



Nanoindentation study of indium nitride thin films grown using RF plasma-assisted molecular beam epitaxy

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ABSTRACT

In this study, we used an RF plasma-assisted molecular beam epitaxy (RF-MBE) system to grow single-crystalline indium nitride (InN) films onto aluminum nitride (AlN) buffer layers on Si(111) substrates. We then used nanoindentation techniques and reflection high-energy electron diffraction (RHEED) to study the influence of the *c*-axis-oriented InN films on the mechanical performance. From morphological observations, we compared the stiffness and resistance against contact-induced damage of the InN films in the presented shrinkage of the area. InN films prepared at growth temperatures of 440, 470, and 500 °C had nanohardnesses (*H*) of 3.6 ± 0.2 , 4.5 ± 0.25 , and 9.1 ± 0.8 GPa, respectively, and Young's moduli (*E*) of 97.4 ± 1.2 , 147.7 ± 1.8 , and 176.0 ± 2.3 GPa, respectively.

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1. Introduction

The group-III nitride compounds indium nitride (InN), aluminum nitride (AlN), and gallium nitride (GaN) have attracted great attention for both fundamental research and practical applications [1,2]. Indium nitride films are particularly interesting materials because of their direct and narrow band gap, high thermal conductivity, excellent mechanical properties, high electrical resistivity, rapid sound propagation, and relatively large piezoelectric constants [3,4]. Nevertheless, high-purity single-crystalline InN is very difficult to produce because of its low dissociation temperature and high nitrogen equilibrium partial pressure [5,6]. Although Si has been used widely as a substrate for InN growth, the resulting film quality is typically very poor. In 1994, Yamamoto et al. [7] attempted to use metalorganic vapor phase epitaxy (MOVPE) to grow InN on a Si substrate, but they were unsuccessful because an amorphous SiN_x layer formed on the substrate surface through unintentional nitridation of the substrate surface during the growth process. Growth temperatures below 400 °C result in polycrystalline InN films, due to decreases in the migration of the deposited materials on Si surface and/or decomposition rate of the raw materials. Silicon is a suitable semiconductor substrate material for InN because of the lower lattice mismatch [8% for InN(0001)/Si(111)] compared

with that of sapphire, a more commonly used insulating substrate [25% for InN(0001)/a-Al₂O₃(0001)]. The InN layers formed on Si(111) and Si(001) also exhibit strong photoluminescence (PL) emissions at room temperature [8,9]. Because epitaxial InN is available only when deposited at relatively low temperatures, measurements of the mechanical properties of InN are complicated by the limited thicknesses of the materials. As a result, based on weak atomic bonds and readily generated defects, group-III nitride compound materials typically display poor mechanical performance.

To investigate defects and deformations generated in thin films, nanometer-scale nanoindentation is often employed to provide controllable crack information relating to mechanical characteristics of the solid surfaces, including hardness (*H*) and Young's modulus (*E*) [10–15]. In this perspective, to promote their advantage from nanoindentation instrument by means of contact loading cycle is necessary for presented work. Nevertheless, the mechanism responsible for InN film growth remains poorly understood in terms of effect on resulting mechanical characteristics.

In this study, we used nanoindentation techniques and atomic force microscopy (AFM) to investigate pressure-induced impairment and surface roughness of InN films grown on AlN buffer/Si substrates.

2. Experimental details

InN films were grown on Si(111) substrates using RF plasma-assisted molecular beam epitaxy (RF-MBE), with a background

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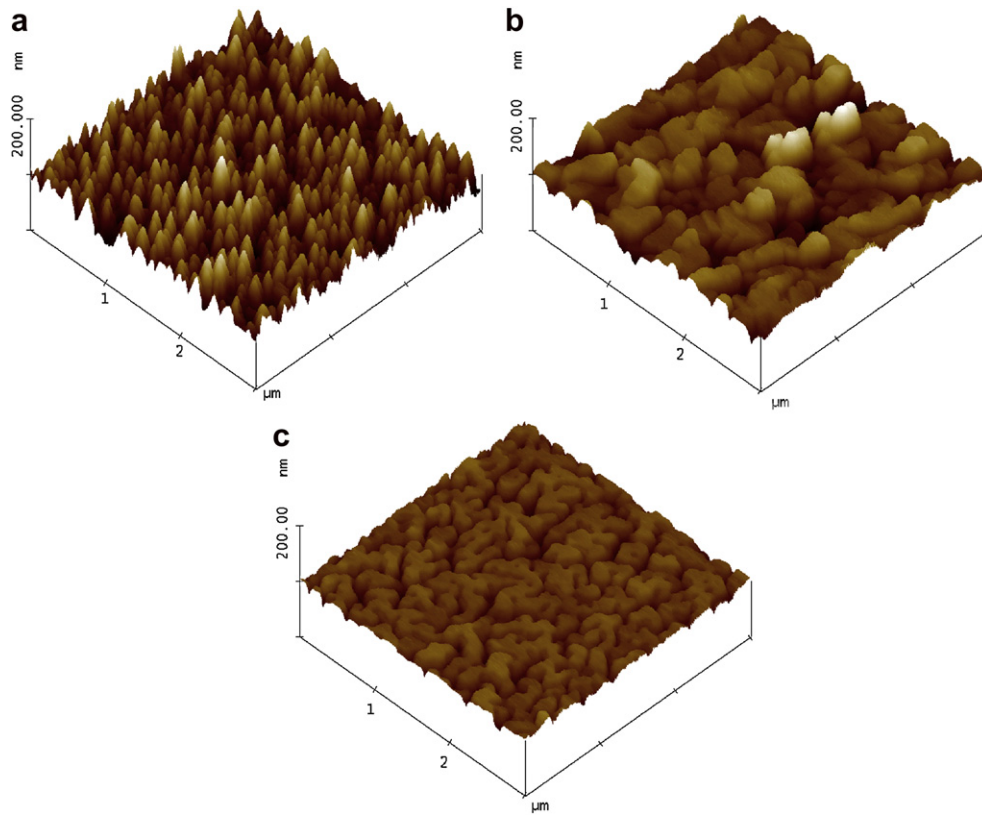


Fig. 1. Three-dimensional (3D) AFM surface topographies of InN films grown at (a) 440, (b) 470, and (c) 500 °C; average surface roughnesses: (a) 4.5, (b) 11.9, and (c) 13.4 nm.

pressure in the growth chamber of approximately 1×10^{-10} torr. The application of the common two-step growth method allowed the growth of flat, good-quality films of InN and AlN on the Si substrate [16]. The AlN buffer layer was grown on Si substrate by supplying nitrogen radicals under Al atomic beam exposure; the temperature during the growth of the AlN buffer layer was controlled at approximately 850 °C. Three types of InN samples (A, B, and C) were deposited with film thicknesses of 400, 450, and 550 nm, respectively. Surface structures at different stages of growth were determined using in situ reflection high-energy electron diffraction (RHEED). The surface roughness and microstructure are analyzed using AFM (Veeco Dimension 5000,

Scanning Probe Microscopy, D5000). Film thicknesses and morphologies were determined using a JEOL JSM-7001F field-emission scanning electron microscope. The polarity of InN was determined after wet etching (10 M KOH, 90 min).

The hardness (H) and Young's modulus (E) of InN samples were analyzed using a Nanoindenter XP instrument (MTS, Nano Instruments Innovation Center, TN, USA) equipped with a 50 nm diamond Berkovich indenter tip under continuous stiffness measurement (CSM) mode. Prior to measuring the H and E of InN, the instrument was calibrated using a standard fused silica sample. The method for calculating the values of H and E was developed by Oliver and Pharr [17].

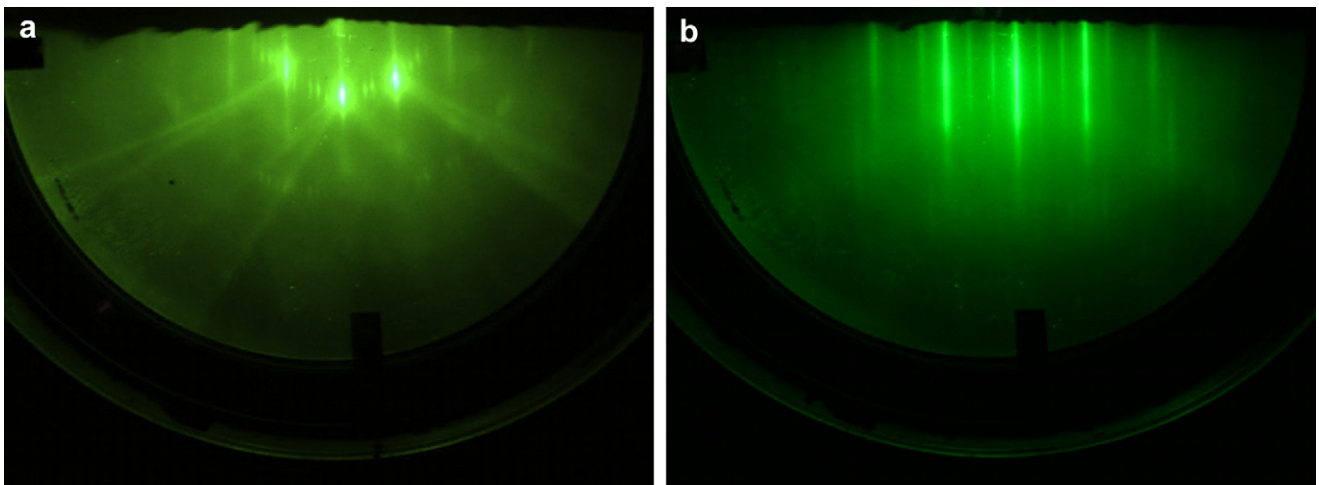


Fig. 2. RHEED patterns of (a) the Si substrate and (b) The AlN buffer layer.

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