

## Designing SiC Based CMUT Structures: An Original Approach and Related Material Issues

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**Abstract.** We present an epitaxy-based approach for realizing a 3C-SiC Capacitive Micromachined Ultrasonic Transducer (CMUT). The design requires to grow a 3C-SiC/Si/3C-SiC heterostructure on a Si substrate. This requires different growth steps of SiC on Si and Si on SiC. We present some specific growth related issues, namely the control of selectively grown Si on a masked SiC(100) layer and the further regrowth of 3C-SiC on a Si(110) layer. The final release of the SiC membrane, to define a CMUT, is also presented using a simple thermal treatment to reduce the total number of technological steps.

### Introduction

Capacitive Ultrasonic Micromachined Transducers (CMUTs) are a family of MEMS devices widely used for medical ultrasonic imaging and non-destructive sensing (biology, gas industry, etc.). Their base principle relies on the monitoring of capacitance changes between two parallel electrodes, one of them being suspended above a sealed cavity [1]. If such a device is widely used for consumer applications, the development of SiC-based sensors could address a growing demand for applications in harsh environments (nuclear, space, etc...), according to physical and chemical properties comparing very favorably to commonly used materials (PZT, silicon) in terms of higher chemical inertness and hardness to radiation. Thus, we propose in this work to consider a 3C-SiC/Si/3C-SiC heterostructure on a Si(100) substrate as a base platform for designing a SiC-based CMUT structure. We discuss presently some of the different issues encountered in the fabrication of such an heterostructure, using Chemical Vapor Deposition (CVD) for targeting the formation of crystalline epilayers, whose utilization is expected to improve device performances according to better mechanical properties than amorphous forms.

### Experimental Details

**1. Targeted heterostructure.** Fig. 1 summarizes the different steps, developed in our lab, to achieve a 3C-SiC layer forming a suspended membrane, separated from an underlying 3C-SiC base layer by an insulating film (final step ⑥). The whole process requires both epitaxial and technological steps. We presently only focus on the epitaxial steps ④ and ⑤ according to their special interest in terms of epitaxial growth. The achievement of step ⑥, based on a thermal approach, will be also discussed.

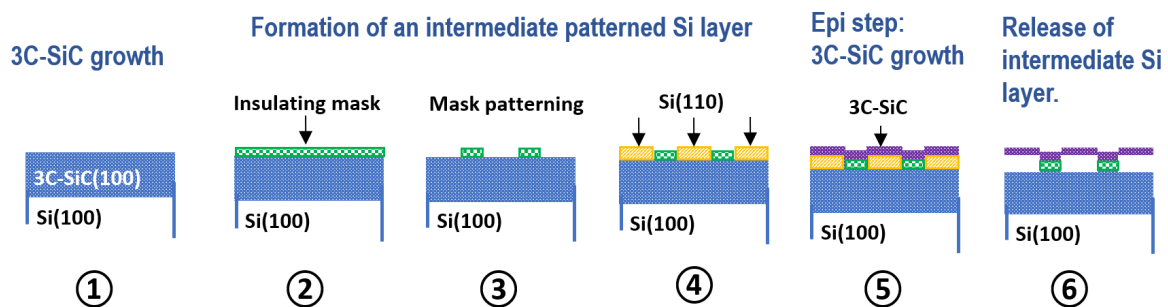
**2. CVD growth steps.** Both SiC and Si epilayers have been grown in a home-made resistively heated hot wall horizontal SiC reactor, using  $C_3H_8$  and  $SiH_4$  for C and Si precursors, diluted in  $H_2$ . In addition to precursors, hydrogen chloride (HCl) has been used for step ④.

In step ①, a 3C-SiC(100) layer is grown on 2 inch Si(100) using a “classical” two stages process (carbonization under  $C_3H_8$  during 10min at 1150°C followed by an epitaxial plateau at 1350°C, 200mbar, C/Si=1.2) with a typical thickness close to 5 $\mu$ m. See [2] for a review on SiC growth on Si.

Steps ② to ④ form a key sequence to obtain a patterned surface with silicon mesas separated by an insulating material, formed in steps ② and ③ and detailed after. Step ④ is a subsequent growth step, required to selectively grow Si on the masked 3C-SiC(100) surface. We have already reported about the growth of Si on non-patterned 3C-SiC(100) surfaces, with a single step process using  $SiH_4$  diluted in  $H_2$  at 950°C, 900mbar [3]. Presently, we assume that the selective character of the growth can be triggered by the addition of chlorine in the gas phase. Taking as base process that developed on non-patterned 3C-SiC(100) surfaces, we added HCl to  $SiH_4$  with Cl/Si ratio equal to 0 (no HCl), 0.4, 0.8, 1 and 2. Different growth temperatures have been explored, ranging from 850°C to 1025°C.

Step ⑤ requires the grow of SiC on a patterned Si(110) surface. Few papers in the literature address specifically the control of the final orientation of SiC grown on Si(110) [4,5] but it is known that the growth of SiC on Si(110) surface can lead to different SiC orientations. Presently, we focus on the role of the introduction temperature of the propane during the thermal ramp up prior to the epitaxial stage. This step is composed of a 5-minute annealing under hydrogen at 1100°C, 200mbar, followed by a short cooling down to 800°C; heating is then turned on again and  $C_3H_8$  is introduced during the temperature ramp-up precisely at a given temperature ranging from 850 to 950°C and flows up to the epitaxial temperature (1320°C). Comparative growths have also been realised on Si(110) substrate for benchmarking on a “defect free” surface.

**3. Insulating mask and patterning.**  $SiO_2$  has been employed as an insulating mask. 200nm thick  $SiO_2$  films have been deposited using cathodic sputtering (②). Following deposition, formation of a micrometer-sized patterning, compatible with a CMUT design, has been realized using photolithography, etching (ICP process using  $C_4F_8$  as etchants), and lift-off process for step ③.

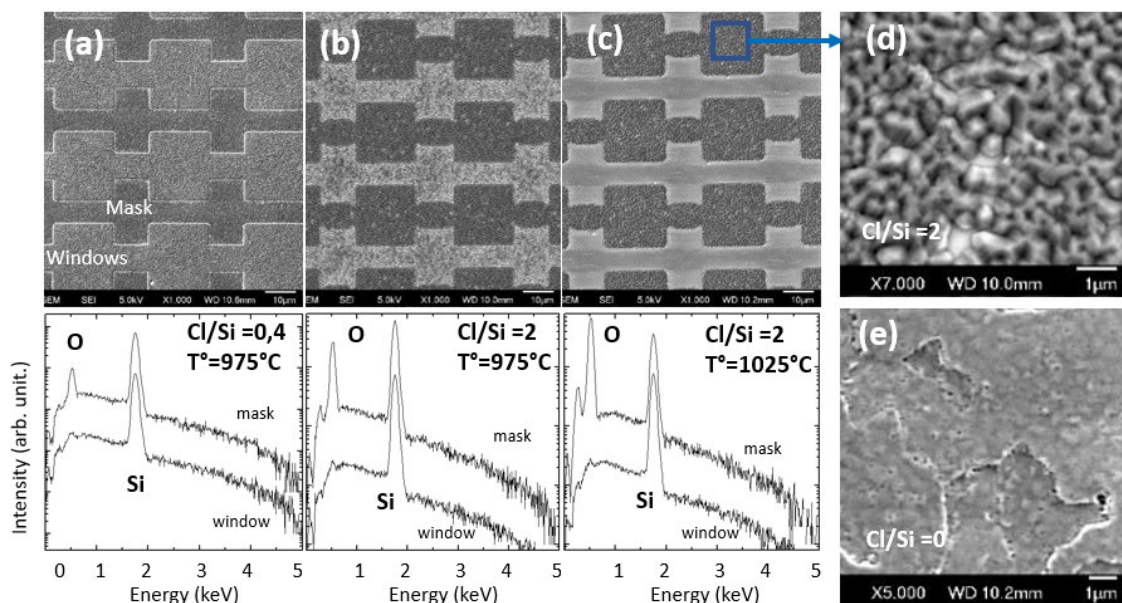


**Figure 1.** Schematic workplan for SiC/Si/SiC/Si heterostructure elaboration, base structure of a CMUT device.

## Results and Discussion

**1. Selective growth of silicon.** Fig.2a-c shows Scanning Electron Microscope (SEM) micrographs recorded after Si growth on a  $SiO_2$  masked 3C-SiC(100) surface (step ) under different conditions. The 20x20 $\mu$ m<sup>2</sup> square shaped structures, linked by rectangular areas, observed on the SEM images correspond to the initial 3C-SiC(100) windows. Areas between these square shaped zones correspond to the initial  $SiO_2$  mask. In addition to the SEM morphological observations, Energy Dispersive Xray Spectroscopy (EDS) has been used to characterize the chemical nature of the surface coverage. EDS spectra have been systematically acquired for all samples on both “mask” and “window” zones (Fig.2). An evolution of the morphology is observed. On the initial SiC windows, the surface presents a quite rough character independent on the Cl/Si or growth  $T^\circ$ . A higher magnified image of such a surface is presented in Fig.2d where small grains can be seen. Significant changes were noticed on the masked regions according to the growth conditions. At “low” Cl/Si ratio and growth  $T^\circ$  values

(Fig2.a) the masked regions present a rough character which totally disappear for the highest Cl/Si value we used and for higher temperatures (Fig2.c). In the same time, EDS measurements indicates the presence of a Si film on the mask in the former case, whereas only stoichiometric SiO<sub>2</sub> (checked by performing quantitative analysis – not shown) is measured in the last one.

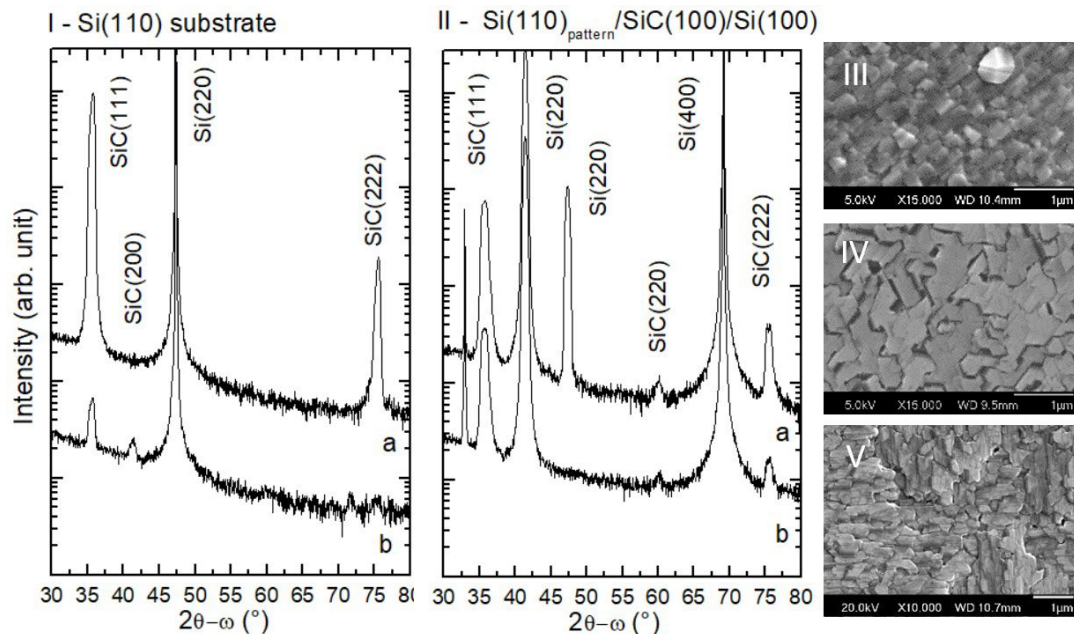


**Figure 2.** SEM images recorded on SiO<sub>2</sub> masked 3C-SiC(100) surface after Si growth under different conditions. EDS spectra recorded both on mask and window regions. (d)-(e) SEM on chlorinated - non-chlorinated growth respectively.

This clearly reveals a selective growth character for the last case. Selectivity has been widely discussed and documented for the growth of Si on Si substrate [6] but, to the best of our knowledge, no publication has reported on this mechanism for Si on 3C-SiC. According to the large dependence of the selective character on the experimental parameters for the Si on Si case (pressure, chemistry employed for the growth, geometry of the patterns...) our results cannot definitely state the most critical parameters for achieving the selectivity. However, the importance of both high Cl/Si ratio and high growth temperature for favouring the selectivity with a SiO<sub>2</sub> mask is presently demonstrated. Another point of interest is related to the effect of HCl addition on the morphology of the grown Si film. As illustrated in Fig.2d-e, a clearly different morphology is obtained according to whether the process is chlorinated or not. For a non-chlorinated growth (Fig.2e), a quite smooth Si film is developing on the SiC(100) surface, characterized by the presence of anti-phase domains as already reported elsewhere [3]. However, the addition of HCl lead to a strongly faceted Si film (Fig.2d). This has been observed even for the non-null lowest Cl/Si values used (0.4).

**2. Growth of SiC on Si(110).** Fig.3 presents X-rays diffraction (XRD) spectra recorded both on reference Si(110) substrates (I) and on patterned Si(110)/3C-SiC(100)/Si(100) heterostructures (II) after ~ 100nm growth of SiC. On Si(110) substrate, a comparison is made between samples grown with propane introduced at 870°C (a) and 930°C (b). It can be seen that the SiC film grown with the 870°C introduction is clearly (111) oriented, whereas both SiC(111) and SiC(200) diffraction lines are observed for 930°C introduction, revealing a polycrystalline nature. The same growth process was applied to patterned Si(110)/3C-SiC(100)/Si(100) heterostructures, with a 870°C propane introduction. For that case, SiC(111) diffraction is clearly observed but we notice a weak SiC(220) diffraction (Fig.3-II-a), attesting to a lower degree of organization than on the Si substrate. Fig.3 also presents the typical surface morphology observed for the different samples. The sample grown with propane introduction at 870°C shows a regular 3-fold symmetry which can be related to twinned domains according to the possibility for 3C-SiC(111) to grow on a Si(110) surface with two equivalent *in plane* positioning [7]. A higher roughness is observed after growth with propane introduction at a higher temperature, as well as in the case of growth on the thin Si(110) layer formed on the 3C-SiC(100)/Si(100) heterostructure. In the last case, this could be attributed to the lower

quality of the Si grown film, favouring the development of misoriented domains, with respect to the very high quality of the Si substrate. We previously assumed that surface roughness could trigger the final SiC crystalline orientation [8]. The present results also show that both temperature of  $C_3H_8$  introduction and quality of the initial Si(110) impact the SiC regrowth.



**Figure 3.** XRD (I) SiC on Si substrate: (a) intro 870°C, (b) intro 930°C; (II) SiC on heterostructure: (a) non-annealed, (b) annealed; SEM on  $C_3H_8$  intro 930°C surface (III),  $C_3H_8$  intro 870°C (IV), heterostructure (V).

**3. Thermal annealing.** Finally, the last issue presently addressed is the way of achieving a silicon dissolution for creating a SiC suspended membrane on a cavity (step ⑥). In order to avoid a chemical approach requiring different processing steps (opening holes in the uppermost part of SiC, dissolving Si and closing the holes), a thermal annealing at 1200°C under  $H_2$  during 30-60minutes at 200mbar has been performed. According to the relative low thickness of SiC, the suppression of the intermediate Si has been obtained by this method, as confirmed by SEM (not shown) and XRD where Si(220) diffraction can be suppressed after such annealing (Fig. 3-II-b). SEM performed in cross section configuration also tends to reveal that this suppression mechanism does not lead to a membrane thickening and let's assumes that Si removal could preferentially be due to the formation of  $SiH_x$  volatile species able to diffuse throughout the defective thin SiC layer and be released in the gas phase. The point is of importance for two reasons: (i) the thermal annealing is efficient to achieve a suspended SiC membrane, but (ii) it shows that such membrane is featured by a porosity due to defects, requiring to further address this potentially problematic point.

## Conclusion

We present some epitaxy-related issues for an ongoing research project aiming to develop crystalline SiC-Si stacked heterostructures for CMUT fabrication. We emphasized some steps which can be helpful for a better understanding of mechanisms encountered during the different steps of the heterostructure elaboration.

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**References**

- [1] A. S. Ergun, G. G. Yaralioglu, B. T. Khuri-Yakub, J. Aerosp. Eng., 16, pp 76-84 (2003)
- [2] G. Ferro, Crit. Rev. in Sol. State and Rel. Mat., 40, pp 56-76 (2015)
- [3] R. Khazaka, M. Portail, P. Vennéguès, D. Alquier, J-F. Michaud, Acta Materialia 98, pp336-342 (2015)
- [4] T. Nishiguchi, M. Nakamura, K. Nishio, T. Isshiki, S. Nishino, Appl. Phys. Lett. 84, pp3082-3084 (2004)
- [5] S. Sambonsuge, L. N. Nikitina, Y. Y. Hervieu, M. Suemitsu, S. N. Filimonov, Russ. Phys. J. 56, pp1439-1444 (2013)
- [6] M. R. Goulding, Mat. Sci. Eng. B 17, pp47-67 (1993)
- [7] T. Nishiguchi, *et al.*, Mat. Sci. Forum 483-485, pp193-196 (2005)
- [8] R. Khazaka, J-F. Michaud, P. Vennéguès, L. Nguyen, D. Alquier, M. Portail. J. Appl. Phys. 120, 185306 (2016)