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Cathodoluminescence microanalysis of porous GaP and InP structures

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Abstract. Electron microscopy and cathodoluminescence (CL) microanalysis were used for a comparative study of porous layers fabricated by electrochemical etching of n-GaP and n-InP substrates in aqueous solutions of sulfuric and hydrochloric acids. Both the CL and morphology of porous layers were found to depend upon the anodic current density. At high current density (100 mA/cm^2) anodization leads to the formation of so-called current-line oriented pores while at low current densities the pores grow along ⟨111⟩ crystallographic directions. The porosity relief was found to give rise to spatial modulation of the CL intensity. The composition microanalysis proved the stoichiometry of porous GaP and InP skeletons, although we found considerable traces of oxygen in porous GaP layers. Self-induced voltage oscillations giving rise to a synchronous modulation of the diameter of pores and CL intensity were evidenced.

PACS. 68.37.Hk Scanning electron microscopy (SEM) (including EBIC) – 78.60.Hk Cathodoluminescence, ionoluminescence – 81.07.Bc Nanocrystalline materials

1 Introduction

Porosity is an effective tool for engineering basic parameters of semiconductor materials [1]. In particular, porous III-V compounds were found to exhibit Fröhlich-type surface-related vibrations with porosity-tunable frequencies and efficient optical second harmonic generation [2–5]. So far, most experiments investigating emission characteristics of porous III-V materials have been restricted to photoluminescence (PL). The PL of porous GaP, GaAs and InP at energies above the band gap of the bulk material has been attributed to quantum size effects [6–10]. In this work, we study the morphology and CL characteristics of porous layers obtained by electrochemical dissolution of n-GaP and n-InP substrates. We report drastic modification in both morphology and CL intensity of porous GaP and InP layers with changing anodization conditions. Self-induced voltage oscillations were observed during anodic etching at high current density and their impact on pore morphology and CL intensity was evidenced. The results of the CL microanalysis indicate the possibility of controlling the spatial distribution of emission in porous GaP and InP.

2 Experimental

(100)-oriented n-GaP:S and n-InP:Si wafers cut from Czochralsky-grown ingots were used in this work. The free electron concentration in the as-grown substrates was $(0.5-1)\times10^{18}$ cm^{-3} at 300 K. The anodic etching was carried out in aqueous electrolytes of H_2SO_4 and HCl in galvanostatical regime.

The CL experiments were performed in a Scanning Electron Microscope (SEM) equipped with *Oxford Instruments* MonoCL2 cathodoluminescence imaging and spectral analysis system, and cryogenic specimen stages. CL and SEM images were taken from the same sample areas for comparison. The CL was excited with a continuous electron beam at normal incidence, and measured using a retractable parabolic mirror collector. CL spectra were collected over the wavelength range 250–900 nm using a *Hamamatsu* R943-02 high sensitivity photomultiplier with a 1200 line/mm grating, blazed at 550 nm. The CL spectra were collected over a range of beam energies (E_b = 15–30 keV) and beam currents (I_b = 0.25–100 nA). EDX microanalysis was used to study the chemical composition of porous layers.

3 Results and discussion

Figure 1 shows SEM and panchromatic CL images in cross-section taken from a GaP sample subjected to successive anodization steps at the current densities: j_1 = 100 mA/cm^2 for 30 min (Figure 1 a) and j_2 = 1 mA/cm^2 for 240 min (Figure 1 d). Electrochemical etching at

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