

Ionic-Liquid-Based Synthesis of GaN Nanoparticles

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Analytical Equipment

Transmission electron microscopy (TEM). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) were conducted with a FEI Osiris microscope operated at 200 kV and an aberration-corrected FEI Titan³ 80-300 microscope operated at 300 kV. TEM samples were prepared by evaporating GaN suspensions in acetonitrile on an ultrathin amorphous carbon film (3 nm) on holey carbon support film mounted on a 400 μm mesh Cu grid (Ted Pella Inc.). The deposition of the samples was performed under argon atmosphere in a glovebox. Subsequently after preparation the GaN samples were slowly heated to 130 °C under vacuum to remove adhered solvent molecules. The grids were thereafter transferred with a suitable vacuum/inert gas transfer module into the transmission electron microscope without any contact to air.

High-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) combined with energy dispersive X-ray spectroscopy (EDXS) experiments were performed with the FEI Osiris microscope at 200 kV, which is equipped with a Bruker Quantax system (XFlash detector) for EDXS. EDX spectra obtained by scanning of a rectangular area within a nanoparticle were used to determine the chemical composition of nanoparticles. The quantification of the Ga and N content was performed on the basis of the Ga–K series and the N–K $_{\alpha}$ line using the FEI software package “TEM imaging and analysis” (TIA) version 4.7 SP3. Element concentrations were calculated from a refined Kramers’ law model, which includes corrections for detector absorption and background subtraction. Standard-less quantification, i.e. by means of theoretical sensitivity factors, without thickness correction was applied. In addition, EDXS elemental maps of Ga (Ga–K $_{\alpha 1}$ line) and N (N–K $_{\alpha}$ line) reveal the Ga and N distribution within nanoparticle ensembles. The EDXS maps were analyzed by using the ESPRIT software (version 1.9) from Bruker.

X-ray powder diffraction (XRD). X-ray powder diffraction (XRD) was performed with a STOE STADI-MP diffractometer operating with Ge-monochromatized Cu-K $_{\alpha}$ -radiation ($\lambda=1.54178$ Å). GaN samples were prepared in glass capillaries under argon. Since the scattering power of the small-sized metal nanoparticles (diameter ≤ 10 nm) is low, certain non-specific background was observed. This nonspecific scattering was fitted by background correction (Win-XPOW, 1.2v).

Fourier-transform infrared (FT-IR) spectroscopy. Fourier-transform infrared (FT-IR) spectra were recorded with a Vertex 70 FT-IR spectrometer from Bruker Optics. The transmittance of pellets consisting of 300 mg of dried KBr and 0.8 mg of the GaN nanoparticle sample was measured.

Ultraviolet/visible (UV/Vis) spectroscopy. Ultraviolet/visible (UV/Vis) spectra were recorded using a UV-2700 (Shimadzu) equipped with an Ulbricht sphere. GaN nanoparticle samples were measured in reflection as powders (3 mg of sample and 200 mg of dried BaSO₄).

Fluorescence spectroscopy. A Horiba Jobin Yvon Spex Fluorolog 3.2 spectrometer was used to obtain excitation and emission spectra of GaN powder samples. The spectrometer was equipped with a 450 W Xenon lamp, an integrating sphere (Ulbricht sphere) as well as double grating excitation/emission monochromators as well as a photomultiplier as detector.

Determination of the quantum yield. The absolute quantum yield was performed and calculated according to Friend.^{S1} To this concern, the diffuse reflection of the GaN sample was determined at certain excitation wavelength followed by measuring the emission under excitation at the same wavelength. The relation of the number of reflected and emitted photons by using the integrating sphere resulted in the absolute quantum yield.

References

(S1) J. C. de Mello, H. F. Wittmann and R. H. Friend, *Adv. Mater.*, 1997, **9**, 230.