III–VI Chalcogenide Semiconductor Crystals for Broadband Tunable THz Sources and Sensors

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Abstract—The layered chalcogenide semiconductor GaSe has been grown under various crystal growth conditions for optimum performance for tunable terahertz (THz) wave generation and broadband THz detection. Low-temperature photoluminescence (PL), Raman spectroscopy, optical absorption/transmission, electrical charge transport property measurements, and THz time-domain spectroscopy (TDS) have been used to characterize the grown crystals. It is observed that indium doping enhances hardness of the grown GaSe crystals, which is very useful for processing and fabricating large-area devices. GaSe crystals have demonstrated promising characteristics with good optical quality (absorption coefficient $\leq 0.1 \text{ cm}^{-1}$ in the spectral range of 0.62–18 μ m), high dark resistivity ($\geq 10^9 \Omega$ cm), wide bandgap (2.01 eV at 300 K), good anisotropic (|| and \perp) electrical transport properties $(\mu_{e/h}, \tau_{e/h}, \text{ and } \mu \tau_{e/h})$ and long-term stability. The THz emission measurements have shown that the GaSe crystals are highly efficient for broadband tunable THz sources (up to 40 THz), and sensors (up to 100 THz). Additionally, new THz frequencies (0.1-3 THz) have been observed for the first time from an anisotropic binary and a ternary semiconductor crystal. Details of characterizations as well as optimum crystal growth conditions including simulation and computer modeling are described in this paper.

Index Terms—Crystal growth, optical characterization, terahertz (THz), time-domain measurements.

I. INTRODUCTION

AYERED chalcogenide semiconductors have been studied for a long time due to their unique properties coming from their layered structures. Their anisotropic properties result from strong covalent bonding within the layer planes and weak van der Waals type bonding between them [1]. Among various chalcogenides, the III–VI semiconductor GaSe has been studied for a long time due to its large nonlinear optical coefficient $(d_{22} = 75 \text{ pm/V})$ and its high structural anisotropy [1]–[4].

The nonlinear optical effects of GaSe can be utilized for generation and detection of broadband tunable terahertz (THz)

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radiation [5], [6]. In spite of its interesting characteristics, GaSe has not been widely studied due to the difficulty of growing and processing of large crystals resulting from its low mechanical hardness [7]. In order to address this issue, we have studied Indoped GaSe and Ge-doped GaTe crystals for THz applications. The crystals have been grown based on numerical simulation for optimizing THz wave generation and detection properties.

One of the primary difficulties in GaSe single crystal growth is its extremely low thermal conductivity (0.37 W/mK) along the c-axis near the melting temperature. Also, the thermal conductivity is anisotropic. The radial conduction in the solid is more efficient for heat removal from the growth interface compared to axial conduction in GaSe growth. Anisotropy and liquid/solid conductivity ratios are expected to strongly influence the interface shape, which, in turn, affects twinning and other defects. Furthermore, the Prandtl number of GaSe is about 2.8, which results in strongly coupled melt flow and heat transfer. It is expected that any disturbance of melt flow from the pulling rate and/or rotation rate will significantly affect the temperature distribution, and consequently, the interface shape. It is, therefore, extremely important to properly control the melt flow, growth interface, and solute transport. The crystal growth parameters were determined based on simulation and modeling studies using a numerical model, multizone adaptive scheme for transport and phase-change processes (MASTRAPP). We have also investigated two other chalcogenide semiconductors, GaTe and $GaSe_{x}Te_{1-x}$, as new THz sources for the first time. In this article, we report details of crystal growth based on numerical modeling and simulation, characterizations of the grown crystals, and the THz results for broadband tunable THz sources and sensors.

II. EXPERIMENTAL

Opto-electronic properties of THz crystals are strongly and negatively influenced by the presence of trace levels of residual impurities, since they substantially reduce charge carrier transport and optical absorption/transmission properties of the grown crystal. We have grown GaSe, GaTe, and GaSe_{0.5}Te_{0.5} crystals from stoichiometric amounts of high-purity (7 N, Alfa Aesar) Ga, and vacuum-distilled and zone-refined (\geq 7 N) Se or Te. For GaSe and GaTe crystal growth, 1000 ppm indium (In) and 0.8 wt% germanium (Ge) were added as dopants, respectively. For growing high-quality large single crystals, it is important to control heat transfer and the fluid flow pattern in the furnace. In order to predict heat transfer in the furnace, numerical modeling and simulation were conducted by MASTRAPP [8]–[11]. Based on simulation results, the temperature distributions in

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Fig. 1. (a) Stream function and temperature distributions in a quartz ampoule for a small diameter GaSe growth. (b) Thermal stress during GaTe crystal growth.

EIC's Bridgman furnace during GaSe and GaTe crystal growth operations were predicted as reported in our previous work [12]. Then, stream function and temperature distributions in a quartz ampoule of ~ 1 inch (~ 2.5 cm) diameter GaSe crystal growth were calculated. As shown in Fig. 1(a), the temperature distribution and fluid flow in the solid and melt phases are strongly affected by the length of the crystal and melt convection in the ampoule. Temperatures at the center and on the edge of a grown crystal are very different. A curved interface generally results and varies with the crystal length.

A thermo-elastic stress analysis was performed for a cylindrical crystal using a displacement-based model by the equilibrium equations:

$$\frac{1}{r}\frac{\partial}{\partial r}(r\sigma_{rr}) + \frac{\partial\sigma_{rz}}{\partial z} - \frac{\sigma_{\varphi\varphi}}{r} = 0 \quad \frac{1}{r}\frac{\partial}{\partial r}(r\sigma_{rz}) + \frac{\partial}{\partial z}\sigma_{zz} = 0$$

where σ_{rr} , σ_{zz} , and $\sigma_{\varphi\varphi}$ are the normal stress components in the radial, axial, and azimuthal directions, respectively, and σ_{rz} is the shear stress component. Thermal stresses were calculated from the Hooke's stress–strain relations for an isotropic material. The preliminary results of thermal stress in a GaTe crystal are shown in Fig. 1(b). Based on this modeling work, the crystal growth parameters were set. The crystal growth has been carried out using a modified vertical Bridgman growth method described earlier [3], [8]–[12].

The grown crystals were processed, and then, characterized by Raman spectroscopy, photoluminescence (PL), optical absorption, Hall effect, and current-voltage (I-V) measurements for determining structural, optical, charge-transport, and electrical properties. The mobility of the grown GaSe crystals were measured both along and perpendicular to the layer planes between 77 and 300 K at magnetic fields up to 0.5 T. Then, the crystals were characterized for THz applications as sources and sensors. For studying the emission and detection characteristics of THz radiation from the grown crystals, THz time-domain spectroscopy (THz TDS) systems at Rensselaer Polytechnic Institute and Yale University were used. The details of the RPI testing setup were discussed in our previous report [12]. For THz emission measurements at Yale University as shown in Fig. 2, a regeneratively amplified Ti-sapphire laser produced 100 fs, 800 nm pulses at 1 kHz with 800 mW average power, and the generated pulse was split into two beams with a non-



Fig. 2. Schematic experimental setup for THz TDS system at Yale University.

polarizing beam splitter, one to photoexcite the sample, and the other to detect the EM transient. The vertically polarized excitation pulse reached the sample after traversing a variable delay line that determined the time at which the sample was excited. A paper beam block ensured that any visible power not absorbed by the sample did not reach the detector. The beam block was transparent in the far infrared (FIR) region of the spectrum, and therefore, allowed the generated THz pulse to pass through. The other portion of the visible pulse was used to detect the electromagnetic (EM) transient via free-space electro-optic sampling (FSEOS) [13] in a 0.5-mm-thick $\langle 110 \rangle$ ZnTe crystal. The entire THz waveform was mapped out by scanning the variable delay line with a step size equivalent to 5-10 fs, and determining the value of the transient electric field at each moment in time. The sample could be rotated azimuthally about the surface normal, and/or about vertical or horizontal axes if oblique incidence was required.

III. RESULTS AND DISCUSSION

From the crystal growth procedures described earlier, we have been able to grow up to 10-cm-long and 2.5-cm-diameter ingots. It is reported in the literature that by doping 0.1-3 mass% In to GaSe, increased mechanical hardness was observed [14]. We have also measured significant increase of microhardness from 7.8 to \geq 14 kg/mm² by doping GaSe crystals with In. The enhanced microhardness of In-doped crystals allow us to cut and polish optical faces in any directions. The GaTe crystal was much harder than In-doped GaSe crystals and could be cut easily into various shapes and sizes. After processing the GaSe crystals, PL and Raman spectra were taken. Low-temperature photoluminescence spectra at 10 K of GaSe and GaSe:In crystals are shown in Fig. 3(a). The dominant peak of GaSe is the exciton bound to an acceptor (A^0, X) . The ground state free exciton $(X)_{n=1}$ and one-LO-phonon replica of the indirect free excitonic recombination (IFE) and the radiative recombination of indirect bound excitons to deep neutral-acceptor center (IBE) have been identified [15]. It is interesting to see that the $(A^0,$ X) peak almost disappears in GaSe:In, while a strong broad peak associated with indium emerges. The peak is attributed to a donor-acceptor pair (DAP) mainly, and electron to acceptor partly. It is generally accepted that the major acceptors in



Fig. 3. (a) Photoluminescence spectrum of as-grown GaSe crystal at 10 K. (b) Raman spectrum of GaSe crystal at room temperature.



Fig. 4. (a) Optical absorption spectrum of GaSe crystal. (b) Terahertz spectra generated from 180 μ m GaSe, the detector was 30 μ m GaSe.

undoped GaSe are V_{Ga} . Doped with indium, most of gallium vacancies are occupied by indium, and the In_{Ga} may be a nonradiative center. Therefore, the (A^0, X) peak decreases significantly while the $(X)_{n=1}$ peak becomes dominant in GaSe:In. It is worth noting that the acceptor levels observed by DLTS are deeper than those measured by PL [16]. It is also noted that the temperature dependence of the peak energy showed a similar trend as the literature, but there was slight shift in the peak energies. The observed Raman modes in Fig. 3(b) matched well with the reported and identified modes in the literature with a slight lower frequency shift presumably due to measurement temperature difference. However, E'(TO) and 2E' (LO) modes were not observed in our crystal [17].

Following the PL and Raman spectra measurements, optical absorption measurements were carried out. The optical absorption measurement data in Fig. 4(a) showed good optical quality (absorption coefficient $<0.1 \text{ cm}^{-1}$ in the spectral range of 0.62–18 μ m) with the bandgap of 2.01 eV at 300 K. The hole mobility measurements showed that $\mu_{\parallel} = 190-210 \,\mathrm{cm}^2/\mathrm{V}\cdot\mathrm{s}$ and $\mu_{\perp} = 120 - 130 \,\mathrm{cm}^2/\mathrm{V} \cdot \mathrm{s}$ at 300 K, while these values increased to 585–590 $\rm cm^2/V \cdot s$ and 260–270 $\rm cm^2/V \cdot s$ near 77 K. The electron mobility at 300 K was $\mu_{\parallel} = 80 \,\mathrm{cm}^2/\mathrm{V}\cdot\mathrm{s}$ and $\mu_{\perp} = 300 \,\mathrm{cm}^2/\mathrm{V} \cdot \mathrm{s}$, respectively. Both hole and electron mobility measurements showed anisotropic electrical transport properties of GaSe. Then, hole and electron mobility-lifetime products were measured using Hecht analysis that showed comparatively similar values with $\mu \tau_{\rm h} \sim 1.5 \times 10^{-5} \, {\rm cm}^2 / {\rm V}$ and $\mu \tau_{\rm e} \sim 1.4 \times 10^{-5} \, {\rm cm}^2 / {\rm V}$. The current–voltage measurements showed that the resistivity of GaSe crystal was $\geq 10^9 \Omega$ cm. The Ge-doped GaTe showed a resistivity in the similar range with



Fig. 5. Frequency domain spectrum. (a) In:GaSe. (b) Ge:GaTe crystals.



Fig. 6. Time-domain waveforms of GaSe_{0.5}Te_{0.5} crystal.

slightly lower values. The high resistivity of GaTe is attributed to Ge doping and high-purity precursors [18], [19].

The crystals were then evaluated as THz detectors and/or emitters using THz–TDS systems. For undoped GaSe crystals, the GaSe crystals showed emission up to 40 THz and also detection capabilities up to 40 THz, as shown in Fig. 4(b). The In-doped GaSe crystals and Ge-doped GaTe crystals showed terahertz signals in 100 GHz–2 THz range, as shown in Fig. 5. The THz emission characteristics are tunable by rotating the crystals [20], [21].

An EIC-grown $GaSe_{0.5}Te_{0.5}$ crystal was also tested as a possible new terahertz source. Fig. 6 shows the time-domain waveforms of this new crystal. From Fig. 6, it is clear that the $GaSe_{0.5}Te_{0.5}$ crystal can work as a strong terahertz emitter. As far as the authors' knowledge and thorough literature search are concerned, it is the first report of THz emission from this ternary chalcogenide crystal. The $GaSe_{0.5}Te_{0.5}$ crystal is interesting because it has a potential of tuning the THz emission and detection characteristics by controlling stoichiometry of the grown crystal.

IV. CONCLUSION

We report crystal growth and characterization of doped GaSe and GaTe crystals for broadband THz sources and sensors. The grown crystals based on simulation results showed good material properties. It was observed that In doping improved the mechanical hardness of the GaSe crystal without noticeable changes in other material properties. The enhanced mechanical properties will allow us for scaling up, leading to higher efficiency and output power.

The emission results in the THz range for the doped GaSe and GaTe crystals show that they are promising as THz sources. Additionally, THz emission from a ternary $GaSe_{0.5}Te_{0.5}$ crystal was observed. This new THz crystal can give us the capability of tuning THz spectra by controlling the stoichiometry. The grown THz crystals are promising for various applications including biochemical and trace explosive vapor identification, security screening, and medical imaging.

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