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Evaluation of Strain in 3C-SiC/Si Epiwafers from X-Ray Diffraction Measurements

Marcin Zielinski[1,a*], Marc Bussel[1,b], Hugues Mank[1,c], Sylvain Monnoye[1,d]
Marc Portail[2,e], Adrien Michon[2,f], Yvon Cordier[2,g],
Viviana Scuderi[3,h], Francesco La Via[3,i]

[1]NOVASiC; Savoie Technolac; 73375 Le Bourget du Lac cedex; France

[2]CRHEA, CNRS-UPR10, Université Côte d'Azur, rue Bernard Grégory, 06560 Valbonne, France

[3]IMM-CNR, VIII Strada, 5, 95121 Catania, Italy

[a]mzielinski@novasic.com, [b]mbussel@novasic.com, [c]hmank@novasic.com,
[d]smonnoye@novasic.com, [e]mpo@crhea.cnrs.fr, [f]am@crhea.cnrs.fr, [g]yc@crhea.cnrs.fr,
[h]viviana.scuderi@imm.cnr.it, [i]francesco.lavia@imm.cnr.it

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Abstract. X-Ray diffraction measurements of lattice parameter were performed for (111) and (100) oriented 3C-SiC/Si epiwafers. Strain of 3C-SiC epilayer and Si substrate were estimated and the result was compared with routine wafer deformation measurements. An unexpected discrepancy was observed between XRD and curvature measurements for (100) oriented samples.

Introduction

The cubic silicon carbide (3C-SiC) layers on silicon (or silicon-based) substrates are considered as an interesting material for template applications (for nitride, diamond or graphene growth) and for sensor/MEMS fabrication purposes. The problem of stress control within the 3C-SiC film is crucial for both families of applications.

On one hand, the planarity of the 3C-SiC/Si epiwafer has to be preserved to insure further processing. This planarity supposed to be directly influenced by the mean residual stress within the 3C-SiC film. From this point of view, the presence of stress can easily become detrimental for the material. On the other hand, for multiple MEMS resonators, the existance of stress within the film improves the quality factor of the resonance vibration – presence of stress turns out to be desirable. As for the stress gradient, not important from the point of view of "template" application, it may be responsible for buckling/bending of self-supported microstructures which are the components of MEMS device.

In the past, several investigations were performed to quantify the stress/strain in 3C-SiC/Si system, some of them by the co-authors of this contribution. This includes the studies based on epiwafer / cantilever curvature demonstrating the correlations between the growth conditions and final wafer / beam deformation (see [1] and refs inside). The dependence of system deformation on stress distribution can be quantitatively described, as demonstrated in [2, 3].

European project PicoGEO offered a unique opportunity to reexamine previous experimental results and proposed models by applying various stress/strain determination approaches on the same samples. Thus, routine wafer deformation measurements were completed by strain gauge approach [4], plain view and cross-section Raman measurements [5] and X-ray diffraction (XRD) determination of lattice parameters.

The present contribution, focalized on determination of lattice strain from XRD measurements is supposed to bring an additional brick to the understanding of complex strain/stress behavior in 3C-SiC/Si (100) and (111) system.

Experimental Details

CVD setup and sample growth. 3C-SiC thin films were deposited in previously described horizontal, low pressure, resistively heated hot wall CVD system with rotating sample holder [6]. Standard chemistry was used with purified hydrogen (H_2) as carrier gas, high purity (5N) silane (SiH_4) and propane (C_3H_8) as principal precursors. Controlled epilayer doping was performed using Nitrogen (N_2) for n-type and Tri-Methyl-Aluminum (TMA) as Al source for p-type 3C-SiC films. Finally, hydrogen chloride (HCl) diluted in H_2 was introduced to CVD process to boost the growth rate without increasing the surface defects density and allow fine adjustment of Al incorporation in p-type films [7]. Classic 3C-SiC growth approach was applied, with Si substrate annealing under H_2, Si carbonization under C_3H_8/H_2 mixture to create oriented 3C-SiC seed and CVD thickening of 3C-SiC layer.

3C-SiC film characteristics. It is well known that the Full Width at Half Maximum (FWHM) of X-Ray Diffraction (XRD) 3C-SiC peaks increases with reducing the film thickness (detailed data for NOVASiC CVD setup are given in [8]). The peak broadening concerns mainly ω-scans (rocking curves) but affects also 2θ-ω scans used for lattice parameter determination. In order to allow precise enough determination of lattice parameter, we focalized in this study on relatively thick 3C-SiC films with well-defined XRD peaks: 3C-SiC films with thickness up to 20μm were grown on Si(100) substrates. To quantify the influence of doping on lattice parameter, samples were non-intentionally doped (NID, dopant incorporation below $10^{16}cm^{-3}$), strongly n-type doped ([N]>$10^{19}cm^{-3}$) or strongly p-type doped ([Al]> $10^{19}cm^{-3}$).

It would be extremely interesting to prepare equivalent 3C-SiC/Si(111) sample set and allow a complete comparison between two orientations. However, we have to remind that in 3C-SiC/Si(111) an extremely strong concave bow is observed even for very thin 3C-SiC films (<1μm). Also, the critical thickness of crack formation is in the range 0.5-1μm for the films grown in NOVASiC setup. The preparation of thick layers is complicated – the strong concave bowing appears during the deposition process, leads potentially to perturbation of gas flow near the sample surface and surface defect creation. Wafer breaking during growth / cooling down was also observed. Finally, strong curvature of the epiwafer affects the precision of XRD measurements, due to peak broadening and difficulty of precise sample surface positioning with respect to X-ray beam. Consequently, we decided to perform only punctual verification of lattice parameter in 3C-SiC/Si(111): 3μm and 8μm thick 3C-SiC(111) layers, cracked, were prepared for the needs of the study.

Characterizations. Epiwafer deformation profiles were measured using optical microscope with x-y-z calibrated stage and automatic measurement of z position with ±1μm precision.

XRD measurements (2θ-ω scans of various basal plane and out of plane reflections) were performed in triple axis configuration using a Panalytical X'Pert Pro MRD diffractometer equipped with a Cu X-ray source emitting at the wavelength Kα1 of copper (0.15406 nm) and with a Ge(220) crystal monochromator and analyzer. In plane and out of plane lattice parameter were measured for 3C-SiC film, front-side of underlying Si substrate and backside of Si substrate. Measured values were compared with reference result, obtained on a fully relaxed bulk 3C-SiC crystal, grown by PVT in FAU, Erlangen [9] and commercial bulk Si wafer.

Strain Evaluation

We suppose that under the effect of biaxial stress, cubic (100) lattice becomes tetragonal, with different in-plane (a) and out of plane (c) lattice parameter. For (111) orientation, the cubic lattice under stress turns into rhombohedral one, characterized by rhombohedron dimension (a) and side angle (α). Side angles higher than 90° correspond to in-plane extension of the lattice (biaxial tension) and contraction of out of plane lattice dimension.

To allow an easy and straightforward comparison of the results obtained on (100) and (111) samples we propose the following approach. Experimentally determined position of 2θ-ω peak for (hkl) plane family allows to assess the interplanar spacing d_{hkl} directly from Bragg's law. For each

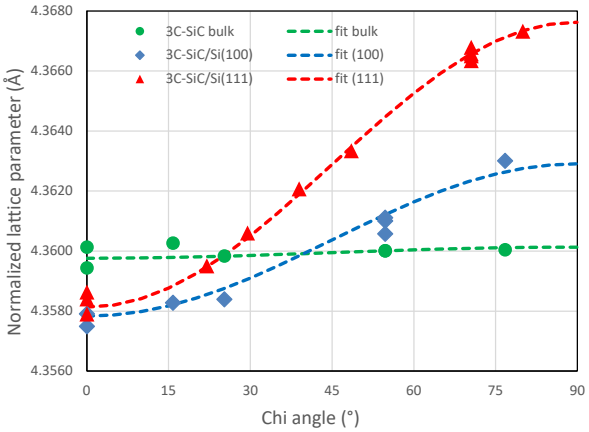

Figure 1. Example of evolution of 3C-SiC normalized lattice parameter with angle with χ angle, for bulk 3C-SiC, 3C-SiC/Si(100) and 3C-SiC/Si(111)

plane family (hkl) we introduce a "normalized lattice parameter", a_{hkl}, calculated from interplanar spacing using the simple formula:

$$a_{hkl} = d_{hkl} \cdot \sqrt{h^2 + k^2 + l^2} \tag{1}$$

Obviously, for relaxed, cubic lattice, same a_{hkl} value is found for any (hkl) plane, equal to cubic lattice parameter a. For the strained lattice, a_{hkl} will depend on χ_{hkl} angle between the (hkl) plane and basal plane of the sample ((001) or (111)):

$$a_{hkl} = \sqrt{\left(a_{\perp} \cdot cos(\chi_{hkl})\right)^2 + \left(a_{\parallel} \cdot sin(\chi_{hkl})\right)^2} \tag{2}$$

Here, $a_{\perp}$ is the normalized lattice parameter along the growth direction and $a_{\parallel}$ - within the basal plane. For (100) samples with tetragonal lattice deformation, $a_{\perp}$ and $a_{\parallel}$ are equivalent to tetragonal lattice parameters c and a, respectively. For given sample, the values of $a_{\perp}$ and $a_{\parallel}$ can be determined by fitting the experimental dependence $a_{hkl}(\chi_{hkl})$.

Finally, the strain of the lattice can be estimated from $a_{\perp}$ and $a_{\parallel}$ as

$$\varepsilon = (a_{\parallel} - a_{\perp})/(a_{\parallel} + a_{\perp}) \tag{3}$$

Experimental Results

Typical XRD result. Figure 1 shows the normalized lattice parameter evolution with angle χ for three samples: bulk 3C-SiC(100) crystal from Ref. [9], 10µm thick 3C-SiC film on Si(100) and 3µm thick 3C-SiC film on Si(111). The 3C-SiC/Si(100) epiwafer presents slightly concave bow (-70µm). The bow of 3C-SiC/Si(111) wafer is also concave but much more pronounced: above -1mm. The table attached to the figure shows the values of $a_{\perp}$, $a_{\parallel}$ and ε deduced for each sample from XRD measurement. The bulk 3C-SiC sample is almost completely relaxed while both 3C-SiC epilayers present a tensile strain ($a_{\parallel} > a_{\perp}$). Strain is more important in the case of (111) oriented sample.

Case of convex 3C-SiC/Si(100) epiwafers. In the past contributions we demonstrated that by adjusting the growth conditions (temperature, growth rate and growth duration) we may control the shape (concave / flat / convex) and (to some extent) the value of final bow of 3C-SiC/Si(100) epiwafers. We also established the correlation between 3C-SiC doping and final epiwafer shape: strong Al doping lead systematically to concave deformation while strong N doping resulted in convex shape of the wafer.

One would assume that XRD strain analysis performed on convex 3C-SiC/Si(100) epiwafer should result in determination of compressive strain of the epilayer ($a_{\parallel} < a_{\perp}$). The experimental result is

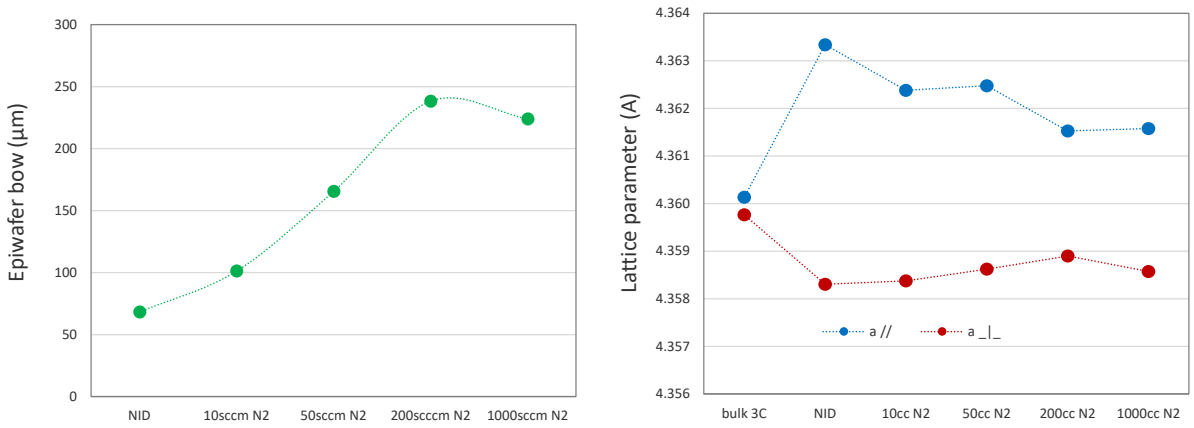

Figure 2. (a) Bow of 3C-SiC/Si(100) epiwafers with various nitrogen doping of 3C-SiC epilayer. (b) in-plane and out-of-plane lattice parameter of these epiwafers.

unexpected – despite the convex epiwafer shape, we observe tensile strain of 3C-SiC epilayer. Figure 2a shows the bow of a series of epiwafers with 3.6µm thick 3C-SiC film grown on 525µm Si (100mm diameter) under different N_2 flow conditions (0, 10, 50, 200, 1000 sccm). Nitrogen incorporation varies between few $10^{17}cm^{-3}$ (10sccm) and few $10^{19}cm^{-3}$ (1000sccm). Convex bow up to 240µm is observed. According to Stoney's formula it should correspond to a compressive stress close to 440MPa and in-plane lattice strain close to ~ -1×10^{-3}. In Figure 2b, we show the result of lattice parameter XRD measurements on the same samples: the in-plane lattice parameter is systematically larger than out of plane, indicating that 3C-SiC is under tension. As the nitrogen incorporation increases, the tensile strain is slightly reducing.

Leaving aside the lack of coherence between the XRD (tension) and bow (convex) results, we wanted to verify the origins of convex wafer deformation: is it an 3C-SiC/Si interface related effect or is it due to the presence of nitrogen in the volume of the epilayer. For this purpose, we prepared a series of thick, strongly doped films. Each epilayer was composed of 0.5µm thick buffer (NID or N-doped) and 10µm thick drift layer (NID or N-doped). For NID and N-doped layers, the nitrogen incorporation was respectively $[N] < 10^{16}$ cm^{-3} and $[N] > 10^{19}$ cm^{-3}.

For both samples with 10µm thick NID drift layer slightly concave bow (~ -50µm) was observed, independently on buffer doping (NID or N). Similarly, the convex bow (~ 400µm) of samples with N-doped drift layer was not affected by the doping of the buffer. We conclude from this cross-test that the convex bowing of 3C-SiC:N/Si(100) epiwafers is related to the presence of nitrogen in the volume of the 3C-SiC film.

Strain of underlying Si(100) substrate. We performed also the measurements of the silicon lattice parameter on both sides of Si substrate. An example of results is presented in **Figure 5a**. The top-side of silicon (near the 3C-SiC/Si interface) is under slight compressive strain $-1 \times 10^{-4} < \varepsilon < 0$. On the back side of the Si substrate slight tension is found (obviously, we have to keep in mind that observed Si peak shift is very small with respect to the diffractometer resolution so this experimental observation should be considered as qualitative only). Such behavior is expected for the substrate under tensely stressed film [2, 3], but remains inconsistent with the convex epiwafer bow observed in several samples.

Conclusion

The evaluation of lattice strain from XRD measurements of 3C-SiC/Si(111) epiwafers give the results that remain coherent with routine wafer deformation measurements: tensile strain of 3C-SiC(111) epilayer is observed, in agreement with strongly concave epiwafer shape. Same protocol applied to (100) oriented epiwafers gives an unexpected result: it turns out from present contribution that in 3C-SiC(100) epilayers on Si, the in-plane lattice parameter is systematically larger than out of plane parameter indicating that the 3C-SiC is under tension, independently on the shape (convex or

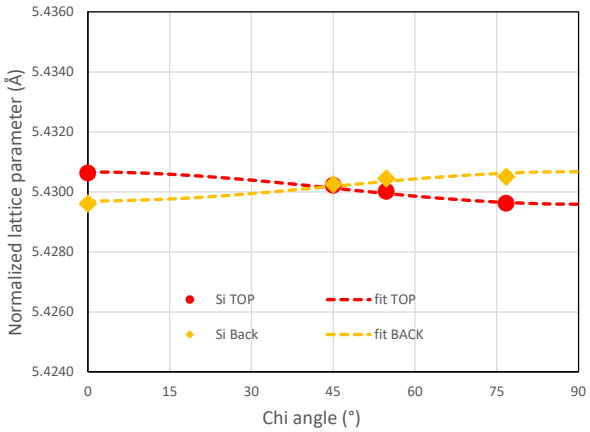

Figure 3. Evolution with χ angle of normalized lattice parameter of Si on top side and back side of silicon substrate indicating, respectively, compressive and tensile strain of the Si lattice.

concave) of 3C-SiC/Si(100) epiwafer. Such observation suggests high importance of creep effects during 3C-SiC/Si growth, which should be the key to explain the apparent lack of coherence between wafer curvature and XRD measurements. We expect putting more light on these effects by performing Raman measurements on cross-section of the samples in order to reveal the profiles of the stress across the epilayer thickness.

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