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Γ_7 valence band symmetry related hole fine splitting of bound excitons in ZnO observed in magneto-optical studies

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The symmetry ordering of the valence bands in ZnO is derived from high-resolution magneto-optical measurements of bound excitons. We report on the experimental observation of a hole state related fine splitting for bound excitons in the Voigt configuration. This splitting is related to a nonzero Landé g value $g_\perp$ for hole states from the A valence band. Based on theoretical considerations, the symmetry of the uppermost valence band is doubtlessly identified as $\mathbf{\Gamma_7}$. This attribution is confirmed by polarization and angular resolved magnetophotoluminescence spectroscopy.

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The symmetry ordering of the valence bands in wurtzite ZnO has been subject to extensive discussions[1–7] since the first investigations of the valence band maximum fine structure by Thomas in 1960.[1] The upper valence bands of ZnO are composed of the $2p^6$ states of the oxygen atoms in the ZnO lattice. The crystal field and spin-orbit interaction lead to a reduction in the degeneracy of the p-like states at the Γ point which result in a splitting of the valence band into three subbands. These bands are labeled as A, B, and C from the highest energy to the lowest one. Numerous reports have emerged over the past 50 years postulating either Γ_7 symmetry[1,4,6–22] or Γ_9 symmetry[2,3,5,23–28] for the uppermost A valence band. Based on polarization-dependent reflection and absorption spectra, Thomas came to the conclusion that the symmetry of the valence bands from the A, B, and C band is Γ_7, Γ_9, and Γ_7 rather than Γ_9, Γ_7, and Γ_7 and therefore anomalous compared to the usual ordering in other wurtzite II-VI materials such as ZnSe and CdS.[1] Theoretical calculations explained the reversed order of the top valence bands by a negative spin-orbit splitting[29] which originates from the contribution of Zn $3d$ bands.[30] The impact of these $3d$ bands, including variations in their energetic positions on the size of the spin-orbit splitting has been investigated by Lambrecht *et al.*[6] These calculations led to the conclusion that a negative spin-orbit splitting and consequently Γ_7 symmetry for the A valence band is mandatory. Furthermore, previous experimental results by Reynolds *et al.*[5] assuming Γ_9 symmetry could also be theoretically explained with Γ_7 symmetry for the A valence band. This interpretation was supported by an experimental investigation of the angular-dependent Zeeman splitting of the neutral bound excitons I_4 and I_9, and the ionized bound excitons I_2/I_3 by Rodina *et al.*[7] Nevertheless, a variety of new publications has emerged in recent years which assert Γ_9 symmetry for the top valence band in ZnO (Refs. 26–28) or avoid a clear statement concerning the valence band ordering in ZnO.[31–34]

In the present work, we present comprehensive magneto-optical data of the commonly observed bound exciton transitions I_{6a}, I_7, and I_8 in high quality lithium doped ZnO grown by chemical-vapor deposition (CVD) on Tokyo Denpa substrates and on the I_4 and I_8 lines in nominally undoped ZnO substrate from EaglePicher grown by the seeded chemical vapor transport technique. We report on the experimental observation of a nonzero hole g value in $\boldsymbol{B} \perp \boldsymbol{c}$ which results in an additional fine splitting of narrow bound exciton transitions. The experimental data including polarization- and angular-dependent measurements are analyzed for bound exciton level schemes assuming Γ_7 and Γ_9 valence band symmetry. Possible arguments objecting to the Γ_7 symmetry of the A valence band are extensively discussed and confuted. In addition to the existing arguments in the literature, the newly observed hole fine splitting for $\boldsymbol{B} \perp \boldsymbol{c}$ and polarization patterns in this contribution provide conclusive evidence for the Γ_7 symmetry of the A valence band.

The magnetophotoluminescence measurements were performed in a split-coil cryostat at a temperature of 2 K. The luminescence was excited by the 325 nm line of a HeCd laser with an output power of 40 mW. The emitted light was dispersed by a 1 m double monochromator in fourth order and detected by a bi-alkali photomultiplier tube. The spectral resolution of the setup was approximately 50 μeV. The PL measurements in the external magnetic field were performed

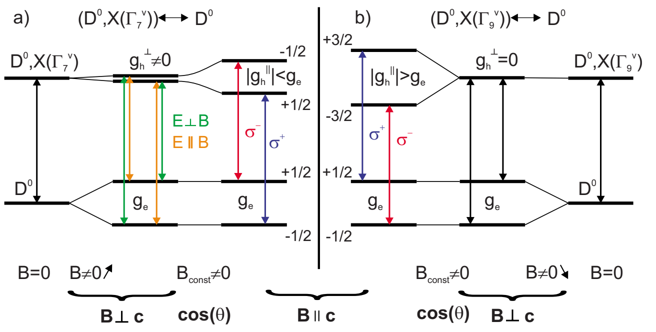

FIG. 1. (Color online) Level scheme and splitting of donor bound exciton states under the influence of external magnetic fields. Holes originating from valence bands with different symmetry representations lead to variations in the hole g values as expressed by the size of the Zeeman splitting.

in Faraday configuration, Voigt configuration, and for variable angles between the direction of the magnetic field $\boldsymbol{B}$ and the crystal $\boldsymbol{c}$ axis. The circular polarization of the detected light was analyzed using a $\lambda/4$ plate and a linear crystal polarizer.

Figure 1 displays the energy-level scheme of donor bound excitons involving hole states from the Γ_7 [Fig. 1(a)] and Γ_9 [Fig. 1(b)] valence bands under the influence of an external magnetic field. The excited state in this level scheme is represented by an exciton bound to a neutral donor, whereas the ground state is characterized by the donor level without the exciton. In the case of a constant magnetic field, the Zeeman splitting of the ground state does not change as function of the orientation of the magnetic field $\boldsymbol{B}$ to the $\boldsymbol{c}$ axis as determined by the (in first approximation) isotropic electron g value g_e. In contrast, the hole g value g_h, which determines the size of the Zeeman splitting in the excited state of a donor bound exciton complex, is anisotropic due to the discrimination of the expectation values of the orbital angular momenta for the p_x/p_y and p_z like states. For acceptor bound excitons, the ground states and excited states in Fig. 1 have to be reversed.[7]

Let us now consider the influence of the valence band symmetry on the Zeeman splitting of bound excitons in ZnO. In the Voigt configuration ($\boldsymbol{B}\perp\boldsymbol{c}$) the hole g value is zero $g_h^{\perp}=0$ in case of an exciton involving a Γ_9 hole state and small but nonzero for a hole originating from a Γ_7 valence band. Consequently, the magnetic field does not only lift the degeneracy of the ground state of the donor bound exciton complex in the case of Γ_7 symmetry, but also results in a small splitting of the excited state [Fig. 1(a)]. The additional splitting of the excited state engenders a fourfold splitting of the bound exciton recombination line due to the nonzero g values of electron and hole in $\boldsymbol{B}\perp\boldsymbol{c}$. This is not the case if the exciton hole corresponds to a state with Γ_9 symmetry [Fig. 1(b)] since no splitting of the excited state exists ($g_h^{\perp}=0$). Therefore, only a twofold splitting determined by the electron g-value g_e occurs.

In order to determine the symmetry of the hole states involved in the bound exciton complexes in ZnO, the photoluminescence (PL) was studied under the influence of an external magnetic field. Figure 2 shows the magneto-PL spectra of the dominant lines I_{6a}, I_7, and I_8 in Faraday configuration (a) for various angles θ between the directions of $\boldsymbol{B}$ and $\boldsymbol{c}$ (b) and in the Voigt configuration (c). The zero-field PL spectrum of the sample shows very narrow bound exciton lines with a full width at half maximum (FWHM) of $\Gamma \leq 80$ μeV. Thus, the different Zeeman components of the transition lines can already be resolved at low magnetic fields of $B=1$ T. Similar exciton spectra with narrow half widths of the I_{6a} and I_8 bound excitons were also obtained from epilayers grown on Zn polar CrysTec substrates.[35,36]

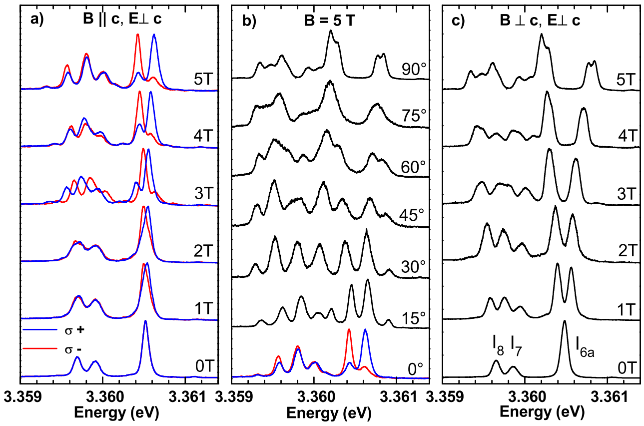

FIG. 2. (Color online) Photoluminescence spectra of the I_{6a}, I_7, and I_8 bound exciton lines at 2 K for different magnetic fields, orientations and angles. (a) Faraday configuration ($\boldsymbol{B}\|\boldsymbol{c}\|\boldsymbol{k}$), (b) angles $\theta=0$, 15, 30, 45, 60, 75, 90 between $\boldsymbol{B}$ and $\boldsymbol{c}$ with $B=5$ T, and (c) Voigt configuration ($\boldsymbol{B}\perp\boldsymbol{c}\|\boldsymbol{k}$). The dark (blue) and light (red) lines (a) indicate the right σ^{+} and left σ^{-} polarized light, respectively.

First, we investigate the splitting behavior in $\boldsymbol{B}\perp\boldsymbol{c}$ in order to determine the charge state of the impurities. All lines exhibit a linear splitting which is expected for neutral bound excitons. Consequently, the bound exciton transitions I_4, I_{6a}, I_7, and I_8 originate from neutral bound excitons which is well established in the literature.[2,11,15,37,38] By contrast, the splitting of ionized impurity bound excitons does not scale linearly with the magnetic field due to a zero-field exchange splitting which leads to a mixing of states and the appearance of previously forbidden transitions.[39,40] Such behavior has been observed in ZnO for the I_0 to I_2/I_3 bound excitons.[7,38,41]

Figure 3 displays the energetic position of the bound exciton lines as function of the magnetic field B and angle θ. Since the bound excitons transitions I_4, I_{6a}, I_7, and I_8 exhibit a comparable Zeeman splitting in all configurations, only the I_{6a} line is shown for clarity. In addition, the I_{6a} exhibits the highest intensity, smallest FWHM, and a sufficient spectral separation to the I_7 and I_8 bound exciton lines so that the evaluation of its shifting and splitting behavior in a magnetic field is not affected by the overlapping of adjacent lines in close energetic proximity. With increasing magnetic field strength in $\boldsymbol{B}\perp\boldsymbol{c}$, a fourfold splitting of the I_{6a} bound exciton can be observed [Figs. 2(c) and 3(c)]. This splitting can only be explained if not only the donor ground state splits in a magnetic field with the electron g factor g_e but also requires a splitting of the excited state (Fig. 1). Since the splitting of the excited state of a neutral donor bound exciton is deter-

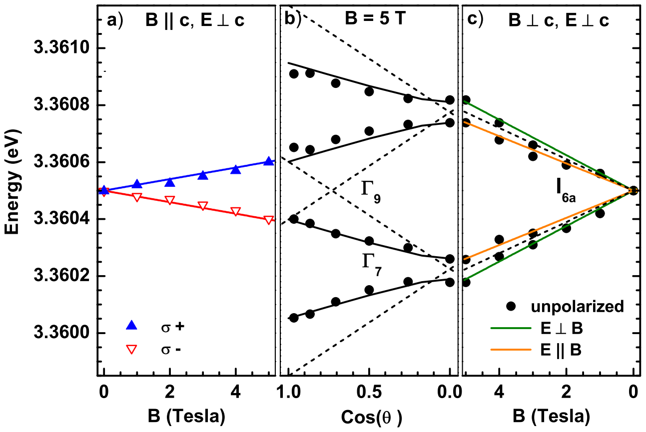

FIG. 3. (Color online) Zeeman splitting of the I_{6a} bound exciton line at 2 K. (a) Faraday configuration ($\boldsymbol{B}\|\boldsymbol{c}\|\boldsymbol{k}$), (b) various angles θ between $\boldsymbol{B}$ and $\boldsymbol{c}$ with $B=5$ T, and (c) Voigt configuration ($\boldsymbol{B}\perp\boldsymbol{c}\|\boldsymbol{k}$). Solid (blue) and open (red) triangles (a) indicate the peak position for σ^+ and σ^- polarized light, respectively. Solid black dots show the peak energies in the unpolarized measurements. Outer (green) and inner (orange) lines (c) represent theoretical fits for $\boldsymbol{E}\perp\boldsymbol{B}$ and $\boldsymbol{E}\|\boldsymbol{B}$, respectively.

mined by the hole g value $g_h^{\perp}$ in $\boldsymbol{B}\perp\boldsymbol{c}$, the experimental data requires a nonzero hole g-value $g_h^{\perp}\neq 0$. Applying group theoretical considerations, one can easily determine for which valence band symmetries zero or nonzero g factor are expected. A magnetic field leads to a splitting of a certain state if the group representation of the state (Γ_i) or those of the magnetic field (Γ_B) is contained in their Kronecker product:

$$g \neq 0 \Leftrightarrow (\Gamma_B \vee \Gamma_i) \in \Gamma_i \otimes \Gamma_B. \tag{1}$$

The group representation of a magnetic field perpendicular to the $\boldsymbol{c}$ axis $(\Gamma_{B\perp})$ is Γ_5, a magnetic field parallel to the $\boldsymbol{c}$ axis $(\Gamma_{B\|})$ transforms like Γ_2. For a perpendicular magnetic field, we therefore derive the following Kronecker products for hole states with Γ_7 and Γ_9 symmetries from Eq. (1) according to Koster *et al.*:[42]

$$\Gamma_7 \otimes \Gamma_5 = \Gamma_7 + \Gamma_9 \Leftrightarrow g_h^{\perp}(\Gamma_7) \neq 0, \tag{2}$$

$$\Gamma_9 \otimes \Gamma_5 = \Gamma_7 + \Gamma_8 \Leftrightarrow g_h^{\perp}(\Gamma_9) = 0. \tag{3}$$

Hence, the observed nonzero hole g value $g_h^{\perp}$ can only be explained for a hole originating from the Γ_7 valence band. Consequently, the topmost valence band in the investigates samples must have Γ_7 symmetry, which is in agreement with the originally proposed valence band ordering of Γ_7, Γ_9, and Γ_7.[1,8] However, the observation of the $g_h^{\perp}$ induced fine splitting may often be hindered by a larger FWHM of the excitonic emission lines or an insufficient spectral resolution at typical magnetic field strengths. In the case of GaN, magnetoreflection spectra up to 27 T revealed a fourfold splitting of the B and C exciton, whereas the A exciton only split into two Zeeman components.[43] Since the valence band ordering of the upper two bands in GaN (Γ_9, Γ_7, and Γ_7) is reversed compared to ZnO (Γ_7, Γ_9, and Γ_7), the observed fourfold splitting of the bound excitons in ZnO involving hole states from the A valence band is in perfect agreement with the splitting of the B and C excitons with Γ_7 valence band symmetry in GaN.

Additional evidence for the Γ_7, Γ_9, Γ_7 valence band ordering in ZnO is given by the angular resolved spectra in Fig. 2(b) and their respective peak positions in Fig. 3(b). The solid lines in Fig. 3 represent fits for Γ_7 hole states, whereas the dashed lines are related to Γ_9 states. Since the hole g value for a Γ_9 state in $B\|c$ would be larger than the electron g value g_e,[6,7] a crossing of the inner Zeeman components in Fig. 3(b) should occur for a Γ_9 hole. This behavior has been observed for neutral bound excitons in other wurtzite semiconductors such as, e.g., CdS,[40,44] where the A valence band has Γ_9 symmetry. Evidently, this is not the case in the here presented data which is in agreement with previously obtained results for the bound excitons I_4 and I_9 by Rodina *et al.*[7] From the fits in Fig. 3, we derived parameters of $g_e=1.9$, $g_h^{\|}=-1.2$, and $g_h^{\perp}=0.25$ for the electron and hole g values. Previously, experimental values of $g_h^{\perp}$ around 0.1 (Refs. 7 and 22) as well as $g_h^{\perp}=0.3$ (Ref. 18) were reported for the Γ_7 hole in ZnO. Theoretical calculations within the quasicubic approximation[45] with the effective mass parameters of ZnO obtained from first-principles calculations[6] predict the values $g_h^{\|}=-1.23$ and $g_h^{\perp}=0.14$ for the upper Γ_7 hole in bound excitons. These values are in good agreement with our experimental results.

So far, we have discussed the fourfold splitting in the $\boldsymbol{B}\perp\boldsymbol{c}$ configuration without considering the polarization of these transitions. Apart from the circular polarized spectra in the $\boldsymbol{B}\|\boldsymbol{c}$ orientation, the spectra and peak position in the Figs. 2(b), 2(c), 3(b), and 3(c) originate from unpolarized measurements. However, another important theoretical prediction in the framework of the quasicubical model can be established concerning the polarization of the Zeeman components in the Voigt configuration. Using the Γ_7 basis functions and Zeeman terms described in Refs. 6 and 45, one can predict a linear polarization of the inner transitions with $\boldsymbol{E}\|\boldsymbol{B}$ and of the outer transitions with $\boldsymbol{E}\perp\boldsymbol{B}$ in the geometry $\boldsymbol{B}\perp\boldsymbol{k}\|c$ as indicated by the orange and green lines in Fig. 3(c). This theoretical prediction is experimentally observed for the I_4 and I_8 lines in the nominally undoped ZnO substrates from EaglePicher (Fig. 4). For clarity, only the energy regime of the I_4 bound exciton is displayed. Apparently, the inner Zeeman components are linear polarized with $\boldsymbol{E}\|\boldsymbol{B}$, whereas the outer components show an increased oscillator strength in the $\boldsymbol{E}\perp\boldsymbol{B}$ polarization. These experimental data are in excellent agreement with the theoretical considerations and provide an additional strong evidence for the Γ_7 symmetry of the hole. Note that for an opposite sign of $g_h^{\perp}$, the $\boldsymbol{E}\|\boldsymbol{B}$ polarization would be observed for the outer Zeeman components instead of the inner ones.

Possible effects and counter-arguments which might contradict our interpretation of the A valence band possessing Γ_7 symmetry shall now be discussed and excluded.

(i) The observed bound excitons I_4 and I_{6a} may result from a B valence band and therefore shows the behavior of a Γ_7 hole state: This explanation can be excluded since no I_4 and I_{6a} related exciton transitions are observed at lower energies with a typical A-B energy spacing of 4.9 meV.[37] Furthermore, the I_4 and I_{6a} bound excitons are well established in the literature and are also the dominant excitonic recom-

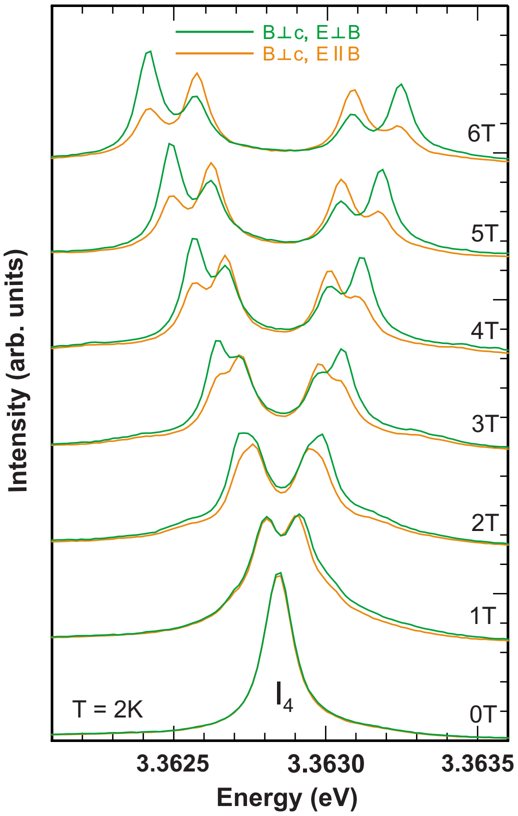

FIG. 4. (Color online) Photoluminescence spectra of the I_4 bound exciton line at 2 K for magnetic fields of 0–6 T recorded in the Voigt configuration ($\boldsymbol{B}\perp\boldsymbol{c}$). Dark (green) and light (orange) lines represent linear polarized measurements in the $\boldsymbol{E}\perp\boldsymbol{B}$ and $\boldsymbol{E}\parallel\boldsymbol{B}$ orientation, respectively.

binations in these samples which should involve holes from the topmost valence band.

(ii) The valence band ordering in the investigated samples might be altered due to the presence of large strain fields: it was pointed out by Gil *et al.*[46–48] and Shan *et al.*[49] that the valence band ordering in GaN and ZnO is rather sensitive to strain which can lead to a reversal of the usual symmetry ordering. However, this is not the case in the present study since no significant strain induced shift of the nonpolar E_2(high) Raman mode was observed from its relaxed value of 437 cm^{-1}.[50] The absence of a strain induced energy shift also applies for the energetic positions of the free and bound excitons in the PL spectra which deviate by less than 0.15 meV from the values provided by Meyer *et al.*[37] In addition, *ab initio* calculations by Schleife *et al.* revealed that only the crystal field determined A-C splitting is strongly affected by biaxial strain which would, even in the case of a A-C valence band crossing for large strain fields, lead to a top valence band with Γ_7 symmetry.[51]

(iii) The additional splitting in the Voigt configuration could also appear in the case of a Γ_9 hole state if an anisotropic in-plane stress perpendicular to the c axis is present in the sample. This stress would not change the ordering of the valence subbands but result in the admixture of the Γ_7 character to the Γ_9 hole. Therefore, a nonzero perpendicular hole g factor could appear even for a Γ_9 hole. For example, it was shown that the simultaneous application of strong in-plane stress and uniaxial stress along the c axis may even lead to a nearly isotropic g-factor tensor of the Γ_9 hole.[52,53] However, the presence of uniaxial in-plane stress would lead to a reduction in the crystal lattice symmetry, which is described by the point group C_{6v} in the unstrained case and C_{2v} in the case of uniaxial stress perpendicular to the $\boldsymbol{c}$ axis. Therefore, a splitting of the zero-field exciton lines should appear which was observed by Langer *et al.*[54,55] and Wrzesinski *et al.*[56] under applied uniaxial pressure $\boldsymbol{P}\perp\boldsymbol{c}$. Without external pressure or magnetic fields, Reynolds *et al.*[57] observed a fourfold splitting for ZnO grown from the gas phase using ZnSe as starting material but no splitting when ZnS was used. They explained the splitting by the combined effect of spin-exchange and local strain due to the presence of residual selenium in the ZnO lattice based on the theoretical description by Akimoto and Hasegawa.[58] The splitting was modified in an magnetic field applied perpendicular to the c axis due to the additional Zeeman splitting of the electrons. The observed fourfold splitting was significantly different for two different donor centers which additionally indicated the influence of the local potential. By contrast, no indications of uniaxial in-plane stress such as line splittings or line-shape broadenings in zero-field are observed in the present study although the spectral resolution was sufficient to resolve these effects. In addition, no residual Se contamination leading to large local strain fields is expected in the here investigated ZnO epilayers and substrates. Consequently, anisotropic uniaxial in-plane stress can be excluded as possible explanation for the nonzero hole g-value $g_h^{\perp}\neq 0$.

(iv) The crystal $\boldsymbol{c}$ axis might be improperly aligned to the direction of the $\boldsymbol{B}$ field which would lead to a deviation from the Voigt geometry and allow the appearance of two additional lines due to the angular dependence of the hole g value (Fig. 1): two possible reasons for such a misalignment are conceivable. First, the orientation of the turnable sample holder might deviate from the perpendicular setting with respect to the direction of the magnetic field and second, the sample might exhibit a tilting of the c axis due to lattice imperfections and microcrystal formations. However, both possibilities can be excluded. The crystal orientation is calibrated by the back reflection of the HeCd laser spot. The error in the angular orientation at 0° and 90° is smaller than 0.5°. Hence, an additional splitting of the observed size could not occur. The second possible explanation involving a tilted c axis also has to be discarded as demonstrated by high-resolution x-ray diffraction. No signs of a tilted c axis could be found and the (0002) reflection shows a perfectly symmetric shape with a full width at half maximum of 17 arcsec in the ω scan.

(v) Finally, one could argue that the ordering of the valence bands might be different from the ordering of the corresponding excitons due to the mixing and interaction of states. Concerning this objection we refer to the extensive exciton fine structure calculations of Lambrecht *et al.*[6] Within this work, it could be shown that the excitons closely maintain their principal valence band character even when band mixing effects are considered. The same results confirming the Γ_7 character of the upper valence band and of the upper A exciton were obtained later by *ab initio* calculations of excitons in ZnO.[19]

In summary, we have observed an hole state related fine splitting for bound excitons in ZnO in the Voigt configuration. Theoretical considerations clearly demonstrate that the splitting is caused by the Γ_7 symmetry of the hole state. This assignment is doubtlessly confirmed by angular- and polarization-dependent measurements. The parallel and perpendicular hole g values were obtained in good agreement with earlier first-principles calculations. Possible countereffects that might lead to alternative explanations of the observed data including uniaxial and biaxial strain effects, misalignments, and the involvement of B valence band holes were extensively discussed and excluded. In conclusion of these arguments, we are most confident that the observed fine splitting provides conclusive proof that the uppermost A valence band in ZnO possesses Γ_7 symmetry.

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