Thickness dependence of a giant nonlinear saturable absorption response in GeSb₄Te₇ thin films

Jie Fang¹, Jun Wang¹, Xinran Cao¹, Yixin Man¹, Chunmin Liu¹, Ling Cheng¹, Xia Zhang¹, Haibin Zhao¹,², Hao Zhang¹,² and Jing Li¹,²

¹ Shanghai Engineering Research Center of Ultra-Precision Optical Manufacturing, Department of Optical Science and Engineering, Fudan University, Shanghai, 200433, People’s Republic of China
² Laser Research Institute, Qufu Normal University, Shandong 273165, People’s Republic of China
³ Key Laboratory of Micro and Nano Photonic Structure (Ministry of Education), Fudan University, Shanghai 200433, People’s Republic of China

E-mail: lijing@fudan.edu.cn

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Abstract
Nonlinear optical absorption (NOA) materials with large saturable absorption response and high damage threshold are useful for a variety of applications such as ultrafast nonlinear optical devices and laser mode lockers. In this study, a suite of GeSb₄Te₇ thin films with different thicknesses were prepared, and the surface morphology, structure and linear optical absorption of these films were investigated in detail. Through an open aperture Z-scan technique with femtosecond pulses at 800 nm wavelength, it is found that effective NOA coefficient as high as $-93$ 009 cm G W⁻¹ can be achieved in GeSb₄Te₇ thin films. Besides, GeSb₄Te₇ thin films exhibit a giant and ultrafast third-order nonlinear optical saturable absorption response due to significant band filling effect. Especially, NOA response, coefficient and damage threshold can be well controlled by adjusting the film thickness. Both stability and efficiency can be acquired by thermal treatment.

1. Introduction
Nonlinear optical absorption (NOA) materials are widely used in many photonic devices [1–3], such as optical limiting [4], super-resolution imaging [5] and multiphoton pumped lasing [6]. In such a scenario, finding and investigating advanced NOA materials as well as understanding their basic physical mechanisms are of great importance. As one major candidate, phase-change materials (PCMs) have attracted great attention due to their proper band gap [7–10].

The series of germanium antimony telluride (Ge–Sb–Te) compounds is an important family of PCMs, which is basically a pseudo-binary alloy, and the exact composition can be described by the GeTe–Sb₂Te₃ binary phase diagram. Although Ge–Sb–Te compounds are famous for their excellent phase-change properties, they have also been reported to have excellent NOA properties [9]. GeSb₄Te₇ is a direct-band-gap semiconductor. Among three most common ratios of Ge–Sb–Te compounds, i.e. 2:1 (Ge₂Sb₂Te₅), 1:1 (GeSb₂Te₄) and 1:2 (GeSb₄Te₇), GeSb₄Te₇ is the most active one with the fastest phase change speed and the lowest annealing temperature due to its high-proportioned Sb₂Te₃ [7].

Till now, many PCMs containing Sb₂Te₃ have been reported to have giant NOA. For example, AgInSb₂Te₃ thin films, which are used in the recording layer and mask layer of phase change memorizers, are reported to have an NOA coefficient up to $3.5 \times 10^{-2}$ m W⁻¹ caused by the free carrier absorption [8]. Ge₂Sb₂Te₅ thin films are the key material in optical recording as well as super-resolution imaging reading and meanwhile have an NOA coefficient up to $4.95 \times 10^{-2}$ m W⁻¹ [9]. The crystalline Sb₂Te₃ thin films themselves are used in the model structure and the layer mask, and also possess giant saturation absorption (SA) caused by the saturated direct-band-gap transition [10]. Due to their fast phase change speed and giant NOA response, PCMs containing...
Sb$_2$Te$_3$ have been widely applied as high density optical recording medium as well as mask layer of super-resolution near-field structure. However, most previous reports on GeSb$_4$Te$_7$ thin films only focused on linear optical and electrical applications [11–13]. To our knowledge, there are still little descriptions in the literature of three-order nonlinearity to date. Moreover, considering its topological insulating behavior currently demonstrated [14], Ge–Sb–Te compound is such an interesting material that is worth investigating deeply.

In this study, we prepare a suite of GeSb$_4$Te$_7$ thin films with different thicknesses and study their NOA properties using the OA Z-scan technique. Moreover, the giant NOA response of GeSb$_4$Te$_7$ thin films have been investigated deeply in detail. It is found that there is a positive correlation between NOA response and thickness.

2. Experimental

A suite of GeSb$_4$Te$_7$ film samples were deposited on fused quartz substrates by the radio-frequency (RF) magnetron sputtering method using a 99.99% GeSb$_4$Te$_7$ target. Working pressure is $2.8 \times 10^{-3}$ mbar. During the sputtering process, substrates were at room temperature and the sputtering rate was constant. Therefore, films with different thicknesses were prepared by varying deposition times, i.e. 100 s, 150 s, 200 s, 400 s, 600 s, 800 s, labeled as S1, S2, S3, S4, S5, S6 respectively. To realize hexagonal close-packed (HCP) crystalline structures, all the films were annealed in flowing nitrogen at 503 K for 60 min and then cooled down to room temperature.

First, atomic force microscopy (AFM) (Shimadzu SPM-9500J3) was employed to measure the surface morphology and approximate thickness of the samples. Then, ellipsometer measurement data were fitted with the Lorentz oscillator model to determine the exact thickness, refractive index and extinction coefficient. Moreover, absorption spectra were measured by a double light-path UV–vis–NIR spectrophotometer (Shimadzu UV-3600), in which an identical fused quartz substrate was set in a reference light path to offset the absorption of substrate. The structural analysis was carried out using an x-ray diffractometer (Bruker D8 ADVANCE), with a Cu-K$_\alpha$, $(\lambda = 1.540 56 \text{ Å})$ radiation source in a $2\theta$ range of 10$^\circ$–70$^\circ$.

As for the nonlinear optical absorption properties of GeSb$_4$Te$_7$ thin films, single beam Z-scan technique was performed. The setup of our Z-scan system was shown in our previous paper [15]. The excitation pulses were generated by a Ti:sapphire regenerative amplifier system (Spectra Physics, Spitfire Ace) at the wavelength of 800 nm with 100 fs pulse duration and 1 kHz repetition rate. In the light path, the incident beam was focused by a convex lens $(f = 300 \text{ mm})$ and then vertically irradiated onto GeSb$_4$Te$_7$ thin films, which can be moved along the light direction by a stepping motor. The beam radius $\omega$ at the focal point is $\sim 32 \text{ µm}$ measured by repeated knife-edge method. The Rayleigh length $(Z_R = (\pi \omega^2)/\lambda)$ is $\sim 4 \text{ mm}$, which is large enough for the validity of the Z-scan theory [16] compared to the thickness of each sample and substrate.

3. Results and discussion

3.1. Morphological and structural analysis

In figure 1, the AFM images for the surface topographies of annealed samples indicate that crystallized GeSb$_4$Te$_7$ thin films have homogeneous surfaces with fine surface roughness. With the increase of the thickness, the grains become bigger and more compact with each other gradually, and the surface roughness increases from 0.253 to 0.562 nm. Thereby, in the case of GeSb$_4$Te$_7$ thin film (~10–100 nm scale), thicker films have better crystallization, leading to higher free carrier concentration.

Moreover, based on AFM step measurement and through ellipsometric data fitting, the thicknesses of films were measured to be 14 nm, 25 nm, 41 nm, 80 nm, 125 nm and 165 nm respectively, corresponding to S1, S2, S3, S4, S5 and S6 samples.

During the thermal treatment, GeSb$_4$Te$_7$ thin films have two phase change processes including (1) at about 410 K, thin films transform from amorphous to cubic close packing (FCC) phase, which is called as the metastable state. (2) At about 485 K, thin films further transform into the HCP phase, which is called as the stable state [12].

Figure 2 is the XRD patterns of the as-deposited and annealed GeSb$_4$Te$_7$ thin films. The amorphous nature of the as-deposited film was unveiled by the lack of diffraction peaks. After annealing at 503 K, all the diffraction peak positions are clear and exhibit preferred (103), (106), (210) and (204) orientations as previously reported [17, 18] revealing that all the annealed samples have crystallized into HCP phase. However, the peaks in S1–S3 are relatively less acuminate compared to those in S4–S6. This is due to a higher percentage of defects in the thinner films (S1–S3). With the increase of film deposition time, the proportion of defects in the films decreases while the films (S4–S6) become thicker.
3.2. Linear optical properties

In order to understand the nonlinear optical properties of GeSb$_4$Te$_7$ thin films, the characterization of linear optics is necessary. By fitting the ellipsometric data using the Lorentz oscillator model, the dependences of refractive index $n$ and extinction coefficient $k$ on wavelengths $\lambda$ are shown in figure 3. The patterns are basically consistent with the previous results [19] and thus give us reliable values for Tauc plot method and NOA coefficient calculation.

The linear optical absorption spectra of annealed GeSb$_4$Te$_7$ thin films are shown in figure 4. The films have relatively small absorption at the excitation wavelength of 800 nm and express a plot reverse from the visible to the near infrared, indicating the presence of defects, such as vacancies, stacking faults and surface defects in GeSb$_4$Te$_7$ nanostructures [12, 13]. These defects will increase the concentration of free carriers. Especially in S1–
S3, fluctuating trends are notable. Such a fluctuation means a high concentration of defects and it may cause absorption jump, which usually results in a good damage-endurable ability. Moreover, while S1 and S2 have a similar trend, the variations of S3–S6 are also comparable. Therefore, between S2 and S3, the influence of thinner films on deposit defect proportion decreases. Based on the fluctuation of defect ratios, the overlap of the curves of S2 and S3 may come from the competition of intrinsic absorption and defect absorption. Although defects are unwanted in bulk materials because of the harmful impact on structure and device performance [20], for thin films, it is found that the existence of defects should significantly augment the performance diversity through the thickness dependence.

Besides, the difference among the absorption spectra can also be explained by the influence of film thickness on the optical band gap. Therefore, the Tauc plot method is applied. The dependence of $\alpha h \nu$ on $h \nu$ will give a straight line in the linear region and extrapolating the linear portion will give a horizontal axis intercept, which is the value of the optical band gap for the allowed direct transition [21]. Herein $\alpha$ is the linear optical absorption coefficient and $h \nu$ is the photon energy. As demonstrated in figure 5, band gap decreases from 0.92 to 0.72 eV with increasing thickness. The optical band gaps obtained by Tauc plot method are in good agreement with reported values [12, 22], although the presence of defects may usually make band gaps slightly larger [23]. Moreover, a comparable thickness dependence of optical band gap is also observed in our previous work [15], which is found to originate from the quantum-confinement effect [24, 25]. Then the variation of optical band gap, containing the effect of both defect and physical energy gap, further affects the variation of NOA in the samples with different thicknesses.

3.3. Nonlinear optical properties

The nonlinear optical properties of both amorphous and crystalline GeSb$_4$Te$_7$ films have been investigated by using the single beam OA mode Z-scan method with 800 nm femtosecond laser pulses. Importantly, the fused quartz substrates have little NOA response until the focal point power density $I_0 = 3600$ GW cm$^{-2}$, and during the test the applied $I_0$ was only 120 GW cm$^{-2}$. Therefore, the influence of the substrates can be ignored. Also, it should be noted that with such low-intensity pulses, both high-order nonlinearity and nonlinear scattering are avoided. The fitting curves of recorded normalized data are shown in figures 6(a)–(f). The clear normalized transmittance peaks reveal the presence of SA. All the curves of GeSb$_4$Te$_7$ films are repeatable and in symmetry with respect to the focal point. Hence, the results mainly come from strong light-induced NOA rather than phase change or structure-induced one [8].
Moreover, the modulation depth (peak height) of crystallized GeSb4Te7 films enhances as the film thickness increases and it can be explained by the variation of optical band gap. As discussed in figure 5, defects effectively blur the energy gap and the optical band gap represents the total impact of both defect and physical band gap. With the same incident photon energy, a smaller optical band gap will lead to a greater SA response as it is easier to saturate. Since optical band gap decreases with thickness increasing in figure 5, modulation depth thus shows a positive dependence on thickness. Similar thickness dependence of nonlinear SA responses also appears in amorphous films, but much weaker. Actually, the small SA increase in amorphous films is mainly caused by the increase of light–matter interaction length when sample gets thicker, which is also included in crystallized samples but negligible compared to the thickness-related variation of defects and free carriers. Just as shown in figures 6(a)–(f), the crystalline GeSb4Te7 thin film has an increasingly larger modulation depth than that of the amorphous one when the thickness increases. This is due to the higher free carrier concentration in thicker film samples when the crystallinity increases as discussed in figure 1. Therefore, we find thermal treatment beneficial for the effectiveness and diversity of GeSb4Te7 thin films in NOA properties.

Generally, chalcogenide semiconductors have a large NOA response because of their suitable band gaps. Herein this study, the GeSb4Te7 thin film even shows a one-order-of-magnitude-higher modulation depth than other semiconductors [9], which is up to 20. Such a giant NOA response can be explained by the significant band filling effect near Fermi level due to high–proportioned Sb2Te3 in GeSb4Te7. Jun-Woo Park et al investigated GeTe, Ge2Sb2Te5, GeSb2Te4, GeSb4Te7, Sb2Te3 [22], and suggested that the properties of GST materials change significantly with increasing Sb2Te3 proportion because of the notably different chemical activeness of GeTe and
Sb₂Te₃ near Fermi level. Especially, when the Sb₂Te₃ proportion increases from 1/2 (GeSb₂Te₄) to 2/3 (GeSb₄Te₇), the band structure of crystalline GST changes from indirect gap to direct gap, which gives GeSb₄Te₇ outstanding linear optical properties. We believe that it also gives GeSb₄Te₇ excellent NOA properties.

Near Fermi level, compared to that of Sb₂Te₃, the contribution of GeTe to the bonding of GeSb₄Te₇ is negligible. Hence, we can mainly consider the impact of Sb₂Te₃ on the overall performance of GeSb₄Te₇. For Sb₂Te₃, its covalent bond nature and significant band filling effect near Fermi level have been proved in [10]. Covalent bond usually means good damage-endurable ability and significant band filling effect will induce great NOA responses. We assume that when the GeSb₄Te₇ thin films are irradiated by an 800 nm femtosecond laser pulse, free carriers are generated based on the total effect of intrinsic absorption and defect absorption, which can be represented by optical band gap. The photo-induced carriers continually occupy the conduction band. The photo-induced carriers continually occupy the conduction band, although the laser intensity increases. To test the validity of the investigation mentioned above for GeSb₄Te₇, we put measured optical band gaps into the following equation for the absorption coefficient change [10]

\[
\Delta \alpha_d(h\omega) = -\frac{16\pi^2}{3} \frac{\alpha^2 h^2}{m_e} \left( \frac{m_e}{m_i} \right)^{\frac{3}{2}} \frac{1}{\hbar^2} E_g^3 \exp \left[ -\frac{\mu (h\omega - E_g)}{m_i k_B T} \right]
\]

Considering photon energy h\omega and optical band gaps E_g, we find the pump--induced absorption coefficient change \( \Delta \alpha \) is negative, which is consistent with the SA in figure 6.

Theoretically, the NOA response of Sb₂Te₃ may be little greater than that of GeSb₄Te₇. However, our results of GeSb₄Te₇ using femtosecond laser pulse in figure 6 are greater than that of Sb₂Te₃ using picosecond laser pulse [10]. This is because the reduction of the laser pulse width can improve the NOA response, which has been pointed out in another research [8]. Afterall, the significant thickness dependence of NOA responses in GeSb₄Te₇ has not been observed in Sb₂Te₃, which indicates more diversified applications of GeSb₄Te₇.

3.4. Damage threshold and effective NOA coefficient

For application purpose, the damage thresholds of GeSb₄Te₇ thin films were investigated. As shown in figure 7, damage thresholds can be increased by adjusting thickness, which is up to 303.4 GW cm⁻² when the sample is 165 nm thick. This is pretty good compared to the common low damage thresholds of other semiconductor nonlinear materials because of their narrow band gaps. Such a good damage-endurable ability comes from the specific defects of GeSb₄Te₇ thin films as mentioned in figure 4 and the stability of covalent bonds.

Also from the view of practicality, the amorphous GeSb₄Te₇ thin films are unstable especially when ambient issues break, and show a poor effectiveness and diversity. Therefore, we concentrate on the effective NOA coefficient \( \beta_{\text{eff}} \) of crystallized GeSb₄Te₇ thin films at 800 nm wavelength. Based on Sheik-Behaes’s OA Z-scan theory, \( \beta_{\text{eff}} \), defined as \( \alpha = \alpha_0 + \beta_{\text{eff}} \cdot I \), is calculated by fitting equation (1) [10]

\[
T_{\text{OA}} = \sum_{m=0}^{\infty} \frac{(- \beta_{\text{eff}} L_{\text{eff}} / (1 + z^2/z_0^2))^{m+1}}{(m + 1)^3/2},
\]

where \( T_{\text{OA}} \) is the normalized transmittance, \( L_{\text{eff}} = (1 - e^{-\alpha_1 L}) / \alpha_0 \) is the effective thickness of the thin film, \( \alpha_0 \) is the linear absorption coefficient at 800 nm wavelength, \( L \) is the physical thickness of the thin film, \( z \) is the light direction displacement of the sample, \( z_0 \) is the Rayleigh length. Typically, only several leading terms of the polynomial are necessary for numerical calculation, but the larger \( \beta_{\text{eff}} \) is, the more terms are required. For the \( \beta_{\text{eff}} \) of GeSb₄Te₇ thin films, we find that, with \( m = 4 \), enough accuracy can be acquired.

Both sign and magnitude of \( \beta_{\text{eff}} \) can be acquired through the fitting curves (\( \beta_{\text{eff}} < 0 \) in the case of SA). For crystallized samples, the \( \beta_{\text{eff}} \) values of S1, S2, S3, S4, S5 and S6 at the wavelength of 800 nm are shown in table 1,
which are pretty larger than those of other semiconductor thin films, such as In$_2$Te$_3$ [15], ZnO [26], CdO nanomorphotypes under 800 nm femtosecond pulse excitation. Compared with ferroelectric thin films such as Bi$_{3.15}$Nd$_{0.85}$Ti$_3$O$_{12}$ [27] as well as some organic materials such as CdSe-doped PMMA [28] and graphene oxide metal porphyrin films, the advantage of GeSb$_4$Te$_7$ thin films is also imposing [29]. Although its damage threshold is smaller than those of organic materials as a weakness of semiconductor, GeSb$_4$Te$_7$ thin film does much better than most semiconductors. Concisely, general modulations of the giant NOA response, coefficient and damage threshold of GeSb$_4$Te$_7$ thin films by thickness dependence are its major advantages. Along with the combination of both stability and effectiveness after thermal treatment, the potential applications of GeSb$_4$Te$_7$ thin films in ultrafast nonlinear devices are foreseeable.

### 4. Conclusion

In summary, GeSb$_4$Te$_7$ thin films with different thicknesses were prepared by the RF magnetron sputtering method. Surface morphology, structure and linear optical properties were investigated and the results provide crucial values for nonlinear analysis and calculation. NOA properties were then studied by OA Z-scan method. Surface morphology, structure and linear optical properties were investigated and the results provide excellent NOA material in future applications like ultrafast nonlinear optical devices and laser mode lockers.

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### ORCID iDs

Jing Li [https://orcid.org/0000-0001-9019-7449](https://orcid.org/0000-0001-9019-7449)

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