Optical and Dielectric Properties of Chalcogenide Glasses at Terahertz Frequencies

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Terahertz time-domain spectroscopy has been used to study the optical and dielectric properties of three chalcogenide glasses: $Ge_{30}As_8Ga_2Se_{60}$, $Ge_{35}Ga_5Se_{60}$, and $Ge_{10}As_{20}S_{70}$. The absorption coefficients $\alpha(\nu)$, complex refractive index $n(\nu)$, and complex dielectric constants $\varepsilon(\nu)$ were measured in a frequency range from 0.3 THz to 1.5 THz. The measured real refractive indices were fitted using a Sellmeier equation. The results show that the Sellmeier equation fits well with the data throughout the frequency range and imply that the phonon modes of glasses vary with the glass compositions. The theory of far-infrared absorption in amorphous materials is used to analyze the results and to understand the differences in THz absorption among the sample glasses.

Keywords: Chalcogenide glass, terahertz time-domain spectroscopy, complex refractive index, complex dielectric constant, absorption coefficient.

I. Introduction

Terahertz (THz) radiation encompasses a range of frequencies in the electronic spectrum between 0.1 THz and 10 THz. Although the THz radiation emitted from interstellar gases has been studied for the last 30 years in astronomy, the research of the THz region for various applications is relatively recent due to difficulties in the generation and detection of THz waves. The past decades have seen a revolution in THz technology, as advanced materials and devices research provided new and higher power sources, and the potential of THz technology for physics research and commercial applications was demonstrated. With the recently developed THz pulse beam sources, generating subpicosecond pulses of THz radiation, new and wide frequency range investigations of materials are available [1]-[3]. The THz pulses consist of a single cycle over a pulse duration of typically 100 fs to 700 fs, and are analyzed using a phase and transform-limited white spectrum in the frequency range of 0.2 THz to 5 THz. THz time-domain spectroscopy is a nonionizing, coherent quasi-optic, and phase sensitive modality, and has been used in the characterization of a wide variety of materials. Some of the prominent phonon resonances of various materials fall in the THz frequency band [4]. Thus, THz time-domain spectroscopy is an advantageous approach to investigate the low-frequency optical and dielectric properties of materials [5]-[7].

A few research groups have studied the optical and dielectric properties of various glasses. Naftaly and Miles's group obtained THz properties of amorphous silica, Pyrex, chalcogenide, and borosilicate glasses [8], [9]. Kojima and others studied the low energy excitations called boson peaks in organic and inorganic glasses using THz time-domain

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spectroscopy [10].

Because their optical and electrical properties make them very attractive materials, chalcogenide glasses have been widely studied for many years. Chalcogenide glasses are based on the chalcogen elements S, Se, and Te, and the addition of other elements, such as Ge, As, and Sb, leads to the formation of stable glasses. Chalcogenide glasses have a lower phonon energy (< 350 cm^{-1}) than that of oxide or fluoride glasses (> 500 cm^{-1}) and possess a prolonged transmission window of up to approx. 10 μm [11], [12]. Because the chalcogenide glasses transmit to a longer IR (up to a far IR of approx. 20 μm for telluride glasses) than silica and fluoride glasses, they have been used as materials for night vision, chemical sensors, mid-infrared light delivery, thermal imaging, environmental monitoring, and integrated optics [13], [14]. There are a number of potential applications in the civil, medical, and military areas. Recent progress in the technology for the less explored THz region has received a lot of attention for the development of new THz materials and devices for various applications.

In the THz or far-infrared range, optical and dielectric constants are mainly sensitive to the phonon frequency of the materials. The optical and dielectric properties of chalcogenide glasses in the THz range have not been systematically investigated yet. A spectroscopic study of chalcogenide glasses is necessary to characterize their optical and dielectric properties over a broad frequency range and to further explore their potential applications in THz technology areas.

In this paper, we report on the measurement of the complex refractive index, absorption coefficients, and complex dielectric constants of three types of chalcogenide glasses using THz time-domain spectroscopy (TDS).

II. Data Analysis Method

Synchronized subpicosecond photoconductive THz pulse generation and an optical sampling technique are used to directly measure the temporal profile of the electric field of each THz pulse trace. THz pulses are recorded in the time domain with and without the sample, $E_{\text{sam}}(t)$ and $E_{\text{ref}}(t)$, placed at an intermediate plane in the THz beam path of the THz-TDS system, and THz pulse waveforms are Fourier-transformed into the complex amplitude, $E_{\text{sam}}(\omega)$ and $E_{\text{ref}}(\omega)$, in the frequency domain, respectively. The ratio of $E_{\text{sam}}(\omega)$ and $E_{\text{ref}}(\omega)$ is given in [15] by

$$\frac{E_{\text{sam}}(\omega)}{E_{\text{ref}}(\omega)} = \frac{4\tilde{n}(\omega)}{(1+\tilde{n}(\omega))^2} e^{-j\frac{\omega d}{c}(\tilde{n}(\omega)-1)} = \rho(\omega)e^{-j\Delta\phi(\omega)}, \quad (1)$$

where $\tilde{n}(\omega) = n(\omega) - i\kappa(\omega)$ is the complex refractive index, d is the thickness of the sample, $\Delta\phi(\omega)$ is the intrinsic phase shift, and c is the speed of light in a vacuum. If the THz power absorption of our samples is low, we can neglect the influence of $\kappa(\omega)$; thus,

$$\rho(\omega) = \frac{4n(\omega)}{[n(\omega)+1]^2} e^{-\kappa(\omega)d\omega/c}, \quad (2)$$

$$\Delta\phi(\omega) = \frac{[n(\omega)-1]\omega d}{c}. \quad (3)$$

The refractive index, extinction coefficient, and absorption coefficient are

$$n(\omega) = \Delta\phi(\omega)\frac{c}{\omega d} + 1, \quad (4)$$

$$\kappa(\omega) = \ln\left[\frac{4n(\omega)}{\rho(\omega)[n(\omega)+1]^2}\right]\frac{c}{\omega d}, \quad (5)$$

$$\alpha(\omega) = \frac{2\kappa(\omega)\omega}{c} = \frac{2}{d}\ln\left[\frac{4n(\omega)}{\rho(\omega)[n(\omega)+1]^2}\right]. \quad (6)$$

III. Experimental Setup

Glass samples were prepared from Ge, Ga, As, S, and Se powders with purities of better than 99.999%. Approximately 15 g batches were weighed in an Ar-purged glove box with O_2 and OH- concentrations of less than 2 ppm and 5 ppm, respectively. Silica ampoules containing the starting materials were sealed, and melted in a rocking furnace. The temperature was first increased to 500°C with a heating rate of 2°C/min and then subsequently elevated to 1000°C by 1°C/min. After the melting at 1000°C for 12 h, ampoules with the melt inside were removed from the furnace and quenched in water. The samples were annealed at around their glass transition temperature (about 360°C) for two hours [16], [17]. Approximately 2 mm thick discs were sliced and optically polished for the experiments.

The optoelectronic THz-TDS system used for our experiments is shown in Fig. 1. The key component for generating and detecting THz pulse radiation is a photoconductive dipole antenna irradiated with 30 fs to 50 fs laser pulses coming at a rate of 80 MHz from a Ti:sapphire laser. We used a dipole antenna fabricated on a low-temperature-grown GaAs (LT-GaAs) film with a 5 μm gap and 30 μm length, as shown in Fig. 1(a). This antenna is DC biased from 10 V to 20 V and generates THz pulses with a majority of the spectral energy density at frequencies between 0.2 THz and 3 THz. The emitted THz pulse is collimated using a highly resistive Si hyper-hemispherical lens with a diameter of 10 mm and gold-coated parabolic mirror into a highly directional beam.

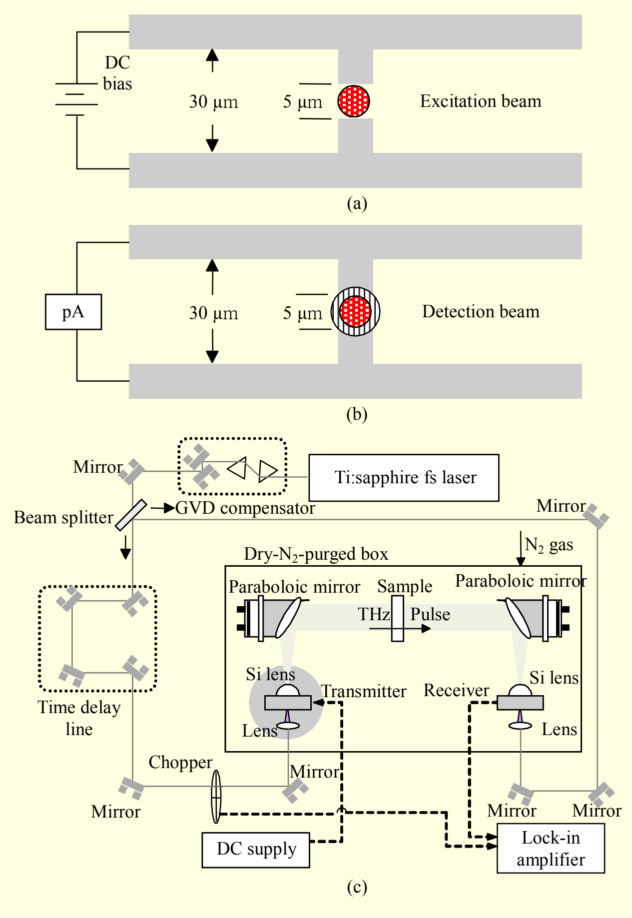

Fig. 1. (a) Photoconductive transmitting antenna used to generate a THz pulse, (b) receiving antenna used to detect a THz pulse, and (c) schematic diagram of the THz-TDS system.

After passing a 40 cm propagation distance, a THz pulse beam is focused using an identical combination of parabolic mirror and Si lens onto the receiving antenna, shown in Fig. 1(b), that was fabricated on an LT-GaAs film and consists of a coplanar transmission line structure with a 5 µm gap and a 30 µm length. The electric field of the incident THz pulses induces a voltage across the 5 µm wide antenna gap and is measured by photoconductively shorting the gap with laser pulses. The receiver antenna is gated by the probe laser pulse for the field-sensitive detection of the THz pulse. When gated by a probe laser pulse, a current proportional to the instantaneous field strength of the incident THz pulse can be measured by a phase sensitive detection technique using an optical chopper and lock-in amplifier. The time delay between the pump and the probe laser pulse is mechanically scanned by moving a retroreflector with a computer-controlled motor stage. By changing the time delay, a THz pulse waveform can be obtained with both the amplitude and phase information. The THz-TDS system was enclosed in a N_2-purged box to eliminate THz power absorption due to residual H_2O vapor in the THz beam path. The chalcogenide glass samples, polished on both sides, are freestanding glasses that are 8 mm in diameter and 2 mm thick. The samples were attached to a sample holder that has an optical aperture 7 mm in diameter. An identical clear aperture was used as a reference.

IV. Measurements

The THz-TDS system has been used to characterize the optical and dielectric properties of chalcogenide glasses in the THz region. In our experiment, a reference THz pulse was first measured in the absence of a sample in the THz beam path. Subsequently, the sample was placed in the THz beam path and a second set of THz pulse waveforms was measured to register the interaction of the THz pulse with the sample.

The electrical field of THz pulses transmitted through the samples and the reference were measured in the time domain, and the corresponding frequency spectra were obtained by fast Fourier transformation. The measured reference and sample pulses are presented in Fig. 2(a), where the THz pulse traces are separated vertically for clarity. The THz pulses measured with the samples show a main pulse that is due to the first transmission through the samples, but they contain smaller pulses caused by the internal Fabry-Perot reflection in the samples. The echoes of the THz pulse are temporally well separated. These echoes were removed from the measured THz waveforms and replaced by zeros prior to the parameter extraction process. The normalized THz spectra are shown in Fig. 2(b). The dynamic range of the data is approximately 1,000:1.

V. Results and Discussion

The frequency-dependent absorption coefficients and complex refractive index of the samples were obtained using a Fourier analysis of the measured transmitted THz pulses through the reference and samples. The real refractive index n_r is obtained directly using the phase difference between the reference and sample pulses. The open figures in Fig. 3 show the measured absorption coefficients and complex refractive index as a function of frequency varying from 0.3 THz to 1.5 THz.

As can be seen in Fig. 3, the measured absorption coefficients and complex refractive index increase with increasing frequency. No remarkable absorption peaks are observed, which is an indication that no noticeable change occurs in the refractive index. The fluctuation feature shown in the absorption coefficients and refractive index arise from the insufficient THz beam power and increasing absorption of

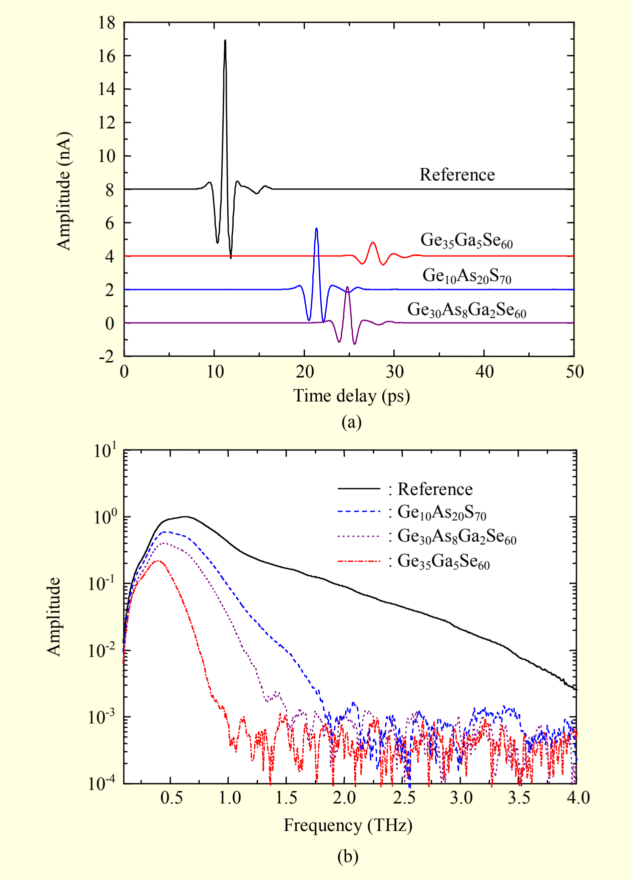

Fig. 2. (a) THz pulse wave forms of the reference and those transmitted through the chalcogenide glasses and (b) their normalized spectrum.

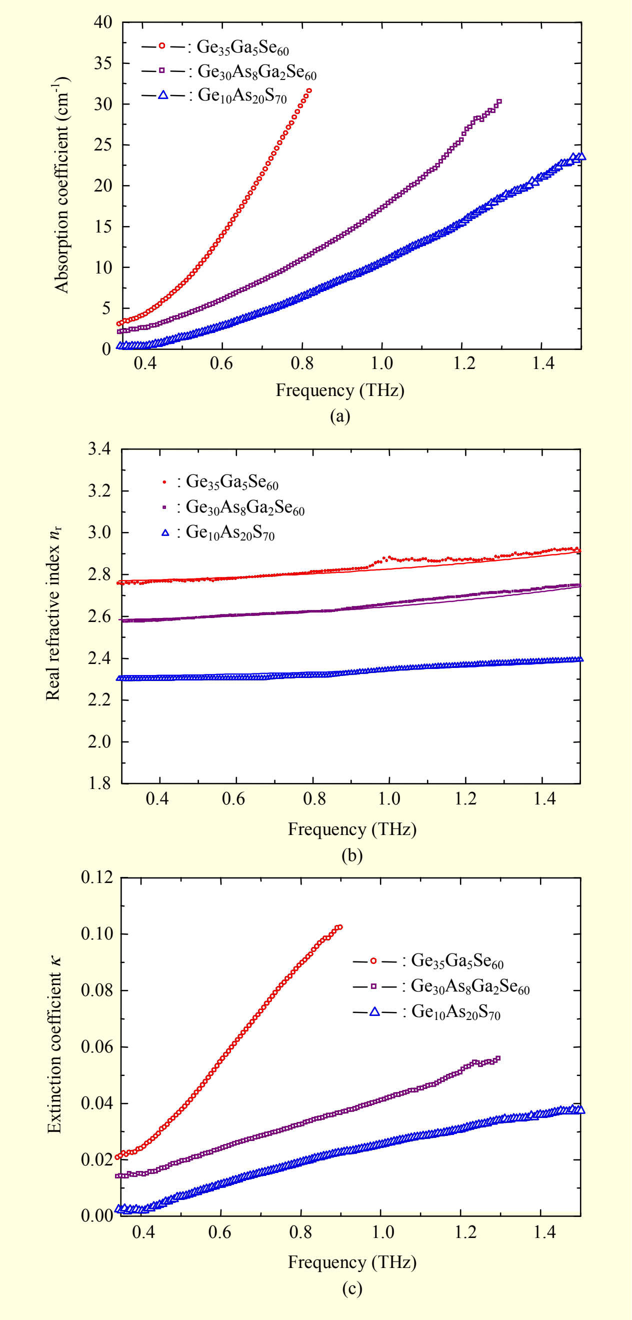

Fig. 3. (a) Measured absorption coefficients of the chalcogenide glasses as a function of frequency, (b) the frequency dependence of the real refractive index (the solid curves represent the Sellmeier fitting), and (c) the frequency dependence of the extinction coefficients κ of the chalcogenide glasses.

chalcogenide glasses at higher frequencies. The measured frequency range differs among the glasses because the dynamic range of the THz-TDS system restricts the available frequencies. Both the dynamic range and absorption coefficients are strongly frequency-dependent; therefore, more absorbent glasses have a narrower data range [18]. As can be seen Fig. 3(c), the measured extinction coefficients are much smaller than the real refractive index. Also, a thicker sample implies a lower contribution to the error of the optical constants from the approximation ($\tilde{n}(\omega) \approx n(\omega)$). A thick sample enhances the interaction between the THz pulse and the sample, as indicated by exponential terms in (1). Therefore, the error of the parameter extraction from the approximation can be considered negligible.

Chalcogenide glasses have a structure that is different from that of other glasses, and their bonding is weaker [11]. As shown in Fig. 3(a), within the frequency range of 0.3 THz to 1.5 THz, $Ge_{30}As_8Ga_2Se_{60}$, $Ge_{35}Ga_5Se_{60}$, and $Ge_{10}As_{20}S_{70}$ have different absorption properties. These three glasses have different components and structure; therefore, they are not directly comparable. It is worth pointing out that chalcogenide glasses have smaller absorption coefficients than that of borosilicate [8], [9], which can be explained by the absence of ionically bonded compounds in their compositions. However, they have much larger absorption coefficients than that of SiO_2 [8], [9], which can be ascribed to their weaker bonding and more disordered structure [19]. Figure 3(b) shows the real

Table 1. Sellmeier coefficients A and λ_1 for chalcogenide glasses.

	A (no units)	λ_1 (μm)
$Ge_{30}As_8Ga_2Se_{60}$	5.60	73
$Ge_{35}Ga_5Se_{60}$	6.65	66
$Ge_{10}As_{20}S_{70}$	4.35	54

refractive indices of the chalcogenide glasses in the frequency range of 0.3 THz to 1.5 THz. In this figure, the real refractive indices of the glasses increase with an increase in frequency. The weak dispersion observed in the refractive indices of all glass samples can be explained by the fact that THz frequencies lie far away from the bandgap or resonances of these glasses. The Sellmeier equation is an empirical equation representing the wavelength dependence of the real refractive index n_r and has a form $n_r^2=1+\Sigma_i A_i\lambda^2/(\lambda^2-\lambda_i^2)$, where λ_i denotes the absorption wavelengths, and A_i denotes constants related to the strengths of the absorption wavelengths. From the relation $\lambda=c/\nu$, the Sellmeier equation can be written as $n_r^2=1+\Sigma_i A_i c^2/(c^2-\lambda_i^2\nu^2)$. The solid curves in Fig. 3(b) are the fits of the Sellmeier equation of the form $n_r^2=1+Ac^2/(c^2-\lambda_1^2\nu^2)$ to the data. The results show that the Sellmeier equation fits the data well throughout the frequency range. The fitting parameters are shown in Table 1.

Employing the measured data of absorption coefficients and complex refractive index, we obtain a frequency-dependent complex dielectric function. The complex dielectric constants of the chalcogenide glasses are calculated using the measured data of the complex refractive index through the relationship $\varepsilon(\omega)=(n_r+i\kappa)^2$, while the imaginary part of the refractive index κ is related to the absorption coefficients as $\kappa=\alpha\lambda_0/4\pi$. As a result, the real and imaginary parts of the dielectric function are given as $\varepsilon_r=n_r^2-(\alpha\lambda_0/4\pi)^2$ and $\varepsilon_i=\alpha n_r\lambda_0/2\pi$, respectively [2]-[5].

The open figures shown in Fig. 4 represent the measured data of the complex dielectric constants. Both real and imaginary parts of the dielectric constants increase with an increase in frequency for all glasses. In Fig. 4(a), the real dielectric constants show a feature that is the square of the refractive index n_r because the power absorption by the glasses is relatively small in the frequency range concerned, and because the contribution of the absorption coefficient to the real dielectric constant is negligible. The imaginary dielectric constants plotted in Fig. 4(b) show a proportional feature of the dielectric loss of the resonance process.

The characteristics of THz and far-infrared transmission in various glasses are of particular interest because power absorption is mediated by phonon processes; therefore, it is related to numerous material properties. A model developed by Schlömann [20] and Strom [21] in the 1970s describes the far-IR absorption in amorphous materials in terms of disorder-induced coupling of radiation into the acoustic phonon modes of the material. Quantitatively, the effect of glass composition on THz transmission can be written in light of the power-law relations [20], [21]:

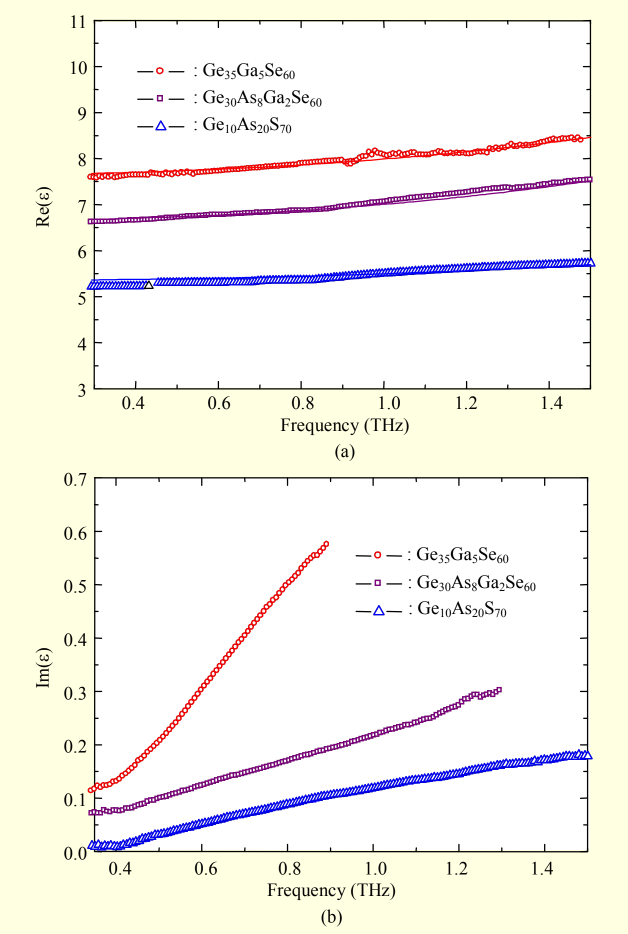

Fig. 4. Measured frequency dependence of the (a) real and (b) imaginary parts of the complex dielectric constants of chalcogenide glasses.

$$n(\nu)\alpha(\nu)=K(h\nu)^{\beta}, \qquad (7)$$

where h is the Plank constant; β is an exponent, approximately 2 in glassy materials; and K is a factor that increases the density of charge fluctuations in the material and with the refractive index. To prove this relationship, and to determine the K and β parameters, Fig. 5 plots the product $n(\nu)\alpha(\nu)$ against frequency on a log-log scale.

Figure 5 shows the agreement between the data and power relationship given by (7). The values of K and β are listed in Table 2. The value of β is 2.0 for all sample glasses, in agreement with [8] and [21]. It is notable that THz absorption is higher in those glasses that also have a high THz refractive index. As

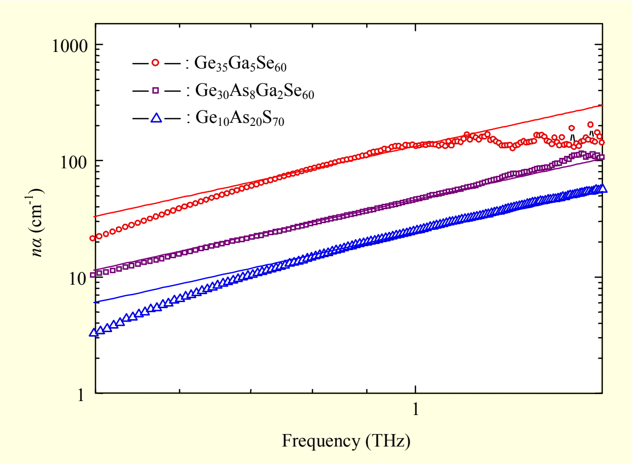

Fig. 5. Frequency dependence of the product $n\alpha$ for the chalcogenide glasses. The solid line represents the theoretical fitting.

Table 2. K and β parameters of chalcogenide glasses.

Glass	n_r (0.8 THz)	K (10^{43}J^{-1}·cm^{-1})	β
$Ge_{30}As_8Ga_2Se_{60}$	2.62	10.5	2
$Ge_{35}Ga_5Se_{60}$	2.81	30.2	2
$Ge_{10}As_{20}S_{70}$	2.31	5.5	2

seen in Table 2, K does actually increase with the refractive index. As seen Fig. 5, at the extrema of the frequency range, the data shows a relatively large deviation of the theoretical fitting. At low and high frequency ranges, the THz signal is noisy due to the dramatic reduction of THz signal amplitude. We think that this amplitude reduction affects the quality of the extracted optical constants. However, the measured data for chalcogenide glasses was found to be in good agreement with the theory of far-IR absorption, and sufficient correlations were observed with optical properties.

VI. Conclusion

The optical and dielectric properties of chalcogenide glasses have been investigated in the THz frequency region using THz time-domain spectroscopy. The complex refractive index n, absorption coefficients, and complex dielectric constants for $Ge_{30}As_8Ga_2Se_{60}$, $Ge_{35}Ga_5Se_{60}$, and $Ge_{10}As_{20}S_{70}$ were obtained in the frequency range from 0.3 THz to 1.5 THz. The measured real refractive indices were fitted by the Sellmeier equation, which fits the data well throughout the frequency range. The Sellmeier coefficients can be obtained and imply that the phonon modes of glasses change with the compositions of the glasses. The theory of far-infrared absorption in amorphous materials was used to analyze the results and to understand the differences in THz absorption among the sample glasses. The results show good agreement between the data and theory of far-IR absorption in chalcogenide glasses.

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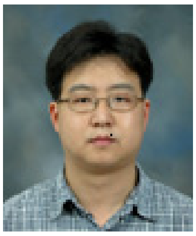

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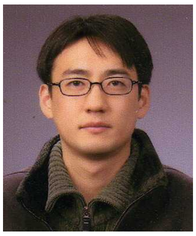

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