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Temperature-dependent optical properties of GaSe layered single crystals

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ABSTRACT

Optical properties of GaSe single crystals have been investigated using temperature-dependent transmission and room temperature reflection measurements in the wavelength range of 380–1100 nm. The analysis of the absorption data at room temperature showed the existence of indirect transitions in the crystal with energy band gap of 1.98 eV. Temperature dependence of the transmission measurements revealed the shift of the absorption edge toward lower energy as temperature is increased from 10 to 280 K. The rate of change of the indirect band gap was found as $\gamma = -6.6 \times 10^{-4}$ eV/K from the analysis of experimental data under the light of theoretical relation giving the band gap energy as a function of temperature. The absolute zero value of the band gap energy and Debye temperature were calculated from the same analysis. The Wemple–DiDomenico single-effective-oscillator model applied to refractive index dispersion data was used to determine the oscillator energy, dispersion energy, oscillator strength and zero-frequency refractive index values.

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Semiconductors; optical properties; crystals

1. Introduction

$A^{III}B^{VI}$ type semiconducting compounds have become very attractive due to their structural, optical and electrical properties. GaSe, one of the members of this family, has been characterised in both experimental [1–4] and theoretical [4–6] studies to investigate its great possibilities in the areas of optoelectronic devices, nonlinear optical technology and solar cells. The studies concerning the technological applications of the crystals showed that GaSe have potentials to be used as light modulator for He–Ne laser [7], ultrathin layer transistors [8], a member of heterojunctions [9] and far infrared conversion applications [10], second harmonic generator of CO_2 laser pulses [11].

GaSe layered crystals are characterised by highly anisotropic forces due to the fact that bonding within the layers is rather stronger than bonding perpendicular to layers. GaSe crystal structure consists of four monatomic sheets in the order of Se–Ga–Ga–Se. A single layer is hexagonally ordered and *c*-axis is perpendicular to the layers. The lattice parameters

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of the hexagonal structure were reported as $a = 0.375$ nm and $c = 1.595$ nm [12]. The optical properties of GaSe single crystals have been previously investigated by absorption, photoluminescence and ellipsometry measurements. Direct and indirect band gap energies were found as 2.117 and 2.169 eV, respectively, from the analyses of absorption measurements carried out at temperature of 77 K [13]. The room temperature band gap energy of the GaSe crystals was reported as 1.988 eV obtained from the analysis of piezoreflectance measurements [14]. Photoluminescence (PL) spectrum of GaSe exhibited two peaks with energies of 2.086 and 2.067 eV [15]. Temperature-dependent PL spectra pointed out the band gap energy as 2.112 eV. Temperature dependence of higher interband transitions in the 3–4 eV spectral region was also revealed using transmission measurements [16]. Three critical points at 3.39, 3.59 and 3.81 eV were revealed in this study. Above band gap optical properties of GaSe crystals were investigated using spectroscopic ellipsometry measurements in the 0.73–6.45 eV range at room temperature [17]. The analysis of the second derivative of real and imaginary parts of the dielectric function resulted in 7 interband transitions at energies ranging from 3.23 to 5.72 eV.

Our research group reported the optical properties of GaS_xSe_{1-x} mixed crystals formed from the combination of $A^{III}B^{VI}$ type semiconducting compounds, GaSe and GaS, by transmission, reflection and ellipsometry experiments. Analyses of room temperature transmission and reflection measurements showed the presence of indirect and direct transitions with 2.28 and 2.38 eV in the $GaS_{0.5}Se_{0.5}$ single crystals formed from the GaSe and GaS [18]. Spectral and compositional dependence of real and imaginary components of pseudodielectric function, refractive index and extinction coefficient were investigated in GaS_xSe_{1-x} mixed crystal ($0 \le x \le 1$) using ellipsometry measurements [19]. In this study, aiming to find the critical point energies in the band structure of the mixed crystals, shift of the studied optical parameters to higher energy values as sulphur concentration is increased were observed.

The technological effectiveness of $A^{III}B^{VI}$ type semiconductors forces researchers to get more detailed information about these materials. In the present work, our aim is to expand the optical characterisation studies of the GaSe single crystals by carrying out the temperature-dependent transmission measurements in the 10–280 K temperature region and in the wavelength range of 380–1100 nm. The analysis of temperature-dependent band gap energies was accomplished and absolute zero value of the band gap energy, rate of change in the band gap energy with temperature and Debye temperature were obtained. Moreover, refractive index, oscillator energy and strength, dispersion energy, zero frequency dielectric constant and refractive index were found from the analysis of room temperature reflection experimental data.

2. Experimental details

Gallium selenide single crystals were grown by Bridgman method using high purity elements (at least 99.999%). The samples were prepared by easy cleavage of an ingot parallel to the crystal layer (perpendicular to the *c*-axis). Transmission and reflection measurements were accomplished in the 380–1100 nm spectral range using Shimadzu UV 1201 model spectrophotometer with resolution of 5 nm, which consisted of a 20 W halogen lamp, a holographic grating and a silicon photodiode. Transmission measurements were carried out under normal incidence of light with a polarisation direction along the (0 0 1) plane. This

plane is perpendicular to the *c*-axis of the crystal. For the reflection experiments, a specular reflectance measurement attachment with a 5° incident angle was used. Temperature-dependent transmission measurements were performed in the 10–280 K range. Advanced Research Systems, Model CSW-202 closed cycle helium cryostat was used to cool the sample. Accuracy of the cooling system was 0.5 K. Technical reasons did not allow us to perform the reflection measurements at low temperatures.

3. Results and discussion

Temperature dependence of the transmittance (T) spectra of GaSe single crystals in the range of 10–280 K is shown in Figure 1. Although the experiments were carried out in the wavelength range of 380–1100 nm, figures presented in this paper were restricted to the spectral range in which analysis methods are acceptable. For the wavelength region below the absorption edge, transmittance, reflectance and refractive index spectra display nearly linear behaviour. Therefore, this region was not focused in the analyses. Room temperature reflection experiments were performed using samples with natural cleavage planes and the thickness (d) such that $\alpha d >> 1$ (inset of Figure 1). Absorption coefficient (α) and refractive index (n) were calculated using the transmittance and reflectance (R) spectra by the help of expressions [20].

$$\alpha = \frac{1}{d} \ln \left\{ \frac{(1-R)^2}{2T} + \left[\frac{(1-R)^4}{4T^2} + R^2 \right]^{1/2} \right\} \tag{1}$$

$$n = \frac{1+R}{1-R} + \left[\frac{4R}{(1-R^2)} - \left(\frac{\alpha \lambda}{4\pi} \right)^2 \right]^{1/2} \tag{2}$$

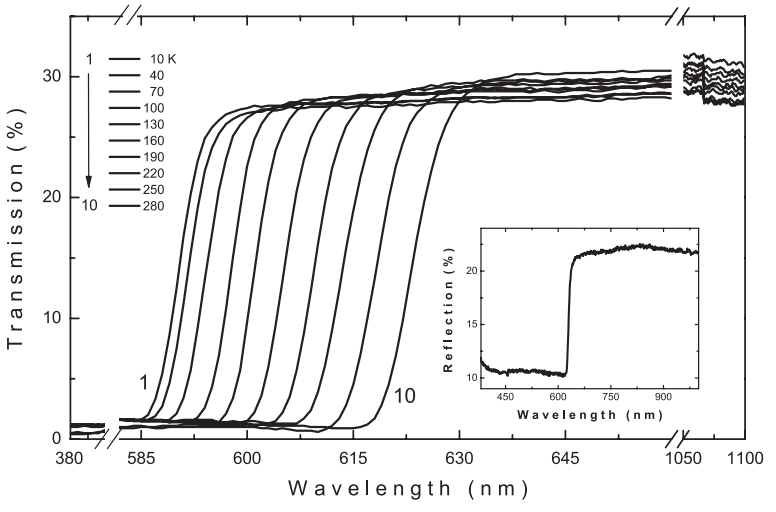

Figure 1. The spectral dependence of transmission for GaSe crystal in the temperature range of 10–280 K. Inset: The spectral dependence of reflectivity at room temperature.

Thickness of the employed sample for temperature-dependent transmission experiments was measured as 80 μm. In order to get information about the band gap energy of the crystal, dependence of α on photon energy ($h\nu$) described as [20]

$$(\alpha h\nu) = A(h\nu - E_g)^p \tag{3}$$

was used in the high absorption region. In Equation (3), A is a constant depending on transition probability and p is an index which is equal to 2 and 1/2 for indirect and direct transitions, respectively. The absorption coefficient should be equal to zero below the band gap energy. However, our spectra do not satisfy this theoretical evidence. At present time, the origin of this below band gap feature for GaSe crystals is unclear. Earlier, the absorption tails below the fundamental gap edge were also observed in the study on optical properties of $CuIn_5Se_8$ and $CuGa_5Se_8$ and $CuAl_xIn_{1-x}Se_2$ crystals [21,22]. According to the authors, these tails are result of intrinsic contributions (e.g. alloy disorder) and deviation from stoichiometry. In GaSe crystals, one of the possible reasons may be the deviation from stoichiometry during the crystal growth process.

Analysis of the experimental data revealed that ($\alpha h\nu$) and ($h\nu - E_g$) relation gives a good coherence for $p = 2$ corresponding to indirect band gap transitions (see Figure 2). The parameter p can also be determined from the slope of $\ln(\alpha h\nu)$ vs. $\ln(h\nu - E_g)$. Inset of Figure 2 presents the corresponding plot and its linear fit resulted with the slope of 2.03,

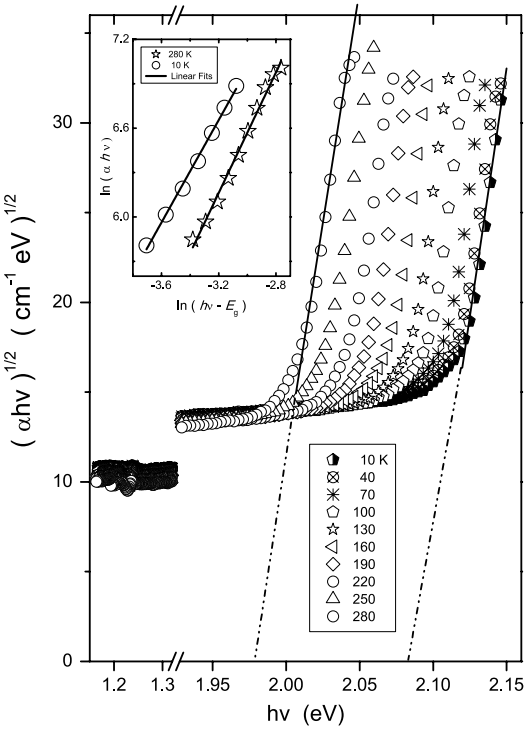

Figure 2. The dependence of $(ahv)^{1/2}$ on photon energy in the temperature range of 10–280 K. Inset: $\ln(ahv)$ vs. $\ln(hv - E_g)$ and its linear fit.

which implies the existence of indirect transition in the crystal. The indirect band gap energies (E_{gi}) at studied temperatures were found from intersection point of fitted line with photon energy axis. Temperature-dependent reflection measurements could not be carried out in our experimental set-up. Therefore, spectral dependence of room temperature reflectivity was uniformly shifted in energy according to the blue shift of the absorption edge for calculation of absorption coefficient. E_{gi} values increase from 1.98 to 2.10 eV as the temperature decreases from 280 to 10 K (Figure 3). It will be worthwhile to compare the obtained band gap energy value with previously reported values and correlate with results on GaSe – GaS mixed crystals which are the interest of our research group. The obtained room temperature band gap energy value of 1.98 eV shows a good agreement with previously reported value of 1.988 eV revealed from the analysis of piezoreflectance measurements [14]. The indirect band gap energies of GaS and $GaS_{0.5}Se_{0.5}$ single crystals were reported as 2.59 eV [23] and 2.28 eV [18], respectively. When our revealed energy of 1.98 eV for GaSe single crystal was compared with above given values for members of GaS_xSe_{1-x} mixed crystals, it can be concluded that the band gap energies of GaS_xSe_{1-x} mixed crystals increases linearly as composition x is raised from 0 to 1.

The derivative analysis of the absorption coefficient, transmission and reflection spectra was also used to find the band gap energy. The relation between absorption coefficient and photon energy given by Equation (3) can also be rewritten as [24]

$$\frac{\mathrm{d}\ln(\alpha h\nu)}{\mathrm{d}(h\nu)} = \frac{p}{h\nu - E_g}. \tag{4}$$

This equation points out that $\mathrm{d}(\ln(\alpha h\nu))/\mathrm{d}(h\nu)$ vs. $(h\nu)$ plot exhibits a peak when $h\nu$ is equal to E_g. Figure 4(a) presents the corresponding plot which shows a peak at 1.99 eV. The absorption band edge of the crystal was also obtained from the first derivative of

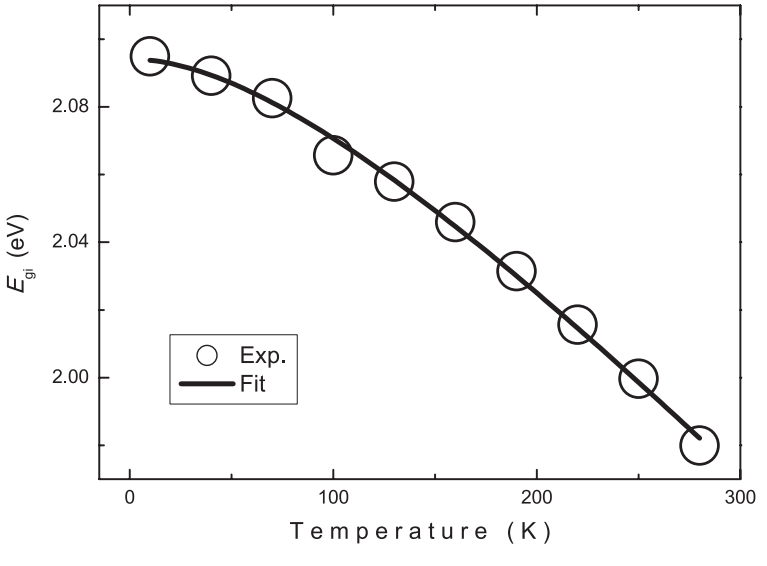

Figure 3. The indirect band gap energy as a function of temperature. The circles and solid line are experimental data and fitted line, respectively.

transmission and reflection spectra [25–27]. Figure 4(b) and (c) shows the photon energy dependence of dT/dλ and dR/dλ which present peaks at 1.99 and 1.97 eV, respectively. The estimated values from derivative analysis method show a good agreement with band gap energy obtained from Equation (3). Temperature dependence of the band gap energy (E_{gi}) was analysed to obtain absolute zero value of band gap (E_{gi} (0)), rate of change of the band gap with temperature (γ) and Debye temperature (β) using the expression [20]

$$E_{gi}(T) = E_{gi}(0) + \frac{\gamma T^2}{T + \beta}. \tag{5}$$

The fit (solid line) of experimental data (open circles) under the light of the above given relation gives successful result as can be seen from Figure 3. The fitting parameters were found as E_{gi} (0) = 2.095 eV, $\gamma = -6.6 \times 10^{-4}$ eV/K and β = 181 K.

Figure 5 shows the spectral dependence of the refractive index calculated by Equation (2). As seen from the figure, the refractive index value decreases rapidly with decreasing wavelength in the resonance energy region of 600–650 nm. This extraordinary (abnormal) behaviour in the refractive index spectrum occurs consistently in the vicinity of absorption bands in the absorption spectrum of crystal. Refractive index of GaSe slightly changes

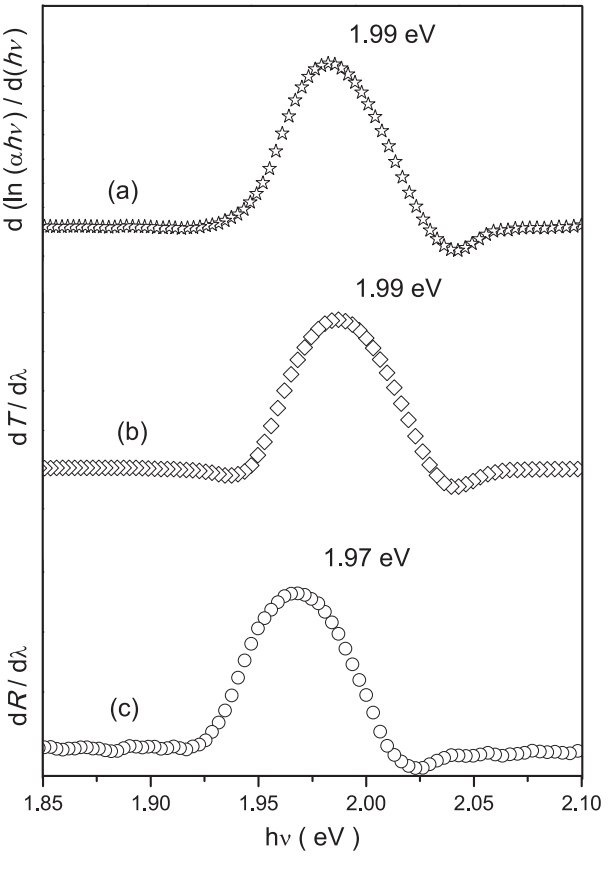

Figure 4. The dependence of (a) $d(\ln(ahv))/d(hv)$, (b) $dT/d\lambda$ and (c) $dR/d\lambda$ on photon energy for GaSe crystal at room temperature.

between 2.51 and 2.54 in the $h\nu < E_g$ region. Refractive index spectrum was fitted using the Cauchy model which relates n and λ in the low energy region as [28].

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4} \tag{6}$$

Inset of the Figure 5 demonstrates the theoretical fit (solid line) using Equation (6). A, B and C parameters were obtained to be 2.51, -7.16×10^4 nm^2, and 6.7×10^{10} nm^4, respectively.

The single effective oscillator model suggested by Wemple and DiDomenico relates the refractive index to photon energy in the $h\nu < E_g$ region. In this model, $h\nu$ dependence is given by [29]

$$n^2(h\nu) = 1 + \frac{E_{so}E_d}{E_{so}^2 - (h\nu)^2} \tag{7}$$

where E_{so} and E_d represent the single oscillator energy and dispersion energy, respectively. The parameter E_d is a measure of the strength of the inter-band optical transition. Wemple and DiDomenico related this parameter to the coordination number for anion and the valence electron number per anion. The oscillator energy E_{so} is average gap energy and, to fair approximation, it is associated empirically with the indirect band gap by the relation $E_{so} \approx 2.5\ E_{gi}$ [30]. Figure 6 shows the linear fit (solid line) of the experimental data (open circles) under the light of Equation (7). The oscillator parameters were found as $E_{so} = 5.2$ eV and $E_d = 26.1$ eV from the result of fitting process. The ratio E_{so} / E_{gi} for GaSe crystals was found in the present work as 2.63. The zero-frequency refractive index (n_0) and dielectric constant (ε_0) were also calculated using Equation (7) by replacing $h\nu = 0$ as $n_0 = 2.45$ and $\varepsilon_0 = n_0^2 = 6.02$.

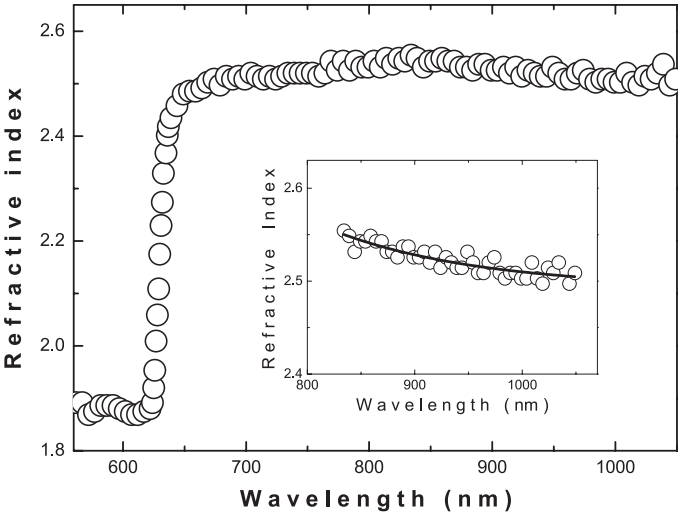

Figure 5. The variation in refractive index as a function of wavelength at room temperature. Inset: The circles and solid line show experimental data and fitted line according to Equation (6) in the low energy range, respectively.

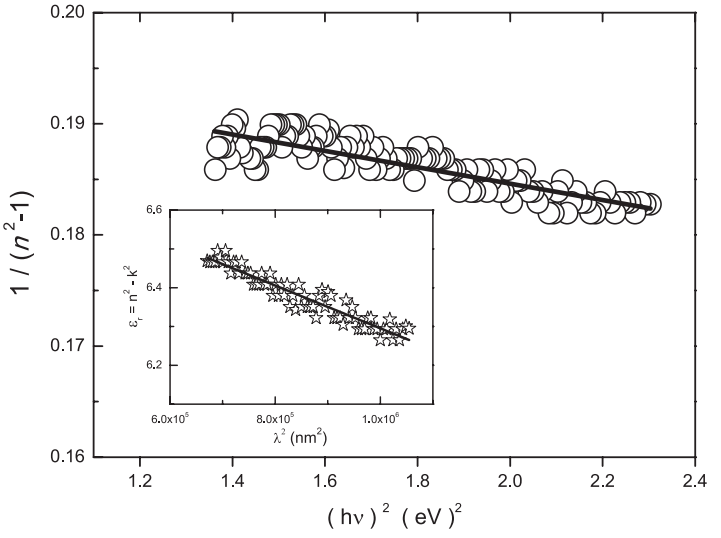

Figure 6. The plot of $(n^2-1)^{-1}$ vs. $(h\nu)^2$ in the $h\nu < E_g$ range. Circles are experimental data and solid line represents the linear fit. Inset: Plot of ε_1 vs. λ^2. Stars are experimental data and solid line shows the linear fit.

The oscillator strength (S_{so}) and wavelength (λ_{so}) of GaSe single crystals were calculated from analysis of the refractive index in the $h\nu < E_g$ region using single Sellmeier oscillator model. The refractive index is expressed in this model as [31]

$$(n^2-1)^{-1} = \frac{1}{S_{so}\lambda_{so}^2} - \frac{1}{S_{so}\lambda^2} \tag{8}$$

S_{so} and λ_{so} values were obtained from the $(n^2-1)^{-1}$ vs. λ^{-2} curve as 8.85×10^{13} m^{-2} and 2.38×10^{-7} m, respectively. The wavelength dependence of real component (ε_1) of dielectric function was analysed using Spitzer–Fan model in which ε_1 is defined as [20]

$$\varepsilon_1 = n^2 - k^2 = \varepsilon_\infty - \left[\frac{e^2}{\pi c^2}\right]\left(\frac{N}{m^*}\right)\lambda^2 \tag{9}$$

where ε_∞ is the high-frequency dielectric constant in the absence of any contribution from free carriers, N is the carrier concentration, m^* is the effective mass, c is the speed of light and e is the electronic charge. Inset of Figure 6 shows the linear fit (solid line) of experimental data (open circles). ε_∞ and N/m^* were obtained from the intersection of fitted line with ε_1-axis and slope of the linear fit, respectively, as $\varepsilon_\infty = 6.85$ and $N/m^* = 6.75 \times 10^{47}$ g^{-1} cm^{-3}. High- frequency dielectric constant was previously reported for GaSe crystals as 7.44 [32].

4. Conclusion

Temperature-dependent transmissions and room temperature reflection measurements have been carried out on GaSe single crystal to investigate its optical properties. The indirect band gap energy of the crystal at room temperature was found as 1.98 eV from the photon energy dependence of absorption coefficient. Analysis of temperature-dependent

transmission measurements revealed that band gap energy increases from 1.98 to 2.10 eV as the temperature decreases from 280 to 10 K. Wemple–DiDomenico single-effective-oscillator model was applied to refractive index dispersion data. The oscillator energy, dispersion energy, oscillator strength and zero-frequency refractive index values were found from the analysis as $E_{so} = 5.2$ eV, $E_d = 26.1$ eV, $S_{so} = 8.85 \times 10^{13}$ m^{-2} and $n_0 = 2.45$, respectively.

Disclosure statement

No potential conflict of interest was reported by the authors.

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