EELS analysis of Ni seeds used as promoters of MBE growth of GaN Nanowires.

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In this work we report a STEM investigation of gallium nitride nanowires (NWs) grown by radio frequency plasma assisted molecular beam epitaxy (MBE). In order to promote the NW growth, Ni seeds were deposited on the sapphire substrate before growth as described in ref [1]. A typical section through an array of NWs is shown in Fig.1a). All the analyses were performed on nanowires firstly removed from the substrate and then deposited on lacey carbon film supported by standard TEM Cu grids as in Fig.1b). In particular, examination of the composition of nickel seeds used to promote the nanowire type growth was made with the aim of understanding the growth mechanism.

Electron microscopy analysis was performed using the aberration-corrected Scanning Transmission Electron Microscope at the SuperSTEM Laboratory in UK. This instrument is based on a 100 keV VG HB501 with a cold-field emission source, equipped with a Gatan Enfina parallel channel Electron Energy Loss Spectrometry (EELS) system and a Nion Mark II spherical aberration corrector.

The bright field image of Fig.2a) shows a typical growth tip end of a nanowire revealing the presence of the Ni seed with lattice resolution. The lattice spacing measured in the GaN nanowire body of Fig.2 yielded a value of \(2.59\pm0.02\) Å in good agreement with GaN (0002) spacing [2]. Measured values of lattice spacing from the seed are attributable to the lattice spacing of either (002) NiO or (111) GaNi\(_3\).

EELS analyses of the same nanowire were performed to elucidate the phase of nickel based seeds. Line scans and spectrum images were quantified assuming a single power law decay for the background subtraction. EELS edges were integrated choosing spectral windows accordingly to Liu and Brown [3]. The electronic partial cross sections for inelastic scattering were calculated using the Hartree-Slater model for each specific core loss feature. This analysis, shown in Fig. 3, yielded the expected 1:1 ratio for the Ga and N elemental compositions within the nanowire body. Nickel seed composition exhibited a metallic core with gallium and nickel concentrations consistent with the presence of the equilibrium phase \(\alpha'\) GaNi\(_3\) [4]. Virtually no nitrogen was detected within the seeds. This observation is consistent with the instability of the Ni\(_3\)N and Ni\(_4\)N phases at the growth temperature of 730°C [5]. Nickel seeds also showed a gallium-doped nickel oxide shell which is attributed to oxidation in the ambient, following removal from the MBE growth system.

The implications of these observations on the growth of GaN nanowires will be discussed.
Fig. 1: (a) A typical section through an array of nanowires as seen by SEM. (b) STEM BF image of a sample of “harvested” NWs supported by lacey carbon film on a Cu TEM grid.

Fig. 2: (a) BF image with lattice resolution of Ni-rich seed at the tip of a nanowire. The box inset designates the area of EELS analysis reported in Fig. 3. (b) FFT of the image with the assigned diffraction spots.

Fig. 3: (a) Quantified elemental distributions by EELS analysis from the seed area of Fig. 2a), each point on the x-axis is related to sum spectra within the inset slices of Fig. 3b). (b) Digital Micrograph spectrum image, the white box refers to the first slice and the black to the last slice of Fig. 3a).

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