Nanoindentation of AlN, GaN, and AlGaN films grown by HVPE on SiC/Si hybrid substrates

A S Grashchenko, S A Kukushkin, A V Osipov and Sh Sh Sharofidinov

Institute for Problems in Mechanical Engineering of the Russian Academy of Sciences, 199178, Saint Petersburg, Russia

E-mail: asgrashchenko@bk.ru

Abstract. This paper presents an experimental study of structural and mechanical characteristics of thin films of AlN, GaN, and AlGaN grown on hybrid SiC/Si substrates by the method of HVPE. The surface roughness of the films and substrates has been measured. The thickness and molecular composition of the films have been determined by spectral ellipsometry. An analysis of elastic deformation occurring in the films as a result of indentation has been made. The values of hardness and Young's modulus of the films have been calculated.

1. Introduction

Wide-gap semiconductors such as aluminum nitride (AlN), gallium nitride (GaN), and aluminum gallium nitride (AlGaN) are considered as promising materials for modern micro and optoelectronics. In this respect, in all industrialized countries, scientific research is being conducted on the development of methods for the formation of such high-quality crystal structures and the measurement of their physical characteristics. A special place is occupied by studies of the mechanical properties of thin films of AlN, GaN, and AlGaN compounds since they are already successfully used in the production of LEDs, transistors, sensors, and other micro and optoelectronic products. The thickness of individual layers in modern devices can be in the range of some ten to a hundred nanometers, and a variety of high-precision diagnostic methods are being used to analyze such films. One of the promising methods to study the mechanical properties of crystals, thin films, and their crystalline quality is nanoindentation [1]. However, the study of samples with a layer of a thin film, and sometimes with several layers of thin films, by the nanoindentation method is a rather nontrivial problem. The complexity of this problem is associated with the fact that, due to the small thickness of the films under study, it is difficult to separate the useful signal directly from the film from the general reaction of the structure to the penetrating indenter since the substrate makes a significant contribution. At the moment, a number of theories and methods have been developed that make it possible to calculate the elastic modulus, hardness, and other parameters of a thin film from experimental data of nanoindentation.

An important problem for manufacturers of micro and optoelectronic components is the integration of new wide-gap semiconductors with silicon substrates. The problem consists in the significant difference in the crystal lattice parameters of silicon (Si) and materials such as AlN, GaN, AlGaN, and other wide-gap semiconductors. This contributes to the appearance of a large number of structural defects in the films. In [1, 2], a method for the synthesis of a nanoscale silicon carbide (SiC) layer inside the surface layers of a silicon wafer was theoretically described and experimentally...
implemented. This method is based on a new physical principle of coordinated replacement of a part of Si atoms by carbon atoms (C). It should be noted that the conversion of Si to SiC occurs uniformly over the entire area of the Si wafer. This process is realized as a result of a topochemical reaction:

$$2Si_{\text{solid}} + CO_{\text{gas}} \rightarrow SiC_{\text{solid}} + SiO_{\text{gas}},$$

(1)

As a result of the synthesis, a hybrid structure is formed [4], consisting of a single-crystal SiC layer 50–250 nm thick, a damper layer of porous Si with SiC inclusions, and a single-crystal silicon base. A depiction of this structure is shown in figure 1a. In [4], the dynamics of pore propagation under a SiC film was investigated by scanning electron microscopy (SEM) as a function of the synthesis time. Due to this specific structure, there are practically no elastic stresses between the Si substrate and thin films of AlN, GaN, AlGaN grown on the hybrid SiC / Si substrates. In this work, we present the results of a study of the structural and mechanical characteristics of thin AlN, GaN, AlGaN films formed by the chloride-hydride vapor phase epitaxy (HVPE) on the SiC / Si hybrid structures.

2. Experimental methods
For the growth of AlN, GaN, and AlGaN layers, we used hybrid SiC/Si substrates synthesized inside Si of KDB-10 grade oriented in the crystallographic direction (111) at a temperature of 1250–1300°C. To avoid the appearance of crystalline defects in GaN and AlGaN films associated with the wettability of GaN on SiC [5], a 10–20 nm thick AlN layer was deposited on the SiC surface before the growth. AlN/SiC/Si, GaN/AlN/SiC/Si, AlGaN/AlN/SiC/Si heterostructures were grown on the hybrid SiC/Si substrates by the HVPE method. Analysis of the surface morphology of the initial SiC layers and AlN, GaN, AlGaN films was carried out using atomic force microscopy (AFM) on the Easy Scan instrument from Nano Surf. The mechanical characteristics of the AlN, GaN, AlGaN layers were measured by the nanoindentation method using a Berkovich diamond tip with a radius of curvature of 100 nm. Experiments on nanoindentation were carried out on the Nano Test 600 multifunctional device from Micromaterials. All experiments on nanoindentation were carried out at a temperature of 22°C. The mechanical characteristics of the AlN, GaN, and AlGaN films were determined by analyzing the experimental dependences of the indentation force $F$ on the tip displacement relative to the initial sample surface $h$ by two methods. In the first case, the initial stage of nanoindentation was analyzed, which has a purely elastic character and is described by the Hertz relation for the interaction of a rigid sphere with an elastic half-space [6]. A comparison of the experimental dependence $F(h)$ with the Hertz relation was carried out using the least-squares method. As a result of the comparison, Young's modulus of the films under study was determined. In the second case, we analyzed a part of the experimental dependence $F(h)$, which was registered at the initial moment of unloading. Using the least-squares method, the tangent to the experimental dependence $F(h)$ at the point with the maximum indentation force was constructed and its slope was calculated, then the hardness and Young's modulus of the films under study were determined from the slope of the tangent by the Oliver and Pharr method. The nanoindentation was carried out with the maximal force of 4 mN. In calculating Young's modulus for all films, the Poisson ratio of 0.22 was used. The thickness and chemical composition of the AlN, GaN, AlGaN layers were determined by optical spectral ellipsometry using M-2000D J.A. Woollam from experimental dependences of dielectric constant on photon energy.

3. Results
The study of the surface morphology of the hybrid SiC / Si substrates, on which the AlN, GaN, AlGaN layers were deposited by the HVPE method, showed that in all cases the homogeneous SiC has a mosaic structure with a total surface roughness of 7–10 nm. Figure 1 shows a typical structure of the SiC / Si hybrid substrates, obtained by SEM from the end of the wafer, and the surface morphology of the SiC layer, recorded using AFM. It should be noted that the data presented in figure 1 were obtained when studying a SiC / Si plate, on which only the AlN film was further grown. According to the data of optical spectral ellipsometry, SiC layers contain an excess concentration of carbon-vacancy...
structures equal to 5–8%. The porosity of the solid solution of Si and SiC under the SiC film is 40–50%, and the average SiC thickness for all hybrid substrates is 100±10 nm.

![Figure 1. SEM images of the SiC/Si hybrid substrate side view (a); AFM image of the SiC film surface (b).](image)

The AFM scanning of the surface of the AlN, GaN, AlGaN films showed that the films are homogeneous and do not have large structural defects. According to AFM data, the surface roughness of the AlN, GaN, and AlGaN films is 11, 12, and 23 nm respectively. Figure 2 shows images of the surface of the AlN, GaN, and AlGaN films obtained using AFM.

![Figure 2. AFM images of surfaces: (a) AlN film (b) GaN film and (c) AlGaN film.](image)

According to spectral ellipsometry data, the AlN film has an excess concentration of Al atoms equal to 1.1%; its structure has no pores or voids. The average AlN film thickness is 1 µm. In the case of a GaN film, an analysis of the experimental dependence of the real and imaginary permittivity on the photon energy gives the following result: the total thickness of the grown GaN film is 1 µm, and the concentration of excess Ga in the film does not exceed 0.5%. The thickness of the AlGaN film cannot be accurately determined from spectral ellipsometry data, but according to the growth parameters and SEM data, it is approximately equal to 3 µm. The main thickness of the film has a concentration of Al and Ga atoms in a ratio of 1 to 4, respectively.

An analysis of the elastic deformation occurring at the initial stage of indentation showed that, in the case of AlN, GaN, and AlGaN films, the experimental dependences of the indentation force on the tip displacement are described by the Hertz ratio with Young’s modulus equal to 350±20, 230±20 and 200±20 GPa, respectively. Figure 3a shows these experimental dependences compared with the Hertz functions with the corresponding parameters. Usually, the elastic deformation is described in the form of the dependence of the average contact pressure on the depth of the tip contact with the test material; in this work, these dependences are shown in figure 3b.
Figure 3. Dependence of the indentation force on the displacement of the indenter (a); dependence of the mean contact pressure on the depth of the indenter contact with a sample (b).

The method of Oliver and Pharr in the case of analysis of the experimental data of elastoplastic nanoindentation to a depth of less than 100 nm shows that the hardness of the AlN, GaN, and AlGaN layers is 20±3, 18±2, and 22±3 GPa, respectively, and Young's modulus is 335±30, 280±40 and 190±30 GPa, respectively.

4. Conclusions

Thus, as a result of the studies carried out, the structural properties were studied and the mechanical characteristics of AlN, GaN, and AlGaN thin films formed by the HVPE method on SiC/Si hybrid substrates were determined. It is shown that the surface roughness of the AlN/SiC/Si, GaN/AlN/SiC/Si, and AlGaN/AlN/SiC/Si heterostructures is 11, 12, and 23 nm, respectively. The analysis of elastic deformation has been carried out, and it has shown that the value of Young's modulus of the AlN, GaN, and AlGaN layers is 350±20, 230±20 and 200±20 GPa, respectively. The Oliver and Pharr method was used to measure the hardness and Young's modulus of AlN, GaN, and AlGaN films in the case of elastic-plastic nanoindentation.

Acknowledgments

The work was carried out as a part of the project of the Russian Science Foundation no. 20-12-00193.

The studies were carried out using the equipment of the unique scientific setup “Physics, chemistry and mechanics of crystals and thin films” of the Institute for Problems in Mechanical Engineering of the Russian Academy of Sciences, St. Petersburg.

References

[1] Fischer-Cripps A C 2011 Springer Nanoindentation
[2] Kukushkin S A and Osipov A V 2008 Physics of the Solid State 50 1238–45
[3] Kukushkin S A and Osipov A V 2013 Journal of Applied Physics 113 024909
[4] Redkov A V et al. 2019 Physics of the Solid State 61 299–306
[5] Kukushkin S A et al. 2018 ECS Journal of Solid State Science and Technology 7 480–6
[6] Hertz H 1881 Journal Fur Die Reine Und Angewandte Mathematik 92 156–71