Dilute nitrides heterostructures grown by liquid phase epitaxy for solar cells applications

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Abstract. In this paper, we present a study on liquid phase epitaxy (LPE) grown dilute nitride GaAsSbN layers and p-i-n heterostructures for use in multijunction solar cells. The composition of the layers and chemical bonding of Sb and N in the compounds were determined by energy-dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscopy (XPS). The electrical properties of the grown samples were characterized by Hall effect measurements. Nominally undoped layers are n-type with Hall carrier concentration of $5 \times 10^{16} \text{cm}^{-3}$. Mg was successfully used as acceptor dopant for obtaining closely compensated layers with electron concentration of $10^{15} \text{cm}^{-3}$ as well as p-type layers with high free hole concentrations in the range $(5-7) \times 10^{18} \text{cm}^{-3}$. Temperature-dependent photoluminescence spectra at low and high excitation were measured to evaluate the optical quality and identify localized states in the grown layers. Non-contact surface photovoltage method provided information about the absorption characteristics of the GaAsSbN layers. A series of GaAs/GaAsSbN/GaAs heterostructures based on closely compensated i-GaAsSbN have been also grown by LPE. The red limit of the structures determined from surface photovoltage measurements was extended down to 1.2 eV. Single junction p-i-n solar cells with area 0.16 cm² were performed based on the grown structures. A power conversion efficiency of 4.1 % was measured for the fabricated cells under AM1.5 air global conditions.

1. Introduction

The growing energy needs in the world combined with ecological issues imply looking for further development of renewable energy sources and increasing their part in energy production. The quantity of solar energy reaching Earth and its availability worldwide makes it an excellent candidate as a renewable energy source, which can be used for increasing global energy production capacities. Therefore, the research and development of photovoltaic materials and devices is a current problem of prime importance. Nowadays silicon cell technologies represent the largest part of the photovoltaic panel market. However, new materials and concepts are under investigation to raise the efficiency of solar cells and decrease their costs. Such material is the dilute nitrides. They are a class of semiconductor alloys, which has emerged from the conventional III-V compounds by incorporating a small amount of nitrogen. The interest in these compounds is due to their potential to tune the bandgap energy and the lattice constant, which creates additional flexibility desired in many applications such as multijunction solar cells, heterojunction bipolar transistors, lasers, and high-speed photodetectors. In photovoltaics, the dilute nitrides are regarded as a perspective material to be used in multijunction solar cells. The idea is to add a dilute nitride-based junction (sub-cell) with a bandgap energy of around 1 eV. This will fill...
the gap between e.g. Ge (0.67eV) and GaAs (1.42 eV) energies and this way will enhance the collection of solar energy in this part of the solar spectrum. The dilute nitride layer has to be thick enough to absorb light and at the same time lattice-matched to the surrounding materials to decrease to a minimum the dislocations and other structural defects. This complicates the growth task because the right composition and growth conditions have to be chosen.

There has been significant progress in the development of dilute nitride InGaAsN materials grown by metalorganic chemical vapour deposition (MOCVD) [1,2]. Another dilute nitride material suitable for solar cell applications as an alternative to InGaAsN is GaAsSbN. It allows for independent tuning of the conduction and valence bands because N primarily reduces the conduction band minimum, while Sb mainly rises the valence band maximum energy. Both of these mechanisms are explained through the band anti-crossing model [3]. This way it is possible to obtain lower bandgap energy with less N content as compared to In containing materials. The incorporation of both Sb and N atoms into the crystal lattice also enables lattice matching with GaAs or Ge. Besides, their incorporation also causes a large concentration of localized states which results in changed electronic and optical properties, and lower device performance, due to reduced carrier collection. Despite the beneficial features of this material it has not been as widely studied as InGaAsN, although interest in GaAsSbN has risen over the last few years [4–8]. Recent solar cells based on GaAsSbN have demonstrated efficiencies of 4.9% for a nonoptimized single-junction solar cell structure, without anti-reflection coating grown by MOCVD [4] and 6% for molecular beam epitaxy (MBE) grown structures [8]. To the best of our knowledge, no other groups have reported on GaAsSbN layers and heterostructures grown by liquid phase epitaxy (LPE).

In this paper, we present a study of dilute nitride GaAsSbN layers and p-i-n GaAs/GaAsSbN/GaAs heterostructures grown on GaAs substrates by low-cost LPE method. The structural, electrical, optical and photoelectrical properties of the GaAsSbN layers are investigated by X-ray photoelectron spectroscopy (XPS), Hall effect measurements, temperature-dependent photoluminescence (PL) and surface photovoltage (SPV) spectroscopy, respectively. Single junction p-i-n GaAs/GaAsSbN/GaAs solar cells are performed based on the grown structures. A power conversion efficiency of 4.1% was measured for the fabricated cells under AM1.5 air global conditions.

2. Experiment

A series of undoped and Mg-doped GaAsSbN monocrystalline layers were grown on n-type and semisolating (100) GaAs substrates by liquid-phase epitaxy. The crystal growth was done in a horizontal quartz tube using a graphite slider-boat technique. A flux of Pd-membrane purified hydrogen at atmospheric pressure was used for experiments. Starting materials for the solutions consisted of 6N pure solvent metals Ga, Sb and polycrystalline GaAs and GaN powder as sources for As and N, respectively. The charged boat was heated at 750°C for 1 h to dissolve the source materials and decrease the contaminants in the melt. Epitaxial growth of GaAsSbN bulk layers is a great challenge because of the huge miscibility gap and the low solubility of nitrogen in the melt. Since the higher growth temperatures limit the N incorporation because of micro-phase segregation [9,10], a low-temperature (T<600°C) variant of the LPE method was used. To grow single crystalline layers without phase separation it is important to find correct solution compositions within a narrow temperature range. The initial supercooling of the melt is also an important parameter, which influences the composition of the grown layers. Since in the best of our knowledge in the literature, there are no reports on LPE growth of GaAsSbN compounds on GaAs substrate many series of experiments were carried out from different initial epitaxy temperatures in the range 570–550°C. The crystallization of the monocrystalline GaAsSbN layer without phase separation was carried out from mixed 95 at.% Ga + 5 at. % Sb solution. The N content in the growth solution was 0.5 at.% for all samples, while the As content was varied in the range (0.08-0.1) at.% depending on the initial epitaxy temperature. The epitaxial layers were grown from a 7°C super-cooled solution at a cooling rate of 0.8°C/min for about 20-30 minutes.

In addition to single epitaxial layers, a series of p-i-n structures p-AlGaAs – p-GaAs – i-GaAsSbN – n-GaAs based on compensated with Mg GaAsSbN layers were grown using the “piston boat”
technique [11]. In this technique, the melt falls into the piston chamber and squeezes through a narrow slot to the substrate, which allows mechanical cleaning of oxides films from the liquid phase and insures a good wetting. The crystallization was carried out from a melt 0.5-1 mm thick. After the growth of the layer, the liquid phase is removed from the substrate by squeezing of the next melt. The substrate surface in this boat after the first wetting is always covered by a melt and this solves difficulties of wetting during the growth of AlGaAs heterostructures in the range 600-400°C. The initial epitaxy temperature was 640°C and GaAsSbN was grown in the temperature interval 558-550°C. The GaAs capping and AlGaAs window layers were grown in the temperature intervals 550-447°C and 447-446°C, respectively. For n- and p-type doping of GaAs and AlGaAs layers Sn and Mg dopant were chosen.

Single junction solar cells with area 4×4 cm² were fabricated based on the grown structures. The metal grid on the photosensitive surface consists of 10 μm width metal lines separated by 90 μm stripe spacing (illuminated window). The front and back metallization of the cell, based on Ni/Ag system was deposited by magnetron sputtering. The schematic structure of the single-junction p-i-n GaAsSbN solar cell investigated in this study is shown in figure 1.

High-resolution scanning electron microscope (SEM)/focused ion beam system LYRA I XMU (Tescan) equipped with energy dispersive X-ray (EDX) microanalyzer (Quantax, Bruker) was used for measurements of the thicknesses and composition of the grown layers. X-ray photoelectron spectroscopy (XPS) was applied to assess the chemical bonding and determine the atom concentrations of Sb and N in the GaAsSbN alloys. The XPS experiments were carried out on AXIS Supra electron spectrometer (Kratos Analytical Ltd., a Shimadzu Group Company) with a base vacuum in the analysis chamber of ~10⁻⁹ mbar. The spectra were measured using a double AlKα/MgKα excitation source. The concentrations (in at. %) of the observed chemical elements were calculated by normalizing the areas of the corresponding photoelectron peaks to their relative sensitivity factors. The procedure included also corrections for the differences in the transmission function and the inelastic mean free paths of the photo-emitted electrons with different kinetic energies. The accuracy of the binding-energy determination was within ± 0.1 eV.

![Figure 1. Schematic cross-section structure of a single-junction p-i-n solar cell based on compensated GaAsSbN.](image)

![Figure 2. Cross-sectional SEM micrograph of LPE grown GaAsSbN layer on GaAs substrate.](image)
The Hall carrier concentration and mobility in the epitaxial layers were measured by the van der Paw Hall technique on 5×5 mm² square samples, with indium ohmic contacts alloyed in hydrogen at 400°C for 5 minutes.

Temperature-dependent PL spectra were measured in the temperature range between 10 and 150 K to investigate the optical properties of the grown structures. The excitation was obtained with laser light with a wavelength of 680 nm and 0.5 mW power. The PL was collected and recorded by a fibre-coupled spectrometer (BWSpec Glacier X) as in Ref. [12]. PL spectra in the temperature range 10-300 K were also measured using laser excitation at 532 nm (Nd:YAG) with a power of 140 mW and a 0.5 m focal length spectrometer (Acton 2300i, Princeton Instruments) with a Si detector. Surface photovoltage (SPV) spectroscopy in metal-insulator-semiconductor (MIS) operation mode technique was applied to study the optical absorption of the structures using the set-up and the measurement procedure described elsewhere [13].

The photovoltaic current density-voltage (I-V) characteristics of the fabricated cells were measured by solar simulator under air-mass (AM) 1.5 global illumination at one sun.

3. Results and discussions

3.1. Structural characterization

Epitaxial GaAsSbN with thicknesses of 0.5-1 µm depending on the initial epitaxy temperature were grown by LPE. Figure 2 shows the SEM image of the cross-section of a 0.5 µm thick GaAsSbN layer grown in the temperature interval 558-550°C on a GaAs substrate. The interface is not ideal, because of dislocations related to the lattice mismatch and partial relaxation of the layer. The Sb content in the GaAsSbN layers was measured in several points on a cross-section by energy-dispersive X-ray spectroscopy (EDX). The average value of Sb concentration was determined to be 3.4 at. %.

XPS spectra are measured at a depth of about 90 nm from the surface after bombardment with 1 keV Ar+ ions. Photoelectron peaks of Ga 3d, Sb 4d and As 3d characterizing a clean GaAsSbN sample are detected in the low binding-energy region and displayed in figure 4(a). The peaks at 31.7 eV and 32.9 eV can be attributed to 4d⁵/₂ and 4d³/₂ peaks of Sb incorporated in the GaAs lattice. The results are in good agreement with the literature data [14–16]. In the high binding-energy region only single Sb 3d⁵/₂ - and Sb 3d³/₂ - peaks have been measured (figure 4(b)) in agreement with the single Sb 4d doublet observation.
in figure 4(a). The calculation of the antimony atoms concentration using the Sb 3d- or Sb 4d-peaks gives close results of 3.6 and 3.2 at. %, respectively. These values correspond to the value found by EDX. Mg Kα excitation source was used to distinguish the N 1s line from the Ga LMM Auger structure. The presence of nitrogen of about 0.1 at. % was detected as a week N 1s peak observed at about 398 eV in the difference spectrum of GaAsSbN and GaAs standard spectra (figure 3(c)). The details on XPS characterization are given in Ref. [6].

### 3.2. Electrical characterization

All as grown intentionally undoped GaAsSbN samples are n-type with Hall carrier concentration in the range \((4 - 5) \times 10^{16} \text{cm}^{-3}\) and room temperature mobility of 3200 cm²V⁻¹s⁻¹. The carrier concentration values are more than one order of magnitude lower as compared to those of InGaAs(Sb)N samples grown previously by LPE from In-rich solution [17]. This makes it possible to controllably grow n- and p-type doped GaAsSbN layers. However, inhomogeneities and defects in the GaAsSbN layer lead to shorter diffusion lengths as compared to GaAs and limit the material quality for solar cell applications. To overcome this problem a p-i-n structure configuration with low background carrier concentration in the i-GaAsSbN layer should be used. For this purpose, i-type epitaxial layers very closely compensated by Mg doping have been grown by carefully choosing the Mg quantity in the growth melt. Hall electron concentration as low as \(8.7 \times 10^{14} \text{cm}^{-3}\) has been obtained for layers grown from a melt with 0.025 at.% Mg content. Mg is a lately used p-type dopant in all type epitaxial growth methods in the development of GaAs-based optoelectronic devices such as lasers, solar cells, and detectors [18–20]. It has high solubility, shallow acceptor level [21] and lower vapour pressure in comparison with the commonly used Zn-dopant. P-type layers with hole concentrations up to about \((5 - 6) \times 10^{18} \text{cm}^{-3}\) have been measured for GaAsSbN layers grown from a melt with Mg content below 0.1 at.%. Figure 4 shows Hall carrier concentration of n- and p-type charge carriers as a function of Mg content in the melt.

![Figure 4. Hall carrier concentration as a function of Mg content in the growth melt.](image)

### 3.3. Optical characterization

Figure 5 presents the normalized PL peaks of a GaAsSbN layer measured at different temperatures under low and high excitation power. The PL peak energy evolution with temperature obtained under low power excitation (figure 5(a)) exhibits S-shaped like behaviour. This behaviour is a characteristic of carrier localization effects associated with band-tail states induced by compositional fluctuations [22–24]. The PL emission energy experiences a blue shift as the temperature increases from 40 to about 70 K. This indicates that as the temperature increases the excitons gain sufficient thermal energy to transfer to higher-energy localized levels, thus increasing the emission energy. With further increase of the temperature (beyond 70 K) the shallower energy localized states are gradually saturated, the excitons
become almost delocalized and the PL peak energy decreases as a function of temperature due to reduction of the bandgap.

![Figure 5. Temperature-dependent PL spectra of GaAsSbN layer measured at laser excitation power of 0.5 mW (a) and 140 mW (b).](image)

As the excitation power increases (figure 5(b)), the lower-energy states in the band-tail are gradually filled until reaching saturation and the recombination of free carriers starts to dominate. In this case, the PL peak energies can be approximated quite well by Varshni’s relation $E_g(T) = E_0 - aT^2/(T+b)$ with $E_0 = 1.32$ eV, $a = 5 \times 10^{-4}$ eV.K$^{-1}$ and $b = 276$ K. Figure 6 presents the temperature behaviour of the PL peak energies of GaAsSbN for the two different excitation powers of 0.5 mW and 140 mW. Localization energy of about 27 meV is estimated from the magnitude of the shift between the Varshni’s curve and the experimental points at low temperatures and low excitation.

![Figure 6. Temperature dependence of the PL peak energy of GaAsSbN at laser excitation power of 0.5 mW (squares) and 140 mW (circles). The line is Varshni’s fit to the data.](image)

![Figure 7. Surface photovoltage amplitude spectra of GaAsSb (triangles) and GaAsSbN (circles). The inset shows the same normalized spectra](image)
SPV spectroscopy was applied to study the optical absorption characteristics of the layers since the SPV amplitude spectrum is considered as emulating the optical absorption [13]. Figure 7 compares the SPV amplitude spectra of GaAsSbN and GaAsSb layers with the same amount of Sb. The spectrum of GaAsSbN shows larger signal by a factor, which increases from 3 for 800 nm to 6.5 for 1010 nm indicating better photo-response. The improvement is more pronounced for photon energies below the GaAs band-gap as can be seen also from the inset, which presents the normalized SPV amplitude spectra of the two samples. Therefore, adding nitrogen into GaAsSb even at ultra-low quantity improves the photo-response of the material. This is explained by the role of N to partially passivate Sb-related localized states [6]. The bandgap energy of GaAsSbN assessed from Tauc plot is 1.24 eV, which is 180 meV lower than the bandgap of GaAs (1.42 eV) and ~20 meV lower than that of the GaAsSb sample. Applying the band-anticrossing model [25], this N-induced red shift corresponds to N content of ≈ 0.1% in agreement with the XPS result. SPV measurements were performed also on p-i-n structures GaAs/ i-GaAsSbN/n-GaAs, which revealed an absorption edge extended down to 1.2 eV [7].

3.4. I-V measurements of solar cells
A typical AM1.5 I-V curve for a GaAsSbN p-i-n solar cell is presented in figure 8 along with the corresponding dark I-V characteristic of the same cell. The measured short circuit current density $I_{sc}$, open-circuit voltage $V_{oc}$, fill factor FF and photovoltaic conversion efficiency $\eta$ are 18.7 mA/cm$^2$, 0.43 V, 51 % and 4.1 %, respectively. This is comparable to the efficiency reported for single-junction 1.25 eV GaAsSbN solar cells with 600 nm thick GaAsSbN base layer grown by MOCVD [4]. Higher efficiency of about 6% has been achieved for 1.15 eV MBE grown GaAsSbN solar cells [8]. In both cases, rapid thermal annealing (RTA) at 800°C of the solar cells was performed, which significantly increased the open-circuit voltage values to 0.5 - 0.6 V due to the decrease of the localized state density. However, typical values of $V_{oc}$ measured in our cells based on as-grown LPE structures without RTA are in the range of 0.40 – 0.44 V.

![Figure 8. Light and dark I-V characteristics of a p-i-n single-junction GaAsSbN solar cell.](image)

$V_{oc}$ corresponds to the forward bias voltage, at which the photocurrent $I_{ph}$ compensates the dark current $I_0$. It depends on both $I_{ph}$ and $I_0$ and can be calculated from the following equation:

$$V_{oc} = \frac{BkT}{e} \ln \left( \frac{I_{ph}}{I_0} + 1 \right) \approx \frac{BkT}{e} \ln \left( \frac{I_{ph}}{I_0} \right)$$

(1)
where \( I_{\text{ph}} \gg I_0 \). While \( I_{\text{ph}} \) typically has small variations, the key effect comes from the saturation dark current, since it may vary by orders of magnitude depending on the sample quality. To achieve maximum sensitivity, it is necessary to minimize the dark current. The saturation dark current depends on the recombination in the depletion region of the solar cell. Therefore, \( V_{\text{oc}} \) is a measure of the amount of recombination in the device. A decrease in \( V_{\text{oc}} \) is expected to be correlated with an increase in the dark current. The saturated dark current density of the cell obtained from the presented dark I-V characteristic is around \( 10^{-5} \) mA/cm². This value is about three orders of magnitude higher than those for the high-quality solar cells which is due to the carrier recombination via N-induced traps in the GaAsSbN layer. It is known that the incorporation even of a small quantity of nitrogen (\(~0.1\%) into GaAs creates a high concentration of minority carriers traps which sufficiently increases the dark current. Besides, the lowering in \( V_{\text{oc}} \) values could be explained by a change in the position of the quasi-Fermi levels from the carrier traps, occurred by the incorporation of N and Sb into the crystal lattice \([26]\).

The open-circuit voltage of solar cells is based upon the difference between the electron \( (F_n) \) and hole \( (F_p) \) quasi-Fermi levels under illumination \([27]\). 

\[
e_{V_{\text{oc}}} = F_n - F_p
\]  

The quasi-Fermi levels are given by \( F_n = E_c - kT \ln (N_c/n) \) and \( F_p = E_v + kT \ln (N_v/p) \). Here \( n \) and \( p \) are the electron and hole concentrations in the base, while \( N_c \) and \( N_v \) are the effective density of states in the conduction and the valence band, respectively. Therefore

\[
e_{V_{\text{oc}}} = F_n - F_p = E_c - E_v - kT \ln \left( \frac{N_c}{n} \right) = E_g - kT \ln \left( \frac{N_c}{n} \right) .
\]  

The logarithmic term in this equation calculated for n- or p-type emitter of GaAs solar cells is about 0.33-0.40 V. Experimentally obtained \( V_{\text{oc}} \) values for the best solar cells based on high-quality GaAs-like compounds are nearly equal to \( E_g - 0.4 \) V. If electron traps due to N-incorporation are present in high concentrations the quasi-Fermi level depends on the trap energy \( E_t \), the concentration of electrons \( n_t \) on the trap level, and the trap concentration \( N_t \), according to the following relation (for \( E_t - F_n > 2kT \) \([26]\)):

\[
F_n = E_t + kT \ln \left( \frac{n_t}{N_t} \right) .
\]

Therefore, the \( V_{\text{oc}} \) values will reduce with increasing the activation energy (lowering \( E_t \)) of the traps. The addition of Sb further reduces \( V_{\text{oc}} \) by an amount that is proportional to the activation energy of trap states introduced by the Sb atoms.

4. **Conclusions**

Dilute nitride GaAsSbN layers were grown on GaAs substrate by low-temperature LPE. The Sb content of 3.4 at.% in the alloy was measured by EDX. This value was confirmed by XPS spectra obtained at a depth of about 90 nm from the surface. To detect N 1s line in the compound Mg K\( \alpha \) radiation leading to the displacement of the Ga LMM structure to low binding-energies was used. The concentration of N atoms were determined to be around and 0.1 at. %. SPV spectroscopy of the GaAsSbN layers at room temperature revealed band gap value of 1.24 eV, which is about 20 meV lower than the bandgap of the reference GaAsSb layers with the same Sb content but not containing nitrogen.

Temperature-dependent PL measurements at low excitation power of 0.5 mW showed a blue shift of the GaAsSbN PL emission energy at low temperatures from 10 K to about 70 K due to localization of excitons in potential fluctuations. The localization energy estimated from the magnitude of the maximum blue shift at low temperatures is about 27 meV. PL spectra measured under higher excitation up to room temperatures revealed negligible S-shaped like behaviour of the PL peak energy with temperature indicating the filling of the localized states.
Single heterojunction p-i-n GaAs/GaAsSbN/GaAs photovoltaic cells based on compensated GaAsSbN were performed and studied. Photovoltaic parameters were measured under AM1.5 global air conditions at one sun. The best cells without antireflection coating demonstrate a power conversion efficiency of 4.1%, an open-circuit voltage of 0.43 V, short-circuit current of 18.7 mA/cm² and fill factor of 51%.

The obtained results reveal the capacity of the LPE method for growing of GaAsSbN for photovoltaic applications and can be used as a basis for further development of technology to implement GaAsSbN based solar cells with improved parameters.

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