Spectral dependences of extrinsic optical absorption in sillenite crystals

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Abstract. **The influence of laser irradiation at wavelengths of 532 and 655 nm and annealing in air at temperatures from 200 to 370 °C on optical absorption spectra of undoped bismuth silicon oxide and bismuth germanium oxide and aluminium-doped bismuth titanium oxide crystals has been studied experimentally. The experimental data have been interpreted in terms of a model for extrinsic absorption that takes into account not only the contribution of the photoexcitation of electrons from deep donor centres with a normal distribution of their concentration with respect to ionisation energy but also that of intracentre transitions.**

1. Introduction

Interest in $Bi_{12}SiO_{20}$ (BSO), $Bi_{12}GeO_{20}$ (BGO) and $Bi_{12}TiO_{20}$ (BTO) cubic sillenite crystals is due to their high photosensitivity, which shows up as photoconductivity and photorefractive and photochromic effects [1–7]. Owing to the advantageous combination of the magnitude and speed of their photorefractive response and the possibility of producing variously oriented samples of controlled size, these materials are widely used in studies of various dynamic holography effects and applications [2, 5, 6, 8–10]. Moreover, sillenite crystals have been demonstrated experimentally to exhibit the optical generation of steady-state holographic currents [11], non-steady-state photovoltage [12], space charge waves [7, 13, 14] and photorefractive surface waves [15]. These materials have been used in studies of the self-action of light beams, the formation and interaction of spatial optical solitons [16–19] and photorefractive nonlinear optical effects. The above effects, as well as the performance of devices that take advantage of them, are significantly influenced by optical absorption and the photochromic effect: photoinduced changes in optical absorption spectra [1, 3, 4].

The short-wavelength edges of the fundamental absorption bands of BSO, BGO and BTO crystals at a temperature of 300 K lie at $\lambda_f = 385$, 385 and 403 nm, respectively, as deter-

mined from their band gaps: 3.22, 3.22 and 3.08 eV [20]. Near the edges, there are strong absorption bands – referred to as 'shoulders' in the literature – where the absorption coefficient in BSO, BGO and BTO at a wavelength $\lambda = 420$ nm is about 20, 40 and 100 cm^{-1}, respectively [1]. The range of laser wavelengths that are most often employed to investigate photorefractive effects in the sillenites and for various applications extends from 442 (see for example Attard [21]) to 1064 nm [22] and is part of the extrinsic absorption region. At the same time, absorption in the extrinsic region of the sillenites is known to be primarily due to intrinsic structural defects [1, 3, 4]. One optical absorption mechanism in this region is the photoexcitation of electrons from defects forming deep donor centres to the conduction band. Subsequent electron recombination at traps whose photoionisation cross section exceeds that of the donors increases absorption in some spectral region, i.e. leads to a photochromic effect [23, 24].

As shown by Tolstik et al. [25], laser pulses at $\lambda = 532$ nm increase the absorption coefficient of bismuth titanium oxide crystals in the spectral range 492–840 nm, and the relaxation time of such reversible photoinduced changes exceeds 60 h. For a theoretical description of the observed spectral dependences, Tolstik et al. [25] proposed a model for extrinsic absorption that took into account the contribution of only the photoexcitation of electrons from several donor centres, each characterised by its own normal distribution of concentration N_n with respect to ionisation energy E_i. The model allowed their experimental data to be interpreted under the assumption that photoexcited electrons were trapped at two types of centres, with average ionisation energies $E_{i1} \sim 1.6$ eV and $E_{i2} \sim 2.57$ eV. The filling of the centre with an average energy $E_{i1} \sim 1.6$ eV by electrons was noted to increase the photorefractive sensitivity of the illuminated BTO crystal in the near-IR spectral region, an effect that was previously demonstrated and studied experimentally by Odoulov et al. [22] and dos Santos et al. [26]. Analysis in the proposed model [25] suggests that, from photoinduced absorption, one can estimate the increase in both the sensitivity and speed of BTO photorefractive elements at their working wavelength upon exposure to green light.

Another mechanism that makes a considerable contribution to extrinsic absorption in sillenite crystals but was left out of consideration in the above model [25] involves intracentre transitions [27–31]. The contribution of intracentre transitions to absorption in BSO and BTO:Al crystals was found at room temperature [29–31] from resonance behaviour of spectral dependences of the changes induced in the optical absorption of the crystals by annealing in air at temperatures from 230 to 375 °C and exposure to laser radiation at various wavelengths in the visible and near-IR spectral

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regions. To analyse the spectral dependence of the absorption coefficient for BSO and BSO:Mo crystals, in which photoinduced changes in absorption were also shown to exhibit resonance behaviour, Panchenko and Strelets [28] decomposed the absorption coefficient into individual Gaussian components, an approach only applicable in the case of intracentre transitions.

The changes that are induced in the optical absorption spectra of sillenite-type crystals by exposure to laser radiation and thermal annealing and are due to an electron redistribution over deep donor centres should modify the photorefractive properties of the crystals, essential for studies of dynamic holography and photorefractive nonlinear optical effects, as well as for their applications. At the same time, evaluation of spectral changes caused by the influence of external factors on the contribution of intracentre transitions allows one to determine energy parameters of the corresponding structural defects and assess the feasibility of utilising them as luminescence centres. It is worth pointing out that structural defects in sillenite crystals are commonly assumed to be related to the bismuth ions [1, 3, 32] and that the nature and parameters of Bi centres in glasses and some single crystals are now receiving considerable research attention [33, 34].

In connection with this, there is interest in data on limiting optical absorption coefficients that can be reached in sillenites under various external influences in a particular spectral range. Experimental spectral dependences of optical absorption coefficients for sillenites in their extrinsic region should be described using a model that takes into account the contributions from both the photoexcitation of electrons from deep donor centres to the conduction band and intracentre transitions. Interpretation of observed changes in absorption spectra in terms of such a model might be helpful in assessing the nature and parameters of defect centres some of which participate in a photoinduced charge redistribution and the formation of photorefractive holograms (deep donor centres) and others are responsible for intracentre transitions (potential luminescence centres).

In this paper, we present a study and comparative analysis of spectral dependences of optical absorption coefficients in the range $\lambda = 486 – 1000$ nm for undoped BSO and BGO crystals and an aluminium-doped BTO (BTO:Al) crystal under external influences (exposure to laser light at $\lambda = 532$ or 655 nm and annealing in air at temperatures from 200 to 370 °C). We obtained spectra with minimum and maximum absorption coefficients, which were fitted by an extrinsic absorption model that took into account the contributions from both the photoexcitation of electrons from deep donor centres (with a normal distribution of concentration with respect to ionisation energy) to the conduction band and intracentre transitions (with Gaussian frequency dependences of the absorption coefficient). This study has enabled a quantitative comparison of the spectral dependences of extrinsic absorption coefficients for the BSO, BGO and BTO:Al crystals and demonstrated that the contribution of the intracentre transitions for the crystals can be represented by a combination of five Gaussians close in energy parameters.

2. Experimental procedure and results

To investigate spectral dependences of optical absorption coefficients in the range $\lambda = 486 – 1000$ nm, where the absorp-

tion coefficient can range from 0.04 to 10 cm^{-1}, and their changes in response to external influences, in our experiments we used sufficiently thick BSO, BGO and BTO:Al samples: $d = 8.0$, 6.9 and 6.6 mm, respectively. The samples had optically polished (100)-oriented faces with cross-sectional dimensions that allowed their optical transmittance to be measured using SF-56 and Genesys-2 spectrophotometers, without taking additional measures to limit the diameter of the light beams used in the instruments. Spectral dependences of absorption coefficients were calculated using optical transmission data, a known technique [35] and the wavelength dependences of the refractive index for the BSO and BGO crystals, which were determined using the Sellmeier formula in a two-oscillator approximation, with dispersion parameters presented in Ref. [4]. In the calculations for the BTO:Al crystal, we used experimental data on the spectral dependence of its reflectivity at normal incidence on its input face.

The BSO, BGO and BTO:Al crystals were subjected to various influences, such as illumination with cw laser light at $\lambda = 532$ and 655 nm to fluences within 160 and 140 J cm^{-2}, respectively, and annealing in air for 30 – 60 min at temperatures from 200 to 370 °C. The optical transmission spectra measured at a temperature of 300 K in the range $\lambda = 486 – 1000$ nm showed that the thermal annealing reduced the absorption coefficient of the BSO and BTO:Al crystals, whereas the exposure to the green light increased it. However, the changes were not always reproducible in repeat measurements, which was possibly due to the complex structure of the defect centres and their metastable states in the crystals and the effect of their prior history on the thermally or photoinduced changes. Illumination at $\lambda = 655$ nm caused insignificant changes in the absorption spectrum of the BSO crystal in all cases, whereas it bleached the unannealed BTO:Al sample over the entire spectral range studied. None of the above influences produced significant changes in the optical absorption spectrum of the BGO crystal in the range $\lambda = 486 – 1000$ nm.

Our experimental studies allowed us to obtain spectra with minimum and maximum absorption coefficients of the crystals, $k_{min}(\lambda)$ and $k_{max}(\lambda)$ (Figs 1, 2). The spectra of the BGO crystal in Figs 1 and 2 are identical because, as mentioned above, it is insensitive to the external influences in question. From the spectra of the BSO and BTO:Al crystals, we can obtain spectral dependences of changes in their optical absorption coefficient: $\Delta k(\lambda) = k_{max}(\lambda) - k_{min}(\lambda)$ (Fig. 3).

The nonmonotonic behaviour of the spectral dependences for the BGO and BSO samples in Figs 1 and 2 in the range $\lambda = 640 – 900$ nm (1.94 to 1.38 eV) suggests that the optical absorption is here contributed by an effect unrelated to the photoexcitation of electrons to the conduction band, a process that always leads to photoconduction. Indeed, the experimental spectral dependences of room-temperature photoconductivity obtained by Panchenko [36] for a BSO crystal in this range are monotonic, with characteristic steps, which made it possible to identify extrinsic photosensitivity bands and estimate the optical activation energy for transitions from local donor levels. The spectral dependence $\Delta k(\lambda)$ for the BSO crystal in Fig. 3, with a resonance nature, has four well-defined maxima and can be interpreted as the result of a decrease in the contribution to the absorption coefficient change for four intracentre transitions when the sample is brought into a minimum absorption state.
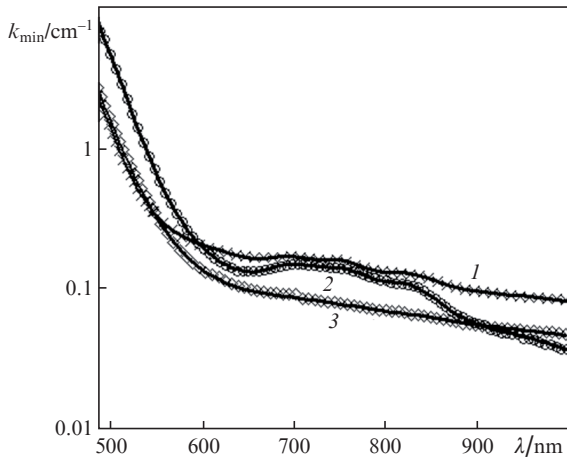
Figure 1. Optical absorption spectra of the (*1*) BGO, (*2*) BSO and (*3*) BTO : Al crystals in their minimum absorption states. The points represent the experimental data and the solid lines show a theoretical fit to the data.

Figure 2. Optical absorption spectra of the (*1*) BGO, (*2*) BSO and (*3*) BTO : Al crystals in their maximum absorption states. The points represent the experimental data and the solid lines show a theoretical fit to the data.
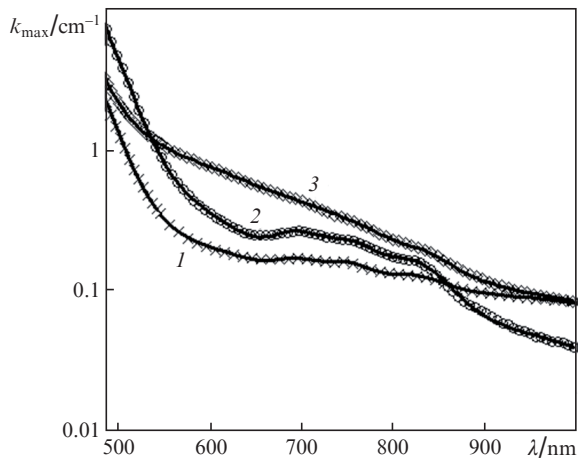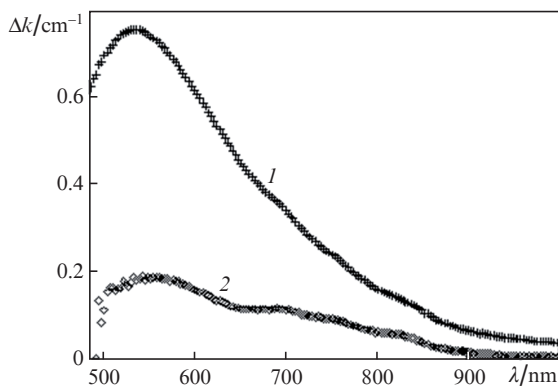

Figure 3. Spectral dependences of changes in the measured optical absorption coefficients of the (*1*) BTO : Al and (*2*) BSO crystals.

Even though the spectral dependences $k_{min}(\lambda)$ and $k_{max}(\lambda)$ for the BTO:Al crystal [Figs 1, 2, spectra (*3*)] are monotonic, the difference spectrum $\Delta k(\lambda)$ [Fig. 3, spectrum (*1*)] demonstrates a resonance nature of absorption, due to intracentre transitions, with maxima similar in position to those of the BSO sample. It is worth noting that the resonance dependence $\Delta k(\lambda)$ for changes in the optical absorption coefficient of the BTO : Al crystal is similar to those reported previously for this sample [30, 31]. The spectral dependences $k_{min}(\lambda)$, $k_{max}(\lambda)$ and $\Delta k(\lambda)$ in Figs 1 – 3 are similar to those obtained previously for BSO samples 2.9 and 10.7 mm thick [29]. The small thickness of the former sample made it possible to obtain data in the range $\lambda = 450 – 900$ nm. Data for the latter were obtained in the range $\lambda = 480 – 2500$ nm.

3. Analysis of the spectral dependences of extrinsic absorption

In interpreting the experimentally observed spectral dependences of the absorption coefficient in the extrinsic region, $k(\omega)$, we assume that the contributions of the photoexcitation of electrons from deep donor centres to the conduction band [$k_{dd}(\omega)$], intracentre transitions [$k_{ic}(\omega)$] and Urbach edge absorption [1] are additive:

$$k(\omega) = k_{dd}(\omega) + k_{ic}(\omega) + k_0 \exp\left[-\frac{\sigma_0(\hbar\omega_0 - \hbar\omega)}{k_B T}\right], \quad (1)$$

where the parameters k_0, σ_0 and $\hbar\omega_0$ determine the band edge absorption coefficient, slope and position; k_B is the Boltzmann constant; and T is the absolute temperature.

The total absorption coefficient $k_{dd}(\omega)$, related to the photoexcitation of electrons from deep donor centres to the conduction band can be represented as the following integral [25]:

$$k_{dd}(\omega) = \int_0^{\hbar\omega} B(E_i)\frac{\sqrt{\hbar\omega/E_i - 1}}{(\hbar\omega/E_i)^3}\left[1 + \exp\left(\frac{E_F - E_i}{k_B T}\right)\right]^{-1} dE_i, \quad (2)$$

where $B(E_i) = \sum_n B_n(E_i)$; $B_n(E_i) = S_n N_n(E_i)$ determines the contribution of the nth type of centre to the absorption coefficient per unit energy interval; S_n and $N_n(E_i)$ are the photoionisation cross section and the distribution of concentration with respect to ionisation energy E_i for this type of centre; and E_F is the energy separation between the Fermi level and the conduction band bottom.

To take into account in (2) that the electron occupation number of the nth type of centre, $b_n(t)$, is time-dependent, because of the electron redistribution caused by thermally and photoinduced effects and relaxation to metastable states, use can be made of the relation [25]

$$B(E_i, t) = \sum_n b_n(t)\exp\left[-\frac{(E_n - E_i)^2}{\Delta E_n^2}\right], \quad (3)$$

which stems from the assumption that the energy parameters of the normal concentration distribution, $N_n(E_i)$, remain unchanged. Here, E_n is the energy corresponding to the maximum of the normal distribution and ΔE_n is its spectral half-width.

The contribution of intracentre transitions to optical absorption can be described by decomposing $k_{ic}(\omega)$ into Gaussian components (see e.g. [37]), also under the assumption that their energy parameters remain unchanged:

$$k_{ic}(\omega, t) = \sum_m k_m^{ic}(t) \exp\left[-\frac{(E_m^{ic} - \hbar\omega)^2}{(\Delta E_m^{ic})^2}\right], \qquad (4)$$

where $k_m^{ic}(t)$ determines the contribution to the absorption coefficient from the mth component with a spectral half-width ΔE_m^{ic} at its maximum at a photon energy E_m^{ic}.

The fitting results obtained using relations (1)–(4) for the experimental spectral dependences of the absorption coefficient $k(\lambda)$ for the BSO, BGO and BTO:Al crystals are shown in Figs 1 and 2 by solid lines. The model parameters obtained by least squares fitting for intracentre transitions and the photoexcitation of electrons from deep donor centres are listed in Tables 1 and 2, respectively. Our estimates suggest that, in the spectral range under consideration, 486–1000 nm, Urbach edge absorption makes no significant contribution to the total absorption coefficient. Calculations for the BGO crystal allow only four intracentre transitions to be identified, whereas five such transitions can readily be identified for BSO and BTO:Al owing to significant changes induced in their optical absorption (Table 1). To describe the contribution to absorption from the photoexcitation of electrons from deep donor centres to the conduction band in the BTO:Al crystal, it is sufficient to take into account four types of such centres; for BGO and BSO, five deep centres, ranging in ionisation energy from 1.07 to 2.74 eV, should be taken into account (Table 2).

Table 1. Model parameters for the contribution of intracentre transitions to extrinsic optical absorption in the BGO, BSO and BTO:Al crystals.

Crystal	E_m^{ic}/eV	ΔE_m^{ic}/eV	k_m^{ic}/cm^{-1}	
			minimum absorption	maximum absorption
	1.510	0.082	0.031	0.031
	1.642	0.072	0.040	0.040
BGO	1.770	0.100	0.042	0.042
	–	–	–	–
	2.415	0.171	0.078	0.078
	1.510	0.082	0.039	0.082
	1.635	0.074	0.040	0.090
BSO	1.765	0.101	0.041	0.107
	2.156	0.319	0	0.161
	2.414	0.155	0	0.062
	1.513	0.088	0	0.040
	1.632	0.070	0	0.039
BTO:Al	1.774	0.132	0.001	0.070
	2.170	0.314	0	0.185
	2.450	0.248	0.230	0.301

It is seen from Figs 1 and 2 that the above model for extrinsic absorption adequately describes the experimental dependences of the absorption coefficient $k(\lambda)$ in the spectral range 486–1000 nm for the BSO, BGO and BTO:Al sillenite-type crystals. It allows one to estimate parameters of defect centres that determine the photoconductivity of sillenite crystals and their photorefractive sensitivity and speed in dynamic hologram recording at various laser wavelengths. Moreover, applying this model to crystals subjected to various external influences, one can evaluate time-dependent electron occupation numbers of donor centres and the relative density of

Table 2. Model parameters for the contribution of electron transitions from deep donor centres to the conduction band to extrinsic optical absorption in the BGO, BSO and BTO:Al crystals.

Crystal	E/eV	ΔE_n/eV	b_n/cm^{-1} eV^{-1}	
			minimum absorption	maximum absorption
	1.070	0.200	1.149	1.150
	1.603	0.182	0.584	0.584
BGO	1.908	0.206	1.424	1.424
	2.194	0.096	1.045	1.045
	2.740	0.287	171.5	171.5
	1.170	0.202	0.664	0.730
	1.602	0.170	0.884	0.940
BSO	1.946	0.142	0	0.135
	2.166	0.246	3.30	3.34
	2.700	0.280	368.0	367.0
	1.080	0.431	0.406	0.648
	1.600	0.332	0.103	0.416
BTO:Al	1.901	0.429	0.444	3.64
	–	–	–	–
	2.750	0.284	146.9	141.4

defects (potential luminescence centres) in which intracentre transitions are observed.

4. Discussion

Our experiments and the interpretation of their results in terms of the model under consideration demonstrate that, in sillenite crystals, optical absorption in the extrinsic region can be contributed by both the photoexcitation of electrons to the conduction band and intracentre transitions. Thermal annealing in air at temperatures from 200 to 370 °C leads to bleaching of the BSO and BTO:Al crystals in the spectral range studied, 486–1000 nm, primarily because of the reduction in the electron occupation numbers of the three shallowest donor centres (Table 2). Such annealing causes complete depletion of the centre with an ionisation energy $E_{i3} = 1.946$ eV in the BSO crystal, whereas the electron occupation numbers of the shallowest centres ($E_{i1} = 1.17$ eV and $E_{i2} = 1.602$ eV) decrease only slightly. In the BTO:Al crystal, annealing leads to considerable depletion of the donor centres with an intermediate position in the band gap: $E_{i2} = 1.6$ eV and $E_{i3} = 1.9$ eV.

Another cause of the annealing-induced bleaching of the BSO and BTO:Al crystals is the reduction in the concentration of defect centres in which intracentre transitions occur (Table 1). The only transition observed in the BTO:Al crystal after annealing is represented in the model under consideration by a Gaussian absorption line peaking at a photon energy $E_5^{ic} = 2.45$ eV, with a spectral half-width $\Delta E_5^{ic} = 0.248$ eV. Annealing the BSO crystal eliminated the absorption bands at $E_4^{ic} = 2.516$ eV ($\Delta E_4^{ic} = 0.319$ eV) and $E_5^{ic} = 2.414$ eV ($\Delta E_5^{ic} = 0.155$ eV).

As pointed out above, the external influences in question produced no changes in the optical absorption spectrum of the BGO crystal. At the same time, using the proposed model we were able to identify contributions from four intracentre transitions (Table 1) and five deep donor centres (Table 2) in its spectrum [Figs 1, 2, spectra (1)].

Analysis of the data presented in Table 1 indicates that the defect centres in the three crystals are close in the energy parameters E_{1-3}^{ic} and ΔE_{1-3}^{ic} of intracentre transitions and that, in addition, BSO and BTO:Al are close $E_{4,5}^{ic}$ and ΔE_4^{ic}. In the case of electron transitions from deep donor centres to the conduction band in the sillenite-type crystals studied, it also should be noted that, in some cases, their energy characteristics E_n and ΔE_n differ only slightly (Table 2). The observed proximity in energy parameters gives grounds to believe that the two types of centres, one responsible for the contribution to extrinsic absorption from the photoexcitation of electrons to the conduction band, and the other, for the contribution from intracentre transitions, are native structural defects bound to bismuth ions in the sillenites. Examples of such defects in $Bi_{12}MO_{20}$ (M = Ge, Si, Ti) crystals include Bi_M antisite defects (Bi^{3+} ions on the tetrahedral site of the M cation [3, 32]) and the complex bismuth ion BiO_7 [1, 3]. However, the existing models for defect centres in sillenites are not generally accepted [3] and further investigation is needed, in particular by optical spectroscopy techniques, to assess the exact nature of such centres.

5. Conclusions

The optical absorption coefficients of BSO, BGO and BTO:Al sillenite-type crystals exposed to laser light at λ = 532 or 655 nm and annealed in air at temperatures from 200 to 370 °C have been measured in the spectral range λ = 486–1000 nm. The results demonstrate that neither laser radiation nor annealing produced any changes in the optical absorption spectrum of the BGO crystal. For the BSO and BTO:Al crystals, we have obtained spectra with minimum and maximum absorption coefficients. These spectra, lying in the extrinsic region of the crystals, have been interpreted and quantitatively compared in terms of a model that takes into account the contributions to the absorption coefficient from the photoexcitation of electrons from deep donor centres to the conduction band and intracentre transitions. The proposed model and the parameters of defect centres found in this study can be used to analyse the photorefractive sensitivity and speed in dynamic hologram recording in sillenite crystals by visible and near-IR laser light, and also to identify luminescence centres and evaluate their parameters.

The comparative analysis carried out in this study indicates that the two types of defect centres existing in the sillenite crystals – one responsible for the contribution to extrinsic absorption from the photoexcitation of electrons to the conduction band (deep donor centres), and the other, for the contribution from intracentre transitions (potential luminescence centres) – have similar energy parameters in the BSO, BGO and BTO:Al crystals. This leads us to assume that these centres are native structural defects bound to bismuth ions in the sillenites.

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