Lasing behaviour from the condensation of polaronic excitons in a ZnO nanowire*

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Atoms under optical and magnetic trapping in a limited space at a very low temperature can lead to Bose–Einstein condensation (BEC), even in a one-dimensional (1D) optical lattice. However, can the confinment of dense excitons in a 1D semiconductor microstructure easily reach the excitonic BEC? A lightly Mn(II)-doped ZnO nanowire under a femtosecond laser pulse pump at room temperature produces single-mode lasing from coherent bipolaronic excitons, which is much like a macroscopic quantum state due to the condensation of the bipoaronic excitons if not real BEC. In this process, longitudinal biphonon binding with the exciton plays an important role. We revisit this system and propose possibility of bipolaronic exciton condensation. More studies are needed for this condensation phenomenon in 1D microcavity systems.

Keywords: Bose–Einstein condensation, exciton, biphonon, ZnO nanowire

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1. Introduction

Bose–Einstein condensation (BEC) is a quantum phase transition from Bose gaseous state to Bose liquid; the latter, containing many Bose particles, behaves like one particle. Bose particles (ever since the issue of excitonic BEC was suggested by Keldysh and Kozlov[1]), excitons and their interactions with each other have been extensively studied.[2−9] The success of atomic BEC obtained by optical and magnetic trapping techniques in a limited space has given rise to the expectation of exciton BEC because excitons may condense at a slightly higher temperature. A great deal of time has been spent attempting to find the substance and structures of a possible BEC, like Cu_2O and the coupled multi-quantum wells (MQW) with numerous theoretical[2−4] and experimental[5−7] indications. In those reports a lot of difficulties, such as the strong repulsion caused by dipole-paralleled excitons standing in a two-dimensional (2D) layer,[8,9] exciton concentration decreases due to exciton expansion and migration, relaxation due to collision with carriers, exciton binding induced weakened superfluidity and the possibility of high density electron hole plasma (EHP), has caused much confusion. Until now, the reported BEC phenomena observed in a coupled MQW is under suspiction due to its evidence of superfluidity.[10] Another example is the bilayer electron system under strong magnetic field,[9] which is able to overcome the repulsion forces and to separate electrons at levels; the bilayer excitons in such a strong magnetic field also need to exhibit their 2D collective and coherent motion experimentally for BEC. Even more interestingly, the 1D BEC is theoretically predicted to be possible.[11] Although polariton, phonon and magnon BEC have been reported recently, exciton BEC study is still ongoing.

For an exciton system, the exciton concentration, stability, phase and spatial population are correlated to influence their high density behaviours, as well as the scattering process in their motions. All these factors can be reflected in the microstructures of special materials. Clean excitonic BEC may need a special exciton system in a microstructure, which can exclude the other state contributions of close energy and afford enough high stability excitons. With regard to the BEC phenomenon itself, we cannot avoid the behaviours of ultracold gases in traps. Although the mean-field theory predicts that BEC cannot occur in a 1D structure, recent results indicate its existence[11,12] and experimental realization in an

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optical lattice.[13−14] Exciton condensation in coupled QWs indicates 2D coherent exciton transport,[6,7] but exciton diffusion and expansion in a 2D plane still cannot be excluded due to their Coulomb repulsions. To avoid exciton diffusion and expansion it may be meaningful to try 1D exciton-confined structures, which satisfy the following conditions: 1) There is a limited 1D trap for excitons, just like atomic condensation in an optical lattice[13,14] or in an optical and magnetic trap with a spindle or T shape of several micrometers;[15] 2) They excite high-density excitons coherently and maintain a finite lifetime; 3) They stabilize high density excitons and queue them up in order. In contrast to exciton orientation in 2D structure, the excitonic dipoles may be in line in the 1D structure, which may favour their correlation and coherency.[8]

Firstly, in order to realize the BEC of excitons, highly stable excitons are needed to fight against thermal dissociation and collision, that is why BEC occurs at low temperatures. II–VI semiconductors with large exciton binding energy might be suitable, together with the easy production of high-density coherent excitons by the fs laser.[16] The only issue in such a system is the short lifetime of the excitons due to the dipole allowed transition rule. So this kind of exciton BEC in such a 1D system should be in a quasi-equilibrium state, which can give a single mode lasing, but the superfluidity is seemingly difficult to see.

Based on the above analysis, excitons in semiconductor 1D micro- or nano-structures may work as a chamber to realize exciton BEC. In this paper, a ZnO nanowire doped with minimal Mn(II) was prepared, its photoluminescence spectra by fs laser pulse excitation were investigated at room temperature and a single mode lasing behaviour like an excitonic BEC was observed at an intermediately high excitation.

Generally, the exciton in an intrinsic semiconductor cannot produce BEC due to scattering, diffusion and relaxation at even low temperatures. Improving exciton stability and correlation in a low-dimensional structure is one way to reach BEC. For example, the carriers in a low-dimensional semiconductor with enhanced with electron–phonon coupling and rich quantum behaviours (like EHP, Q-Hall effect etc.) are detected; layered cuprates produce high-T_c superconductivity. These examples indicate that the low-dimensional structures and tuned compositions might favour macroscopic quantum states in their electronic phase diagram. Moreover, excitons in transition metal ions-doped semiconductors have been reported to possess a long lifetime when it is excited by fs lasers under a magnetic field.[17] These results provide us with a new idea for exciton BEC if no excess carriers are involved.

2. Experimental

The Mn(II)-doped ZnO nanowires were synthesized via chemical reduction techniques. The detailed preparation process is described in Ref. [18]. The scanning electron microscope (SEM) morphology and high resolution transmission electron microscope (HRTEM) images of the samples were also reported in Ref. [18]. SEM images of teh ZnO nanowires indicated that the nanowire length was over 10 μm and the average diameter 500 nm with regular hexagonal cross sections. HRTEM observation indicated that all the nanowires grew along the [001] direction in a good crystallization and the *c*-axis growth direction was marked by an arrow. The chemical composition was also checked by a energy dispersive spectroscopy (EDS) detector attached in the TEM system, but no Mn was detectable. Only Zn and O elements could be detected, but about 0.6 ppm to 25 ppm Mn was detected by the inductively coupled plasma-mass spectrometry (Thermo element X7) for these samples.

The experimental setup for laser excitation and emission probe is shown schematically in Fig. 1. The photoluminescence (PL) was obtained by excitation of ∼fs pulsed UV line at room temperature. The laser excitation source, derived from a regenerative amplifier (Spitfire, Spectra Physics), was seeded by a mode-locked Ti-sapphire laser (Tsunami, Spectra Physics) delivered laser pulses at 800 nm (∼120 fs, 0.7 mJ, 1 kHz), which was frequency doubled by a BBO crystal to generate second harmonic line at 400 nm. It then passed through another BBO crystal, in which the mixed signal was generated at 266 nm. The highest output energy could be 10.5 μJ/pulse. A variable neutral-density filter was used in front of the first BBO to calibrate the output power. The PL data were collected by the microscope objectives and then focused into a polychromator. The detector was aligned to the longitudinal direction of nanowire. The PL decay was detected using a photon counting streak camera (Hamamatsu C2909), the time resolution was 20 ps and the spectral resolution of polychromator with 150 lines grating was 0.2 nm.

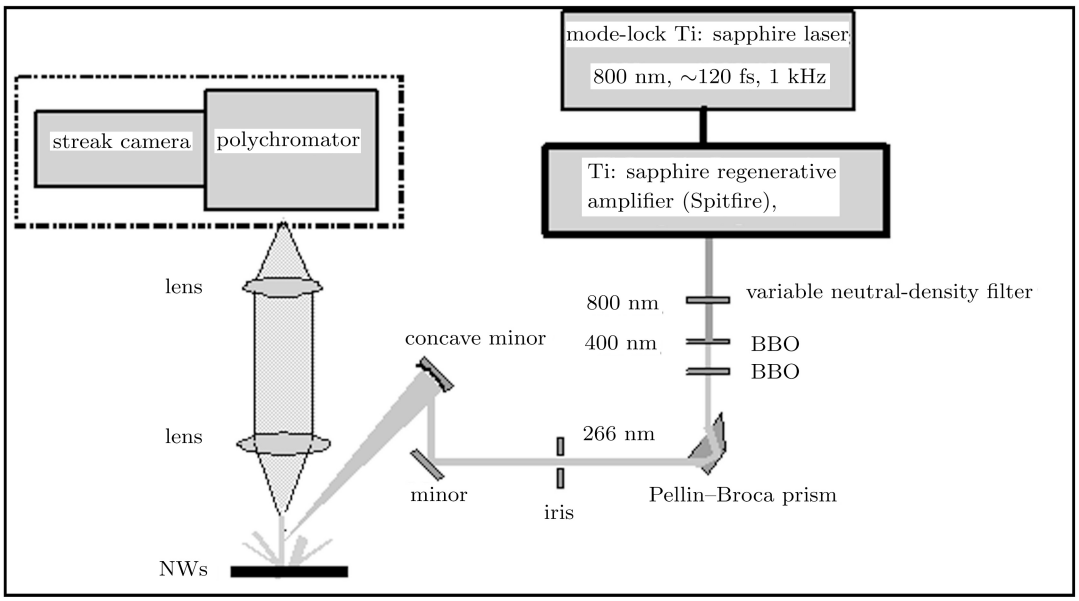
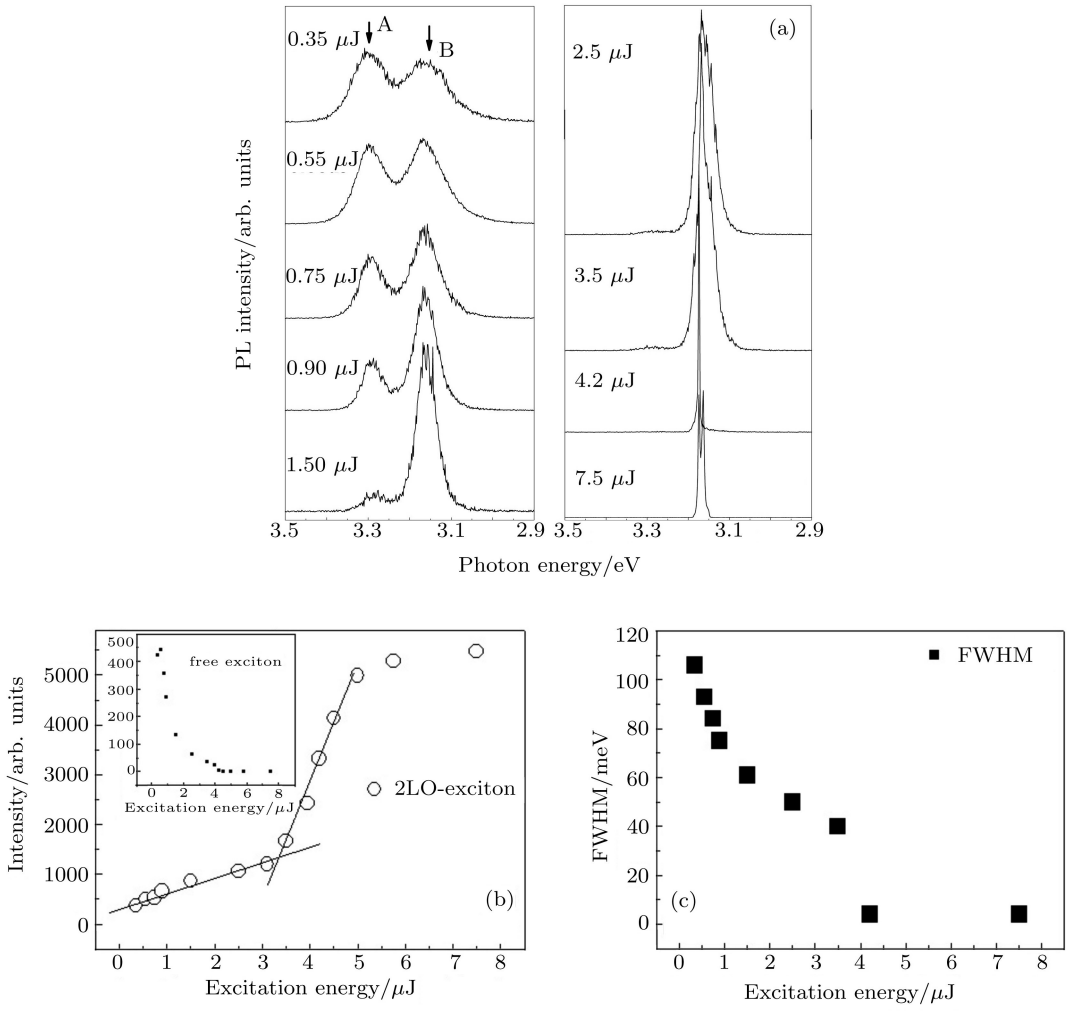


Fig. 1. The setup for PL and lasing experiments of the nanowires. The 800-nm beam is generated by a regeneratively amplified Ti-sapphire oscillator (120-fs pulse width, 1 kHz). A streak camera system is used to detect the PL and lasing signals generated from nanowires excited by the 266-nm fs pulse.



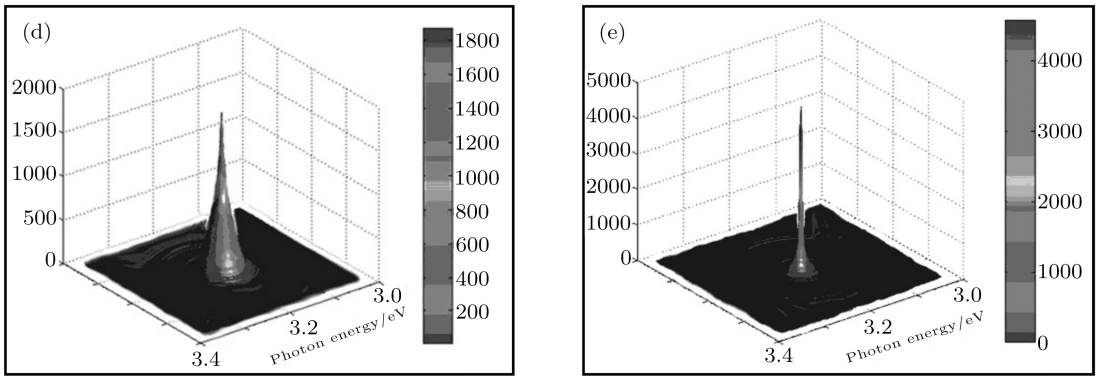


Fig. 2. (a) Photoluminescence spectra at various pumping energies of ZnO nanowires. b) The luminescence intensity versus laser excitation energy. c) The luminescence B band FWHM versus laser excitation energy. The energy profile of the luminescence line (d) at 3.5 μJ, and (e) at 4 μJ.

3. Results and discussion

The photoluminescence (PL) spectra of these doped ZnO nanowires under rising excitation powers are shown in Figs. 2(a)–2(c). In the low pump power region, two PL bands are observed for this nanowire, one from free exciton (arrowhead A) and another companion-band (arrowhead B), at 3.305 eV and 3.168 eV, respectively. The energy span between them is 137 meV. This span is surprisingly equal to the energy of two-phonon (the overtone of A1 LO mode), so the B band originates from polaronic excitons (bound excitons).[19] With rising excitation power, B band increases with a superlinear rate (Fig. 2(b)), but free exciton (A) band shows a slight increase at the starting stage of rising power, then decreases swiftly in the range of 0.3 μJ/pulse–3 μJ/pulse. Then the A band will diminish until pump fluence reaches 3.5 μJ/pulse, whenever B band increases with rising power (Fig. 2(b)). In the high power range, only the B band is left, with fast reduction of band full width at half maximum (FWHM) (Fig. 2(c)). Moreover, the whole PL curve becomes single mode stimulated emission line without any background (spontaneous radiation) after the excitation power reaches 3.5 μJ/pulse. The lasing line of B band is blue-shifted at about 50 cm^{-1} from its wavelength at low excitation, as shown in Fig. 3(a). The shift is slightly analogous to the phase space filling effects of excitons at high excitation[20] which represent an exciton repulsion but not a carrier effect. When the excitation pulse reaches about 4.0 μJ, the lasing band of nanowire becomes much narrower than that at low excitation. Only a super-narrow line of 3-meV FWHM exists in the emission spectra, which reflects a complete coherent emission. Further increase of the excitation power leads to no change of emission profile except intensity, but a second line occurs at its red-side with an energy span of 10.5 meV at 7.5 μJ. This splitting may come from the dipole–dipole interactions of excitons out of single mode coherence. In this entire excitation and emission process, the FWHM of the A and B bands become narrower with increasing excitation power (Fig. 3(c)), and finally the two emission bands converge to a line near B band (Fig. 2(c)). The single mode emission differs from previous lasing phenomena from bound excitons in ZnO nanostructures, but much like an emission of BEC excitons.[21]

For a heavier doped nanowire Mn ion (not more than hundreds ppm), we could observe the dominant B band with a very small shoulder of A band at low excitation. The emission behaviours of these samples at high excitation is similar to that of the nanowire (less doped) sample, and the threshold power to produce stimulated emission is slightly lower than that of the less doped nanowires. For over 200 ppm Mn-doped ZnO nanowire, we can observe strong lasing emission at about 386 nm in 28 μJ/cm^2 to 56 μJ/cm^2, then multimode lasing at 56 μJ/cm^2–76 μJ/cm^2, similar to that of Fabry–Perot cavity effect by the electron–hole plasma, which have been reported in Ref. [18].

The A band (free exciton) of the ZnO nanowire (at low Mn doping) decreases at low excitation and disappears at high excitation, which is related to the exciton couplings in this system. The large exciton binding energy (60 meV), scattering and strong electron–phonon coupling in ZnO are critical factors to free exciton absorption and the emission bands at room temperature. The free exciton (the A band) can be prevailing due to the high binding energy

(60 meV) under low excitations for pure crystal.[18] For enhanced exciton–phonon coupling in doped systems, the exciton–phonon replica may occur. The B band originates from the significant exciton–phonon coupling,[19] i.e., the two A_1 phonons couple with exciton (polaronic exciton), which will be discussed in the next section. Such a coupling state is a type of bound exciton, whose mass will increase due to the coupling with biphonons, and whose size will be close to 2 times the lattice cell, but still movable like a heavy exciton at room temperature. This coupled state can be induced by incorporating imperfections or impurities[19] to a definite amount, just like Mn ion doping in this nanowire.[18] Overdoped system may produce ingap carrier state and cause more scatterings.

This phonon–exciton complex possesses a higher binding energy than that of free excitons, i.e., the sum (197 meV) of 60 meV (free exciton binding energy) and 137 meV (two-phonon binding), hence this 2LO-exciton state becomes the lowest exciton level in this system (see Fig. 3), the large energy difference (137 meV) between 2LO-exciton and the free exciton indicates their independences at low density because of the sparse doping and weak coupling. So band A and band B could coexist for their high stabilities against thermal energy (26 meV) at room temperature.

The B band appears in ZnO nanowire upon the ion doping and enhanced exciton–phonon coupling, i.e., therein even higher stability and smaller mobility. If slightly more Mn ions are doped, B band will prevail instead of free exciton (A band). Even more Mn ions would lead to the disappearance of ultraviolet light emission at low excitation due to lots of trapped states within bandgap.[18] The emission spectra without one-phonon sideband reflect the forbiddance selection rule for one-phonon transition and allowance of two-phonon transition[22] and a significant nonlinear phonon–phonon coupling in 1D structure. The nonlinear coupling enhancement should be due to the 1D transport of A1 phonons along the axial direction. This strong nonlinear coupling would lead to 2LO phonon process, whose coupling to exciton, which facilitates the three- or four-particle process, is like that of two phonons with one exciton, or two phonons with two excitons, for all of their 1D boson nature. When the last free exciton is combined with the two-phonon propagating packet, the bipolaronic excitons suddenly stop and completely turn into a coherent state (BEC)[23] in a nanowire at about 4 $\mu J/cm^2$. Clearly the population reduction of free excitons with rising density is due to their binding with two-phonon, as proved by the A-band red-shifting and B-band dominating in Figs. 2(b) and 3, indicating an attraction force between free excitons. The fast enhancement and the narrowing of band B and its gradual and slight blueshift with rising density reflect the coherent two-phonon–two-exciton formation and condensation, because the excitons in paired or condensed state show repulsive forces. In this process the carrier screening effect due to the enhanced Coulomb potential and band-gap renormalization is not available. Only the axial phonon wave and exciton wave exist within the nanowire after photo-excitaion. Their coupling overcomes the repulsion of excitons and leads to the BEC state.[23] From Fig. 2(b) and Fig. 4 the abrupt disappearance of free excitons and the sudden shift stopping of bipolaronic exciton state is much like a phase transition from exciton gas to an exciton liquid at the critical concentration.

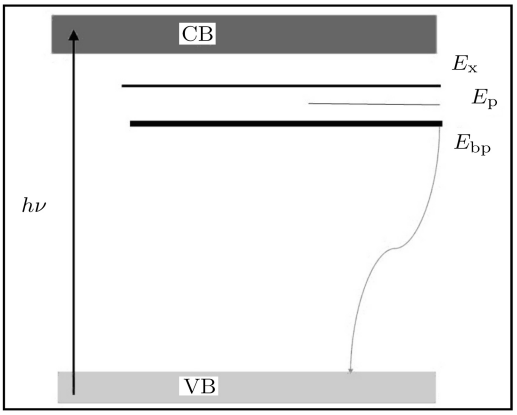


Fig. 3. The electronic states and optical processes of ZnO:Mn nanowires.

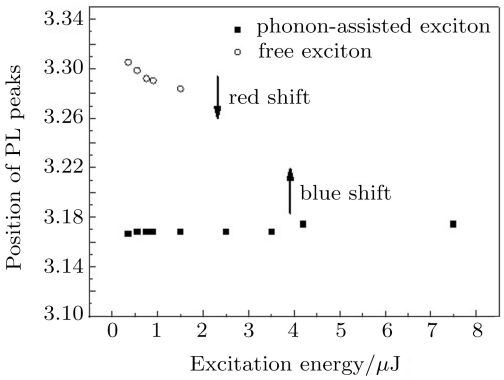


Fig. 4. The dependence of peak positions of the photoluminescence from free exciton and 2LO phonon-assisted complex on the excitation energy corresponding to the carrier density in nanowire.

There are another three reasons for the free excitons transforming efficiently to bipolaronic exciton (coherent B band) besides the stability. One is that the fs laser could produce coherent and high density excitonic states, which can coherently travel very long distances[21] in nanowire. The second is that the 1D transport and side crystal face reflection could confine and afford high density excitons in nanowire, which reduce or remove the diffusion or expansion effect. The third one is that aggregation of free excitons with different orientations is assisted and corrected by the longitudinal coherent 2LO A1 mode[8] at high density, which reduces the moving speed of the exciton and favours uniform orientation and coherency of excitons in the longitudinal direction. If 1D orientation is dominant, then head-to-tail connection between exciton dipoles leads to the reduction of exciton energy in their attraction interactions, which may be avoided by biphonon assistance. If the parallel orientation of the exciton is dominant, the exciton energy increases due to dipole repulsion of excitons. The data in Fig. 4 clearly indicate an attraction force (redshift) in the rising density of free excitons. The 1D population is dominant in the low excitation range. For the biphonon, coupled excitons aggregate at critical density and their repulsion interaction indicates an increasing parallel orientation. Finally, the high density bipolaronic excitons emit light coherently without other states (Figs. 3 and 4). This reflects a characteristic of a dynamic BEC state.

The BEC-like state does not occur for the intrinsic excitons but bipolaronic excitons, two A_1 phonons participate in the aggregation and condensation process, just like Cooper pairs in a superconductor. In Fig. 5 we plot the diagram of the exciton to bipolaronic exciton transformation and later aggregation or condensation with the pulse fluence, in which 1D biphonon chain (photoinduced) plays an important role in binding excitons together to form condensate with repulsed bipolaronic excitons. Clearly the precondition for BEC is that no ingap state from defects or carriers exists within the nanowire, otherwise the ingap state will increase the scattering effect and reduce the population in bipolaronic state.

The force between bipolaronic excitons at even high density (over 7.5 J/cm^2) is a little complicated with a minor splitting of coherent emission as indicated by the two lasing lines in Fig. 2(a), which may represent a nonlinear interaction between condensates after saturation. Lower than this power, only one mode lasing is observed, showing a clean condensate emission.

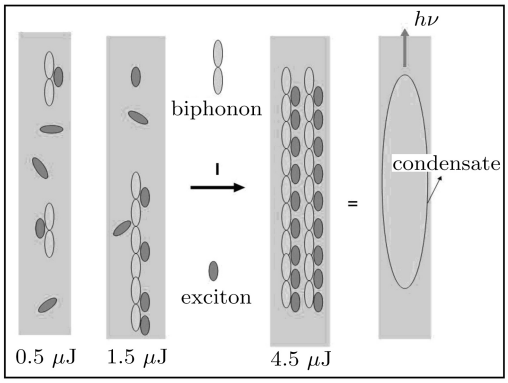


Fig. 5. A diagram of exciton and bipolaronic exciton formation and condensation process with rising laser fluence (each ellipse represents a dipole; the big one is for phonon, small one for exciton).

In Figs. 2(a) and 2(c) the B band exhibites a tendency to keep exciton coherency with rising excitation power. This differs from the lasing from an electron–hole plasma (EHP),[24–28] in which spectral band broadening and red-shift could be generally observed with single- and multi-mode lasing over a broad background.

This coherency of excitons occurs at room temperature. The reason lies in the contribution of 1D biphonons (two A1 modes), which trap excitons and let them queue in order. Moreover, both the phonon and the exciton are bosons, the binding of them leads to a stable composite boson with high order in 1D orientation. This composite boson can only move along c axis of ZnO nanowire at a much slower rate than that of free excitons. So they can find enough time and space to couple together.

In this system, the A and B bands coexist only at low excitation. Figure 6 shows the decay profiles of the A and B bands, respectively. Their lifetime data are listed in Table 1. The different PL decay profiles are also correlated with their different relaxation processes. The lifetime comparison at low power reflects slightly higher stability of B band as shown in Fig. 6. The free exciton emission decay shows a reduced lifetime when it is excited by increasing power (Table 1); moreover, the free exciton signal emission can hardly be detected at intermediate fluences. The ∼ps decay time corresponds to the free-exciton recombination rate in the bulk. This fact shows that no other quasi-particle and relaxation channel take part in the free exciton relaxation except for the inner conversion

before the critical excitation. The fast components in relaxation become dominant at high power. The lifetime reduction reflects the increasing rate of exciton coupling to biphonon and then being stabilized, or coherent conversion to bipolaronic excitons, but not a collective emission. The B band decay has a longer lifetime at low power, but its lifetime reduction with fluences goes much faster than that of A band. The driving force is the nonlinear coupling of biphonon with free excitons, later 1D packing and therein coherent emission. This emission has a linear dependence on the laser fluence, which is in contrast to the superradiance[29] in ZnO nanowire for their different nature.

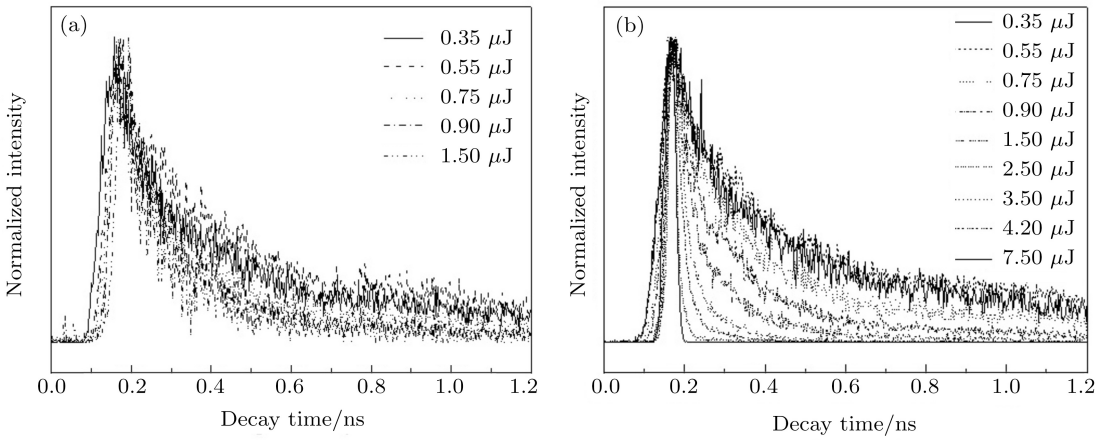


Fig. 6. Decay time of photoluminescence with various excitation energies: (a) free exciton and (b) the complex with 2LO phonon.

It is well known that Mn-ion-doped ZnO has ferromagnetic properties[30,31] as the hole doping level or Mn ion partial oxidation is above a critical value. The PL emission of bound exciton by Mn doped ZnO was reported to be around 370 nm–420 nm.[32−34] These reports indicate that doping impurities might lead to a significant change of the electronic structures and properties of ZnO,[32,35−37] which includes electron–phonon coupling enhancement, p–d hybridization and magnetic correlation. In fact, these properties could be obtained in heavy-doped systems in which the doped carriers play an important role in their properties. But the variable electronic structure in a lightly doped ZnO nanostructure has seldom been studied in detail, especially for the dimension effect. In a 1D system the biphonon coupled exciton dominates the excitonic behaviours, the multi-phonon mode may involve Mn ions. Despite the low doping in this case, the photoinduced magnetic or spin coupling between Mn ions may be positive to help to bind these excitons together to form condensate if the spins couple with excitons in 1D orientation.[30,31] Magnon related BEC has been recently reported in a rare earth metal oxide,[38] which indicates the importance of 1D correlation and spin coupling. The decay of an exciton in a cavity has also been simulated, which demonstrates that the excitons decay into a very narrow mode when exciton–cavity coupling becomes strong enough.[39]

Table 1. Decay time for the two bands under different excitation energies.

	Decay time of free exciton	Decay time of 2LO-assisited excition
0.35 μJ	t_1 = 133 ps, 67%	t_1 = 155 ps, 63%
	t_2 = 898 ps, 33%	t_2 = 1.36 ns, 37%
0.55 μJ	t_1 = 124 ps, 69%	t_1 = 128 ps, 67%
	t_2 = 756 ps, 31%	t_2 = 797 ps, 34%
0.75 μJ	t_1 = 110 ps, 68%	t_1 = 103 ps, 66%
	t_2 = 706 ps, 32%	t_2 = 731 ps, 33%
0.9 μJ	t_1 = 80 ps, 74%	t_1 = 35 ps, 69%
	t_2 = 318 ps, 26%	t_2 = 179 ps, 31%
1.5 μJ	t_1 = 32 ps, 67%	t_1 = 31 ps, 74%
	t_2 = 192 ps, 33%	t_2 = 142 ps, 26%
2.5 μJ	unable to be detected	t_1 = 26 ps, 89%
		t_2 = 108 ps, 11%

The above single mode lasing emissions at high excitation density occur at fs pulse pumping, which do not happen under the excitation of ns and ps laser

pulses. The latter is caused by the photothermal effect, which may dissociate the exciton and produce a broad emission band. Leung *et al.*[40] studied the time-resolved stimulated emission in ZnO and observed the formation time of exciton at about 5 ps, during which exciton-scattering happened. In fs excitation, the exciton behaves as itself. When the fs laser pulse excites the nanowire in ∼120 fs, at least $\sim 10^{19}$ excitons/pulse could be created according to the photon scaling if the various losses are subtracted. For a nanowire of 10 μm in length and 500 nm thick, the number of excitons in a 1D wire could reach about 10^4, while their 3D packing could afford 10^6 excitons. The strong fs laser pulse can coherently produce enough excitons in the nanowire for condensation. Due to the limitation of the experimental condition we cannot detect the exact dynamic times for exciton generation, condensates formation and relaxation directly. The condensate might not possess a long lifetime due to its allowed transition rule and fast propagation within the nanowire. If excitons condense, it is hard to observe their superfluidity in the nanowire.

It is supposed that a critical size of nanowire may be suitable for exciton BEC. Firstly, there is a critical density of excitons at quasi-equilibrium, so a limited volume in nanowire can avoid the diffusion and expansion of excitons, and produce a standing wave of exciton propagation, hence act as a 1D exciton trap. Secondly, the exciton binding energy should overcome the thermal effect. Thirdly, propagating phonons will couple with excitons in a quasi-one-dimensional structure, reduce the exciton moving speed and fix the 1D orientation of coupled excitons. Such a 1D semiconductor structure is able to supply a confined space for excitons to move only along the 1D axis coherently. Then the fs pulse can produce coherent and dense excitons in it. For atomic condensation at an optical lattice,[13,14] a limited number of atoms condenses in a space like this limited 1D trap. Hence this nanowire could limit the expansion of excitons in two directions and facilitate the combination of the 2LO phonon mode[41] with excitons,[22] and further to condensate.

The fs pulse is another advantage in producing coherent excitons and realising temporal Bose condensation. In the 120-fs excitation duration and thereafter relaxation, not many excitons could dephase due to expansion or collision and other non-radiative pathways before condensation. A large number of excitons are created in a short time to reach the critical density for exciton condensation. The other advantage is that fs pulses do almost no to damage to the sample because of the short interaction time; so the lattice temperature would not increase significantly and swiftly. The high lattice temperature usually is the origin of exciton dissociation and decoherence. For exciton–phonon coupling in a 1D structure, the exciton phase diagram, and especially the behaviours of magnetic exciton, is not yet clear.

4. Conclusion

We have synthesized a ZnO nanowire doped with Mn ions in ppm concentration. When it is resonantly excited by a fs laser pulse, the gradual transformation of free exciton to bipolaronic exciton is observed in the low fluence range; a sudden jump into the bipolaronic state occurs at the intermediate fluence region, which gives rise to a coherent emission line like a macroscopic quantum state. From the consideration of the excitonic interactions, exciton density, exciton–phonon coupling, bipolaron exciton condensate in 1D structure, we find a dynamic Bose condensate of excitons realized in this nanowire via a BCS-type interaction.

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