ELSEVIER

Micron 36 (2005) 281–284

micron

www.elsevier.com/locate/micron

Preparation of GaN-based cross-sectional TEM specimens by laser lift-off

Li Zilan[a,b,*], Hu Xiaodong[a,b], Chen Ke[a], Nie Ruijuan[a], Luo Xuhui[a], Zhang Xiaoping[a], Yu Tongjun[a,b], Zhang Bei[a,b], Chen Song[a], Yang Zhijian[a,b], Chen Zhizhong[a,b], Zhang Guoyi[a,b]

[a]*State Key Laboratory for Artificial Microstructure and Mesoscopic Physics, Department of Physics, Peking University, Beijing 100871, China*
[b]*Research Center for Wide Gap Semiconductors of Peking University, NR. 468, Physics Building, Beijing 100871, China*

Received 16 October 2003; revised 28 November 2004; accepted 4 December 2004

Abstract

Laser lift-off (LLO) technology is successfully used to prepare GaN-based TEM cross-sectional specimens. Detailed procedures of the method to prepare the specimens are demonstrated. Large thin areas suitable for TEM analysis were obtained. TEM images of the resulting GaN interface are studied, and the changes in structural quality are confined to approximately the first 250 nm of the epilayer. Clear TEM images of the whole epilayer and the InGaN quantum wells and the HRTEM images of the superlattice layer are demonstrated, showing that LLO is a quick and ideal method to study the crystal structure of the epilayer, especially if only the upper layers are of interest.

Keywords: GaN; TEM; Laser lift-off; InGaN LD; HRTEM; Specimen preparation

1. Introduction

In the past several years, the prospect of using GaN and related alloys in electronic and optoelectronic devices has drawn the attention of scientists and engineers alike (Pearton et al., 2000). Despite the remarkable success of these materials to date, significant problems remain in the creation of GaN-based materials with high crystal quality. Due to the different properties between GaN-based materials and the substrates used, a high density of defects is introduced in the GaN-based epilayer. The crystal defects greatly influence the properties of these materials and their related devices. Much attention has been paid to the crystal defects in these materials. For instance, TEM and EDX have been widely employed to study the defects and to analyze the composition of different layers in the GaN-based epilayer.

Additionally, there are several major problems in preparing GaN-based TEM samples due to the hardness and mechanical instability of the sapphire substrates, which are the most widely used. First of all, since sapphire is very hard, cutting, polishing, dimpling and sputtering times are very long. Secondly, sapphire is easy to crack. It may crack during any step of the above mentioned preparation procedures. Thirdly, during the long process of sputtering, the copper support ring and the glue are readily sputtered away. So, it is really ideal to lift the sapphire substrate off without introducing obvious crystal quality degradation.

Recently, Kelly et al. (1996) and Wong et al. (1998) demonstrated a laser lift-off (LLO) technique with which the GaN-based epilayer and the sapphire substrate can be separated. Their method takes advantage of the different bandgaps of GaN (3.4 eV) and sapphire (8.1–8.3 eV) to locally decompose nitride materials at the interface between the nitride films and the sapphire substrates. Stach et al. (2000) have studied the lifted-off membranes by cross-sectional TEM. They prepared their sample with seven free-standing membranes originally deposited by HVPE to a thickness of 7 μm. In our experience, very thin free-standing GaN-based membranes readily crack and are very difficult to handle. Due to their serious mechanical instability, it is very difficult to adjust the membranes if they are glued in place, which is a necessity to ensure that observations are along desired directions.

In this work, we demonstrate a new method to prepare GaN cross-sectional TEM specimens by laser lift-off. We characterize the structural integrity of the lifted-off

* Corresponding author. Tel.: +86 1062759726; fax: +86 1062751732.
E-mail address: zealan@water.pku.edu.cn (L. Zilan).

0968-4328/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.micron.2004.12.001

films and demonstrate that only slight crystal quality degradation is induced near the irradiated surface. Conventional cross-sectional TEM images and HRTEM images are presented. These images suggest that our method is nearly ideal for GaN-based specimen preparation suitable for cross-section TEM analysis, especially if only the upper layers of the films are of interest.

2. Experimental procedure

The sample utilized here was a piece of InGaN multi-quantum-wells (MQW) Laser diode (LD) wafer grown by MOCVD on a 430 μm thick sapphire substrate. The InGaN LD structure consisted of a 25 nm GaN buffer layer, a 3 μm thick layer of n-type GaN:Si, a 0.1 μm thick layer of n-type $In_{0.1}Ga_{0.9}N$:Si, an $Al_{0.14}Ga_{0.86}N$:GaN strained-layer superlattices (SLSs) cladding layer consisting of 240 2.5 nm thick Si-doped GaN layers separated by 2.5 nm thick undoped $Al_{0.14}Ga_{0.86}N$ layers, a 0.1 μm thick layer of Si-doped GaN, an $In_{0.15}Ga_{0.85}N$:$In_{0.02}Ga_{0.98}N$ MQW structure consisting of four 3.5 nm thick undoped $In_{0.15}Ga_{0.85}N$ well layers forming a gain medium separated by 10.5 nm thick Si-doped $In_{0.02}Ga_{0.98}N$ barrier layers, a 200 nm thick layer of p-type $Al_{0.2}Ga_{0.8}N$:Mg, a 0.1 μm thick layer of Mg-doped GaN, an $Al_{0.14}Ga_{0.86}N$:GaN SLS cladding layer consisting of 120 2.5 nm thick Mg-doped GaN layers separated by 2.5 nm thick undoped $Al_{0.14}Ga_{0.86}N$ layers and a 0.05 μm thick layer of p-type GaN:Mg.

The 8 mm×7 mm wafer sample was mounted face down on a 12 mm×10 mm×0.8 mm piece of silicon wafer with epoxy. The pieces were clamped together and heated to solidify the epoxy. The sample was then irradiated from the backside of the sapphire substrate with a KrF excimer laser. The photon energy (5.0 eV) is less than the bandgap of sapphire (8.1–8.3 eV) but larger than the bandgap of GaN (3.4 eV). The laser pulses thus pass through the sapphire substrate and are absorbed at the interface between the GaN-based epilayer and the sapphire substrate. The laser irradiation induced the decomposition of the interfacial GaN into Ga and N_2 gas with a single 600 mJ/cm^2 laser pulse. The entire sample was transferred by rastering a 0.01 cm^2 spot across it. The sapphire substrate was then removed by heating the sample in 40 °C to melt the Ga-rich interface. A thin Ga-rich layer on the surface of the exposed interface was easily removed with a 1:1 solution of HCl and deionized water.

The Si/adhesive/GaN structure specimen was then cut into 2.5 mm×4 mm pieces (Fig. 1). A piece of the specimen was then bonded to another one by epoxy. The sample was subsequently clamped and heated to solidify it, forming a stable Si/adhesive/GaN-based membrane/adhesive/GaN-based membrane/Adhesive/Si structure. Then the sample was cross-sectionally cut into 0.5 mm thick pieces. One piece of the samples was mounted on a steel holder with low melting point wax, and then wet polished below 20 μm with progressively finer grit diamond lapping films. The film

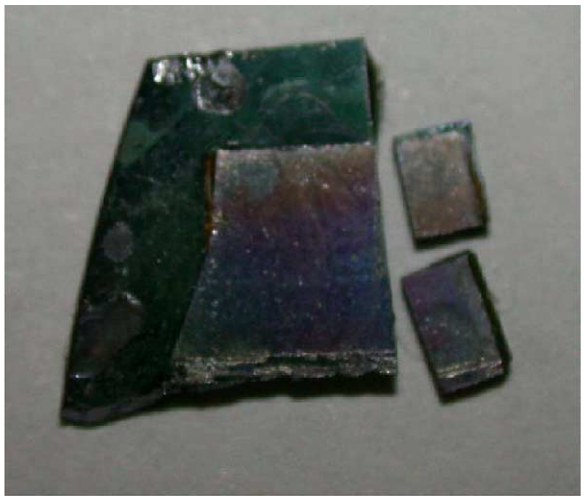

Fig. 1. The Si/adhesive/GaN structure specimen prepared by laser lift-off.

mounted on the holder was carefully glued to a copper ring with epoxy, leaving the hole of the ring uncontaminated by the epoxy. The holder was heated to solidify the epoxy and to melt the wax. Then the sample was carefully removed from the hot holder to filter paper, where the wax was removed with alcohol. The sample was then ion milled from both sides. Milling was done for about 50 min at a voltage of 5 kV and a current of 22 μA, with a tilt angle of 10°. Because the thick and hard sapphire substrate was substituted with Si, the whole preparation time was greatly reduced and the possibility to successfully prepare a specimen was enhanced. The TEM investigation was carried out on a Tecnai F30 operated at 300 kV.

3. Result and discussion

Successful TEM specimens of the InGaN LD epilayer were obtained with large thin areas suitable for TEM observation. Fig. 2 shows the cross-sectional bright field

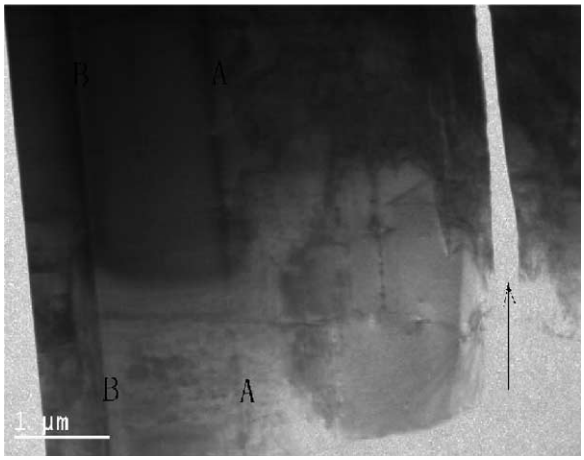

Fig. 2. Cross-sectional bright field TEM image of the InGaN LD films. The arrow indicates the epoxy gap between the newly created GaN surfaces.