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Analyses of Semiconductor Materials by Spectrometric Full-color Cathodoluminescence Microscopy

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The luminescence released by a photon incidence, so-called photoluminescence (PL), provides information of electronic states of the specimen such as the band gap of semiconductors or the highest occupied molecular orbital (HOMO)-lowest unoccupied MO (LUMO) of organic materials. However, the band-gap of recent semiconductor materials for optoelectronic devices is as wide as several eV, and the excitation of such materials becomes impossible by regular light source including discharge lamps for PL study use. Focusing and scanning of probing photons on the specimen contains another difficulty if microscopic observation is tried by PL microscopy. The luminescence upon electron incidence, cathodoluminescence (CL) can overcome all above difficulties. Our new spectrometric full-color CL microscope [1] collects CL spectra of each observing point, and draws full-color CL and corresponding secondary electron micrographs of 512 x 512 pixels in 8 s, which enabled us to observe irradiation-sensitive materials such as organic or biological materials. Since CL is excited by the incident narrow-focused electron beam in SEM, most electronic states are excited and luminescence light down to 200 nm wavelength can be released. Spatial resolution of CL micrographs may become worse than that of SEM due to scattering of incident electrons in the specimen. Delayed CL emission is another cause of poor resolution. Both result in practical CL resolution around 100 nm. By choosing lower incident energy and slow image scan speed, the spatial resolution of CL observation is significantly improved, and in our system 25 nm resolution was verified with 6 kV beam and 40 s/frame. In the present study, we summarize previous studies and show new observations on growth and qualification of GaN/InGaN layers for laser diode fabricated by metalorganic vapor-phase epitaxy (MOVPE) method [2], deep-UV emission from grain boundaries of ZnO varistor [3], and cyanine dye J-aggregates. The block diagram of the system is shown in Fig. 1. In the SEM image of Fig. 2, we noticed two white lines (pointed by green arrows and noted as “D” (D_1, D_2) in the corresponding CL image) run. They are assumed to be defects being created on the substrate surface upon epitaxy and kept growth across hetero interfaces of InGaN/GaN layers. CL spectra along the white lines show sharp emission at about 560-570 nm that is absent in the spectra of other parts marked by “N” (N_1-N_3). Hence, the 560-570 nm peak is characteristic to the defect structure. A survey of CL spectra throughout the specimen revealed another points of the characteristic 560-570 nm peak (marked by “H”). In the SEM, no special contrast is seen along “H”, which suggests the defect lying deep from the surface. Luminescence light can escape deep from the surface whereas secondary electrons hardly do. Fig. 4 is a study of grain boundaries of ZnO varistor [3]. CL observation across the boundary revealed that, from the boundary, 500 nm peak appears first, then 510 nm peak rises slightly inside

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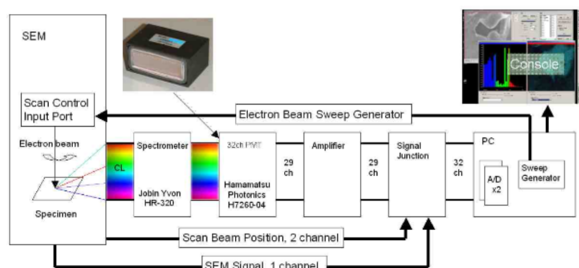

Fig. 1. System block diagram of spectrometric full-color cathodoluminescnece microscope.

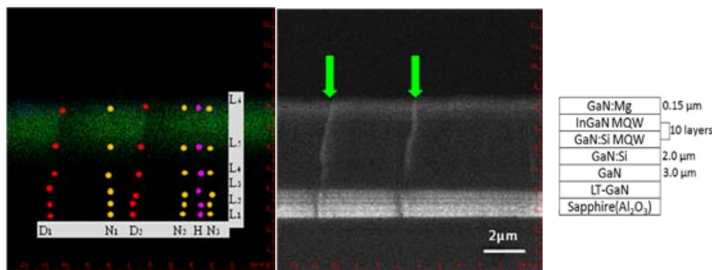

Fig. 2. CL and SE images of InGaN/GaN wafer. Color dots are the points of CL spectra in Fig. 3

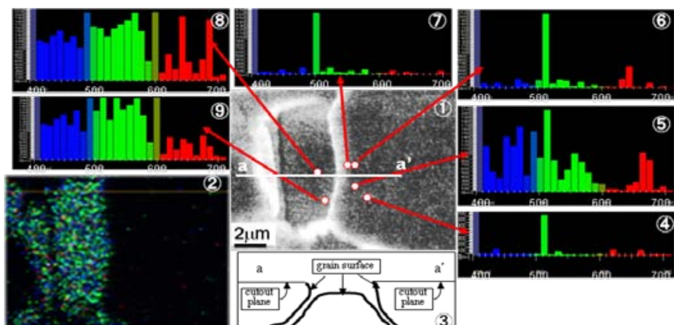

Fig. 4. Spectral variation near the grain boundaries of ZnO varistor. 1 and 2: SEM and full color CL micrograph. 3: Schematic diagram of the cross section, a-a'.4-9: CL spectra of points marked in 1.

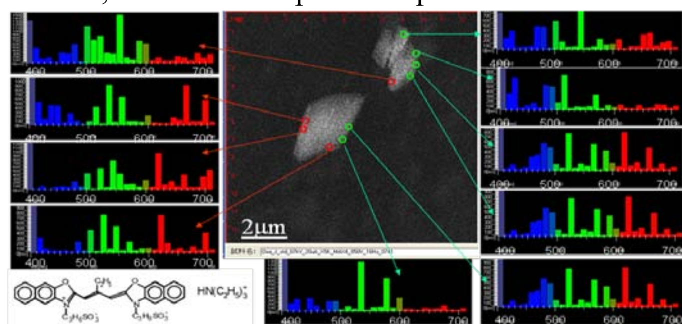

Fig. 5 Spectral variation of CL from Oxacarbo-cyanine J-aggregates grown in water.

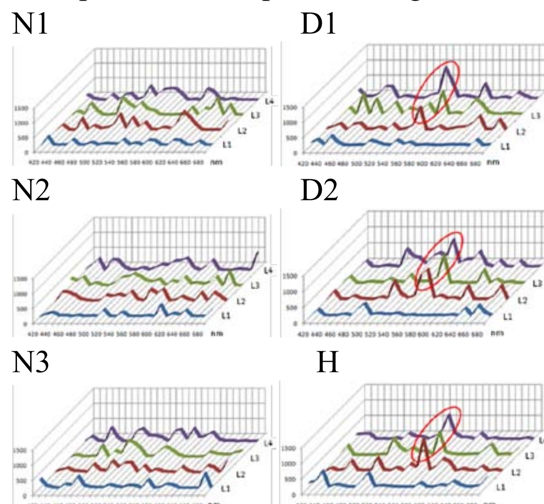

Fig. 3. Spectral variation of layers L_1, L_2, L_3 and L_4 at columns N_1, H, N_2, D_1, N_3 and D_2

the grain. Then, 490 nm peak twins with 510 nm peak. The spectral variation suggests the change of elemental composition across the boundary. This material also emits deep-UV luminescence at about 210 nm[5].

Cyanine dye molecules gather taking special arrangement each other and form so-called J-aggregates, and the aggregate emits sharp and strong luminescence. Fig. 5 shows the spectral variation of CL depending on the position and angle of dye molecules inside aggregates.

Our new spectrometric full-color CL microscope showed successfully the ability of detection such as hidden defects and structural variation near boundaries, and heterogeneous luminescence emission.

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