Ultrafast Spectroscopy and Red Emission from β-Ga$_2$O$_3$/β-Ga$_2$S$_3$ Nanowires

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Abstract

Ultrafast pump-probe and transient photoluminescence spectroscopy were used to investigate carrier dynamics in β-Ga$_2$O$_3$ nanowires converted to β-Ga$_2$O$_3$/Ga$_2$S$_3$ under H$_2$S between 400 to 600 °C. The β-Ga$_2$O$_3$ nanowires exhibited broad blue emission with a lifetime of 2.4 ns which was strongly suppressed after processing at 500–600 °C given rise to red emission centered at 680 nm with a lifetime of 19 μs. Differential absorption spectroscopy reveals that state filling occurs in states located below the conduction band edge before sulfurization, but free carrier absorption is dominant in the β-Ga$_2$O$_3$/Ga$_2$S$_3$ nanowires processed at 500 to 600 °C for probing wavelengths >500 nm related to secondary excitation of the photo-generated carriers from the mid-gap states into the conduction band of Ga$_2$S$_3$.

Keywords: β-Ga$_2$O$_3$/Ga$_2$S$_3$ nanowires; Pump-probe spectroscopy; Carrier dynamics; Photoluminescence

Background

Metal oxide (MO) semiconductor nanowires (NWs) such as SnO$_2$, In$_2$O$_3$, and Sn-doped In$_2$O$_3$ have been investigated extensively for the fabrication of nanoscale devices such as sensors and nanowire solar cells [1, 2]. With the incorporation of MO NWs in such devices that are subjected to various gasses such as NH$_3$, H$_2$S, or liquids containing S, Na$_2$S requires an understanding of their effect on the electrical, optical, and structural properties of the MO NWs. In the past, we investigated the growth and properties of SnO$_2$ [3], In$_2$O$_3$ [4], Sn-doped In$_2$O$_3$ [5], and β-Ga$_2$O$_3$ NWs and found that they exhibit photoluminescence at ≈2.5 eV due to oxygen vacancies and states residing energetically in the upper half of the energy band gap as shown by ultrafast absorption spectroscopy [6].

More recently, we showed that post growth processing of SnO$_2$ and Sn-doped In$_2$O$_3$ NWs under H$_2$S between 300 to 600 °C resulted in an increase of conductivity and band edge emission, respectively. However, there are no investigations into the conversion of β-Ga$_2$O$_3$ into β-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs under H$_2$S. Ga$_2$S$_3$ is a III-VI semiconductor which can have the monoclinic, hexagonal, or cubic crystal structure [7]. Among these, the most stable crystal structure is monoclinic of Ga$_2$S$_3$ which has a direct wide-band gap of 3.4 eV [8, 9] but contains many vacancies in its Ga sub lattice and consequently exhibits defect emission similar to oxides [10, 11]. Despite this, Ga$_2$S$_3$ has been investigated for light-emitting diodes and photovoltaic devices while recently, it was shown that Ga$_2$S$_3$ has a large second-harmonic generation efficiency ideally suited for nonlinear optics [12]. The optical properties of single crystals and epitaxial layers of Ga$_2$S$_3$ have been investigated using steady-state absorption-transmission and photoluminescence (PL) spectroscopy and exhibit blue and red emission at 10 K due to deep donor-to-acceptor transitions related to S and Ga vacancies [8, 10, 11]. In contrast, one-dimensional GaS or Ga$_2$S$_3$ nanotubes so far have been shown to exhibit PL between 450–600 nm [13–15], but there are no detailed investigations on the optical properties of GaS or Ga$_2$S$_3$ NWs using steady-state or transient optical spectroscopy which could provide insight into their fundamental properties and potential device applications.

Consequently, we have undertaken an investigation into the post growth processing of β-Ga$_2$O$_3$ NWs under H$_2$S between 400–600 °C and its effect on the optical properties using ultrafast absorption spectroscopy in conjunction with steady-state and time-resolved PL. We find that the as-grown β-Ga$_2$O$_3$ NWs exhibit state filling following carrier excitation at 266 nm ($\lambda_p$) for probing wavelengths ($\lambda_p$) between 340 to 450 nm corresponding to states lying energetically below but near the conduction
band edge. In addition, the $\beta$-Ga$_2$O$_3$ NWs exhibited broad PL with a maximum at 435 nm and lifetime of 2.4 ns. Similar results have also been observed for the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs after post growth processing under H$_2$S at 400 °C. In contrast, the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained under H$_2$S at 500 to 600 °C exhibit free carrier absorption for $\lambda_p > 500$ nm and state filling for $\lambda_p < 450$ nm related to the formation of Ga$_2$S$_3$. This is accompanied by a suppression of the broad PL at 435 nm and the emergence of strong emission at $\approx 680$ nm with a much longer lifetime of 19 μs. In addition, at low temperature, a relative narrow emission at 428 nm emerges resulting from near band edge states to the acceptor states located near the valence band of the $\beta$-Ga$_2$S$_3$.

**Methods**

$\beta$-Ga$_2$O$_3$ NWs were grown by low-pressure chemical vapor deposition at 800 °C and 1mBar for 60 min on Si(001) using a 1-nm layer of Au as catalyst and exactly the same growth conditions used for Sn-doped In$_2$O$_3$ NWs as described in detail elsewhere [5]. The morphology of the NWs were then examined with a TESCAN scanning electron microscope (SEM) while their crystal structure and phase purity were investigated using a SHIMADZU, XRD-6000, X-ray diffractometer with Cu-Kα source, by performing a scan of θ-2θ in the range between 10° and 80°. The dynamic behavior of carriers within the NWs was investigated through the temporal behavior of ultrafast time-resolved differential absorption obtained from simultaneous measurements of time-resolved differential transmission and reflection [16, 17]. The experiments were carried out using a Ti: Sapphire ultrafast amplifier system generating 100-fs pulses at 800 nm and running at a repetition rate of 1 kHz. Non-linear crystals were used to generate 266 nm for the purpose of exciting the NWs whereas part of the fundamental and second-harmonic generation at 400 nm was used to generate a super continuum light for probing different energy states. Measurements were carried out using a typical pump-probe optical setup in a non-collinear configuration. Photoluminescence were carried out using a spectrometer equipped with an intensified charge-coupled device camera. The low-temperature measurements were carried out using a closed cycle refrigerator capable of cooling the sample to temperatures below 10 K.

**Results and Discussion**

A typical SEM image of the $\beta$-Ga$_2$O$_3$ NWs which had diameters of 50–100 nm and lengths of up to 100 μm is shown as an inset in Fig. 1a.

The $\beta$-Ga$_2$O$_3$ NWs have a monoclinic crystal structure similar to that obtained previously [6] and were exposed to 50 sccm of H$_2$S at 400, 500, and 600 °C for 60 min using a ramp rate of 10 °C/min which resulted in the formation of the $\beta$-Ga$_2$O$_3$/$\beta$-Ga$_2$S$_3$ NWs as shown by the XRD of Fig. 1a. In particular, we observe peaks belonging to $\beta$-Ga$_2$O$_3$ but also the emergence of monoclinic $\beta$-Ga$_2$S$_3$ due to the diffusion of S into $\beta$-Ga$_2$O$_3$. Ga$_2$S$_3$ is a III-VI defect semiconductor which can have the monoclinic, hexagonal, or cubic crystal structure. Among these, the most stable crystal structure of Ga$_2$S$_3$ is the monoclinic phase. We observe a suppression of most peaks belonging to $\beta$-Ga$_2$O$_3$ with increasing temperature although a few remain at 600 °C which means that high temperatures are necessary to achieve complete conversion into Ga$_2$S$_3$. Interestingly, the $\beta$-Ga$_2$O$_3$/$\beta$-Ga$_2$S$_3$ NWs remained one-dimensional up to 600 °C in contrast to SnO$_2$ and Sn-doped In$_2$O$_3$ NWs which were eliminated under H$_2$S due to their reduction by H$_2$ [18, 19]. Here, we should point out that post growth processing of MO NWs
such as Sn-doped In$_2$O$_3$ at elevated temperatures resulted in the formation of crystals on the surface and not a conformal or epitaxial like shell [19]. The lattice constants of β-Ga$_2$O$_3$ are $a = 12.23$ Å, $b = 3.04$ Å, and $c = 5.80$ Å with β = 103.7° while β-Ga$_2$S$_3$ have lattice constants of $a = 11.11$ Å, $b = 9.58$ Å, and $c = 6.4$ Å with β = 141.15°, so one expects interfacial stress due to the lattice mismatch between the β-Ga$_2$O$_3$ and β-Ga$_2$S$_3$. The band line-up and the effect of strain on the β-Ga$_2$O$_3$/Ga$_2$S$_3$ heterojunction is not known, but the energy band gap and work function of Ga$_2$O$_3$ is 4.5 eV and 4.1 eV, respectively [20], while the energy band gap of Ga$_2$S$_3$ is smaller and equal to 3.42 eV. Assuming that the work functions are similar, the β-Ga$_2$O$_3$/Ga$_2$S$_3$ heterojunction is expected to be type I. It is also useful to note that F.Säuberlich et al. [21] showed that the large interface dipole moments exist between metal oxides and chalcogenides resulting into small conduction band discontinuities. Steady-state absorption measurements (Fig 1b) through the NWs grown on fused silica suggest a band gap at $≈ 4.55$ eV for the β-Ga$_2$O$_3$ and 400 °C NWs which decreased to 3.1 eV after post growth processing under H$_2$S at 500 and 600 °C consistent with the formation of β-Ga$_2$S$_3$. Furthermore, there appears to be a broad absorption below the band edge for all the samples most likely due to the defect states present. Unfortunately, the absorption spectra for the β-Ga$_2$O$_3$/β-Ga$_2$S$_3$ NWs do not provide any clear evidence of the type of band offset that may exist between this hetero-structure [22]. Nevertheless, transient PL and differential absorption measurements indicate that the main contribution of both signals is coming from Ga$_2$S$_3$ suggesting a type I heterojunction. This is confirmed since the observed time constants in the decays in these signals remain the same with increasing post growth processing temperature from 500 to 600 °C and higher where the NW structure is completely converted to Ga$_2$S$_3$.

Photoluminescence of the as-grown β-Ga$_2$O$_3$ and β-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained at 400, 500, and 600 ° C is shown in Fig 2a. The as-grown β-Ga$_2$O$_3$ NWs exhibit broad PL with a maximum at $≈ 435$ nm, which is attributed to transitions between donor-like states related to oxygen vacancies (V$_O$) and acceptor-like states due to gallium vacancies (V$_Ga$) or gallium-oxygen vacancy pairs (V$_Ga$-V$_O$) [23]. We observe a small red shift of the PL from 2.9 to 2.8 eV after post growth processing under H$_2$S at 400 °C and a tenfold reduction in intensity. However, the suppression of the PL at $≈ 2.9$ eV was accompanied by the emergence of red emission at $≈ 1.8$ eV following post growth processing of the β-Ga$_2$O$_3$ NWs under H$_2$S at 500 and 600 °C as shown in Fig 2a. This red emission is attributed to transitions between donor-like states, located $≈ 1.1$ eV below the conduction band edge, to acceptor states $= 0.4$ eV above the valance band edge in Ga$_2$S$_3$ [10, 11]. It should be noted that the PL of the as-grown β-Ga$_2$O$_3$ NWs increased by a factor of two upon decreasing the temperature down to 10 K (inset Fig 2a). On the other hand, we observed a sixfold increase of the 680-nm peak for the NWs processed under H$_2$S at 500 and 600 °C, whereas new peaks appear at 428 nm (2.9 eV) and 620 nm (2.0 eV) for temperatures below 100 K as shown in Fig. 2b.

The temperature dependence of the PL peak intensity can be described by [24]

$$ I(T) = I(0) \left[ 1 + C \exp \left( \frac{-E_{act}}{k_B T} \right) \right] $$

where $I(T)$ is the intensity as a function of temperature $T$, $C$ is a constant, $k_B$ is Boltzmann constant, and $E_{act}$ is
the activation energy. A fit of this equation to the data provided an activation energy of 18 meV for the 428-nm PL peak.

Given the activation energy, we believe that the emission occurs from states just below the band edge to acceptor states located around the 0.4 eV band. Our findings are consistent with those described by Yoon et al. [10] for $\text{Ga}_2\text{S}_3$ who identified at 10 K two transitions of 1.93 and 2.9 eV between an acceptor state at 0.42 eV above the valence band edge and donor states at 0.1 eV and 1.1 eV below the conduction band. Furthermore, it appears from our findings that the occupation of the lower-end energy states in the 0.4 eV band at low temperatures results in a substantial increase in the 680-nm line as well as the new emission emerging at 620 nm at $<100$ K. These trends are consistent with the findings of Ho and Chen [8] who observed PL at 1.99 and 2.79 eV at low temperatures attributed to the formation of sulfur vacancies ($V_\text{S}$) as well as the existence of Ga vacancies ($V_{\text{Ga}}$) which form donor and acceptor levels, respectively. We should point out that in their work on the high quality $\text{Ga}_2\text{S}_3$ crystal [8], they have observed a broad emission around 500 nm at room temperature in contrast to the observed emission at 680 nm from the $\beta$-$\text{Ga}_2\text{O}_3/\text{Ga}_2\text{S}_3$ NWs (Fig. 2). It should be emphasized that all previous investigations on one-dimensional GaS and $\alpha$-$\text{Ga}_2\text{S}_3$ showed PL between 450–600 nm or 2.1–2.8 eV, so here, we show that $\beta$-$\text{Ga}_2\text{O}_3/\text{Ga}_2\text{S}_3$ can exhibit red emission at room temperature which makes them attractive not only for the applications in sensors and non-toxic biomedical imaging but also for down-conversion in solar cells.

In order to gain a better understanding of the optical properties, time-resolved differential absorption measurements were carried out using excitation at 266 nm and probing wavelengths between 340 to 850 nm. The differential absorption versus optical delay acquired from the as-grown $\beta$-$\text{Ga}_2\text{O}_3$ NWs and the $\text{Ga}_2\text{O}_3/\text{Ga}_2\text{S}_3$ NWs obtained after processing with $\text{H}_2\text{S}$ at 400 and 500 °C are shown in Figs. 3, 4, and 5, respectively.

The various traces in each case correspond to different $\lambda_p$ over a time span of 10 to 500 ps and were obtained using a fluence of 115 $\mu$J/cm$^2$. The value of the fluence was chosen for best signal-to-noise ratio with negligible nonlinear recombination as clearly seen from the normalized fluence measurements in the inset of Fig 3b. Similar behavior was observed for the different probing wavelengths and samples for this range of fluence.

For the shortest probing wavelengths, we observe a pulse width-limited, sharp decrease in the induced absorption followed by a recovery toward equilibrium over a time scale of hundreds of ps. These changes in absorption are associated with the excitation of electrons and holes in the $\beta$-$\text{Ga}_2\text{O}_3$ and $\text{Ga}_2\text{O}_3/\text{Ga}_2\text{S}_3$ NWs by photons resulting in the generation of non-equilibrium carriers. These non-equilibrium carriers will re-distribute themselves among energy states that are normally unoccupied under equilibrium conditions. This will appear as a decrease in the absorption (state filling) at the energy states that are being probed. The recovery of this absorption change will be a direct measure of the time required by the photo-generated carriers to move out of the occupied states. In the case of the as-grown $\beta$-$\text{Ga}_2\text{O}_3$ NWs, we observe a strong negative differential absorption for $\lambda_p < 450$ nm corresponding to state filling of the energy states located below the conduction band of the $\beta$-$\text{Ga}_2\text{O}_3$ NWs as shown in Fig. 3. The relaxation of the non-equilibrium carriers can be described by a tri-exponential decay with time constants ($\tau$) and amplitudes ($\alpha$) of $\tau_1 = 2–4$ ps ($\alpha_1 = 28–35 \ %$), $\tau_2 = 68–85$ ps ($\alpha_2 = 31–50 \ %$) and $\tau_3 = 1915–2050$ ps ($\alpha_3 = 20–41 \ %$). Both $\tau_1$ and $\tau_2$ are related to non-radiative, re-distribution of photo-generated carriers in point defect or surface
states, depicted schematically in the energy band diagram shown as an inset in Fig. 3, whereas $\tau_3$ is most likely associated with radiative recombination. For $\lambda_p = 550$ to 600 nm, the observed differential absorption is relatively small and is associated with the Au plasmon resonance [6]. For longer $\lambda_p$, the signal appears to be weaker and is associated with what is known as “free carrier absorption”. We should point out here that free carrier absorption is considered as a competing process to state filling, and it occurs due to secondary excitation of the photo-generated carriers by the probing photons from their initial states to higher energy states. This phenomenon will result in a positive change in the induced absorption.

Given that state filling is observed for $\lambda_p$ corresponding to energies that are smaller than the energy band gap of $\beta$-Ga$_2$O$_3$, it is evident that the $\beta$-Ga$_2$O$_3$ NWs contain states residing energetically in the upper half of the gap consistent with the broad PL shown in Fig. 2.

Similar trends were also observed in Fig. 4 for the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained after processing under H$_2$S at 400 °C which resulted into a slight reduction of differential absorption and a faster decay of electrons occupying energetically higher states, compared to the as-grown $\beta$-Ga$_2$O$_3$ NWs.

The decay times are similar, i.e., $\tau_1 = 3.5–5.5$ ps ($\alpha_1 = 50–52\%$), $\tau_2 = 59–95$ ps ($\alpha_2 = 34–41\%$) and $\tau_3 = 1950–2010$ ps ($\alpha_3 = 10–22\%$), but the strength of $\tau_1$ increased from 30 to 50% consistent with the suppression of radiative recombination and blue PL which occurs upon processing the $\beta$-Ga$_2$O$_3$ NWs at 400 °C as shown in Fig. 2. This is also consistent with the fact that there is noticeable state filling observed for longer $\lambda_p$ in contrast to the as-grown $\beta$-Ga$_2$O$_3$ NWs, and it is clearly due to the formation of defect states.

Differential absorption measurements for the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs after exposure to H$_2$S at 500 °C are shown in Fig. 5. Again, state filling is observed for $\lambda_p < 450$ nm, but in this case, the main signal is coming from the $\beta$-Ga$_2$S$_3$ where the carriers are generated above the conduction band and relaxation takes place on a very short time scale with $\tau_1 = 1$ ps ($\alpha_1 = 25–35\%$), $\tau_2 = 35$ ps ($\alpha_2 = 34–50\%$) and $\tau_3 = 145–167$ ps ($\alpha_3 = 8–20\%$).

For the larger probing wavelengths, $\lambda_p$ free carrier absorption is dominant. It appears that the $\beta$-Ga$_2$S$_3$ formed during the processing of the as-grown $\beta$-Ga$_2$O$_3$ NWs under H$_2$S plays a crucial role in the carrier dynamics. The differential absorption signal for $\lambda_p > 500$ nm appears to be much larger than the contribution from the $\beta$-Ga$_2$O$_3$. The donor states in $\beta$-Ga$_2$S$_3$ [10, 11] which are located approximately 1.1 eV from the conduction band edge are populated following excitation, and as a result, the carriers undergo a secondary excitation by the probing beam back into the conduction band. Further evidence of this was observed for the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained at 600 °C where differential absorption measurements reveal an increase in the free carrier absorption signal in the long wavelength range. The free carrier absorption may be described well with a two exponential function ($\alpha_0 + \alpha_1 e^{-t/\tau_1} + \alpha_2 e^{-t/\tau_2}$) and a constant $\alpha_0$ associated with the very long radiative emission, consistent with the lifetime of the observed red emission (~19 μs) from the 1.1-eV donor state. The time constants have a range of values of $\tau_1 = 1.2–2.9$ ps ($\alpha_1 = 22–25\%$), $\tau_2 = 49–87$ ps ($\alpha_2 = 49–54\%$) and a non-zero $\alpha_0$ constant (21–29%). Here, we should also point out that the low-temperature pump-probe measurements carried out in these samples revealed similar temporal behavior with time constants approximately 10–15 % longer.
In order to investigate the carrier dynamics on a longer time scale in the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs, transient PL measurements were obtained using $\lambda_p = 266$ nm and 100-fs pulses. Room temperature (RT), transient, or time-resolved PL (TRPL) of the as-grown $\beta$-Ga$_2$O$_3$ NWs reveal a single exponential decay with a time constant of 2.4 ns. We should also point out that TRPL at 10 K gave an identical decay and a twofold increase in the PL intensity. Similarly, RT TRPL of the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs exposed to H$_2$S at 400 °C consists of a single exponential decay with a time constant of 2.0 ns. On the other hand, TRPL measurements at room temperature of the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained at 500 °C show a single exponential decay at 680 nm with a much larger time constant of 19 μs (Fig. 6 and Fig. 7a).

The corresponding TRPL at 10 K reveals a new peak at 430 nm following a double exponential decay with time constants 6 ns (62%) and 42 ns (38%) (Fig. 7b). The red PL emission can be expressed by a double exponential decay with time constants of 1.8 μs (32%) and 26.9 μs (68%) (Fig. 7b). Similar results have also been observed for the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs at 600 °C. The 26.9-μs time constant is mainly associated with the red emission, and as expected, it is longer at low temperature whereas the short time constant is most likely associated with carriers moving into another state most likely non-radiative.

**Conclusions**

In conclusion, we have grown $\beta$-Ga$_2$O$_3$ NWs at 800 °C which were converted to $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs by post growth processing under H$_2$S between 400–600 °C for 60 min. We find that the as-grown $\beta$-Ga$_2$O$_3$ NWs exhibit state filling following carrier excitation for probing wavelengths between 340 to 450 nm corresponding to states lying energetically below the conduction band edge. In addition, the $\beta$-Ga$_2$O$_3$ NWs exhibited broad PL with a maximum at 435 nm (2.85 eV) and lifetime of 2.4 ns. Similar results have also been observed in the case of $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained under H$_2$S at 400 °C. In contrast, the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs obtained under H$_2$S at 500 to 600 °C exhibit state filling for $\lambda_p < 450$ nm related to the formation of Ga$_2$S$_3$ and free carrier absorption for $\lambda_p > 500$ nm related secondary excitations from formation mid-gap states in Ga$_2$S$_3$ located 1.1 eV below the conduction band edge. Furthermore, this is accompanied by a suppression of the broad PL at 435 nm and the emergence of strong emission at $\approx 680$ nm (1.8 eV) with a much longer lifetime of 19 μs. This long-life emission is associated with the transition of carriers from the 1.1 eV band to a band located at 0.4 eV above the valence band edge of Ga$_2$S$_3$. Finally, we
like to point out that the room temperature red emission of the $\beta$-Ga$_2$O$_3$/Ga$_2$S$_3$ NWs may be used in various applications including sensors, non-toxic biomedical imaging, and energy down-conversion in nanowire solar cells.

**Abbreviations**
NWs: nanowires; PL: photoluminescence; MO: metal oxide; SEM: scanning electron microscope; $\lambda_x$: excitation wavelength; $\lambda_p$: probing wavelength; RT: room temperature; LT: low temperature.

**Competing Interests**
The authors declare that they have no competing interests.

**Authors’ Contributions**
KWO, CC, and AO were responsible for the ultrafast and PL measurements. MZ was responsible for the growth and structural characterization of the nanowires. All authors have read and approved the final manuscript.

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