Samples were modeled as a three-layered stack comprised of a GaAs substrate, a generalized oscillator (genosc) layer to capture the optical properties of the GaAsBi film, and a surface layer used to account for the effects of roughness and a surface oxide. The genosc layer was comprised of oscillator models which were tuned to fit the absorbing features of the ellipsometric data while maintaining Kramers-Kronig consistency between the complex and real portions of the dielectric function. Oscillator models were specifically assigned to the absorption features indicating the interband critical points $E_0$, $E_0 + \Delta_0$, $E_1$, and $E_1 + \Delta_1$ which have been shown to be visible in ellipsometry data [32]. Modeling of the surface roughness and oxide was achieved by using a layer composed of 50% underlying GaAsBi film optical properties and 50% void in a Bruggeman effective medium approximation as described in [32]. Once GaAsBi layers were modeled using the genosc layer, the optical absorption coefficient of the layer was calculated using the modeled GaAsBi index of refraction, $n$, and extinction coefficient, $k$.

The absorption coefficient of the GaAsBi layer was extracted from the ellipsometry data and used to estimate the band gap energy of the semiconductor and confirm alloy composition, similarly to the method described in Masnadi-Shirazi et al [33]. A linear fit to the square of the absorption coefficient near the film band-edge is extended to the energy axis. This gives an approximate measurement of the band gap. The band gap can be translated into an approximate Bi fraction using an experimentally derived relationship [33] and estimations agree with the HRXRD simulations within 4% on average.

For electrical transport measurements, samples were cleaved into squares and Ohmic contacts were made by soldering indium metal onto the four corners. N-type GaAsBi with $1 \times 10^{18}$ cm$^{-3}$ required an additional rapid thermal anneal at 400 °C for 30 s to ensure the contact would be ohmic. Using a Lake Shore Cryotronics Hall effect system, van der Pauw and Hall effect measurements were used to extract the extrinsic carrier concentration and majority carrier mobility. Samples were excited with 0.9 mA of current in a SSI Inc. rapid thermal processing system using quartz lamps in an N$_2$ environment. Annealing temperatures from 300 °C–500 °C were explored in this work and all samples were annealed for 10 min. Hall specimens were prepared after annealing.

3. Results

Figure 1 shows GaAsBi$_{0.02}$ majority carrier mobility trends as a function of dopant incorporation for the three dopants studied in this work. The right axis shows hole mobility in p-type GaAsBi$_{0.02}$:Be and the values are in line with other authors who have investigated p-type GaAsBi [14]. The extremely low hole mobility in GaAsBi arises from the valence band perturbation by the Bi-impurity state, similar to what is seen in the electron mobility of dilute nitride semiconductors due to perturbation of the conduction band [35]. Hole mobility was measured over a wide range of p-type doping concentrations, $1 \times 10^{16}$–$1 \times 10^{19}$ cm$^{-3}$, and decreased as dopant incorporation increased due to scattering from the ionized impurities.

The electron mobility in n-type GaAsBi$_{0.02}$ doped with Si and Te is shown on the left axis of figure 1. For all measured doping values, the electron mobility of GaAsBi$_{0.02}$:Si was higher than that of GaAsBi$_{0.02}$:Te; the maximum mobility of GaAsBi$_{0.02}$:Si was 2060 cm$^2$ V$^{-1}$ s$^{-1}$ with $N_D = 5.6 \times 10^{17}$ cm$^{-3}$ while the maximum mobility of GaAsBi$_{0.02}$:Te was measured to be 1710 cm$^2$ V$^{-1}$ s$^{-1}$ with...
N\textsubscript{D} = 1.7 \times 10^{17} \text{ cm}^{-3}. For both dopant types, dopant incorporations less than 1 \times 10^{17} \text{ cm}^{-3} could not be achieved, indicated by the gray region in figure 1. Lowering the Si or Te cell temperature to achieve <1 \times 10^{17} \text{ cm}^{-3} based on a previously derived Arrhenius relationship between the cell temperature and the measured dopant incorporation. The GaAs:Te sample grown under conventional GaAs conditions (580 °C and high V/III flux ratio) exhibited a measured donor concentration of 4 \times 10^{16} \text{ cm}^{-3}, while the GaAsBi:Te sample was again too resistive to measure. As it is unlikely that fewer Te atoms incorporated into the GaAsBi film at its much lower growth temperature, this may indicate that the donors were being compensated by the grown-in p-type defects in GaAsBi at this Bi concentration.

It has been shown previously in the literature that GaAsBi films are intrinsically p-type. Pettinari et al. showed that the excess hole concentration in their GaAsBi films increased with Bi incorporation [19]. However, for their extremely thin films (1 = 30-56 nm), their trends show excess hole concentrations of only \sim 1 \times 10^{14} \text{ cm}^{-3} for films of x = 0.02. The data shown in figure 1 indicates that the excess hole concentration is as high as \sim 1 \times 10^{17} \text{ cm}^{-3} at x = 0.02 for 500 nm thick films. These values are more in line with the work of Zhu et al., who grew films of x = 0.037 with an unintentional acceptor concentration of N\textsubscript{A} = 2.8 \times 10^{17} \text{ cm}^{-3} at thicknesses of 450 nm [20].

Mobility as a function of Bi incorporation was studied at a n-type carrier concentration of \sim 2-5 \times 10^{18} \text{ cm}^{-3}. There was a small spread in the measured dopant concentration, as increasing the Bi concentration required retargeting the n-type dopant cell to overcome changing levels of compensating p-type defects. Samples were compared to GaAs grown under conventional growth conditions: 580 °C and high V/III flux ratio. Figure 2 shows the mobility of GaAsBi decreased as a function of Bi concentration for both Si and Te dopants. This is in line with what has been shown previously in the literature for Si, Be, and C dopants [14, 22]. Select samples, shown as black icons, were grown lattice matched to partially relaxed InGaAs buffer layers. This prevents significant strain relaxation at higher Bi concentrations and also prevents droplet phase separation due to Bi saturation [31]. A test sample with a concentration of x = 0.02 shows the mobility of n-type samples grown under small levels of compressive strain on GaAs and lattice matched to InGaAs underlayers were nearly the same. The dotted lines in figure 2 are guides to the eye, rather than a fit to the experimental data.

As figure 2 shows, the mobility of GaAsBi:Te is significantly lower than GaAsBi:Si for low Bi concentrations. The electron mobility of GaAsBi\textsubscript{0.02}:Te is 867 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1} and the electron mobility of GaAsBi\textsubscript{0.02}:Si is 1420 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1}. This represents a 46% and 21% decrease compared to GaAs, respectively. Other authors have shown that increasing the Bi concentration from x = 0 (GaAs) to x = 0.02 results in a \sim 45% decrease in the electron mobility [22]. While there is an advantage to using Si as a dopant for low Bi fractions, the advantage becomes less dramatic at higher Bi fractions. The electron mobility of GaAsBi\textsubscript{0.061}:Te is 579 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1} and the electron mobility of GaAsBi\textsubscript{0.054}:Si is 679 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1}. The 100 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1} difference in mobility between the two samples is likely due to the difference in composition, x = 0.054 for the sample doped with Si and x = 0.061 for the sample doped with Te. Therefore, for low Bi fraction devices, silicon is the more suitable dopant. At higher Bi fractions, such as x = 0.06 used for a 1.0 eV subjunction solar cell, Te may be an equally suitable dopant choice when comparing electron mobility.

Annealing samples of GaAs\textsubscript{0.98}Bi\textsubscript{0.02} doped with Te at concentrations of \sim 3 \times 10^{18} \text{ cm}^{-3} for 10 min showed improvement in mobility across temperatures ranging from 300 °C-500 °C, as shown in figure 3. A 30% increase in mobility from 759 ± 17 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1} to 984 ± 17 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1} was shown by annealing at 350 °C compared to the un-annealed sample. However, mobility did not continue to improve at temperatures higher than 350 °C. The sample annealed at 475 °C became phase separated, as indicated by multiple film peaks present in the 004 2θ-ω HRXRD line scan (not shown). The 004 2θ-ω HRXRD line scans of the sample annealed at 450 °C showed a single film peak; however, other authors have shown that resistive Bi precipitates can form during annealing that do not dramatically impact the HRXRD signal [36]. We assume that the film annealed at 500 °C was also phase separated, although this was not measured by HRXRD. Phase separation and Bi segregation with annealing of bulk GaAsBi has been seen by other authors [37-40] where the optimal annealing temperature can be dependent on growth temperature [41] and Bi.
composition [42]. While improvement is seen by annealing at 350 °C, the mobility of Te doped GaAsBi$_{0.02}$ is still lower than the un-annealed Si doped samples shown in figure 2.

As the Hall effect measurement is a destructive measurement technique, requiring cleaving of the sample and depositing metal contacts, it would be ideal to have a nondestructive method of determining the effects of ex-situ annealing. Photoluminescence measurements have previously been used to characterize improvement in GaAsBi optical quality after in-situ or ex-situ annealing [42]. In this work, we explored whether spectroscopic ellipsometry could also be used as a method of determining optimal annealing temperatures. Figure 4 shows the absorption coefficient calculated from the models of ellipsometry data from GaAsBi$_{0.02}$:Te with $N_D = 3 \times 10^{18}$ cm$^{-3}$ annealed at 300 °C, 350 °C, and 450 °C compared to a piece of the sample that was unannealed. The insets in figure 4 show the squared absorption coefficient at important absorption features associated with interband critical points, including the $E_D$ and $E_V$ points [43], in the doped material. The samples annealed at 300 °C and 350 °C have sharper absorption features and increased absorption around both critical points, indicating sharper interband transitions and improved optical quality. The sample annealed at 450 °C looks very similar to the unannealed sample, suggesting that annealing at 450 °C caused both electrical and optical deterioration of the material, even though the XRD showed no long-range structural changes. The resulting optical properties measured by ellipsometry agree with the Hall effect measurements in indicating that 350 °C was the optimal annealing temperature.

Another significant difference between using Si and Te dopants in GaAsBi arises when Ga-Bi droplets are present on the growth surface. Due to the low V/III ratios and high Bi fluxes necessary for increased Bi incorporation in GaAsBi, metallic droplets can form on the surface during growth. These droplets can be Bi, Ga, or bi-metallic depending on the growth conditions used [44, 45]. While ideally these droplets should be avoided to prevent phase separation from occurring in the films [31, 46], many bismide films with high Bi incorporation at device-relevant thicknesses also have these droplets present [30]. Therefore, it is important to understand the effect their presence has on the dopant incorporation. Tait and Millunchick showed that the local Bi composition can be affected by Ga droplets, as the droplets provide an additional source of Ga adatoms at the growth surface, changing both the local Bi/Ga flux ratio and the local V/III ratio [46]. Field et al found the amphoteric nature of the silicon dopant in GaAsBi to be highly influenced by V/III BEP ratios [25]. All of the p-type GaAsBi films shown in their work were covered in Ga-Bi droplets, while their n-type films were droplet free. Erol et al showed compensated films of GaAsBi$_{0.05}$:Si covered with surface droplets, but that work does not indicate whether the droplets were Bi-only or Ga-Bi bimetallic [24].

GaAsBi$_{0.05}$:Si films were grown using matched GaAsBi growth conditions with and without significant droplet coverage on the surface. Samples were doped in a range of $1.8 \times 10^{18}$ to $5.0 \times 10^{18}$ cm$^{-3}$. All samples were grown with the same V/III BEP ratio, Bi flux conditions, substrate temperatures, and growth rates. The only difference was the choice of underlayer: GaAs or InGaAs with composition chosen to lattice match the GaAsBi. The choice of underlayer determined whether or not droplets were present on the growth surface, as the presence of a partially relaxed InGaAs underlayer been shown to prevent Bi rejection [31]. Figure 5 shows Nomarski images and the resulting Hall data from these samples. GaAsBi films grown with Te as the dopant source were n-type regardless if there were droplets present on the growth surface or not. While the measured mobility of GaAsBi$_{0.02}$:Te with droplets was higher than GaAsBi$_{0.02}$:Te without droplets, we attribute this
to a difference in film Bi composition. XRD measurements of GaAsBi:Te grown with droplets present had multiple film peaks (not shown), indicating compositions of $x = 0.01, 0.037$, and 0.056 throughout the film. The presence of Bi droplets on the surface of the film induced local phase separation by acting as a sink for the Bi adatoms \[31, 47, 48\]. As figure 2 shows that the mobility of GaAsBi:Te decreased with Bi content, the increase in mobility in the presence of surface droplets was likely due to a decrease in overall film Bi content, rather than an improvement of film parameters at $x = 0.061$.

GaAsBi films grown with Si as the dopant source had polarity that changed when droplets were present. Si doped films grown with Ga-Bi surface droplets exhibited p-type electronic behavior and low majority carrier mobilities. These values of mobility were in line with the measurements of hole mobility presented in figure 1 for equivalent levels of Be doping, as well as measurements in the literature for GaAsBi:Be/C \[14, 25\]. While the mobility of the highly doped film was slightly higher than what is shown in figure 1, that can again be attributed to an average decrease of the Bi composition of the film. The film grown without significant Ga-Bi droplet coverage on lattice matched InGaAs underlayers exhibited n-type character with mobility of 679 cm$^2$/V·s$^{-1}$. This indicates that the presence of Ga-Bi droplets, impacting the local V/III ratio, can encourage group-IV dopants to incorporate on the As lattice site rather than Ga in the GaAsBi material system.

4. Conclusions

In this work, n-type GaAs$_{1-x}$Bi$_x$ layers were grown using both silicon and tellurium as dopants. Room temperature Hall effect measurements showed GaAsBi:Te has lower electron mobilities than GaAsBi:Si at low Bi concentrations. However, as Bi content was increased from $x = 0.02$ to $x = 0.06$, this difference was reduced and the mobilities became comparable. Higher Bi content samples required an InGaAs buffer layer to prevent phase separation, but comparison to strained films on GaAs showed this buffer had no effect on the carrier mobility of the subsequent bismide film. Using rapid-thermal annealing for 10 min at 350 °C improved electron mobility by 30%. This improvement was also seen in the absorption coefficient around important optical transitions. Samples using both dopants were grown under conditions known to induce droplet formation on the surface. Due to changes in local V/III ratio, these droplets caused Si to act as amphoteric dopant, consistently resulting in p-type GaAsBi:Si films. However, as a group-VI dopant, Te showed no such behavior and all GaAsBi:Te films grown were found to be n-type. Ultimately, Te is found to be a more reliable choice of dopant for moderate Bi content films, given their increased likelihood of droplet formation and tellurium’s comparable electron mobility.

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