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RADIATION-OPTICAL AND THERMO-RADIATION PHENOMENA IN LASER PHOSPHATE GLASSES COACTIVATED WITH NEODIUM AND CERIUM OXIDES

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Abstract. For laser phosphate glasses activated by pairwise neodymium and cerium ions, a number of radiation-optical effects have been studied: thermal and thermoradiative decolorization in the region of 200-800 nm; jump-like switching of the valence of cerium ions during the valence-coordination change in its oxygen environment. A new conceptual approach is proposed to the mechanism of the thermoradiation effect of gamma irradiation at temperatures of 100-400 °C, leading to the detection of neodymium-neodymium and neodymium-cerium ion-ion coordination interactions in nano-structural complex complexes involving the structure of a glass matrix, Only thermoradiation exposure.

Keywords: Thermoradiation phenomena, laser phosphate glasses, discoloration, switching effect.

Introduction Multicomponent phosphate glasses co-activated with neodymium and cerium oxides have long been used as laser elements in quantum electronics and in dosimetry of ionizing radiation, but have also begun to attract increasing attention in connection with the possibilities in nano-electronic computer technology. Recently, the spectroscopic properties of Nd³⁺ + activated antimony-phosphate glasses for use in micro-chip lasers have been studied in [1]. A number of works are devoted to the study of radiation color centers and the transparency of the matrix in various phosphate glasses: in germanium-phosphate glasses used as special optical filters [2]; in phosphate and fluorine-phosphate glasses [3]; in a fluorine-aluminate glassy matrix with a micro-nonhomogeneously structure that has a small scattering and a wide transparency window in the IR region [4]. To increase the radiation resistance in [4], it is proposed to activate the glass by cerium Ce³⁺ ions and europium Eu³⁺.
The results of a study of the radiation color centers in γ-irradiated glasses of the K2O-Al2O3-PbO-P2O5 model system activated with PbO and Eu³⁺ ions by Raman spectroscopy and electron absorption spectroscopy are presented in [5].

Electron irradiation with an electron energy of 5-50 keV in the near-surface layer of silicate glass produces silver nanoparticles with dimensions less than 20 nm and a gradient optical waveguide is formed with an increase in the refractive index by Δn = 0.01-0.04 [6]. In this work, radiation-stimulated processes that underline the radiation technology of creating devices in photonics, integrated optics and nano-plasmonics are considered.

In [7], the radio and thermo-luminescent properties of laser phosphate glasses activated with Nd2O3 and CeO2 oxides exposed to large doses of 60Co gamma irradiation were investigated. It is shown that nanostructural defects are formed in radiation effects, which can be used in various luminescent radiation dosimeters.

Thermoradiation effects on the absorption spectra of multicomponent phosphate glasses of the type “OPS”-3215 (without additives Nd2O3 and 2 mass.% Ce2O3) and “OPS”-3106 (with additions of 1.6 mass.% Nd2O3 and 1 mass.% Ce2O3) were investigated in [8]. The effects of radiation, thermal, and thermoradiation annealing of induced color centers with appropriate glass treatments have not been clearly identified. These shortcomings were eliminated in subsequent works [9-10] for magneto-optical glasses of the potassium-alum-borate system. We also note that the possible contribution of thermoradiation effects of interest in meta- and nanomaterial studies [11-12] is not taken into account in [2-6].

The purpose of this article is to identify the distinctive features of thermo-radiation treatments for selected multicomponent laser phosphate glasses of the type “KGSS”-2010 and “OPS”-3106, in which the ratio of the concentrations of coactivators varies for the establishment and detection of neodymium-neodymium and neodymium-cerium ion-ion coordination interactions in nanostructured impurity complexes created only by the thermoradiation effect, differing both from irradiations at room temperature, and from thermal treatments to mud after irradiation. In particular, finding the conditions for thermoradiation treatments for their practical use-increasing their radiation resistance and reducing active losses (increasing transparency).

Experimental method Two types of multicomponent laser phosphate glasses “KGSS”-2010 and “OPS”-3106 with dimensions of 10x10x8 mm³ were studied by the State Optical Institute by named S.I. Vavilov (St.Peterburg) with the corresponding marking and digestion numbers, with concentrations of activating additives Nd2O3 and CeO2 together in the composition of glass in mass% (over 100%) C (n) = [Nd2O3]: [Ce2O3] within (“OPS”-3215) - 0; 0/2; 0.2; 0.8; 1.0; 1.3; 1.6; 2.0; 6.6; 8.0; 4/0 and 8/0, as well as glasses without Nd2O3 (“OPS”-3215) and glasses without CeO2 with additives Nd2O3 4 and 8 mass.%.

Such a set of samples would make it possible to detect Nd-Nd and Nd-Ce direct interionic interactions or with the participation of oxygen or phosphate coordination shells at the nano-scale that are possible in thermoradiation processes. Sufficiently detailed fractional radiation-optical and thermoradiation studies in this article were carried out for the following compositions of phosphate glasses of the type “KGSS”-2010 C (n) = 2, [Nd2O3] (4 mass.%), CeO2 (2 mass.%) and “OPS” -3106 (C(n)=6.6, [Nd2O3] = 5 + 1.6 mass.%, CeO2 (1 mass.%). This choice is due to the fact that previous studies have shown that for C(n) = 1.0, 1.3, 1.6 Nd-Ce ion-ion interactions were not detected, and at C (n) = 8/0 and 4.0 in glasses with a large amount of Nd2O3, the phenomenon of concentration quenching causing unsatisfactory laser generation e characteristics [8]. The samples with dimensions of 10x10x8 mm³, 2 mm thick plates were cut with diamond disks, which were then machined (grinding and polishing) to the same plane-parallel plates with dimensions (1.1±0.01) mm for quantitative radiation-optical studies. Absorption spectra were recorded in the range of λ = 290-2000 nm on the EPS-3T spectrophotometer of Hitachi company and the complex spectral computational device “KSVU-23” with the diffractive mono-chromator MDR-2, as well as on a more advanced SF-56 programmed spectrophotometer (LOMO, Russia). The obtained absorption spectra were processed according to the program [19] to obtain differential absorption spectra, which increased the accuracy and reliability of the results.

Thermal and thermoradiation treatment of unified samples was carried out at a temperature close to the glass transition temperature Tg≤(673±0.5) K in the same thermo-regulated cell as part of a
modified installation outside the field and in the $\gamma$-channel with a dose rate of 3000-1000 R/s from the source of $^{60}$Co of the Institute of Nuclear Physics of the Academy of Sciences of Uzbekistan [8]. The temperature regulation of the samples outside and in the $\gamma$-field was carried out remotely (from the outside of the gamma-source channel) by means of an electronic temperature controller with an accuracy of $\pm$ 2 K, and the holding time was monitored by means of an electronic clock with an appropriate signaling of the time interval for samples in the $\gamma$-field with preset modes of thermal annealing and quenching. The optimal dwell time in the $\gamma$-field was chosen within the time $\Delta t$= 30, 60, 90 and 120 min. It was experimentally established that the $\gamma$-field was attenuated inside the cell for thermoradiation treatment to 40% as it passed through the metal housing and the heater wire.

**Experimental results and their discussions**

Consider the results shown in Fig. 1, 2. The absorption spectra in Fig. 1, curves 1 refer to the initial samples. Gamma irradiation at the channel temperature (330 K) of $^{60}$Co sources with doses of $10^4$ Gy (Fig. 1, a, b, curves 2) and $10^5$ Gy (Fig. 1, a, b, curves 3) cause an increase in absorption in the UV region (300-400 nm). Since the glasses under consideration contain 1 mass.% CeO$_2$, an increase in absorption in the UV region can be attributed to the course of oxidation reactions:

$$\text{Ce}^{3+} \rightarrow \text{Ce}^{4+} + e^- \text{ (electron transfer)}; \quad \text{Ce}^{3+} - e^+ \rightarrow \text{Ce}^{4+} \text{ (hole transfer)}.$$  

It should be taken into account that in the initial glass there is always an equilibrium concentration $[\text{Ce}^{4+}]/[\text{Ce}^{3+}] = n_2/n_1 = C(n)<1$. The aforementioned charge transfers with an increase in the C (n) ratio provides a shift in the absorption edge to the DW side. It can be seen that the absorption band with $\lambda_{\text{max}} = 240$ nm of Ce$^{4+}$ ions increases strongly (Fig. 2a, b, curves 1). Although the relative fraction of C(n)<1, however, the electron capture cross section of Ce$^{4+}$ ions is appreciably larger than the ionization cross section of Ce$^{3+}$ ions [20,21]. Therefore, at a certain dose of gamma irradiation, a dynamic equilibrium is achieved between the capture of charge carriers on structural defects and their annihilation with each other. In other words, at high doses of gamma irradiation, the process of saturation with newly created radiation defects and pre-radiative absorption defects in the glass begins. Further irradiation leads to the process of radiation annealing. As a rule, the saturation dose occurs at $\geq 5\cdot10^6$ Gy.

The absorption bands in the 430, 470, 500, 564, 660 nm regions are due to Nd$^{3+}$ f-f transitions, and the neodymium ions do not participate significantly in the absorption spectra (Fig. 1,2) in the capture of charge carriers. However, when comparing the absorption spectra of different brands of LPS, it is noted that with changes in C(n), a qualitative change in the shape and intensities of the Nd$^{3+}$ lines occurs, which indirectly indicates the presence of [Nd$^{3+} - \text{Ce}^{3+}$], [Nd$^{3+} - \text{Ce}^{4+}$] interactions.

Consider the absorption spectra of glasses of “OPS”-3106 grades with a content of (1.6+5) mass. % Nd$_2$O$_3$ and 1 mass.% CeO$_2$, depending on the processing method (Fig. 2a, b). The comparison of curves 1, 2, 3 in Fig. 2a and b show that the changes in the superconductivity in the UV region have a significant value in comparison with the ordinary radiation treatment (curve 2) and in the thermoradiation treatment (curve 3), and in In the latter case, the intensities of radiation-induced absorption bands in the UV region decrease. Absorption in the region of 290-320 nm is due to Ce$^{3+}$ ions [15], whereas a comparatively narrow line, observed during the
Fig.1. Absorption spectra of “OPS”-3106 with (1.6 + 5) mass. % Nd₂O₃ and 1 mass. % CeO₂, depending on the method of treatment (a) and (b) differ in the treatment time τ = 1 and 2 hours outside the field (a) and in γ-field (b): 1- initial sample; 2 - TT outside the field at 673 K; 3-TRT in the γ-field at 673 K. The thickness of the samples is 1 mm.

Fig.2. Absorption spectra of “KGSS”-2010 with 4 mass. % Nd₂O₃ and 2 mass% CeO₂, depending on the method of treatment (a) and (b), differ in treatment time (a) and in γ-field (b): 1- initial sample; 2 - TT outside the field outside the field τ = 1 and 2 hours at 673 K; 3-TRT in the γ-field τ = 1 and 2 hours at 673 K.
thermoriadation treatment (Fig. 2a and b, curve 3), can be connected by radiation-induced migrating hole centers [Ce$^{3+}$(e)], interacting with Nd$^{3+}$ ions.

It can be seen that the radiation dose intensifies the intensity of this line more than 2-fold and reduces the intensity of UV absorption [Ce$^{4+}$]. The changes in the bands 290-320 nm and 360 nm ([Ce$^{3+}$(e)]/[Nd$^{3+}$]) indicate the following oxidation-reduction reactions: [Ce$^{4+}$ + e$^{-}$]→ [Ce$^{3+}$ ]; [Ce$^{4+}$]→ [Ce$^{3+}$ + e$^{-}$ ]; [Ce$^{3+}$ + e$^{-}$]→ [Ce$^{2+}$ ]$^{+}$ ; [Ce$^{4+}$ ]→ [Ce$^{4+}$ + e$^{-}$]. The most probable change in the charge state for radiative TO apparently corresponds to the reaction of [Ce$^{4+}$]→ [Ce$^{4+}$ ]→ [Ce$^{3+}$(e)], and the course of these reactions significantly affects the field of ligands of Nd$^{3+}$ ions.

Since the above reactions are essentially oxidative, by analogy with the state of Fe$^{3+}$ ions in KAB glasses [10] it can be assumed that the thermoradiation conditions are equivalent to the formation of four-coordinated structures {≡ Nd─O─O─Ce ≡}, which can easily decay with trapping holes at subsequent gamma-irradiation (the disappearance of the radiation-induced band in the region of 600 nm and the shift of the absorption edge in the KB side).

The presented mechanism of the course of radiation-optical processes during thermoradiation processing is confirmed by the following considerations: for a phosphate glass matrix (OPS-3215) in the 500-600 nm region, a very wide induced absorption band arises, caused by radical-like defects of the hole type [PO]$^{+}$. On the other hand, according to the [Ce$^{3+}$] → [Ce$^{4+}$] reaction, there corresponds a hole center of 2.4 eV (525 nm) [20-21], and the recovery [Ce$^{4+}$] → [Ce$^{3+}$] corresponds to the center of 1.9 eV (650 nm). Consequently, the processes of conventional and radiation maintenance are not adequate in relation to defect formation and interaction of defects that initiate the course of radiation-quasi-chemical reactions. The sum of the effect depends on the rate of radiation-induced diffusion of defects produced by irradiation and processing temperature.

The obtained differential optical density spectra after mathematical processing are shown in Fig. 3 (a, b) in the form of differential curves $\Delta D = D_{\text{max}} - D_{\text{min}}$. We consider $\Delta D = f(\lambda)$ for glass OPS 3106 (1.6+5) mass.% [Nd$_3$O$_5$], where C(n)=5.6(5.6/1) (Fig.3,a). Curve 1 refers to the heat treatment of the sample at 673±2 K for 1 hour. It is seen that the differential negative absorption in the ultraviolet region is $\Delta D_{\text{max}}=0.4$, with the observed doublet bands of 222 and 250 nm (half-width at half-height of the Gaussian curves of 5.6 and 4.9 nm) are not allowed; there is also an unresolved doublet of bands 285 and 300 nm (with half-widths 4.3 and 4.1 nm).

In addition, a band (322 ± 350 nm) is observed in the UV region. At the end of the UV and visible region, the following bands are observed: (375-525 nm) - $\Delta D$=0; (600-700 nm) - $\Delta D$=0; (750-767 nm) - $\Delta D$=0. All the latter bands have a half-width at half-height of 3.2 and 1.6 nm. Further, the following bands are observed with maxima in the region of 540 nm ($\Delta D_{\text{max}}=0.06$); 592 nm ($\Delta D_{\text{max}}=0.09$); 725 nm ($\Delta D_{\text{max}}=0$); in the region of 775 nm ($\Delta D_{\text{max}}=0.01$). According to the literature data [8-17], the absorption bands of 230, 290, 325 nm relate to Ce$^{3+}$ ions, whereas 590, 750 and 780 nm are related to Nd$^{3+}$ ions. It can be seen that the valence-coordination state of neodymium ions depends little on temperature, whereas the valence-coordination state of cerium ions with respect to oxygen is noticeably dependent on the temperature near the glass transition point of 673 K.

Further, curve 3 (Fig. 3, a) differs from curve 1 only by holding time (2 hours) at an irradiation temperature of 673 K. The bands 240 nm and 294 nm have $\Delta D_{\text{max}} = -0.7$, i.e. become more negative than curve 1. The absorption band at 325 nm corresponds to $\Delta D_{\text{max}} = 0.12$. Absorption bands 350-372 nm; 370-450 nm; 450-600 nm and 600-650 nm correspond to the values of $\Delta D_{\text{max}} = -0.061$; 0.02; +0.15; +0.1. The half-width on the half-height varies from 3.8 to 1.8 nm. Next, the following bands are observed with maxima in the region of 540 nm ($\Delta D_{\text{max}} = +0.06$); 592 nm ($\Delta D_{\text{max}} = +0.09$); 725 nm ($\Delta D_{\text{max}} = 0$); in the region of 775 nm ($\Delta D_{\text{max}} = 0.01$).

We now turn to the consideration of thermo-radiation-treated samples of OPS 3106 (1.6+5) mass.% [Nd$_3$O$_5$] at the same temperature within the gamma field of 673±2 K at the exposure time in the gamma field of 1 hour (curve 2, Fig.3, a) and 2 hours (curve 4, Fig. 3, a). It can be seen that the bands related to Nd$^{3+}$ ions (f-f transitions) [18] here also change weakly, with the exception of the absorption band in the region of 580 nm, which disappears $\Delta D_{\text{max}} = 0$, then as with a two-hour exposure in the gamma field this level becomes the most maximum in amplitude, and Ce$^{3+}$ ions in the UV region have a minimum absorption.

In other words, the absorption band in the region of 580 nm of the Nd-Ce interaction is responsible for the complex. This explanation is also due to the fact that the oxygen coordination in cerium ions changes, which leads to a change in their charge state, Ce$^{3+}$→Ce$^{4+}$→[Ce$^{4+}$]$^+$ and, as shown
above from the absorption spectra (Fig. 1.2), correspond to the transformation of the electron center into a hole center.

The foregoing is explained by the fact that the thermoradiation conditions are equivalent to the formation of four coordinated structures \( \equiv \text{Nd} \cdots \text{O} \cdots \text{O} \cdots \text{Ce} \equiv \), which easily decay during subsequent gamma-ray irradiation with hole capture, in particular, in the 580 nm region, the absorption band disappears and a short-wave shift of the absorption edge at 50 nm. Very broad absorption bands in the 500-600 nm region are due to radical-like defects of \([\text{PO}_4]^{2-}\) in a complex with neodymium and cerium ions, which corresponds to nanoscale coordination complexes around ions of rare earths.

Fig. 3. Differential absorption spectra \( \Delta D = D_{\text{irr}} - D_{\text{init.}} \): a - for glass grade "OPS"-3106 (1.6+5) mass.% [Nd\(_2\)O\(_3\)]; 1 mass.% [CeO\(_2\)]; 1 - TT 673 K, 1 h; 2 - TRT 673 K, 1 h; 3 - TO 673 K, 2 hours; 4 - TRT 673 K, 2 hours; b - for glass grade KGSS2010; C (n) = 2 (4 mass.% [Nd\(_2\)O\(_3\)], 2 mass.% [CeO\(_2\)]); 1 - TT 673 K, 1 hour; 2 - TRT 673 K, 1 hour; 3 - TT 673 K, 2 hours; 4 - TRT 673 K, 2 h.

On the other hand, according to [8, 13-17], a hole center of 525 nm (~2.4 eV) is formed from the Ce\(^{3+} \rightarrow \text{Ce}^{4+}\) oxidation reaction, and the reduction reaction of Ce\(^{4+} \rightarrow \text{Ce}^{3+}\) is 650 nm (~1.9 eV).

Figure 3b shows the \( \Delta D \) dependence for KGSS2010 glass with C (n) = 2 (4 mass.% Nd\(_2\)O\(_3\)) and 2 mass.% CeO\(_2\)). It is seen that in contrast to Fig. 1, a the value of \( \Delta D \) has changed and become noticeably different: In the range \( \Delta D < 0.4 \) wavelengths \( \lambda = 200 \) nm to the "point" of charge transfer \( \lambda_{\text{trans.}} \).

We note the following facts: TT (at 673 K and \( \tau = 1 \) h) under atmospheric conditions in the region 200-\( \lambda_{\text{trans.}} \) nm resulted in smaller (in ~2 times) values of \( \Delta D < 0; \) as well as in Fig. 1a, the switching frequency of the Ce\(^{3+} \rightarrow \text{Ce}^{4+}\) state with \( \lambda_{\text{trans.}} = 332.4 \) nm is observed, whereas for the “OPS”-3106 \( \lambda_{\text{trans.}} \) is 318.2 nm with a shift to the short-wavelength region \( \Delta \lambda_{\text{trans.}} = 14.2 \) nm.

It is interesting that this shift \( \Delta \lambda_{\text{trans.}} \) exclusively depends on the Ce\(^{3+}\) concentration in the glass. When comparing TRT (673 K, \( \tau = 1 \) h, Fig. 3, a, curve 2, and \( \tau = 2 \) h, Fig. 3, a, curve 4) with the same curves in terms of TT and TRT for Fig. 3b, we record that curves 2 and 4 coincide at the point \( \lambda_{\text{trans.}} = 309.7 \) nm. In other words, there is a short-wave shift:

For Fig. 1, a:
\[
\Delta \lambda_{\text{trans.}}^{1,3} = 332.4 - 315.4 = (17 \pm 1) \text{ nm}
\]
\( \text{(CeO}_2 \) - 1 mass.\%\)
\[
332.4 - 310.8 = 21.6 \text{ nm}
\]
For Fig. 1, b:
\[
\Delta \lambda_{\text{trans.}}^{2,4} = 332.4 - 309.7 = (22.5 \pm 1) \text{ nm}
\]
\( \text{(CeO}_2 \) 2 mass.\%\)
Consequently, with TPO, an increase in the short-wavelength shift of the "point" of the charge transfer of Ce$^{3+}$$\rightarrow$Ce$^{4+}$ relative to TT is observed, depending on the increase in C(n). It can also be argued that the long-wave shift is observed in the case of TO with respect to TPO. The time dependence of TT (τ) is weaker than this shift $\Delta \lambda_{\text{trans}}$. Since in both Figures the "point" of charge transfer coincide for τ = 1 and 2 hour treatments (compare curves 1,3 and 2,4 in Figures 1, a and 1, b at the "points" of charge transfer).

In the region behind the charge transfer point $\Delta D \geq 0$ with $\Delta \lambda \geq 0$ up to 800 nm. In this region, 350-1800 nm ($\Delta D > 0$) with the exception of three "points" with $\lambda = 582.1; 597.6$ and 767.6 nm, where $\Delta D = 0$.

Thus, the processes of conventional radiation and thermoradiation processing are not identical with respect to defect formation and the interaction of defects initiating the course of radiation-chemical reactions.

As the result of the combination of thermoradiation processes, an increase in the efficiency of healing of oxygen vacancies present in the initial glass to the switching of the DOB bonds ($\equiv$Nd$^{4+}$O$^{2-}$) [13] according to scheme

\[ [\text{PO}_4]^{3-} \rightarrow [\text{PO}_3^{3-}]^{3-} \rightarrow [\text{PO}_2^{2-}]^{3-} \rightarrow [\equiv\text{Ce}^{3+}]^{3-} \rightarrow [\equiv\text{Ce}^{4+}]^{4+} \]

The total effect depends on the rate of radiation-induced diffusion of defects created by irradiation and high-temperature heat treatment.

The investigated glasses with a certain ratio of C (n) -neodymium and cerium oxides under thermal irradiation treatment near the glass transition temperature of 673 K become radiation sensitive to small doses of gamma or X-ray irradiation (0-10$^3$) P (low-dose effect). Prior to irradiation, the glass, due to thermoradiation treatment, acquires a blue-violet coloration, which, unlike conventional radiation treatment, is easily converted by additional irradiation. The thermoradiation effect is equivalent to strong oxidation conditions, which leads to an increase in the radiation-optical stability of the glass due to the heating of oxygen vacancies, the facilitated rearrangement of the structure of the glass matrix during subsequent radiation treatment.

**Conclusions.** Thermoradiation conditions are equivalent to enhanced oxidation conditions leading to: 1) the interaction of glasses with the atmospheric ozonized oxygen atom (O$^-$); 2) the effective formation of bound electron-hole pairs [PO$_4^{3-}$, [PO$_3^{3-}$]$^{3-}$; 3) the effective decay of cluster structures of the type [P$^5$O$^{10}$]$^{2-}$ existing in the initial glass with the formation of double oxygen bridges (DOB) called peroxide ($\equiv$O$^{2-}$O$^{2-}$). As a result of the combination of these thermoradiation processes at $T \geq 673$ K, a twofold increase in the efficiency of healing of oxygen vacancies present in the initial glass is observed, due to the switching of the DOB bonds. Under ordinary gamma irradiation, at gamma-channel temperatures, as a rule, the number of oxygen vacancies increases due to the rupture of unbridged oxygen bonds in the glass matrix.

In other words, the dynamics of the course of the thermoradiation process at $T \geq 673$ K leads to the formation of 4-coordinated switching structures [=Nd$^{3+}$O$^{2-}$O$^{2-}$Ce$^{4+}$=], which easily decay with hole capture during subsequent gamma irradiation at gamma-channel temperature. This thermoradiation mechanism is observed in the experimental $\Delta D$ curves as the charge transfer [Ce$^{3+}$]$\rightarrow$[Ce$^{3+}$]$\rightarrow$[Ce$^{4+}$], the disappearance of the radiation-induced absorption in the region of ~600 nm and the short-wavelength shift of the absorption edge in the UV region by ~50 nm. The observed and observed radiation-optical effects have both scientific and applied value, in particular, the above-mentioned switching effect can be used to create an optical processor [18] of computer technology.

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