Ultra-fast growth of In nanowires on In-rich InGaN layers by focused ion beam irradiation

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Abstract. We grew In nanowires on In-rich InGaN layers with In content of 80% using focused ion beam system equipped with Ga liquid metal source. The experiments were performed at room temperature without any cooling or heating source, and the whole growth process can be monitored in-situ by field emission scanning electron microscope / FIB dual beam system. The nanowires were observed only at the ion beam irradiation area, and the length and diameter of nanowires were controlled by adjusting accelerating voltage. The energy dispersive x-ray spectroscopy and the electron diffraction pattern of nanowires using transmission electron microscope confirmed that the composition of nanowire was indium, and the In nanowire was grown along the [-1 1 2] direction. This In nanowire induced by FIB irradiation grew with the ultra-fast growth rate as fast as 8 μm/min. The growth of In nanowires was supposed to be caused by ion irradiation-induced phase decomposition.

1. Introduction

One-dimensional nanostructures, including nanowires, have attracted much attention due to their fascinating properties and various applications superior to their bulk counterparts.[1-3] Semiconductor nanowires for optoelectronic and electronic devices and metal nanowires for superconducting devices have been spotlighted.[4-6] Until now, several techniques to grow various types of nanowires have been reported.[7] Most of them are based on the vapor-liquid-solid (VLS) mechanism, in which various metals catalytically enhance the growth of nanowires.[8-10] Moreover, many different approaches to make nanowires using various template materials, e.g., alumina membranes, nanochannel glasses, polymers, mesoporous silica, carbon nanotubes, etc., have been introduced and studied.[11-15]

Recently, Lugstein et al. reported GaSb and Sb nanowires growth using a novel growth technique, focused ion beam (FIB), and the ion beam irradiation on GaSb made spaghetti-like structures caused by FIB-induced phase decomposition of GaSb.[16] This new accomplishment widened the application of FIB technique from tools of various analysis and device fabrications to the growth of one-dimensional nanostructure.[16-20] In this paper, we present another FIB approach to make In nanowires from In-rich InGaN film using FIB-induced phase decomposition.
2. Experiments
The In-rich InGaN layer was used as a target sample for FIB irradiation. The InGaN layer with 80% In content was grown on a GaN/sapphire substrate at 650°C by metal-organic chemical vapor deposition. The layer was 300 nm thick and very porous. The experiments were performed using field emission scanning electron microscope (FE-SEM)/FIB dual beam system equipped with a Ga liquid metal source (FEI Nova NanoLab) at room temperature without cooling or heating. The whole process by FIB irradiation could be monitored in-situ by FE-SEM. Ga ion beam irradiation was carried out at the fixed ion dose per area of 1.2 pC/μm² over the accelerating voltage rage 5 ~ 30 kV. The current density was also varied at fixed accelerating voltages.

Structural properties of nanowires induced by FIB irradiation were confirmed by transmission electron microscope (TEM) and their composition was analyzed by energy dispersive x-ray spectroscopy (EDS). Nanowire samples for TEM observation were prepared by using FIB system. The targeted nanowire was detached by manipulating a probe and attached to a TEM grid using Pt deposition by electron beam irradiation.

3. Results and discussion
The formation of nanowires was confirmed by in-situ FE-SEM/FIB dual beam system. When the surface of an In-rich InGaN layer was irradiated by FIB, the growth of nanowires and nanocrystals were observed, as shown in Fig. 1. Long needle-shaped nanowires were formed, and sometimes, spiral nanowires were also observed. Moreover, the growth was observed only at FIB irradiated area.

Fig. 1 FE-SEM image showing nanowires and nanocrystals induced by FIB at the accelerating voltage of 30 kV and the ion current density of 125 nA/cm²

To identify the composition of nanowires induced by FIB irradiation, TEM measurement was performed. As shown in Fig. 2a, the electron diffraction pattern from a nanowire with (110) zone axis confirmed the single crystalline nature of the In nanowire. It could be indexed in terms of the body-centered tetragonal structure with lattice constants of a = 0.325 nm and c = 0.495 nm, which agreed well with the reported values of indium bulk crystal. Energy dispersive x-ray spectroscopy (EDS) using TEM in Fig. 2b identified nanowires induced by FIB irradiation as indium. Detailed TEM observations indicated that In nanowires grew along the [-1 1 2] direction, as shown in Fig. 2c.
Fig. 2 TEM measurement of a nanowire grown at the condition of 10 kV of accelerating voltage and ion dose per area of 1.2 pC/μm². a) An electron diffraction pattern of the In nanowire with the [110] zone axis. b) Energy dispersive x-ray spectroscopy of the In nanowire. Cu peak was originated by the copper grid for TEM sample preparation. c) High resolution TEM image showing that the In nanowire is grown along [-1 1 2] direction.

The effect of FIB accelerating voltage and ion beam current density on the growth behavior of In nanowires was examined. When FIB accelerating voltage and beam current density were controlled, the length and the diameter of nanowires varied from 100 nm to 80 μm and from 30 nm to 500 nm, respectively. The nanowire size was largely affected by accelerating voltage. As accelerating voltage was changed from 5 kV to 30 kV, the length of the longest nanowires approximately increased from 8 μm to 80 μm, as shown in Fig. 3a. The maximum and the minimum diameter also increased from 80 nm to 400 nm and from 30 nm to 200 nm, respectively, as shown in Fig. 3b. As accelerating voltage increased, higher energy ion beam could supply more In, which was caused by the FIB-induced phase decomposition of the InGaN layer. It was reported that FIB irradiation induced the phase decomposition of some materials, and InGaN is expected to be decomposed by FIB in a similar way.[16, 20]

The continuous increase of beam energy by increasing accelerating voltage and current density made the subtractive process (cutting and etching) superior to the growth process. InGaN layer became milled and cut at high energy ion beam irradiation instead of the growth of In nanowire. At the accelerating voltage of 30 kV and the current density of 1 μA/cm², the growth of nanowires and the milling of InGaN layer occurred together, and the only milling process was observed at higher current densities. The In nanowires grew at very fast growth rates. For example, at the accelerating voltage of 30 kV and the beam current density of 125 nA/cm², 80 μm long In nanowire was grown for 10
minutes, i.e., approximately at 8 μm/min. and other irradiation conditions also showed remarkably fast growth rate of In nanowire. Yi et al. reported that the metallic lead nanowires were grown by electrodeposition using polycarbonate membranes, and the growth rate was estimated at 3.6 μm/min.[21] Nam et al. reported that the 50 μm GaN semiconductor nanowires by VLS mechanism was grown for 2 hours, i.e., 0.42 μm/min.[17] Souplantica et al. introduced another In nanowire growth method, chemically synthesized, but the growth rate was 0.01 μm/min at most.[22] Compared with other representative growth techniques, the In nanowire growth in this study induced by FIB irradiation was an ultra-fast technique.

Fig. 3 a) Effect of accelerating voltage on the length of In nanowire. b) Effect of accelerating voltage on the diameter of In nanowires.

4. Conclusion
In nanowires were grown for the first time by FIB irradiation of In-rich InGaN layers. The growth of In nanowires was monitored in-situ using FE-SEM/FIB dual beam system. The nanowires were identified as In nanowires by using TEM and EDS. The length and diameter of In nanowires were controlled by FIB irradiation conditions. This work provides us a new method of In nanowire growth with controlled length, diameter and position by an FIB system.

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