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Mechanical Properties of Cubic SiC, GaN and AlN Thin Films

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Abstract. Cubic polytypes of SiC, GaN and AlN were grown on silicon by molecular beam epitaxy. The mechanical properties of the epitaxial layers were investigated by nanoindentation. For 3C-SiC grown on Si(111) and Si(100) an extremal behaviour of the indentation modulus in dependence on the surface preparation with germanium prior to the carbonization was obtained. The maximum values of the indentation modulus for both Si orientations were achieved at 1 ML Ge.

Introduction

The knowledge and control of mechanical properties and residual stress are essential for micro- and nanoelectromechanical systems (MEMS and NEMS). MEMS and NEMS cover a wide application range with varying designs from cantilevers/bridges up to membranes. Due to their physical properties, SiC and group III-Nitrides, especially AlN, are preferable materials for MEMS and NEMS applications [1, 2]. Compared to non-piezoelectric materials, the piezoelectricity adds an additional energy dissipation path which might affect the vibrational properties and the quality factor of the MEMS device [3]. From this point of view, cubic modifications of these materials, as non piezoelectric materials, offer the advantage avoiding pronounced piezoelectric effects. The mechanical and dissipative properties are affected by the epitaxial layer quality, their dislocation densities and the residual stress. These quantities in turn depend on the substrate used and the growth technology. The mechanical properties of 3C-SiC are mainly studied on thick epitaxial layers, but advanced applications [4] require thin films exhibiting different mechanical properties [2]. In contrast to 3C-SiC, the growth of cubic III-nitrides on Si substrates is challenging and experimental studies of the mechanical properties of cubic III-N materials are rare [5] so that predominantly theoretical investigations can be found.

We report on the mechanical properties of cubic AlN and GaN grown by plasma assisted molecular beam epitaxial growth on Si(100) substrates, as well as on 3C-SiC grown on Ge modified Si(100) and Si(111).

Experimental

The III-nitride epitaxial growth was carried out on 3C-SiC/Si(100) templates. They were fabricated using UHV-CVD [6]. Reflection high energy electron diffraction (RHEED) of the (0,0) streak was used to control the growth of 3C-GaN(100) and 3C-AlN(100) on 3C-SiC(100)/Si(100) templates [7, 8]. The growth was realized under metal-rich conditions. The thickness of the 3C-AlN was between 100 and 200 nm, whereas the thicknesses of the 3C-GaN layers were 600 and 1000 nm.

The 3C-SiC(100) and 3C-SiC(111) heteroepitaxial layers were grown by solid source molecular beam epitaxy using a specific interface design technique based on the incorporation of Ge into the 3C-SiC/Si interface and into the near interface region [9]. The Ge precoverage was varied between 0 and 2 monolayers (ML) with respect to the silicon surface. The growth process was monitored by *in situ* RHEED and UV-Vis spectroscopic ellipsometry. The thickness of the epitaxial layers was between 100 and 200 nm.

Nanoindentation measurements were carried out to evaluate the mechanical properties of the cubic SiC, AlN and GaN epitaxial layers grown on silicon substrates. The experiments were performed using a Fischerscope® HM2000 equipped with a Vickers pyramid. The maximum load used in the nanoindentation experiments was 50 mN.

Results and Discussion

Measurement technique. In nanoindentation measurements the penetration depth should be equal to or less then one-tenth of the film thickness if the intrinsic properties of a thin film on a substrate are to be measured. For larger depths an effective composite modulus will be acquired, because with increasing penetration depth the indenter deforms not only the film, but also the substrate. It is therefore the value of the composite modulus which is obtained for larger penetration depth. This composite effect increases with increasing load and penetration depth. In addition to the standard load and unload measurement an alternative method can be used to test the mechanical properties of a composite material, here a thin epitaxial layer grown on a single crystalline substrate. The Enhanced Stiffness Procedure (ESP) uses a series of load and partial unload steps [10]. The measurement is carried out in a series of measurement cycles consisting of three steps. In the first step the load is increased to a certain value (pure load step). Subsequently, the load is reduced to a fraction of the maximum load (partial unload step). Afterwards, the load is increased to the previous maximum load and further increased to the next load value (pure load step). Within the measurement procedure each single entire load and partial unload step has a constant time step. Fig. 1 displays the dependence of the indentation depth on the load acquired during an ESP measurement cycle including all loading and unloading steps.

The obtained depth on load dependence was used to determine the Martens hardness and the composite modulus depth dependence of the heteroepitaxial layer – substrate system. An example is shown in Fig. 2. The indentation modulus was obtained for every reversal point of the indentation depth - load curve shown in Fig. 1, after the elastic modulus correction of the tip rounding [10]. The Martens hardness depth dependence was determined using the Martens hardness correction [10].

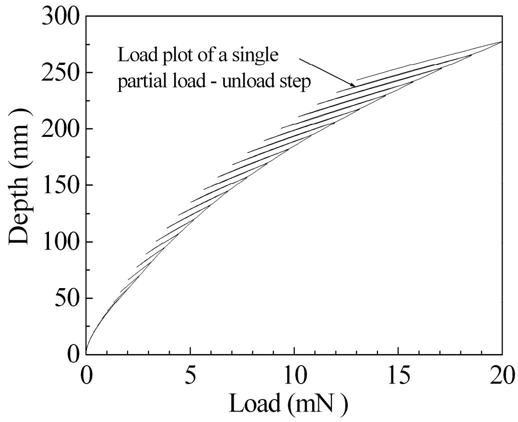

Fig.1 Indentation depth versus applied load of the ESP measurement procedure (163 nm 3C-SiC(100) on Si(100)).

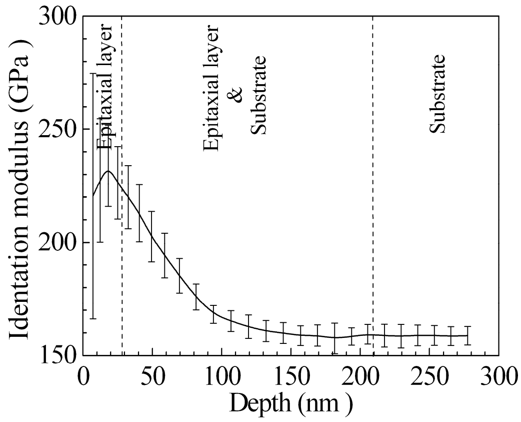

Fig.2 Depth dependence of the indentation modulus determined from indentation depth load curve (163 nm 3C-SiC(100) on Si(100)).

The composite modulus depth dependence shows three distinct regions. At a small indentation depth, below 20 % of the overall layer thickness, the indentation modulus is determined by the epitaxial layer. For intermediate depths a decrease of the indentation modulus from the layer to the

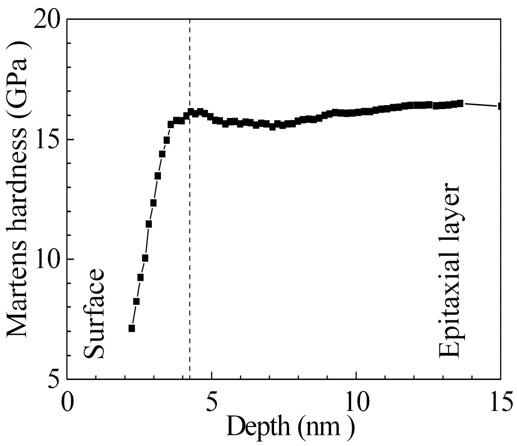

Fig. 3 Martens hardness versus depth for a 3C-SiC(111) layer grown on Si(111) for the depth region determined bv initial ESP measurements.

substrate values can be noticed. Thus, the epitaxial layer and the substrate contribute to the measured indentation modulus. This region extends into the substrate. In this case the indentation modulus or the hardness of the epitaxial layer can be determined using appropriate models for the composite hardness or modulus [11]. Investigations are underway to verify an appropriate model. At larger depth the elastic modulus is determined only by the properties of the substrate.

The ESP measurement procedure was used to identify the depth where the influence of the substrate material on the mechanical properties can be neglected. An example of the nanoindentation test is shown in Fig. 3, where the dependence of the Martens hardness on the indentation depth is given. It is evident that at depths exceeding 5 nm constant Martens hardness was obtained.

Measurement results. The results of the indentation tests for the 3C-SiC(100) on Si(100) and 3C-SiC(111) on Si(111) samples are shown in Figs. 4 and 5. In both cases the indentation modulus increases with increasing Ge coverage below 1ML Ge. At higher Ge coverages the indentation modulus decreases. The observed behaviour correlates with the dependencies of the quality factors and resonance frequencies of doubly clamped resonators fabricated from 3C-SiC heteroepitaxial layers grown on Ge modified Si(100) and Si(111) substrates [12, 13]. An increased indentation modulus can be caused by hardening due to grain boundaries and increased dislocation densities or by an improved material quality. In a previous study it was shown that the full width of the half maximum of the 3C-SiC(111) diffraction peak decreases and the terrace width increases if 3C-SiC is grown on Ge pretreated wafers [9]. Furthermore, the residual stress is reduced at Ge precoverages where the indentation modulus shows increased values [14-16]. Thus, the increased indentation modulus might not be caused by dislocation hardening, but could be related to an improvement of the crystalline structure of the 3C-SiC heteroepitaxial layers.

The results of the nanoindentation tests of 3C-GaN and 3C-AlN are summarized in Table 1. The values reported in Table 1 are averaged values, where at least five nanoindentation measurements were performed. The obtained values are in good agreement with the values obtained in [13] determined using doubly clamped resonator beams. The 3C-GaN(100) elastic modulus was found to be in the in the range between 235 and 250 MPa [13].

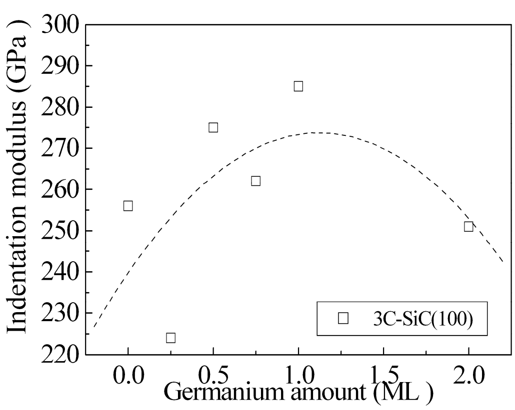

Fig. 4 Indentation modulus versus germanium precoverage prior to 3C-SiC(100) on Si(100) epitaxy. Dotted line represents the least square fit of the data.

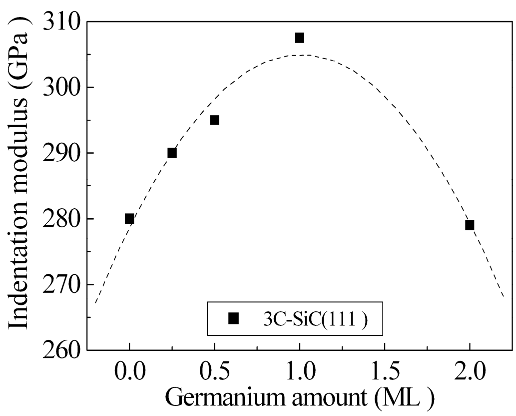

Fig. 5 Indentation modulus versus germanium precoverage prior to 3C-SiC(111) on Si(111) epitaxy. Dotted line represents the least square fit of the data

Table 1 Measured 3C-AlN and 3C-GaN mechanical properties

Material type	Substrate	Thickness [nm]	Hardness [GPa]	Indentation Modlus [GPa]	Compliance [μm/N]
3C-GaN(100)	3C-SiC(100)/Si(100)	600	11.3	220	48.3
3C-GaN(100)	3C-SiC(100)/Si(100)	1000	9.8	262	3.7
3C-AlN(100)	3C-SiC(100)/Si(100)	100	4.2	129	-
3C-AlN(100)	3C-SiC(100)	220	12.8	241	41.4
3C-AlN(111)	3C-SiC(111)/Si(111)	180	12.4	220	46.6

Summary

The mechanical properties of cubic SiC, AlN and GaN were determined using ESP. The obtained results correlate with data extracted from vibration frequencies of MEMS structures.

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