Strength and structural properties of AlN films grown on SiC/Si substrates synthesized by atomic substitution

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Abstract. In the present work, we studied the strength and structural characteristics of the layers of the AlN/SiC/Si heterostructure. The surface morphology of the AlN film and the SiC/Si substrate was studied using atomic force microscopy. The thickness of the AlN and SiC layers was determined by analyzing the data of ellipsometry. The hardness of the films and the substrate was measured by the method of nanoscratch testing. It was experimentally shown that the films under study have a high crystalline quality.

1. Introduction

In the papers [1,2], a unique method was proposed to produce a new type of silicon Si substrates with a single crystal layer of silicon carbide SiC. This method allows nanoscale assembling of the SiC crystal inside the Si matrix simultaneously over the entire area of the substrate. This process occurs due to the chemical reaction of a Si crystal with carbon monoxide CO:

\[ 2\text{Si}(cr) + \text{CO(gas)} = \text{SiC}(cr) + \text{SiO(gas)} \]

In this chemical reaction, a part of the Si atoms is substituted by carbon atoms C, and a nanoscale SiC layer is formed, as a result. Moreover, the SiC films grown in this way are practically unstressed, since the stress in the film relaxes because of a microporous structure that forms under the film during the synthesis [3]. With that, SiC films obtained by the method of substitution of atoms are low-defect, which makes them ideal substrates to grow wide-bandgap semiconductors, such as aluminium nitride AlN and gallium nitride GaN. In this work, we carried out a comprehensive study of structural and strength properties of the hybrid SiC/Si substrate and an AlN film grown on its basis.

2. Experimental methods

The substrate for the growth of the AlN film was synthesized by the method of substitution of atoms inside a silicon wafer of the KDB mark with the (111) crystallographic orientation without deviation from the base plane. The synthesis lasted for 20 minutes at the CO pressure of 0.6 Torr and a temperature of 1250 °C. Then the resulted substrate was cut into two parts. One of the parts was left in its original state, while on the other one, the AlN film was grown by the method of chloride-hydride vapor phase epitaxy (HVPE), according to the procedure described in [4]. Further, both samples were...
investigated identically. The structural properties of the surface of the SiC and AlN films were explored by atomic force microscopy using the Nanosurf EasyScan microscope. The surface morphology of the films was measured in the semicontact mode. The thickness and composition of the SiC and AlN films were analyzed by spectral ellipsometry using the Woollam M-2000D ellipsometer. The strength properties were studied by nanosclerometry on the MicroMaterials NanoTest 600 hardness tester. The nanosclerometry was carried out in a triple scan mode with a conical indenter with a radius of 25 μm. In this mode, the experiment follows three stages. At the first stage, the surface is scanned at the site of the future scratch, this is done to eliminate the influence of the natural inclination of the surface of a sample on the calculations. Then the scratching itself takes place, and, at the third stage, the residual deformation is scanned. The scanning of the surface topography and slope of the sample before and after scratching was carried out with a force of 0.1 mN. As a result of the analysis of the nanosclerometry data, the relative hardness was measured, which is determined as follows:

\[ H_s = H_{ref} \frac{F_s}{F_{ref}} \left( \frac{W_{ref}}{W_s} \right)^2 \]

where \( s \) and \( ref \) are indices related to the test and reference materials, respectively; \( H, F, \) and \( W \) are the hardness, applied force, and scratch width, respectively. The nanosclerometry method allows to estimate the strength properties of brittle materials, which is very important in the case of a microporous substrate.

3. Results

The Fig. 1a,b shows images of a surface of SiC and AlN films with an area of 10 μm², obtained by the atomic force microscope. It is clearly seen that the surface structure of both films is uniform. Morphological analysis of these images shows that the roughness is 22 and 25 nm for the SiC and AlN films, respectively.

The Fig. 2a,b shows the ellipsometric spectra, i.e. the dependences of the real and imaginary parts of the dielectric constant on the photon energy. The solid line is the real part of the dielectric constant, the dotted line is the imaginary part of the dielectric constant. The spectrum of the SiC film on Si is
well described by a two-layer theoretical model, while a single-layer ellipsometric model can’t make the theoretical and experimental curves fit each other. In our case, a layer of pure SiC with a thickness of 135 nm is separated from the layer of Si with voids by a layer of a mixture of SiC with Si having a thickness of 45 nm. Considering the AlN film, the analysis of the ellipsometry data shows that the thickness of the AlN film is 610 nm, it is single-layer and contains 1.5% excess of aluminium.

Figure 2(a, b). Ellipsometric spectra. (a) SiC/Si substrate and (b) AlN/SiC/Si heterostructure. The solid line is the real part, the dotted line is the imaginary part of dielectric permittivity.

The Fig. 3a shows the dependences of the applied normal load on the immersion depth during scratching, taking into account the natural inclination of the samples. The curves 1, 2, 3 correspond to the Si substrate, hybrid SiC/Si substrate, and AlN film.

Figure 3(a, b). (a) Dependence of the applied normal load on the immersion depth during scratching (a) curves 1, 2, 3 correspond to the Si substrate, the SiC/Si substrate and the AlN film; (b) dependencies of hardness on immersion depth, curves 4, 5 correspond to the SiC/Si substrate and the AlN film.

The Fig. 3b shows the dependences of the relative hardness on the immersion depth for the hybrid SiC/Si substrate (curve 4) and AlN film (curve 5) calculated relative to the hardness of the Si substrate, which is 11.3 GPa according to the nanoindentation data. In the case of the SiC film grown
on Si by the method of atomic substitution, it can be seen that at an immersion depth less than 130 nm, the hardness is almost constant and equal to 30.8 ± 2.3 GPa, which is only 7% lower than the hardness of bulk silicon carbide 33 GPa [5]. Then the hardness smoothly decreases to the value of 8.7 GPa, which is 22% less than the hardness of the calibration silicon substrate 11.3 GPa. In the case of the AlN film grown on the hybrid SiC/Si substrate, the hardness in the entire depth range is 20.5 ± 3.5 GPa. Note that, in contrast to nanoindentation [6, 7], the method of nanosclerometry made it possible to explore the strength characteristics of the film formed on the porous substrate over almost the entire thickness of the film.

4. Conclusions

The structural characteristics of an AlN film grown by the HVPE method on a hybrid SiC/Si substrate synthesized by the method of substitution of atoms have been studied. Atomic force microscopy data show that the surface structure of the AlN film and the SiC buffer layer is uniform, and their surface roughness is 22 and 25 nm, respectively. By the method of ellipsometry, it has been determined that the thickness of the AlN film is 610 nm, it is single-layer and contains 1.5% excess of aluminium. The relative hardness of the AlN film has been measured, its value is constant over the entire depth of the film and equal to 20.5 ± 3.5 GPa. Thus, it can be concluded that the AlN film grown on the SiC/Si hybrid substrate is homogeneous in its structural and strength properties, and stoichiometry of its chemical composition is close to ideal.

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