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Matching in-situ and ex-situ recorded stress gradients in an $Al_xGa_{1-x}N$ Heterostructure: Complementary wafer curvature analyses in time and space

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ABSTRACT

In-situ wafer curvature measurements and ex-situ ion-beam layer removal method are used to evaluate residual stress depth profiles in a 1.8 μm thick $Al_xGa_{1-x}N$ heterostructure on Si(111) by evaluating substrate surface curvatures and deflections of stepwise ion-beam milled micro-cantilevers, respectively. Both approaches reveal oscillatory stress depth gradients which correlate excellent in their depth alternations. Differences are found locally in the magnitudes of the stress concentrations, especially in the regions with relatively large stresses, inhomogeneous microstructures and at relatively small thicknesses. The discrepancies are interpreted by local stress relaxations in the growing heterostructure, like dislocation formation and overgrowth by differently stressed regions.

The knowledge of residual stress depth gradients in thin films is of great importance in all stages of their synthesis and applications. The gradients influence decisively mechanical and structural integrity of the films as well as the lifetime and functional parameters of the components [1]. There are numerous experimental techniques to assess the stress gradients ex-situ after film deposition, like X-ray diffraction, Raman spectroscopy, transmission electron microscopy and recently introduced ion beam layer removal (ILR) approach [2,3]. ILR is based on a stepwise focused ion beam (FIB) thinning of micro-cantilevers, which consist of a thin film and a certain portion of a substrate. The stress depth gradient is obtained using finite-element (FE) simulation considering the cantilever deflection changes after the ion milling steps as well as the film and substrate elastic constants. Remarkable features of ILR are (i) the need for the use of a monocrystalline substrate of known mechanical properties, which does not undergo plastic deformation during the FIB milling, and (ii) the fact that the characterization is performed ex-situ after the film deposition in scanning electron microscope (SEM). ILR studies were reported for polycrystalline, epitaxial as well as amorphous as-deposited thin films [4,5].

In-situ characterization of stress development during thin films deposition represents a complementary approach, which is based on in-situ monitoring of wafer curvature (WC) performed usually using optical or capacitance approaches [6]. WC allows assessing “direct” real time stress evolution as a function of time and the actual thin film thickness, whereby the stress magnitudes are evaluated using Stoney's equation and/or quasi-analytically. WC was applied to study fundamental processes in growing thin films like nucleation, island coalescence, the influence of sublayer sequences and surface diffusion [7–9]. WC cannot be used to resolve directly stress gradient changes occurring in already grown film and/or during cooling from deposition to room temperature [10]. Nevertheless among others, WC was extensively used to monitor strain and indirectly also microstructural developments during the growth of group-III-nitride heterostructures, especially GaN, on silicon [11]. Here, the large lattice mismatch of ~17% and the large mismatch of coefficients of thermal expansion (CTEs) of ~ − 56% between Si and GaN result in a generation of pronounced stress gradients, which may result in serious reliability issues [12].

It is obvious that a comparison of in-situ and ex-situ recorded residual stress profiles obtained using WC and ILR methods, respectively, possesses not only a methodological potential to verify the two approaches but could also open a way to analyze stress gradient evolution, which occurs at during film growth at high temperatures and while cooling from deposition to room temperature, e.g. as a result of various diffusion-driven processes, sublayer overgrowth and plastic deformation.

In this study, a residual stress gradient in a 1.8 μm thick $Al_xGa_{1-x}N$ heteroepitaxial structure is evaluated in-situ using WC during metallic-

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organic chemical vapor deposition on Si(111) substrate as well as ex-situ after the deposition using ILR. The aim is to compare both stress profiles, discuss the discrepancies as well as further potential for the application of both complementary approaches.

The investigated $Al_xGa_{1-x}N$ multilayer structure was grown in an Aixtron G5 planetary reactor by using trimethylaluminum (TMA), trimethylgallium (TMG) and ammonia (NH_3) as precursors for aluminum, gallium and nitrogen, respectively. After the reactor heating followed by the annealing and simultaneous degreasing of the Si substrate, a ~30 nm thick low temperature (LT)-AlN nucleation layer was grown at 993 °C. Subsequently the temperature was increased to 1100 °C and a ~120 nm thick high temperature (HT)-AlN layer was deposited. The following ~500 nm thick $Al_{0.25}Ga_{0.75}N$ transition layer was grown at 1086 °C. At the beginning of the ~1130 nm GaN buffer layer small temperature fluctuations levels off at 1100 °C in order to enable a steady layer growth. After the deposition of the final ~20 nm thick $Al_{0.22}Ga_{0.78}N$ barrier layer at 1083 °C, the reactor was cooled down to the room temperature. During the entire deposition process an integrated LAYTEC Epi TT system recorded the actual wafer bow as well as the surface temperature at the center of the wafer (Fig. 1a).

Complementary, the stress gradient in the as-deposited AlN/$Al_{0.25}Ga_{0.75}N$/GaN/$Al_{0.22}Ga_{0.78}N$ heterostructure was characterized using ILR ex-situ. The sample was taken from the same wafer position at which the WC data were collected (Fig. 1). The entire measurement was carried out in a Zeiss Auriga workstation, which combines a gallium operating FIB and a high resolution SEM. Fig. 2a shows the free standing micro-cantilever, which consists of the Si substrate and the $Al_xGa_{1-x}N$ heterostructure, after performing the ILR analysis. In this perspective overview image the top face shows the heterostructure cross-section and the front face belongs to the heterostructure surface. Owing to the residual stress within the heterostructure, the micro-cantilever is bent. During the ILR experiment, the cantilever was gradually thinned in steps of ~100 nm in section A (Fig. 2) and the cantilever deflection was evaluated after every milling step by comparing the mutual positions of two reference markers in section B (Fig. 2). At every milling step, the recorded cantilever deflection corresponds to the curvature of the section A, which depends on the “remaining” residual stress gradient in the heterostructure and the heterostructure thickness. Fig. 2b,c show the initial cantilever cross-section and the corresponding deflection (δ_1), whereas SEM images in Fig. 2d,e show sections A and B after the sixteen milling steps and the cantilever deflection δ_2. In order to avoid any significant sample beam damage, the FIB workstation was operated at a high voltage of 30 kV and relative low ion current of 50 pA during the entire experiment. Finally, the residual stress gradient $\sigma_{ILR}(z)$ as a function of the distance z to the Si substrate in the as-deposited heterostructure was evaluated from the recorded cantilever deflections and the remaining film thickness data by using a three dimensional finite element model (FEM). Schöngrundner et al. have studied the accuracy of the ILR method and found that the obtained stress profiles are usually slightly underestimating the stresses [5]. However, by considering the influences of boundary conditions, cantilever geometry, cantilever fabrication as well as the relatively large FIB milling step size of 100 nm, an absolute error smaller than 10% can be assumed for the presented ILR study [5,13].

Fig. 1 displays the in-situ measured wafer curvature (κ) and the actual deposition temperature (T_D) data as a function of process time (t). In this chart all important process stages are indicated, whereby the grey highlighted regions correspond to the applied changes in the deposition parameters, like the reactor temperature and the partial pressures of the precursors, which were changed between the individual sublayer growth periods. Consequently, the curvature changes within these ramping periods are mainly triggered by process adjustments rather than residual stress changes. The blue curve in Fig. 3 correlates the recorded wafer curvature κ to the actual film thickness h_f, which has been calculated under the assumption of a steady sublayer growth rate. At the beginning of the film growth (at $t \cong 2094$ s) the recorded curvature κ was nonzero and therefore the recorded value of ~13.5 km^{-1} was considered as an offset and subtracted from the entire $\kappa(h_f)$ dependence used for the further evaluation of stresses. Additionally it was assumed that no significant film growth and changes in the heterostructure thickness occurred within the grey highlighted ramping periods.

The magnitudes and the slopes of the $\kappa(h_f)$ dependence in Fig. 3 provide important indications on the stress nature and the stress development in the growing heterostructure. Whereby positive and negative $\kappa(h_f)$ magnitudes indicate the presence of overall tensile and compressive stresses in the growing heterostructure, respectively, positive and negative slopes $\partial\kappa(h_f)/\partial h_f$ correspond to the generation of tensile and compressive stresses at the actual stages of the heterostructure evolution, respectively [14].

Consequently at every process time t, the in-situ recorded curvature data $\kappa(t)$ (Fig. 1) were used to evaluate the actual average residual stresses $\langle\sigma(h_f)\rangle$ as a function of the actual film thickness h_f using the Stoney's equation as follows [6].

$$\langle\sigma(h_f)\rangle = \frac{1}{h_f(t)} \frac{M_s h_s^2}{6} \kappa(t) \tag{1}$$

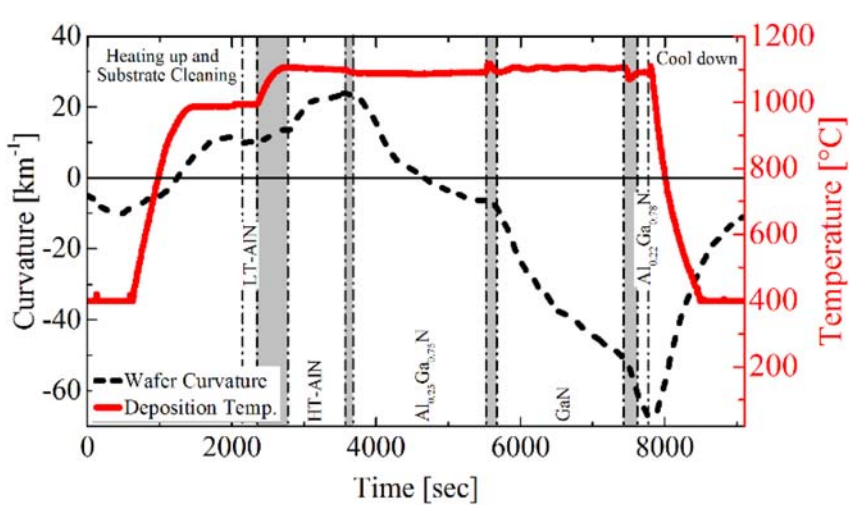

Fig. 1. In-situ recorded wafer curvature (dashed line) and temperature (solid line) data collected during the heterostructure deposition. The grey regions indicate time intervals of changing growth parameters like precursors, composition as well as reactor pressure and temperature.