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Optical properties of Te-doped GaSe crystal

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Abstract: ε-GaSe crystals are grown with the stoichiometric GaSe of 0.05%, 0.1%, 0.5%, 1% and 2% (mass percent) Te and are characterized by GaSe:Te(0.01%, 0.07%, 0.38%, 0.67% and 2.07% (mass percent)) crystals. The transformation of the rigid layer phonon modes with doping is studied for the first time. The absorption peak of the rigid mode $E'^{(2)}$ centered at ~0.59 THz is rising up in the intensity till reaching a maximal value on the first stage of the doping concentration less than 0.38% (mass percent). This process correlates well with the improvement in the optical property. Further doping is resulting in the decrease of the intensity till vanishing the $E'^{(2)}$ absorption peak at 1% (mass percent) Te. Simultaneously with the $E'^{(2)}$ absorption peak decreasing, the absorption peak of the rigid mode $E'^{(2)}$ centered at 1.78 THz is rising up in the intensity. The two processes correlate well with the degradation in the optical quality of GaSe:Te crystal. The doping level that results in the highest intensity of the absorption peak of the rigid layer mode $E'^{(2)}$ is proposed as a criterion in the identification of the optimal Te-doping in GaSe crystal that is confirmed by THz generation via optical rectification.

Key words: GaSe; GaSe:Te crystal; crystal growth; optical property; THz

掺碲硒化镓晶体的光学性能

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摘要:采用水平区熔法生长了碲(Te)掺杂浓度(质量百分比)分别为 0.05%,0.1%,0.5%,1%,2% 的硒化镓(GaSe)晶体,并分别对掺杂浓度为 0.01%,0.07%,0.38%,0.67%,2.07% 的 GaSe:Te 晶体的光学性能进行了表征。首次研究了 GaSe:Te 晶体中刚性层声子模式的转换。吸收光谱测试结果表明:当 Te 掺杂浓度小于 0.38% 时,振动中心位于 0.59 THz 附近的 $E'^{(2)}$ 刚性模式吸收峰强度可达最大值,这一过程与 GaSe:Te 晶体光学性能的提高密切相关。但 Te 掺杂浓度的进一步提高会导致 $E'^{(2)}$ 刚性模式吸收峰强度逐渐减弱,当 Te 掺杂浓度为 1% 时,$E'^{(2)}$ 刚性模式吸收峰基本消失。这两个过程与 GaSe:Te 晶体光学质量的下降密切相关。因此,$E'^{(2)}$ 刚性模式吸收强度达到最高时对应的掺杂浓度即是 GaSe:Te 晶体中 Te 的最佳掺杂浓度,光整流产生太赫兹过程证实了此结论的正确性。

关　键　词:硒化镓;掺碲硒化镓;晶体生长;光学性能;太赫兹

中图分类号:O734; TN304.2　　**文献标识码**:A

1　Introduction

Due to a set of extreme optical properties, GaSe has been successfully employed to generate coherent radiation in the mid-infrared and down to the terahertz (THz) frequency range[1-2]. On the other hand, low hardness and easy cleaving lead to low optical properties that hamper the applications of large-area crystals. Fortunately, GaSe is a good matrix material for doping with various impurities. An original ε-polytypic structure of GaSe is strengthened by doping impurities and other physical properties being responsible for the frequency conversion efficiency and exploitation parameters are also significantly modified[3-9]. Nevertheless, while THz generation by optical rectification and down-conversion in GaSe has been studied in detail[2,10-11], there are very few tasks devoted to the experimental study of THz range optical properties and generation in doped GaSe. Optical properties and efficiency of THz generation is studied experimentally in S-doped GaSe (GaSe:S)[12-17], GaSe:In[15-16,18], GaSe:Er[19-20], GaSe:Al[21] and GaSe: Te[15-16,18,22-23]. The studies of the physical properties and THz generation in the intricate crystals grown from the melt GaSe:$AgGaS_2$ (identified as double doped GaSe:S:Ag) and GaSe:$AgGaSe_2$ (identified as GaSe:Ag) are reported[21,24], relatively.

Beyond mid-IR, the THz absorption of a nonlinear crystal places a practical limit on frequency range of optical rectification and down conversion. The THz absorption of a crystal is usually attributed to infrared-active phonon modes or their combination modes. The impact of the phonon modes including rigid layer mode $E'^{(2)}$ centered at 0.586 THz on refractive indices and THz generation efficiency in GaSe:Er was studied experimentally in Ref. [20]. Second order phonon modes were identified in centimeter-sized high optical quality GaSe:S crystals in Ref. [12-13]. No rigid mode transformation with doping were ever been reported but rising up of the rigid mode $E'^{(2)}$ at 1.78 THz in intensity[14], so as degeneracy of the rigid layer mode $E'^{(2)}$ in low doped GaSe:S[14,17] and GaSe:Te[25]. Thus, the available data on optical properties in centimeter-sized doped GaSe in the THz region are still inconsistent. Besides, no optimal doping by an impurity in GaSe has ever been reported.

This work reports the growth of hexagonal structure GaSe doped with 0.05%, 0.1%, 0.5%, 1%, 2%, 5% and 10% (mass percent) Te or GaSe:Te (0.05%, 0.1%, 0.5%, 1%, 2%, 5% and 10% (mass percent)) in the charge composition and for the fist time in our knowledge experimental study of the rigid modes transformation with doping is carried out. Criterion for determination of the optimal Te-doping is proposed and checked in an optical rectification experiment.

2 Crystal growth and characterization

2.1 Crystal Growth

Creation of $GaSe_{1-x}Te_x$ crystals involves two principle steps. Initially synthesis polycrystalline material with the mass of 120 – 150 g has been produced in a two-zone horizontal furnace by using high purity (99.999 9%) gallium (Ga), selenium (Se), and 99.9% tellurium (Te). A weighted charge of Ga and Se were placed in the boats located at hot and cold ends of the ampoule. Chemical reaction of the reagents up to GaSe formation has been produced on the first stage by sublimation of Se at 690 ℃ and interaction of the vapor with Ga melt at 970 ℃, that is, the GaSe compound is synthesized under selenium vapor pressure in the reaction ampoule. The second stage provides the melt homogenization at 1 000 ℃ due to diffusion, whereas on the third stage the melt is cooled for 36 h and the homogeneous large-block GaSe ingots are grown. The process of GaSe synthesis is performed by three sequential stages at different temperature profiles over the ampoules as it is described in details elsewhere[26-32]. Doping by Te atoms was developed by 0.05%, 0.1%, 0.5%, 1%, 2%, 5% and 10% (mass percent) into the boat with gallium during synthesis of the compound. The temperature gradient at the crystallization front was 10 ℃/cm and crystal pulling rate was 10 mm/d. The samples were prepared by cleaving an as-grown ingot parallel to the *c*-plane layer and used without any additional treatment. The lengths of the specimens prepared and studied were 1.14, 1.0, 1.0, 0.98 and 0.86 mm, respectively.

2.2 Crystal composition and structure

The composition of the GaSe:Te crystal was provided by Z-8000 atomic-absorption spectrometry, Hitachi spectrometer (air-acetylene flame) and by inductively coupled plasma optical emission spectrometry (ICP-OES) with spectrometer iCAP 6500, Thermo Scientific after dissolution of sample weights in nitric acid. Crystals grown from the charge with 0.05%, 0.1%, 0.5%, 1% and 2% (mass percent) of Te are identified as GaSe : Te (0.01%, 0.07%, 0.38%, 0.67% and 2.07% (mass percent)) crystals, respectively. The ε-polytype structure of the observed specimens was identified by the proposed non linear method[33] through φ-angle dependence of a femtosecond Ti:sapphire laser optical rectification. For all crystals, rectified signal versus φ-angle was clear six-petal-flower type which is similar to that in ε-GaSe, as it goes from the relation for efficient nonlinear susceptibility coefficient $d_{\text{eff}} = d_{22}\cos\theta\sin 3\varphi$ for the type-I of three frequency interactions.

2.3 Optical properties

UV-visible transmission of the close length specimens observed was recorded by Cary 100 Scan (Varian, Inc., Austria) spectrometer: wavelength range is 190 900 nm, spectral resolution is 0.2 – 4 nm, wavelength deviation is ± 1 nm. Mid-IR transmission was recorded by FTIR Nicolet 6700 (Thermo Electron Corp.) spectrometer: operation wavelength range is 11 000 – 375 cm^{-1}, spectral resolution is 0.09 cm^{-1}. Selected spectra are presented in Fig. 1(a) and (b).

Absolute values of the attenuation coefficients at maximal mid-IR transparency range were measured at chosen points on the crystal faced with low power ϕ1.0 mm beam at wavelength of 9.6 μm CO_2 laser band to minimize the influence of the surface defects on the measurement results.

Terahertz range absorption coefficient and n_o spectra in the crystals were determined by a home-made THz-TDS spectrometer with 50 fs Ti:sapphire laser system (800 nm) described elsewhere[14]. The THz beam was normally incident to the crystal face. The THz absorption and dispersion spectra are shown in Fig. 1(c) and (d).

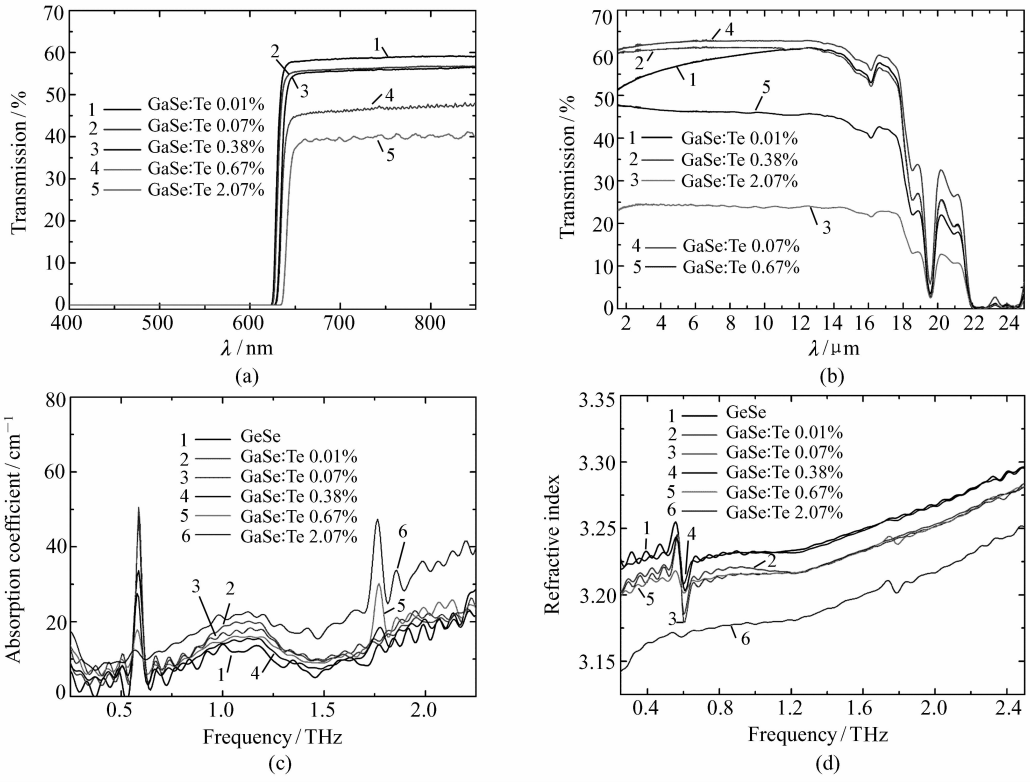

Fig. 1　UV-visible(a), mid-IR(b) transmission, THz o-wave absorption(c) and n_o dispersion spectra(d) in GaSe:Te.

3　Results and discussion

Crystals grown from the charge with 0.05%, 0.1%, 0.5%, 1% and 2% (mass percent) of Te are identified as, respectively, ε-GaSe:Te(0.01%, 0.07%, 0.38%, 0.67% and 2.07% (mass percent)) single crystals that are suitable for THz generation via optical rectification and down conversion. At maximal transparency range of mid-IR GaSe:Te (0.01, 0.07, 0.38 mass%) crystals possesses *o*-wave absorption coefficient α from ~ 0.2 cm^{-1} to 0.5 cm^{-1}. GaSe:Te(0.67%, mass percent) crystals is characterized by dramatically increased $\alpha \geqslant 5$ cm^{-1} mainly due to high density of Te precipitates. Crystals grown from the charge with 5% and 10% (mass percent) Te appear as usefulness polycrystalline low-optical-quality material.

In Fig. 1(a), it is seen that the structure of phonon modes in mid-IR are independent but rigid modes in THz range transforming with Te-doping (Fig. 1(c)). The rigid layer mode $E'^{(2)}$ at ~ 0.59 THz is expected to be the mode in which the layer vibration are as rigid units against each other and that there is no relative displacement of the Ga and Se atoms within a layer. For low Te-doped crystals the center of the $E'^{(2)}$ mode is well in coincidence with the data for pure GaSe in Ref. [34]. Measuring the phonon absorption spectra in mm-length crystals we report $E'^{(2)}$ is rising up till reaching the highest value at Te-doping between 0.07% and 0.38% (mass percent). This process correlates well with the improvement in the optical quality in GaSe:Te due to the decreasing in the number of point and layer stacking defects[35].

The rigid layer mode $E'^{(2)}$ is decreasing in the intensity till degeneracy with further Te-doping. Simultaneously, the absorption peak of the rigid mode $E''^{(2)}$ centered at 1.78 THz is rapidly rising up in the intensity and noticeably influence the n_o dispersion at Te-doping $\geqslant$0.67% (mass percent) (Fig. 1(d)). It is the mode in which the layers vibrate a-

gainst each other with the relative displacement of two Ga-Se sub-layers within a four-atom layer[36]. It was established that both $E'^{(2)}$ rigid layer mode degradation and $E''^{(2)}$ rigid mode rising up correlate well with the decreasing in the crystal optical quality that is due to numerous reasons such as structural defects (polytypism, stacking faults, dislocations)[35,37-38], defect complexes [9,37], exciton phonon and exciton impurity interactions[37], interlayer interstitials[35,39] and internal strains[40].

No a distinct reason was formulated to explain the rising up in intensity of $E''^{(2)}$ rigid mode with Te-doping. Possibly interlayer intercalation of larger size of Te atoms[9,37] leads to the formation of the local strained regions that bond hard Se and Ga layers.

4 Conclusion

The rigid mode transformation in GaSe∶Te crystal was studied for the first time . This process correlates well with the transformation of the optical quality in GaSe∶Te. Doping level that is resulting in the highest intensity of the absorption peak of rigid layer mode $E'^{(2)}$ is proposed as the criterion of optimal Te-doping in GaSe.

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