Available online at www.sciencedirect.com
SCIENCE DIRECT®
Applied Surface Science 252 (2006) 3033–3042
applied surface science
www.elsevier.com/locate/apsusc

Nanomechanical characterizations of InGaN thin films

Sheng-Rui Jian [a], Te-Hua Fang [b], Der-San Chuu [a,*]

[a] *Institute and Department of Electrophysics, National Chiao Tung University, 1001 Ta Hsueh Road, Hsinchu 300, Taiwan*
[b] *Institute of Mechanical and Electrochemical Engineering, National Formosa University, Yunlin 632, Taiwan ROC*

Received 22 April 2005; received in revised form 4 May 2005; accepted 4 May 2005
Available online 13 June 2005

Abstract

$In_xGa_{1-x}N$ thin films with In concentration ranging from 25 to 34 at.% were deposited on sapphire substrate by metal-organic chemical vapor deposition (MOCVD). Crystalline structure and surface morphology of the deposited films were studied by using X-ray diffraction (XRD) and atomic force microscopy (AFM). Hardness, Young's modulus and creep resistance were measured using a nanoindenter. Among the deposited films, $In_{0.25}Ga_{0.75}N$ film exhibits a larger grain size and a higher surface roughness. Results indicate that hardness decreases slightly with increasing In concentration in the $In_xGa_{1-x}N$ films ranged from 16.6 ± 1.1 to 16.1 ± 0.7 GPa and, Young's modulus for the $In_{0.25}Ga_{0.75}N$, $In_{0.3}Ga_{0.7}N$ and $In_{0.34}Ga_{0.66}N$ films are 375.8 ± 23.1, 322.4 ± 13.5 and 373.9 ± 28.6 GPa, respectively. In addition, the time-dependent nanoindentation creep experiments are presented in this article.

Keywords: InGaN; XRD; AFM; Nanoindentation; Creep

1. Introduction

The III-nitride wide-band-gap semiconductors are the most promising materials for constructing optoelectronic and electronic devices [1,2]. Among the materials, ternary InGaN alloys which cover the direct-band-gap energy ranging from 1.9 eV (InN) to 3.4 eV (GaN) are crucial and functional materials for GaN-based blue/UV light emitters/lasers and high-power electronic devices [3,4]. Although many works have been focused on the optoelectronic characteristics of InGaN alloys, however, their mechanical properties are almost ignored. InGaN alloys have been used in the form of thin films in optoelectronic and electronic devices. Mechanical properties of materials are size-dependent. Thin films may have different mechanical properties from their bulk materials. A precise measurement of the mechanical properties of InGaN thin films is required to use them as structural/functional elements in devices. Nanoindentation has been widely used for charactering the mechanical properties of solid surfaces and thin films [5,6]. Among the mechanical properties of interest, hardness, Young's modulus, elastic/plastic deformation

* Corresponding author. Tel.: +886 37261111x56105; fax: +886 35725230.
E-mail address: dschuu@mail.nctu.edu.tw (D.-S. Chuu).

0169-4332/$ – see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.apsusc.2005.05.019

behavior and creep resistance can be obtained from nanoindentation testing.

Recently, the indentation-induced plastic behaviors and mechanisms of III–V semiconductors have been discussed [7] in more detail by means of atomic force microscopy (AFM), plane-view and cross-sectional transmission electron microscopy (XTEM); in particular, the effects of alloy strengthening of $In_xGa_{1-x}As/GaAs$ were also investigated. To our best knowledge, however, there are no reports on the mechanical properties of InGaN thin films have been made until now.

In this study, InGaN thin films with different indium concentrations were deposited by low-pressure metal-organic chemical vapor deposition (MOCVD). We report, for the first time to our knowledge, the hardness, Young's modulus and creep resistance of InGaN thin films. Nanoindentation deformation behavior of the deposited films was studied by analyzing the nanoindentation load–displacement curves and post-indentation imaging of the indentation impressions. Changes in mechanical properties for the deposited films are discussed in conjunction with deposition temperature, crystalline structure, grain size and surface morphology.

2. Experimental details

The $In_xGa_{1-x}N$ films were grown on sapphire substrate by horizontal low-pressure MOCVD. Prior to material growth, the sapphire substrate was annealed/cleaned to remove surface residual stress and impurities in H_2 gas at 1120 °C for 10 min. Trimethylgallium (TMGa), trimethylindium (TMIn) and ammonia (NH_3) were used as source precursors for gallium, indium and nitrogen, respectively. A 25 nm thick GaN nucleation layer was first deposited at 520 °C for 4 min on the sapphire substrate. The substrate temperature was then raised to 1120 °C to grow a 2 μm thick undoped GaN layer. Finally, a 400 nm thick $In_xGa_{1-x}N$ film was grown on the undoped GaN layer. During growth of the $In_xGa_{1-x}N$ thin films, reactor pressure was kept constant at 200 mbar and input flow rates of ammonia, TMGa and TMIn were kept at 12, 26.5 and 25.5 μmol/min, respectively. To obtain different In concentrations, deposition temperature was varied from 730 to 790 °C. The In concentration in the deposited thin films was obtained by separating GaN and InGaN peaks in X-ray diffraction (XRD) spectra using Vegard's law [8].

Crystalline structure of the deposited InGaN films was characterized using a Bede QC200 X-ray diffractometer with Cu Kα irradiation at 40 kV and 0.4 mA. Surface morphology of the deposited films was investigated by using a Veeco/TM CP-R atomic force microscopy (AFM).

Hardness and the elastic modulus were calculated from the load–displacement (P–h) data obtained by nanoindentation using a TroboScope nanomechanical testing systems (Hysitron Inc.). The Hysitron nanoindenter monitors and records the load and displacement of the indenter, a diamond Berkovich three-sided pyramid, with a force resolution of about 1 nN and displacement resolution of about 0.1 nm. All nanoindentation tests were performed when the thermal drift dropped down to 0.01 nm/s. The thermal drift effects were corrected for each test using a holding segment in the air before indentation. The indentation impressions were then imaged in situ using the same indenter tip. Five indentations were made on each specimen. A typical indentation experiment consists of four subsequent steps: approaching the surface; loading to peak load at a loading rate of 10 μN/s; holding the indenter at peak load for 5 s; finally unloading completely. The hold step was included to avoid the influence of creep on the unloading characteristics since the unloading curve was used to obtain the elastic modulus of a material. For more details on nanoindentation experimental techniques, please see references [5,6].

The indentation P–h data obtained at each depth were analyzed to determine the hardness, H, and elastic modulus, E, by using the Oliver and Pharr method [9]. In this method, the hardness and modulus are determined as following.

Nanoindentation hardness is defined as the indentation load divided by the projected contact area of the indentation. It is the mean pressure that a material will support under load. From the P–h curve, hardness can be obtained at the peak load as:

$$H = \frac{P_{\max}}{A} \tag{1}$$

where A is the projected contact area.

The elastic modulus was calculated using the Oliver–Pharr data analysis procedure [9] beginning by