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# Donor-acceptor pair recombination in gallium sulfide

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Low temperature photoluminescence of GaS single crystals shows three broad emission bands below 2.4 eV. Temperature and excitation light intensity dependencies of these bands reveal that all of them originate from close donor-acceptor pair recombination processes. Temperature dependence of the peak energies of two of these bands in the visible range follow, as expected, the band gap energy shift of GaS. However, the temperature dependence of the peak energy of the third band in the near infrared shows complex behavior by blueshifting at low temperatures followed by a redshift at intermediate temperatures and a second blueshift close to room temperature, which could only be explained via a configuration coordinate model. A simple model calculation indicates that the recombination centers are most likely located at the nearest neighbor lattice or interstitial sites. © 2000 American Institute of Physics. [S0021-8979(00)04724-1]

#### I. INTRODUCTION

A wide variety of binary and ternary layered semiconductors attract much interest due to possible optoelectronic applications from ultraviolet to the infrared. For the most part, optoelectronic properties of these materials are dominated by defects of various types and the interactions between them. In spite of the significant experimental and theoretical efforts devoted to the study of these materials, both experimental data and the overall theoretical understanding of them lacks a coherent and complete framework. In fact, even in binary compounds such as GaS, little has been done either experimentally or theoretically.

Photoluminescence (PL) spectroscopy is a very suitable and widely used technique to study the defect structures of semiconductors. Unfortunately, most of the work on layered semiconductors has concentrated on the near band edge emission of these materials. Scarce data exist on deep PL bands with emission energies significantly below the band gap of the semiconductor.

GaS belongs to a family of semiconductor crystals of the A<sup>III</sup>B<sup>VI</sup> type along with GaSe and InSe, which crystallize in a layered structure. In GaS, interlayer interactions are dominated by van der Waals forces, while intralayer-bonding forces are primarily ionic covalent in nature. Due to weak interlayer interaction, GaS can easily be cleaved along these layers. GaS crystallizes in the so-called  $\beta$ -polytype and carries the symmetry properties of the  $D_{6h}^4$  space group. Each layer consists of four atoms stacked along the c axis with a repeating unit of S-Ga-Ga-S. There are two layers in a unit cell.

GaS is a wide band gap semiconductor with an indirect band gap at about 2.59 eV at 300 K.2 A direct band gap exists at approximately 0.45 eV higher in energy. Thus, GaS is considered to be a promising material for near-blue light emitting devices. It exhibits both electroluminescence and PL in the green-blue region.<sup>3-5</sup> PL of both undoped and Zn doped GaS near the fundamental band edge has been studied by various authors.<sup>6,7</sup> Near band edge luminescence attributed to recombination of shallow donors with distant acceptors has been proposed.8 Deep level luminescence was observed by Karaman and Mushinskii.<sup>9</sup> However, there are no PL data on GaS below 77 K and no detailed analysis of deep level luminescence spectra of GaS in the literature.

In this article, we report both the temperature dependence in the 9-280 K range and excitation intensity dependence in the  $10^{-3}$ – $10 \text{ W cm}^{-2}$  range, of the deep level luminescence in GaS.

### **II. EXPERIMENT**

Gallium sulfide polycrystals were synthesized from high purity (at least 99.999%) gallium and sulfur taken in stoichiometric proportions. Single crystals of GaS were grown by the modified Bridgman method. X-ray diffraction analysis of the data showed that GaS crystallizes in the hexagonal unit cell with lattice parameters of a = 0.359 and c = 1.549nm. Crystals suitable for measurements were obtained by easy cleavage along the (001) plane perpendicular to the optical c axis. As-grown GaS is an n-type semiconductor having an indirect energy band gap of 2.591 eV at 77 K and 2.597 eV at 4.2 K.<sup>2</sup>

PL experiments on GaS were made from the cleaved surfaces of the crystals in a backscattering configuration in the wavelength range of 525-860 nm. The 457.9 nm (2.71 eV) line of an argon ion laser was used as the exciting light source. The luminescence was analyzed with a U-1000 Jobin-Yvon double grating monochromator and cooled GaAs photomultiplier tube with the standard photon counting electronics. A CTI-Cryogenics M-22 closed-cycle helium

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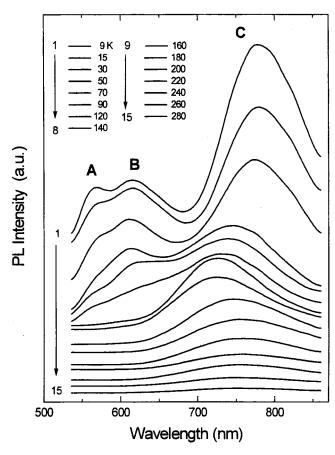


FIG. 1. Temperature dependence of PL spectra from GaS single crystal in the 525–860 nm range. Note that for curves 13–15, intensities have been multiplied by a factor of 2.

cryostat was used to cool the crystals from room temperature down to 9 K. Temperature was controlled to within an accuracy of  $\pm 0.5$  K. A cylindrical lens was used in focusing optics in order to minimize unnecessary heating of the sample by the incident laser beam. Sets of neutral density filters were used to adjust the exciting laser intensity from 0.0007 to 8.0931 W cm<sup>-2</sup>, a factor of more than  $10^4$ . The PL spectra have been corrected for the spectral response of the optical apparatus.

### III. RESULTS

PL spectra of GaS in the 525–860 nm wavelength and 9–280 K temperature range at a constant excitation intensity are shown in three separate graphs for clarity in Fig. 1. We observe three broad PL bands centered at 558 nm (2.22 eV, A band), 614 nm (2.02 eV, B band) and 780 nm (1.59 eV, C band) at 9 K. We note that both the PL intensities and the PL peak energies change as a function of increasing sample temperature. As the temperature of the sample is increased, PL intensity of all three bands decreases. However, while at higher temperatures, the two short wavelength peaks disappear altogether, the intensity of the third band in the near infrared part of the spectrum increases for a narrow range of temperatures from 90 to 150 K, followed by a decrease of the PL intensity. The observed peak energies of the two short wavelength bands at 9 K shift toward the red continuously as

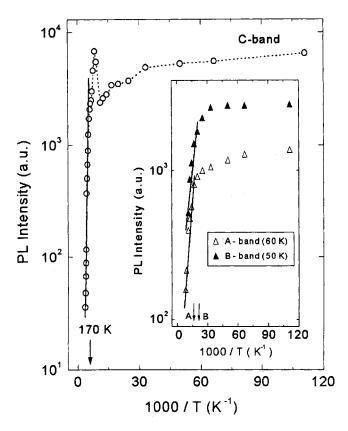


FIG. 2. Temperature dependence of the peak intensity of A, B, and C bands. The activation energies obtained from the fit of Eq. (1) are found to be 0.017, 0.013, and 0.151 eV, for A, B, and C bands, respectively. The arrows show the starting points of the intensive quenching.

the temperature is increased, until they are totally quenched, whereas the third band shifts toward the blue until 130 K followed by a redshift thereafter. Despite this peculiarity of the near infrared band, these features are indicative of donor-acceptor pair transitions observed in many ternary semiconductors. All data have been analyzed by fitting Gaussian line shapes from which peak intensity, peak position, and the full width at half maximum (FWHM) of all three bands were obtained. The variations of the PL peak intensity of all three bands with respect to temperature are plotted in Fig. 2. In the low temperature range, PL intensity of the A and the B bands decreases slowly. Above 60 K for the A band and 50 K for the B band, however, the peak intensity decreases at a much higher rate due to a thermal quenching process. A similar behavior is observed for the C band, which decreases slowly in the 9-90 K range and except for a small range of temperatures between 90 and 150 K where it increases, it decreases much more sharply above 150 K. The small increase in the PL emission intensity between 90 and 150 K is due to redistribution of carriers as both A and B bands totally quench. The activation energies for all three bands have been obtained by fitting to the following equation:

$$I = I_0 \exp(\Delta E/k_B T), \tag{1}$$

where I is the PL intensity,  $I_0$  is a proportionality constant, and  $k_B$  is Boltzmann's constant. The semilog plot of the peak intensity as a function of the reciprocal temperature gives a

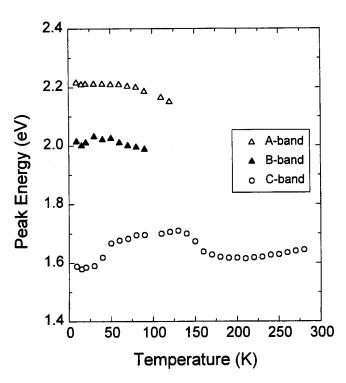


FIG. 3. Temperature dependence of the peak energy of bands A, B, and C.

straight line for all three bands at low temperatures, the slopes of which give the activation energies as 0.017, 0.013, 0.151 eV for *A* band, *B* band, and *C* bands, respectively. Since, as-grown GaS is an *n*-type semiconductor as confirmed by the measurement of the carrier type by the hot probe method, we consider that the activation energies of 0.017 and 0.013 eV are associated with donor levels. As will be shown shortly, the temperature dependencies of the PL peak energy and FWHM of the *C* band are different from the others which lead us to explain the behavior of this band within the configuration coordinate (CC) model. Therefore, the activation energy derived from Fig. 2 for the *C* band is associated with the difference between the minimum of the excited state and the intersection point of the excited and ground state CC curves.

Temperature dependence of the peak energy for all three bands is plotted in Fig. 3. Both A and B bands show a small redshift as the temperature increases. Full temperature dependence of the band gap of GaS is not available in the literature but it is clear that the temperature coefficient of the band gap energy of GaS is negative.<sup>2</sup> Therefore, the peak energy due to donor-acceptor pair recombination should also decrease as the temperature increases. The observed shift of the peak energy position toward lower energies satisfies the temperature dependence expected for donoracceptor recombination for these bands. Similar behavior is observed in many binary and ternary semiconductors. 11,12 As seen in Fig. 3, temperature dependence of the C band, however, shows totally different behavior. Instead of the redshift observed as in the cases of A and B bands, peak energy of the C band, starting at 1.59 eV (at 9 K) blueshifts rapidly between 25 and 50 K and continues to blueshift beyond 50 K until 130 K, albeit at a smaller rate until it reaches a peak value of 1.71 eV. The total blueshift observed is approxi-

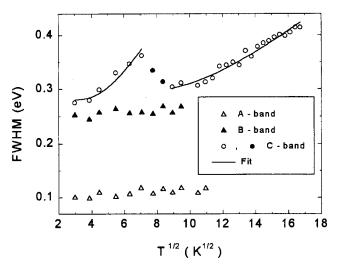


FIG. 4. Linewidths of the A, B, and C bands as a function of the square of temperature. Note the two separate behaviors at low and high temperature regions for the C band. The data points shown with solid circles have not been used in the fitting procedure.

mately 120 meV in this temperature range. Beyond 130 K, peak energy of the *C* band, redshifts back, reaching 1.62 eV at 190 K, larger than what is observed at the lowest temperature. In fact, the peak energy value does show yet a second blueshift beyond 200 K up until 280 K, where the intensity of the *C* band becomes very weak. Blueshifting of the peak energy of an emission from semiconductors has previously been studied within the framework of the configuration coordinate model where a ground state is associated with an acceptor level and an excited state is derived from a donor level situated in the band gap of the crystal. Before we discuss our data within this model, we present the temperature dependence of the FWHM of the *C* band *W*, which appears to follow the CC model equation<sup>10</sup>

$$W = W_0 \left[ \coth(h \nu_e / 2k_B T) \right]^{1/2}, \tag{2}$$

where  $W_0$  is a constant, whose value is equal to W as the temperature approaches 0 K and  $h\nu_e$  is the energy of the vibrational mode of the excited state. The result of the analysis is shown in Fig. 4. The FWHM of the C band shows two distinct behaviors. At lowest temperatures, it has a value of 0.28 eV. As the temperature increases, FWHM shows rapid increase until 50 K where it reaches the maximum value of 0.36 eV followed by a rapid decrease in the FWHM in the 50-80 K range. The value of FWHM at 80 K is 0.30 eV, which is still larger than the starting value of 0.28 eV. Finally, it increases once more, but now at a slower rate than previously, reaching 0.42 eV at 280 K. As is clearly seen from the data, the slopes of low- and high-temperature behavior of the FWHM are different. Using a least squares routine, we fit Eq. (2) to low- and high-temperature parts of the data, separately. The fitted values of  $W_0 = 0.279$  eV,  $h \nu_e = 5.45$  meV for the low-temperature and  $W_0 = 0.294$  eV,  $h\nu_e$ =25.36 meV for high-temperature parts of Fig. 4 are obtained. Clearly, much higher values of  $h\nu_e$  are associated with the high temperature behavior of the width of the C

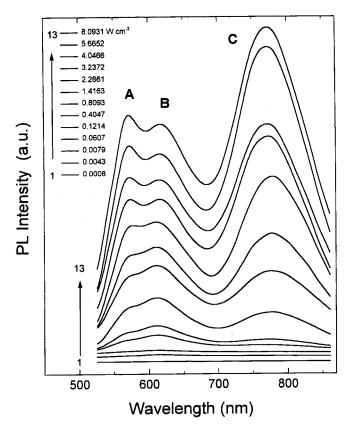


FIG. 5. Excitation intensity dependence of the photoluminescence spectra of GaS at T=9 K.

band. Finally, we have also analyzed the widths of both A and B bands and find that they do not show any significant temperature dependence as does the C band (Fig. 4).

In elucidating the nature of deep level luminescence from semiconductors, excitation laser intensity dependence of the PL spectra is an important consideration. In Fig. 5, we present the excitation laser intensity dependence of the PL spectra of GaS. All three bands are visible even at the lowest excitation intensity and increase in intensity as the excitation intensity increases. Detailed analysis of the peak positions of all three bands, as a function of increasing excitation laser intensity did not yield any variation of the peak energy values of any of the bands. Therefore, we concentrated on the analysis of PL intensity as a function of the excitation laser intensity. Excluding the very high intensity region where there is clear saturation of the PL intensity, analysis has been carried out by fitting the experimental data to a simple power law of the form

$$I \propto L^{\gamma}$$
. (3)

where I is the PL intensity, L is the excitation laser intensity, and  $\gamma$  is a dimensionless exponent. It was found that the maximum PL intensity of the emission band increases sublinearly with respect to the excitation laser intensity for all three bands (Fig. 6). The values of  $\gamma$  were found to be 0.86, 0.80, and 0.88 for A, B, and C bands, respectively. Saturation starts at  $L>0.41~{\rm W\,cm^{-2}}$  for the B band, at  $L>3.24~{\rm W\,cm^{-2}}$  for A and C bands. It is well known that for the excitation laser photon with an energy exceeding the band

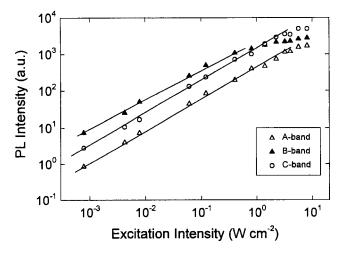


FIG. 6. Excitation intensity dependence of the peak photoluminescence intensity of bands A, B, and C.  $\gamma$ =0.86, 0.80, and 0.88 for A, B, and C bands, respectively.

gap energy  $E_g$ , the coefficient  $\gamma$  is generally  $1 < \gamma < 2$  for the free- and bound-exciton emission, and  $\gamma \le 1$  for free-to-bound and donor-acceptor pair recombinations. Thus, the obtained values of  $\gamma < 1$  for all three bands further confirms our assertion that all three bands in the PL spectra of GaS are due to donor-acceptor pair recombination.

### **IV. DISCUSSION**

From the above data, it is clear that there are two types of behavior in the PL spectrum of GaS: one is the behavior of bands A and B, the other is the behavior of the C band. The two short wavelength bands A and B show quenching at relatively low temperatures of 60 and 50 K, respectively, with no significant change in the width of their bands. Peak energy of both bands shows a small decrease with increasing temperature but does not change with increasing excitation laser intensity. While the former result is typical of donor–acceptor pair recombination process, the latter result rules out the model of "distant" donor–acceptor pair (DAP) recombination process where a shift toward higher energies is expected as the excitation laser intensity is increased. It is known that in general, the emission energy from a DAP separated by a distance of r is obtained from 15

$$E(r) = E_g - (E_A^0 + E_D^0) + \frac{Z_D Z_A e^2}{\epsilon r} - \Gamma(r),$$
 (4)

where  $E_g$  is the band gap energy,  $E_A^0$  and  $E_D^0$  are the acceptor and donor ionization energies,  $\epsilon$  is the dielectric constant,  $Z_D$  and  $Z_A$  are the charges of the donor and acceptor states, respectively.  $\Gamma(r)$  is a quantum mechanical correction representing the interactions at very short distances. Since this term gives only second order correction and only at very short distances, we will neglect it in our discussion. However, the value of  $\Gamma(r)$  at very short distances may be as high as 25 meV in some cases  $^{16}$  so that theoretically calculated and experimentally determined values of Coulombic energies may differ significantly.

From Eq. (4), two features of the observed data become clear. First, as the temperature is increased, thermal emission

of charge carriers bound to donors and acceptor levels should result in longer diffusion lengths, increasing the probability of recombination between pairs with larger separation. Because of this, the peak energies should shift to lower energies in addition to the shrinking of the band gap.<sup>17</sup> Second, it is also clear that as the excitation laser intensity is increased, the contribution of closer pairs will increase, leading to the expected blueshift of the peak energy of the emission. Since this is in contrast with the observed data, we conclude that only close pairs such as the nearest or next-nearest neighbors are involved in the transitions. This assumption is further supported by the fact that observed decrease of the peak energy of both *A* and *B* bands is indeed relatively small, suggesting that pairs far apart contribute little to the observed emission.

Unfortunately, there is little data on the nature of defect states in GaS that will help identify possible defect complexes involved in the observed recombination processes. Aono et al.<sup>5</sup> studied GaS crystals grown by the iodine transport method, where iodine highly substitutes for a sulfur site and acts as a donor and where, from charge neutrality arguments, production of gallium vacancies with two charge states  $V_{\rm Ga}^{+2}$  and  $V_{\rm Ga}^{+1}$  was assumed. Furthermore, the existence of  $V_{\rm Ga}^{+2}$  is understood to be an excited state of  $V_{\rm Ga}^{+2}$  when it releases an electron. Thus, the energy level of  $V_{\rm Ga}^{+2}$  should be higher than that of  $V_{\rm Ga}^{+1}$  above the valence band edge. In general, one should consider all combinations of vacancies and interstitials for both gallium and sulfur in GaS as possible sites of recombination. Due to the binary nature of the crystal, the possible number of such pairs is limited. However, different charge states may increase this number. It is possible that a gallium vacancy with different charge states and sulfur interstitial are involved in these transitions but more data are required before a firm conclusion can be made.

Recently Krustok *et al.*<sup>15</sup> suggested that it may be possible to guess the positions of the defect states involved in DAP recombinations by calculating the energy separation of the observed bands. Assuming that only the nearest neighbor or next-nearest neighbor lattice sites are involved, we compare a pair such as Ga–S with another such as (i)–S, where (i) represents an interstitial position, as an example. Here, Ga and S represent the lattice sites, which behave as acceptor and donor sites. Again, assuming that the acceptor and donor energies as well as the quantum mechanical correction term  $\Gamma(r)$  for both pairs are approximately the same, one finds the energy separation of two bands as

$$\Delta E = \frac{Z_A Z_D e^2}{\epsilon} \left( \frac{1}{r_1} - \frac{1}{r_2} \right),\tag{5}$$

where  $Z_A$  and  $Z_D$  are the charge states of the acceptor and donor sites,  $r_1$  and  $r_2$  represent the pair separation of each pair, e is the electronic charge, and e is a combination of optical and static dielectric constants. However, an exact numerical value for e is hard to predict not only because the exact combination of optical and static dielectric constants are not known, but GaS has two dielectric constants: one parallel to the e axis, the other perpendicular to it. Furthermore, there may be lattice distortion associated with defect sites, which would introduce further errors into the calcula-

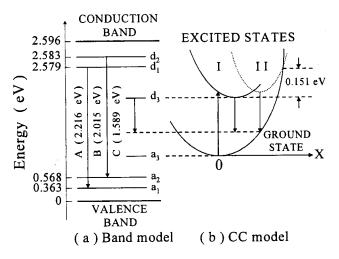


FIG. 7. Proposed model for the donor-acceptor pair recombination processes in GaS at T=9 K. Bands A and B are associated with  $d_1-a_1$  and  $d_2-a_2$  levels in the band gap, whereas band C is associated with the ground and excited states of the CC model derived from acceptor and donor levels, respectively. The energy difference between the bottom of the first excited state and the intersection point of the ground state with the second excited state is the energy required for thermal quenching. This is indicated by the value of 0.151 eV on the diagram.

tions. Despite the uncertainties associated with using Eq. (5), we find that only the nearest neighbor pairs and interstitial sites give reasonably close values to the observed energy separation  $\Delta E$  of 200, 430, and 630 meV between A and B, B and C, and A and C bands, respectively. Comparison of nearest neighbor pairs with distant neighbors results in energy separation values on the order of a few eV, clearly beyond what is observed experimentally. In these calculations, we used  $Z_A = 1$  and  $Z_D = 1$  and the larger of the dielectric constants which is the one perpendicular to the c axis,  $\epsilon = 9.5$ ,  $^{18}$  but using any value smaller than 9.5 would not change our overall conclusion since  $\Delta E$  is inversely proportional to  $\epsilon$ .

Sublinear dependence of the PL intensity on the excitation laser intensity is also consistent with the donor–acceptor pair recombination process since the simultaneous solution of the rate equations involving all possible radiative transitions give sublinear dependence for free-to-bound and donor–acceptor pair recombinations. We summarize the results for A and B bands in Fig. 7. Using the activation energies obtained from the temperature dependence of the PL intensity for n-type GaS, we place donor levels  $d_1$  and  $d_2$  at 0.017 and 0.013 eV below the conduction band. From the peak energy values of the observed transitions, the related acceptor energy levels  $d_1$  and  $d_2$  are then placed at 0.363 and 0.568 eV above the valence band. Thus, our DAPs for  $d_1$  and  $d_2$  bands are composed of deep acceptor and shallow donor states.

The most striking feature of the *C* band is the temperature dependence of its peak energy. As seen in Fig. 3, in contrast with *A* and *B* bands, the peak energy of the *C* band increases by almost 120 meV until 130 K is reached followed by a redshift. Such behavior is classically understood in terms of the configuration coordinate model of defects. In Fig. 7, we present our configuration coordinate model for

the C band. In this model, the ground state of the system is derived from a deep acceptor level while the excited level is associated with a donor level. Since AIIIBVI-type layered crystals are more polar than covalent, 19 the displacement of the excited state minimum is expected to be reasonably large. The quantum mechanical treatment of the configuration coordinate model leads to quantized states associated with each level. The initial blueshift of the peak energy with increasing temperature is understood by the transitions from higher states in the excited level as the occupation of these states increase with temperature. From the bandwidth data we deduce that FWHM and hence the energy of the excited state  $h\nu_e$ , involved in the emission, changes in the temperature range 50–80 K. While  $h\nu_e$  is about 5 meV below 50 K, it becomes 25 meV above 80 K. This suggests the presence of a higher excited state. This is shown as a second excited state next to the first one in Fig. 7. Blueshifting of the peak energy continues to increase up to 130 K in this second excited state. In this model, the redshift beyond 130 K is understood by the filling of the lowest ground state. The slow blueshift following the redshift is also understood yet by further filling of the second excited state. Finally, the thermal quenching occurs when the carrier energy is larger than 0.151 eV, so that it can relax to the ground state with a nonradiative transition at the intersection of the ground and excited state energies. It may be possible to construct more complicated CC models; however, we find that this is the simplest model that seems to account for all the observed features of the PL from GaS.

#### V. CONCLUSIONS

We measured low temperature PL spectra of high quality GaS single crystals grown by the vertical Bridgman method. At 9 K, three PL bands at 2.22 eV (*A* band), 2.02 eV (*B* band), and 1.59 eV (*C* band) have been observed. Both the excitation intensity and temperature dependence of the bands suggest that nearest neighbor pairs are responsible for the emissions. Deep acceptor and shallow donor levels in the band gap of GaS account for the observed emission for *A* and

*B* bands. The peculiar behavior of the *C* band is understood within the framework of a proposed configuration coordinate model in Fig. 7. The changes in the FWHM of the *C* band as a function of temperature imply that there may be two excited states. As the samples are not intentionally doped, donor and acceptor states are thought to originate from point defects. Clearly, more experimental data are required to clarify and develop this model further.

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