Electron-hole liquid and hysteresis in the emission of biexcitons in AgBr

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The intensity of the emission of biexcitons has been studied as a function of the excitation of AgBr single crystals in the temperature range corresponding to the "'biexciton pocket." The excitation intensity was smoothly increased and then reduced to its original level. The resulting curve is loop-shaped, indicating the appearance of an electron-hole liquid in this material. The loop is traced out in the direction opposite that which follows from an analysis of the kinetics of isothermal condensation. The reason for the anomalous behavior of the observed hysteresis loop may be that the condensate droplets undergo self-heating because of the obviously nonequilibrium nature of the phase transition which leads to the stratification of the electron-hole system.

INTRODUCTION

Indirect-gap semiconductors with a polar chemical bond, such as AgBr, provide the most favorable setting for the appearance of an electron-hole liquid. The presence of several energetically equivalent valleys (in AgBr, these are four hole valleys, at the L points in the Brillouin zone) and the strong electron-phonon interaction which is characteristic of these materials stabilizes the liquid phase with respect to the state of free excitons (or biexcitons). Calculations have shown that the condensation energy in AgBr, $\varphi \approx 30$ meV, is significantly higher than that for the electron-hole liquids in Ge and Si, which have been studied most thoroughly.[1]

A luminescence band with properties corresponding to the liquid phase of the carriers was discovered in silver bromide crystals nearly ten years ago.[2,3] The spectroscopic characteristics of this band indicate a significant condensation energy ($\varphi \approx 55$ meV) and a pronounced selective heating of the electron-hole liquid ($T \approx 70$ K) with respect to the reservoir ($T = 26$ K). At the same time, it was reported that an electron-hole liquid and biexcitons coexist in AgBr and that the biexcitons exist in a bounded region in the plane of the carrier density and the temperature (the n-T plane).[2,4] This bounded region in the n-T plane has been named the "biexciton pocket." Its appearance was explained in terms of the formation of an electron-hole liquid. Despite these results, which would appear to be encouraging for a study of condensation, Baba and Masumi[5] have recently disputed the interpretation of the band (the Q band) which was observed in Ref. 2 as a consequence of the recombination of an electron-hole liquid. As a result, some uncertainty has been introduced, and it remains a substantial question mark in the experimental results on condensation in AgBr. Yet a further complication of this research is that the Q band coincides in spectral position with an intense, broad band characteristic of the crystal of this compound, caused by the emission of excitons which are bound at iodine atoms, with the simultaneous emission of different numbers of phonons.[3] This circumstance has forced the use of time-resolved measurements to distinguish the "pure" Q band at relatively low crystal excitation levels. This procedure rules out certain types of experiments which are typical in research on electron-hole liquids.

The experimental results which we will describe below provide what we perceive as convincing evidence in favor of the appearance of an electron-hole liquid in AgBr. A premise underlying these studies was that if the biexciton pocket which was detected in Ref. 2 and observed in our own earlier study[6] is a consequence of the existence of an electron-hole liquid then the hysteresis in the appearance and disappearance of electron-hole liquids would mean that its concentration width should depend on the history of the excitation of the crystal.

It is found that a slow increase in the excitation level, followed by a decrease, gives rise to a hysteresis loop on a plot of the integral intensity of the biexciton emission band in AgBr versus the intensity at which the crystal is excited (on the illumination-intensity characteristic). From the direction in which the observed loop is traced out we can draw conclusions regarding a self-heating of electron-hole droplets as they develop. As we will show, the condensation is nonisothermal in this case because of the distinctly nonequilibrium nature of the phase transition. The extent of the deviation of this transition from equilibrium is determined by the relatively brief ($\sim 10^{-8} - $s) lifetime τ_0 of the carriers which have undergone condensation.

EXPERIMENTAL PROCEDURE

We studied the total intensity I of the emission band of free excitons while varying the intensity I_p of the excitation of the AgBr single crystals ($E_g = 2.7$ eV). Specifically, we smoothly increased I_p [the ascending branch of the $I(I_p)$ curve] and smoothly reduced it (the descending branch). The test samples, with a typical thickness ≈ 1 mm and a surface area $\approx 4 \times 4$ mm^2, were cut from nominally pure single-crystal bars grown by the Bridgman method in bromine vapor. The surfaces of the samples were chemically polished in a sodium hyposulfite solution. After etching, the samples were washed several times in alcohol and in doubly distilled water.

Figure 1 shows a simplified layout of the computer-controlled spectral apparatus. Bulk excitation of the samples, held in a cryostat with automatic temperature regulation within ± 0.01 K, is achieved with the beam from a He–Cd laser ($h\nu_{\text{He-Cd}} = 2.807$ eV). The absorption coefficient of AgBr at the frequency $h\nu_{\text{He-Cd}}$ is ~ 10 cm^{-1}. In order to

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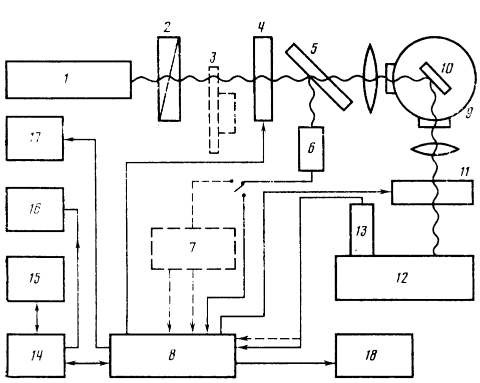

FIG. 1. Block diagram of the computer-controlled spectral apparatus. 1—He-Cd laser; 2—Glan prism; 3—mechanical modulator; 4—polarizer linked to the axle of a stepping motor (ShD1); 5—quartz plate; 6—photomultiplier (FEU1); 7—two-channel pulse generator; 8—CAMAC apparatus; 9—cryostat; 10—test sample; 11—chopper; 12—DFS-12 spectrometer, linked to the axle of a stepping motor (ShD2); 13—photomultiplier (FEU2); 14—computer; 15—alphanumeric display; 16—printer; 17—graphics display; 18—x, y plotter. The dashed lines indicate units which are inserted for time-resolved measurements of the spectra.

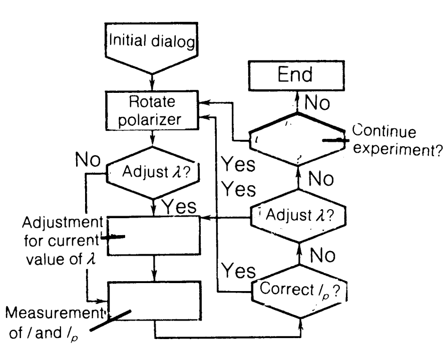

FIG. 2. Flowchart of the program for measuring the ascending and descending branches of the illumination-intensity characteristic of the biexciton emission band in AgBr.

vary I_p smoothly, we used an intensity "variometer" consisting of a Glan prism crossed with a polarizer connected to the axle of a ShD1 stepping motor. This motor was controlled through a CAMAC apparatus by a computer; the smallest attainable rotation angle of the polarizer was $\approx 1'$.

The system consisting of the computer and the CAMAC apparatus scans the emission spectrum under study. The emission is sent through a DFS-12 high-luminosity double monochromator with a diffraction grating which is linked to the axle of the ShD2 stepping motor. Data on the emission intensity arrive at the inputs of the CAMAC counters in digital form and are recorded in memory cells of the computer. In addition to controlling the experiment, the computer serves as a multichannel analyzer. The time interval over which the data arriving at the counters are stored is chosen with the help of a clock-pulse generator and a timer. Figure 2 is a flowchart of the program used by the computer to record the ascending and descending branches of the $I(I_p)$ dependence for the biexcitons.

After the measurements have been completed, the computer carries out the mathematical processing of the spectra smoothing the experimental points, subtracting from the spectra a reference spectrum corresponding to a low excitation level, and integrating the biexciton emission band. The subtraction procedure is included in order to obtain the pure shape of the biexciton band—the shape undistorted by the superposition of bands of different nature. For each value of I_p, the reference spectrum is corrected in a procedure which incorporates the illumination-intensity characteristics of the bands which are being subtracted.

In addition to recording the ascending and descending branches of the illumination-intensity characteristics of the biexciton band, we carry out time-resolved studies of the emission of the AgBr. In this case, the exciting beam is modulated by a mechanical modulator, with parameters which make it possible to produce pulses with a length of 23 μs and a duty factor of 1/3. A coincidence circuit made from CAMAC apparatus is then used to simultaneously record the slow component of the spectrum (for which the time constant of the decay is in the microsecond range) and the overall spectrum. Subtracting the slow component from the overall spectrum, we find the fast component.

EXPERIMENTAL RESULTS AND DISCUSSION

According to the concept of a biexciton pocket, the biexcitons appear in AgBr only when the electron-hole (e-h) system is heated. Figure 3 demonstrates the spectral composition of the photoluminescence of our samples over the interval 463–466 nm for various reservoir temperatures. At $T < 7$ K the spectrum contains bands for which the appearance mechanism was established in Refs. 6–8. These are In^{TO}, which is due to the recombination of an indirect free exciton and the simultaneous emission of a $TO(L)$ phonon; ES, which is due to the inelastic interaction of a free exciton and an exciton which is bound to Cd, accompanied by the emission of a $TO(L)$ phonon; and EX_2^{TO} and EX_3, which result from the recombination of excitons which are captured by centers of unknown nature. As the temperature is raised, the band $EM(\lambda_{\max} = 464.3 \text{ nm})$ appears in the spectra; the spectral position, shape, and temperature depen-

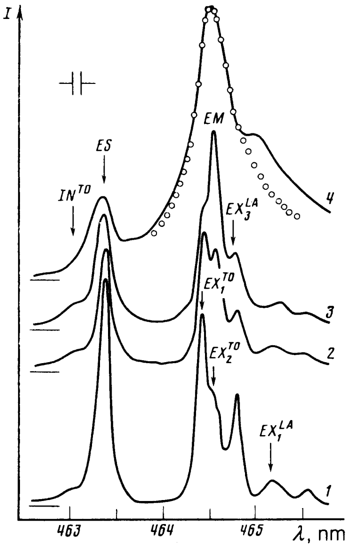

FIG. 3. Photoluminescence spectra of AgBr at various temperatures: 1—4.5 K; 2—7 K 3, 4—10K. Spectra 1, 2, and 3 were measured during excitation of the AgBr by a cw He-Cd laser with $I_p \approx 3 \cdot 10^{18}$ photon/(cm$^2\cdot$s), while spectrum 4 was measured during excitation with a pulses nitrogen laser with $I_p \approx 10^{20}$ photon/cm$^2\cdot$s). The points on spectrum 4 correspond to the spectrum of the emission band of indirect biexcitons according to calculations in Ref. 10 with a theoretical parameter $\Gamma = 1.4$.

dence of this band correspond to the emission (described in Refs. 2 and 9) of free biexcitons with a binding energy $\varepsilon_b \approx 7$ meV. In determining the latter quantity one should bear in mind that the biexciton states in AgBr decay by the following scheme: transition of one of the excitons into one of the valleys at an L point and recombination of the other exciton, accompanied by the simultaneous emission of the $TO(L)$ phonon which conserves the wave vector.[9] The spectra in Fig. 3 thus provide evidence for the presence of a lower limit on the temperature range in which biexcitons exist in our samples. Such a boundary is a necessary attribute of the biexciton pocket.

Figure 4 shows how the luminescence spectra of AgBr near the EM band change with the excitation intensity. The excitation intensity was initially increased smoothly up to the maximum level reached; then it was reduced to its original low value. We can clearly see a hysteresis effect, i.e., a dependence of the spectrum on the history of the excitation of the crystal. The intensity of the EM band at some I_p is seen to depend on how this value was reached. This dependence is shown explicitly in Fig. 5, which shows the ascending and descending branches of the illumination-intensity characteristics of the EM band according to measurements at $T=10$ K. The branches are positioned in such a way that they form a loop which is traced out in the counterclockwise direction. We will refer to a loop with this tracing direction as a "negative" loop, while one traced out in the opposite direction is "positive."

Let us work from the possibility that the electron-hole liquid is involved in the appearance of a biexciton pocket to attempt to explain the observed hysteresis loop on the illumination-intensity characteristic of the luminescence band of free biexcitons in AgBr. Generally speaking, the existence of hysteresis effects in an e-h system as it stratifies into two phases is by itself understandable, stemming—very generally speaking—from the existence of an energetically unfavorable interface between phases and the nucleation of a new phase. Consequently, the appearance of a hysteresis loop on the illumination-intensity characteristic of the emission band of the biexcitons coexisting with the electron-hole liquid, reflecting a change in the concentration of the electron-hole droplets during their nucleation and subsequent growth, is completely natural. What does cause some surprise is another observation: In similar experiments carried out with Ge, we observe a positive loop in the illumination-intensity characteristic, corresponding to the emission of a gas phase.[11]

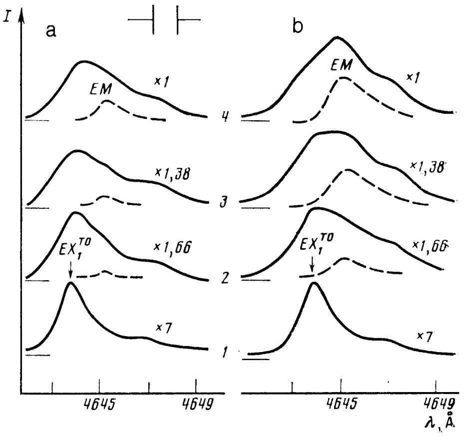

FIG. 4. Photoluminescence spectra of AgBr in the indirect-biexciton region. a: Measured as the excitation intensity I_p is increased smoothly. b: Measured as this intensity is reduced slowly. I_p: 2—$0.53I_0$; 3—$0.65I_0$; 4—$0.9I_0$, where $I_0 \approx 3\cdot 10^{18}$ photons/(cm^2·s), $T=10$ K. 1—Reference spectrum calculated from the spectra 2, 3, and 4 and recorded at a "low" excitation level ($I_p = 0.13I_0$), at which there is no emission of biexcitons. The dashed lines are the results of the subtraction of reference spectrum 1 from spectra 2, 3, and 4.

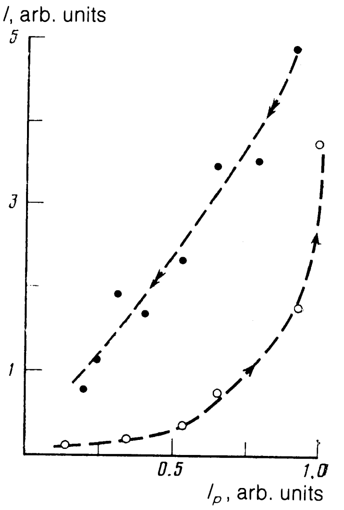

FIG. 5. Experimental illumination-intensity characteristic of the biexciton emission band as the intensity of the excitation of the AgBr is smoothly increased (open circles) and smoothly decreased (filled circles).

This is the situation, however, if the nucleation of the electron-hole liquid is an isothermal process, i.e., if the temperature of a droplet which has appeared remains constant over the entire lifetime fo the droplet. If the droplet instead warms up, the hysteresis loop for the gas phase may become negative. To demonstrate this point, we examine the change in the density of the gas as the radius of the droplet changes.

With AgBr in mind, we treat the e-h system as a mixture of ideal gases consisting of free carriers, excitons, biexcitons, and also droplets of an electron-hole liquid of radius R. This mixture is in equilibrium. In the steady state, the loss of matter from the electron-hole liquid due to evaporation and recombination is balanced by the flux of particles captured from the surrounding gas by the droplets. If we assume for simplicity that the average thermal velocities of the carriers, the excitons, and the biexcitons, are roughly the same, equal to v, we can write the density of the gas above a droplet as

$$n=\sum_i n_i(T)+Rn_0/3\tau_0 v, \qquad (1)$$

where n_0 is the equilibrium density of the electron-hole liquid, and the partial densities $n_i(T)$ are the partial densities of the particles of species $i (i=1, 2, 3)$ corresponding to thermodynamic equilibrium. From the condition for a chemical equilibrium we have

$$n_i(T)=g_i(m_i kT/2\pi\hbar^2)^{3/2}\exp(-\xi_i/kT), \qquad (2)$$

where m_i and g_i are respectively the effective mass of the state density and the statistical weight of the ground state for a particle of species i, and ξ_i is the work function for the escape of a particle of species i from a droplet of radius R. For excitons ($i=1$), biexcitons ($i=2$), and carriers ($i=3$) we have

$$\xi_1=\varphi-2\sigma/n_0R, \tag{3}$$

$$\xi_2=2\varphi-\varepsilon_b-4\sigma/n_0R, \tag{4}$$

$$\xi_3={}^1/_2(\varphi+\varepsilon_{ex})-\sigma/n_0R, \tag{5}$$

where σ is the surface-tension coefficient of the electron-hole liquid. It can be estimated from[12] $\sigma \approx 1/5(\varphi n_0)^{2/3}$ (for AgBr, $n_0 = 8\cdot 10^{18}$ cm^{-3}); ε_{ex} is the exciton binding energy (in AgBr, $\xi_{ex} \approx 28$ meV).

Expressions like (1) were first found by Pokrovskiĭ and Svistunova[13] and were analyzed in Refs. 13 and 14. On the $n(R)$ curve, shown schematically in Fig. 6, the only stable states are those on the right section (to the right of the minimum); the states on the left section of the curve are unstable. Because of the recombination loss, the minimum possible gas density above the droplet, n_m, is always higher than its thermodynamic-equilibrium value $n(T)$.

Let us use Fig. 6 to examine a simple model for the formation of a hysteresis loop in the density of the gas phase. We assume that the level to which the crystal is excited is raised extremely slowly; the gas density will of course also increase. As soon as the degree of supersaturation of the gas reaches a level sufficient for a significant condenstion rate, a nucleating region of electron-hole liquid appears with a critical size R_c, with the same temperature as that of the original phase, T_1. The state of the e-h system at this time is mapped by the point A on the unstable section of the $n(R)$ curve. As I_p is raised further, the radius of the droplet increases, and if its temperature does not change then the droplet will move to the stable section of the $n(R)$ curve. The state of the e-h system in this case is mapped by point B_1. If we now begin to reduce I_p, the droplet will move down the stable section to point C_1. As a result of this trajectory we find a positive hysteresis loop for the property n. If the droplet warms up in the course of the growth, on the other hand, its trajectory in the n, R plane will be different. Let us assume that the droplet temperature at the time at which the increase in I_p is stopped is $T_2 > T_1$. By this time the droplet will then be on the stable section of the $n(R)$ curve, and the state of the system will be mapped by point B_2. As I_p is reduced further, the radius of the droplet will decrease, and we will ultimately reach the state described by point C_2. Such a trajectory corresponds to a negative loop for the gas density. It follows from this analysis that the negative hysteresis loop which we observe on the illumination-intensity characteristic of the biexciton emission band in AgBr probably appears as a result of an increase in the temperature of the electron-hole droplets as they grow.

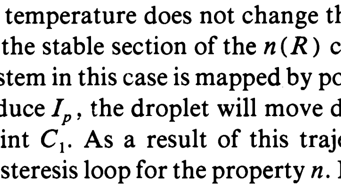

FIG. 6. Changes in the density of the gas above a droplet of the electron-hole liquid as the radius of the droplet changes at the temperatures (1) T_1 and (2) T_2 where $T_2 > T_1$.

We believe that the heating of the electron-hole droplets in AgBr stems from the highly nonequilibrium nature of the phase transition which gives rise to these droplets, and this heating is enhanced by the large condensation energy characteristic of the electron-hole liquid in this compound. Finally, in complete thermodynamic equilibrium, characteristic for systems with $\tau_0 \to \infty$, no heating of any sort can occur, since in this case the flux of particles which are going from the gas phase into the liquid phase is balanced by the returning flux due to evaporation. For an electron-hole liquid, such a equilibrium would be disrupted because of the additional loss of particles within a droplet. In the steady state, the particle flux into the droplets thus exceeds the evaporation by an amount which balances the recombination flux. As a result, additional energy is injected into a droplet, and its temperature should rise. The rise will understandably be more significant, the greater the value of φ and the smaller the value of τ_0.

The temperature of the electron-hole liquid can be estimated from the balance equations for the energy and matter going into and coming out of a droplet:

$$\begin{aligned}&4\pi R^2[\varphi n_1v_1+(2\varphi-\varepsilon_b)n_2v_2+(\varphi+\varepsilon_{ex})n_3v_3]\\&\quad=4\pi R^2[\varphi n_1(T)v_1+(2\varphi-\varepsilon_b)n_2(T)v_2\\&\qquad+(\varphi+\varepsilon_{ex})n_3(T)v_3]+P(T),\end{aligned} \tag{6}$$

$$\begin{aligned}&4\pi R^2\{(n_1v_1+n_2v_2+n_3v_3)-[n_1(T)v_1+n_2(T)v_2+n_3(T)v_3]\}\\&\quad=4\pi R^3n_0/3\tau_0,\end{aligned} \tag{7}$$

where $P(T)$ is the power which is removed from a droplet because of the emission of phonons. Under the conditions—which hold for the electron-hole liquid in AgBr—

$$\overset{*}{\mu}_{e,h}=\mu_{e,h}/T\gtrsim 1, \tag{8}$$

$$(8m_{e,h}s^2\overset{*}{\mu}_{e,h}/T)^{1/2}\ll 1, \tag{9}$$

where $\mu^*_{e,h}$ is the reduced chemical potential of the electrons, holes which have condensed, and s is the sound velocity [in AgBr at $T=0$ we would have $\mu^*_e = 895$ K, $\mu^*_h = 143$ K, $(8m_es^2)^{1/2} \approx 0.1$ K$^{1/2}$ and $(8m_hs^2)^{1/2} \approx 0.3$ K$^{1/2}$). For scattering by the strain potential, the function $P(T)$ is[15]

$$P_{e,h}(T) = \frac{8(T-T_0)T^{1/2}n_0m_{e,h}^{5/2}E_{e,h}^2}{2^{1/2}\pi\hbar^4\rho}\,\frac{F_1(\overset{*}{\mu}_{e,h})}{F_{1/2}(\mu_{e,h}^*)}. \tag{10}$$

Here $E_{e,h}$ is the strain-energy constant for the electrons, holes; ρ is the density of the crystal ($\rho = 6.48$ g/cm^3 for AgBr; T is the temperature of the electron-hole liquid; T_0 is the temperature of the reservoir; and F_1 and $F_{1/2}$ are the Fermi integrals of degrees 1 and 1/2.

Using the simple approximation

$$F_n(\mu^*)=(\mu^*)^{n+1}/(n+1) \tag{11}$$

for the Fermi integrals, which holds in the case of strong degeneracy,[16] and combining (6) and (7), using (10), we find the following expression for the temperature of the electron-hole liquid:

$$\begin{aligned}T=T_0+&\frac{2^{1/2}\pi\hbar^4\rho}{2n_0R(m_e^{5/2}E_e^2\mu_e^{1/2}+m_h^{5/2}E_h^2\mu_h^{1/2})}\\&\times\left\{\frac{Rn_0\varphi}{3\tau_0}+\varepsilon_{ex}v_3[n_3-n_3(T)]+(\varphi-\varepsilon_b)v_2[n_2-n_2(T)]\right\}.\end{aligned} \tag{12}$$

Let us use expression (12) to estimate the temperature of the electron-hole liquid in AgBr. Using only the second term, in which all of the parameters are more or less known, we find that the electron-hole liquid rises in temperature by about 40 K. Such a pronounced heating agrees with the experimental data found in Ref. 2.

Let us go back to the experimental hysteresis loop in Fig. 5. In going from point to point along both branches of this loop we spend a time $\sim 10^2$ s, while in recording the spectrum at each point we take $\sim 10^3$ s. The minimum time interval from the beginning of the recording of the last point on the ascending branch to the time of the recording of the initial point on the descending branch is therefore $\geqslant 10^3$ s. We see that the end of the ascending branch does not coincide with the beginning of the descending branch. This fact indicates that the transition of the system consisting of the gas and the electron-hole droplets to a metastable state with a higher temperature occurs over times which are macroscopic in comparison with the time scales for capture, evaporation, and recombination. To explain the appearance of such a slow dynamics we need to consider the behavior with changing I_p not of a single droplet but of the entire ensemble of droplets. A problem of this sort was solved in Ref. 17, where it was shown that the time required to reach an equilibrium state in such an ensemble as a result of redistribution of matter is astronomically long. In particular, estimates for Ge showed that at $T = 2$ K a lower limit on this time is $\sim 10^{19}$ s. Consequently, the macroscopically long time for the transition of the system to the new state manifested in our experiments seems entirely reasonable.

We conclude with a few comments regarding the emission by the electron-hole liquid itself in AgBr. According to Ref. 3, this emission corresponds to a fast component with a decay time $\sim 10^{-8}$ s, which can be distinguished from the overall spectrum in the region 470–500 nm. Figure 7 shows time-resolution emission spectral recorded in this region for our AgBr samples. We see a fast component in the spectrum, whose violet edge has an energy position which agrees precisely with the value of 55 meV which was found for the condensation energy in Ref. 2. There are apparently several sources contributing to this component, however. One such source, in addition to the electron-hole liquid, might be emission from biexcitons bound to an isoelectronic iodine impurity.[9] At the very least it is clear that a further study of the electron-hole liquid in AgBr will require improvements in the purification of the original material and in the techniques for growing the single crystals.

CONCLUSION

The hysteresis loop in the emission of biexcitons in AgBr, seen in these experiments for the first time, is a good argument in favor of the appearance of an electron-hole liquid. The direction in which this loop is traced out is evidence of a self-heating of the droplets of the *e-h* condensate as they grow. The most likely cause of this heating is the nonequilibrium nature of the phase transition. In addition, the short lifetime τ_0 in AgBr, which intensifies this deviation from equilibrium, results from the existence of a very broad channel for Auger recombination of the condensed carriers. The cross section for this process is determined by the fairly significant density of the electron-hole liquid.[8]

The possibility of nonisothermal formation of an electron-hole droplet has been discussed previously in the literature.[19,20] However, the effect of a finite τ_0 on this process has been totally ignored, and it has been assumed that the deviation from an isothermal situation stems from a net capture of free carriers within the electron-hole droplets. For Ge, with $\tau_0 \sim 10^{-6}$ s and $\xi_{e,h} \approx 3$ meV $> \varphi$, that approach seems to be justified, especially in a study of electron-hole droplets in a microwave field. For AgBr, in contrast, with $\xi_{e,h} \sim 40$ meV $< \varphi$, we can no longer ignore the evaporation of carriers; in this case the heating of the electron-hole droplets is caused primarily by the existence of an addition flux of gas-phase matter (excitons, biexcitons, and carriers). This flux is significant because of the relatively short lifetime of the droplets.

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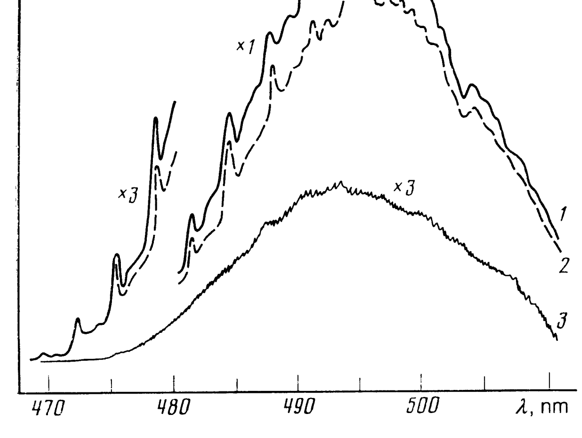

FIG. 7. Time-resolved photoluminescence spectra of AgBr. $I_p \approx 3 \cdot 10^{18}$ photon/(cm$^2 \cdot$s), $T = 10$ K. 1—Overall spectrum; 2—slow component of overall spectrum, with a time constant $\approx 25 \cdot 10^{-6}$ s; 3—fast component of overall spectrum, found by subtracting spectrum 2 from 1.

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