

Article

Optical Properties of Se-Excess $\text{Ge}_2\text{Sb}_2\text{Se}_x\text{Te}_1$ ($4.23 < x < 4.97$) Phase-Change Thin Films

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Abstract

In this study, Se-excess $\text{Ge}_2\text{Sb}_2\text{Se}_x\text{Te}_1$ (GSST) thin films were synthesized via magnetron co-sputtering. The crystal structure, surface morphology, complex refractive index, and bandgap of the Se-excess GSST thin films were systematically investigated. Owing to the formation of higher-energy Sb–Se bonds at an annealing temperature of 350 °C, grain refinement occurred (the average dendrite trunk width decreased from 0.64 μm to 0.37 μm). Consequently, the extinction coefficient (k) in the crystalline state was reduced to 0.17, resulting in a figure of merit (FOM) of 8.76 at the telecommunications C-band wavelength of 1550 nm.

Keywords: phase-change materials; GSST; optical materials and properties; Raman

1. Introduction

Phase-change materials (PCMs) exhibit reversible switching between amorphous and crystalline states, accompanied by pronounced changes in optical constants (refractive index n and extinction coefficient k), which enables nonvolatile data storage and optical modulation in integrated photonic devices [1–2]. The figure of merit ($\text{FOM} = \Delta n / \Delta k$) [3] is widely used to evaluate the optical switching performance [4] of PCMs. A large Δn ensures a high modulation depth (strong phase change), while a small Δk in the crystalline state minimizes optical loss (absorption). Thus, a high FOM translates to low insertion loss and high switching contrast in photonic devices. $\text{Ge}_2\text{Sb}_2\text{Te}_5$ (GST) [5–10] is one of the most extensively studied phase-change materials. However, the high extinction coefficient of crystalline GST (~ 1.49) [11] poses challenges for its application in integrated silicon photonic devices [12–16]. Previous studies [17–18] have demonstrated that partially substituting tellurium (Te) with selenium (Se) in GST ($\text{Ge}_2\text{Sb}_2\text{Te}_5$) yields PCMs with a broad transparency window (1–18.5 μm), such as $\text{Ge}_2\text{Sb}_2\text{Se}_4\text{Te}_1$ (GSST) [17]. The extinction coefficient of crystalline GSST is approximately 0.42, which, while significantly lower than that of GST, still results in considerable optical loss. Consequently, further reducing the extinction coefficient of crystalline GSST has become an issue in urgent need of address. In this work, via Se-excess doping, the extinction coefficient of GSST was improved (to 0.17). Meanwhile, we investigated the effects of excess Se on the refractive index, microstructure, and optical performance of GSST films, ultimately achieving a high figure of merit (8.76).

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2. Materials and Methods

The Se-excess GSST films were prepared at room temperature using an LN-1082FS multifunctional vacuum magnetron sputtering system (Shenyang Lining Vacuum Technology Institute, Shenyang, Liaoning, China). A $\text{Ge}_2\text{Sb}_2\text{Se}_4\text{Te}_1$ (GSST) alloy target and a Se single-element target (Zhongnuo Advanced Material (Beijing) Technology Co., Ltd., Beijing, China; both purity = 99.99%) were co-sputtered onto SiO_2/Si (100) substrates. The GSST2 composition ($\text{Ge}_2\text{Sb}_2\text{Se}_{4.46}\text{Te}_1$) was obtained (EDS spectra of the two films are shown in Fig. S1, while the elemental content data for the other Se-excess films are summarized in Table S1, both in the Supplementary Material). Both targets had a diameter of 2 inches. The base vacuum during sputtering was 2×10^{-4} Pa, with an RF power of 40 W applied to the GSST target and a sputtering time of 2 min. The Se target was powered by a separate RF source, with its power adjusted to regulate the Se content in the GSST films. The argon gas flow rate was maintained at 13 Sccm, and the working pressure was 0.5 Pa. The film thickness was approximately 50 nm (surface morphology and cross-sectional images are inserted as Figure S1a and Supplementary Figure S1b, respectively). The as-deposited amorphous GSST films were annealed at various temperatures in an argon atmosphere to induce crystallization. An XploRA PLUS confocal Raman microscope (HORIBA Ltd., Kyoto, Japan) was employed to characterize the crystal structures of both amorphous and crystalline GSST and GSST2. Surface topography and average surface roughness (R_a) of films under two phase states were measured by an MFP-3D atomic force microscope (Oxford Instruments Asylum Research Inc., Santa Barbara, CA, USA). A Model GES5E spectroscopic ellipsometer (Semilab Semiconductor Physics Laboratory Co. Ltd., Budapest, Hungary) was utilized to measure the ellipsometric parameters as a function of wavelength (increment: 5 nm) at an incidence angle of 70° . The complex refractive indices were extracted by fitting the data using an air-GSST-Si three-layer model and the Tauc-Lorentz function.

3. Results

Figure 1a displays the Raman spectra of the GSST films at different annealing temperatures. At annealing temperatures below 300°C , the GSST films remained amorphous, consistent with the unannealed samples. At 300°C , the GSST films exhibited coexisting amorphous (AM) and crystalline (CR) characteristic peaks, which is consistent with previous reports [19,20]. The AM and CR peaks were located at 160 cm^{-1} and 120 cm^{-1} , respectively. When the annealing temperature reached 320°C , the Raman feature peak at 120 cm^{-1} suggested a dominant Ge-6Se octahedral structure [20,21]. Further increasing the annealing temperature to 350°C did not introduce new Raman features. Figure 1b shows the Raman spectra of GSST2. Similar to GSST, GSST2 transitioned from an amorphous to a crystalline state as the annealing temperature increased. However, the coexistence of AM and CR peaks occurred at 320°C for GSST2, slightly higher than for GSST, suggesting an increase in crystallization temperature for GSST2 [22]. In the amorphous state, the Raman peak of GSST2 was located at 160 cm^{-1} , indicating that both GSST and GSST2 share the same characteristic peak in the AM state, dominated by the Ge-4Se tetrahedral structure [20]. Notably, for the GSST2 film annealed at 350°C , a strong Raman feature appeared at 192 cm^{-1} , which corresponds to the Sb-6Se octahedral structure [20,23], unlike crystalline GSST, which is dominated by the Ge-6Se octahedral structure (120 cm^{-1} peak). Previous studies [17,24] indicate that Se has a higher electronegativity (2.05) than Ge (2.01), and tends to form higher-energy bonds [24]. The formation of Sb-Se bonds requires higher energy than Ge-Se bonds [17,20,21]. This difference in bond energy explains the different crystallization temperatures. As demonstrated by DFT calculations in the supplementary information of Zhang et al. [17], the most energetically favorable atomic configuration (the

cf2 model) exhibits the strongest Sb–Se bonds with a cohesive energy of 3.383 eV/atom. It is precisely because the Sb–Se bond has a higher bond energy that the Sb–6Se octahedral vibration at 192 cm^{-1} only emerges at the higher annealing temperature of $350\text{ }^{\circ}\text{C}$ in the Se excess sample. In contrast, the Ge–6Se vibration at 120 cm^{-1} appears at lower annealing temperatures (e.g., $300\text{--}320\text{ }^{\circ}\text{C}$) in stoichiometric GSST, as the Ge–Se bonds require less energy to form. This temperature-dependent behavior further supports the interpretation that higher energy Sb–Se bonds require more thermal energy to activate.

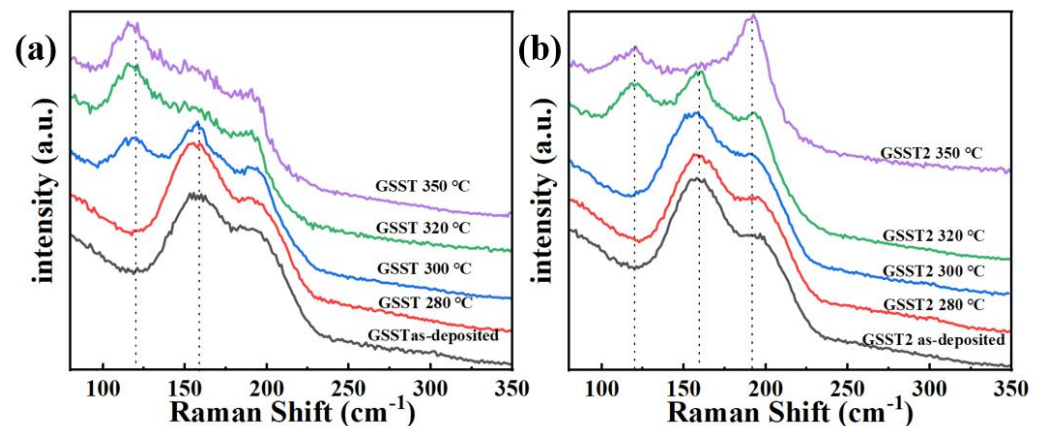


Figure 1. (a) Raman spectra of GSST films; (b) Raman spectra of GSST2 films.

Figure 2 shows the AFM images of the GSST and GSST2 films in the unannealed (amorphous) and $350\text{ }^{\circ}\text{C}$ annealed (crystalline) states. As shown in Figure 2a,c the average surface roughness (R_a) values of the amorphous and crystalline GSST films were 1.419 nm and 5.966 nm , respectively. As shown in Figure 2b,d the R_a values of the amorphous and crystalline GSST2 films were 0.744 nm and 3.599 nm , respectively. Generally, crystallization leads to an increase in grain size and thus surface roughness. Both GSST and GSST2 films exhibited dendritic grains after crystallization at $350\text{ }^{\circ}\text{C}$. However, compared with crystalline GSST, the grains in crystalline GSST2 were significantly refined, with the average dendrite trunk width decreasing from $0.64\text{ }\mu\text{m}$ to $0.37\text{ }\mu\text{m}$ (as statistically verified by $n = 30$ independent measurements for each sample, with the full distribution and standard deviations presented in Supplementary Figure S2). Consequently, the average surface roughness (R_a) of crystalline GSST2 was slightly lower than that of crystalline GSST. Smoother films typically result in lower optical losses [25], which is beneficial for improving the optical properties of GSST2 films.

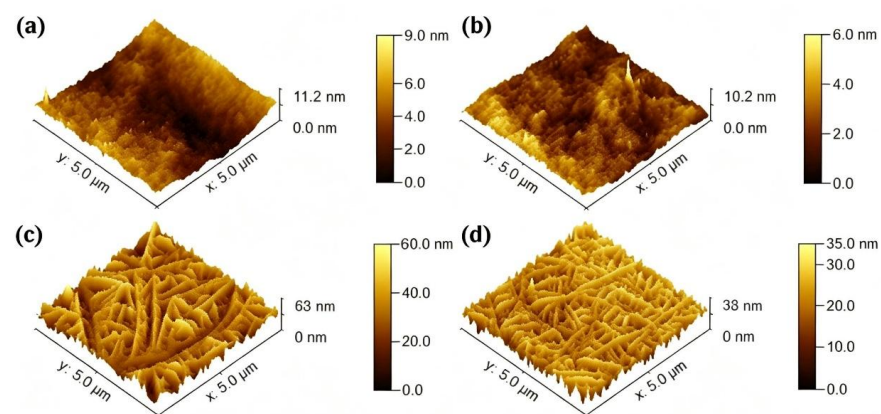


Figure 2. (a) AFM image of GSST in the AM state; (b) AFM image of GSST2 in the AM state; (c) AFM image of GSST in the CR state; (d) AFM image of GSST2 in the CR state.

Figure 3a, b show the complex refractive indices of GSST and GSST2 (data for other Se-excess GSST films are provided in Supplementary Figure S3 and Table 1). The complex refractive index of GSST is closely related to its optical performance, which can be evaluated by the figure of merit (FOM) [2]:

$$FOM = \frac{\Delta n}{\Delta k} \quad (1)$$

where Δn represents the refractive index contrast between the amorphous and crystalline states, and Δk represents the difference in extinction coefficient. The FOM increases with a larger refractive index contrast and a smaller extinction coefficient difference, indicating superior optical performance. In this work, we focus on the telecommunication C-band (1550 nm), as shown in Table 1, while the data for the O-band (1310 nm) is provided in Supplementary Table S2, confirming the broadband applicability of our Se-excess doping strategy.

Figure 3 displays the refractive indices and extinction coefficients of the GSST and GSST2 films in both the amorphous and crystalline states over the wavelength range of 400–1600 nm. At the telecommunication wavelength of 1550 nm, the refractive index of the amorphous GSST2 film was slightly lower than that of the amorphous GSST film (as shown by the black solid lines in Figure 3a,b). Similarly, the extinction coefficient of the amorphous GSST2 film was slightly lower than that of GSST (as shown by the black dotted lines). This can be attributed to the increase in Se content, which decreases both the refractive index and extinction coefficient [22]. In the crystalline state at 1550 nm, the refractive index of GSST2 was slightly lower than that of GSST (as shown by the red dashed lines in Figure 3a,b). Meanwhile, the extinction coefficient of GSST2 decreased significantly compared to GSST (as shown by the red dash-dot lines). The refractive index data for GSST and GSST2 at 1550 nm are listed in Table 1. The complex refractive index of amorphous GSST was $3.21 + 0.01i$, and that of crystalline GSST was $5.02 + 0.42i$, yielding an FOM of 4.41, which is consistent with reported values of 4.17 [17] and 4.2 [18]. The k value of crystalline GSST2 was 0.17, representing a substantial improvement over crystalline GSST, and its FOM reached 8.76.

To comprehensively reveal the evolution of optical properties across the entire composition range ($4 < x < 4.97$), we systematically analyzed the optical constants of all samples at 1550 nm, and the results are summarized in Table 1. The refractive index of a material is fundamentally related to the electronic polarizability, i.e., the response of bonding electrons to an external electric field [26]. Furthermore, electronic polarizability is inherently dependent on the number of molecules per unit volume, N [26]. The refractive index n of a material is fundamentally related to the electronic polarizability, which depends on the number of atoms per unit volume, N . A higher N leads to increased electronic polarizability and, consequently, a higher refractive index [26]. The atomic densities of Ge, Sb, and Te are 5323 kg/m³, 6697 kg/m³, and 6240 kg/m³, respectively, while the atomic density of Se is 4819 kg/m³, which is lower than those of Ge, Sb, and Te. Therefore, the increased Se content reduces the overall atomic density of the material, thereby decreasing electronic polarizability and ultimately leading to a lower refractive index. As a result, the crystalline refractive index (n) significantly decreases with increasing Se content. For example, at 1550 nm, the crystalline refractive index decreases from 5.02 at $x = 4$ to 4.63 at $x = 4.46$, and further to 3.97 at $x = 4.97$. Similarly, the crystalline extinction coefficient (k) also decreases with increasing Se content, reaching a minimum value of 0.11 for the $x = 4.97$ sample. However, the optical contrast (Δn) also diminishes, dropping to 0.87 at $x = 4.97$, which is unfavorable for high ON/OFF ratio optical devices. To evaluate the overall optical switching performance, we

calculated the Figure of Merit ($FOM = \Delta n / \Delta k$). As shown in Supplementary data Figure S4, the optical properties exhibit clear evolution trends with increasing Se content, summarizing the refractive indices, extinction coefficients, and FOM for both the amorphous and crystalline states at $x = 4, 4.23, 4.46$, and 4.97 , and in Table 1, the FOM exhibits a distinct non-monotonic trend with increasing Se content. It rises from 4.41 at $x = 4$ to a maximum of 8.76 at $x = 4.46$, and then slightly decreases to 7.91 at $x = 4.97$ (still higher than the original $x = 4$ composition). These results demonstrate that a moderate excess of Se ($x = 4.46$) effectively balances a large refractive index contrast (Δn) with a low extinction coefficient contrast (Δk), yielding the optimal FOM and confirming the superior optical performance of the GSST2 composition.

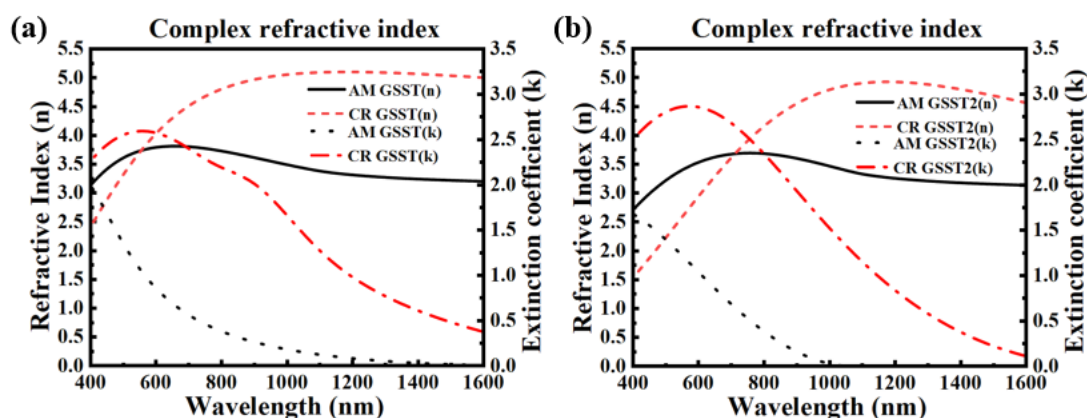


Figure 3. Complex refractive index of (a) GSST and (b) GSST2.

Table 1. Optical constants of GSST at different Se doping concentrations (1550nm).

GSST films	AM	CR	Δn	Δk	FOM
GSST	3.21+0.01 i	5.02+0.42 i	1.81	0.41	4.41
GSST1	3.16+0 i	4.79+0.27 i	1.63	0.27	6.04
GSST2	3.14+0 i	4.63+0.17 i	1.49	0.17	8.76
GSST3	3.10+0 i	3.97+0.11 i	0.87	0.11	7.91

The significant improvement in the k value of crystalline GSST2 can be attributed to a combination of structural and electronic effects. On the one hand, the reduced surface roughness (as shown in Figure 2d) [25] minimizes scattering-induced optical losses. On the other hand, the higher Se content fundamentally alters the material's electronic structure. Specifically, the substitution of Te (electronegativity 2.10) by Se (electronegativity 2.55) enhances the ionic/covalent character of the bonds, which is theoretically expected to widen the optical bandgap. A wider bandgap shifts the absorption edge to higher energies, thereby suppressing intrinsic absorption in the infrared range [17]. To verify this theoretically predicted reduction in absorption, we calculated the absorption coefficient α

$$\alpha = \frac{4\pi k(\lambda)}{\lambda} \quad (2)$$

we obtained high α values ($>10^5 \text{ cm}^{-1}$). Subsequently, the bandgap was analyzed using the Tauc equation, as shown in Figure 4a,b. The Tauc equation describes the relationship between the absorption coefficient and photon energy:

$$(ah\nu)^n = K(h\nu - E_g) \quad (3)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, E_g is the optical bandgap of the sample, K is a constant, and n represents the type of optical transition ($n = 2$ for direct transitions or $n = 1/2$ for indirect transitions). According to the literature [24], $n = 1/2$ was

adopted here, and the absorption coefficient α conforms to the Tauc [27] and Davis–Mott [28,29] relationships.

To further verify the bandgap difference between crystalline GSST and GSST2, the transmittance of the samples was measured using UV-Vis spectrophotometry (Model UV-2550, Shimadzu Corporation, Kyoto, Japan) (detailed data are provided in Supplementary Material Figure S5) and analyzed via Tauc fitting serving as a semi-quantitative verification of the bandgap trend, while the absolute bandgap values are exclusively determined from the ellipsometry measurements. The obtained bandgap values were compared with those calculated from ellipsometry data. The results are shown in Figure 4. As shown in Figure 4a, the bandgap calculated by ellipsometry for crystalline GSST was 0.43 eV, while that for Se-excess GSST2 was 0.52 eV. The significant improvement in the extinction coefficient (k) of crystalline GSST2 can be attributed to a synergistic combination of three factors: (1) Chemical bonding: The excess Se promotes the formation of higher-energy Sb–Se bonds (as evidenced by the Raman peak at 192 cm^{-1}), which require higher thermal energy to form and thus suppress sub-bandgap optical absorption in the crystalline state. (2) Structural refinement: The formation of Sb–Se bonds leads to grain refinement (dendrite trunk width reduced from 0.64 μm to 0.37 μm) and reduced surface roughness (R_a from 5.966 nm to 3.599 nm), which mitigates scattering-induced optical losses. (3) Bandgap widening: The substitution of Te with Se (a more electronegative nonmetal, electronegativity: Se 2.55 vs. Te 2.10) enhances the ionic/covalent character of the bonds, which widens the optical bandgap (from 0.43 eV for crystalline GSST to 0.52 eV for GSST2). A wider bandgap means that the near-infrared photon energy is further below the band edge, thereby reducing both free-carrier absorption and inter-band transitions. This combined effect leads to a substantially reduced k value (0.17 for GSST2 vs. 0.42 for GSST), ultimately yielding a high FOM of 8.76 at the C-band.

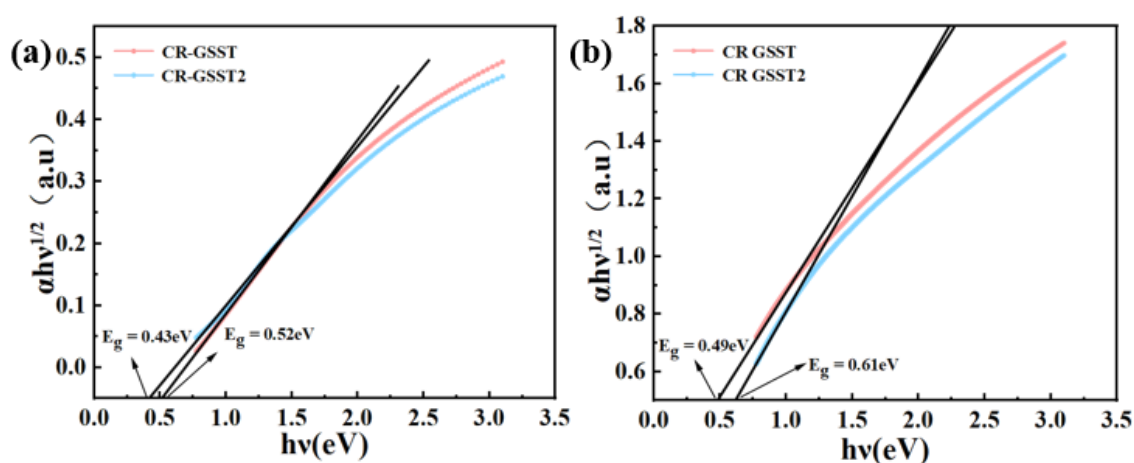


Figure 4. (a) Bandgaps of GSST and GSST2 calculated from refractive index data; (b) bandgaps calculated from UV-Vis spectrophotometry measurements.

4. Conclusions

In summary, Se-excess GSST films were prepared via magnetron co-sputtering. Compared with pristine GSST, the Se-excess GSST films exhibited reduced grain size and lower surface roughness. At an annealing temperature of 350 $^{\circ}\text{C}$, the excess Se resulted in the formation of higher-energy Sb–Se bonds, accompanied by grain refinement and a widened optical bandgap (from 0.43 eV to 0.52 eV), primarily due to the higher electronegativity of Se (2.55) compared to Te (2.10). These three synergistic effects collectively suppressed optical absorption in the crystalline state. As a result, a figure of merit of 8.76 was achieved at the telecommunications C-band (1550 nm). Therefore, Se-excess GSST thin

films present a promising novel phase-change material for optical communication applications.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst16090578/s1>, Figure S1: S1 EDS spectra of GSST (a) and GSST2 (b); Table S1: Elemental ratios of GSST and Se-excess GSST samples; Figure S2: Bar charts showing the individual dendrite trunk width measurements for the GSST and GSST2 samples. The x-axis represents the sample index (1 to 30) for each independent measurement. The y-axis represents the dendrite width (μm). Each bar represents a single measured value, and the error bars denote the standard deviation (or uncertainty) of each respective measurement, providing a detailed view of the data distribution; Table S2: Optical constants of GSST at different Se doping concentrations (1310 nm); Figure S3: Complex refractive index of Se-excess GSST films: (a) refractive index of amorphous films with various Se contents; (b) refractive index of crystalline films with various Se contents; (c) extinction coefficient of amorphous films with various Se contents; (d) extinction coefficient of crystalline films at various Se doping concentrations; Figure S4: Variation in Δn , Δk , and FOM with Se content Figure S5: Transmittance spectra of crystalline GSST and GSST2 characterized by UV–Vis spectrophotometry.

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