

3C-SiC Heteroepitaxial Layers Grown on Silicon Substrates with Various Orientations

FERRO Gabriel^{1,a*}, YEGHOYAN Taguhi^{1,b}, CAUWET François^{1,c},
COINDEAU Stéphane^{2,d}, ENCINAS Thierry^{2,e} and SOULIERE Véronique^{1,f}

¹Laboratoire des Multimatériaux et Interfaces, UMR CNRS 5615, 6 rue Victor Grignard, Lyon 1 University, 69622 Villeurbanne (France)

²CMTC – SIMaP, Université Grenoble-Alpes, 1260 rue de la piscine, 38402 Saint Martin d'Hères (France)

^{a*}gabriel.ferro@univ-lyon1.fr, ^btaguhi.yeghoyan@gmail.com, ^cfrancois.cauwet@univ-lyon1.fr,
^dstephane.coindeau@grenoble-inp.fr, ^ethierry.encinas@grenoble-inp.fr,
^fveronique.souliere@univ-lyon1.fr

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Abstract. This work investigates the 3C-SiC heteroepitaxial growth on silicon substrates having a wide variety of orientations, i.e. (100) on axis and 2°off, (111), (110), (211), (311), (331), (510), (553) and (995). All the 3C-SiC layers were grown using the same two-step CVD process with a growth rate of 2 µm/h. According to X-ray diffraction characterizations, direct heteroepitaxy (layer having exactly the same orientation as the substrate) was successful on most of the Si substrates except for (110) one which was the only orientation leading to obvious polycrystalline deposit. Each layer led to a specific surface morphology, the smoothest being the ones grown on Si(100)2°off, and (995) substrates. None of these layers cracked upon cooling though those grown on Si(111), (211) and (553) substrates were highly bowed.

Introduction

Despite decades of research and hundreds of published articles, the 3C-SiC heteroepitaxially grown on silicon substrate has still not reached sufficient crystalline quality for industrial use in electronics. The growths are usually performed with the (100) or (111) orientation of the Si substrate, both generating their own additional difficulties such as antiphase domains generation for the former case or cracks for the latter case. The use of other substrate orientations, such as (110) and (211), was scarcely investigated despite interesting features in terms of defects density reduction [1, 2], surface morphology [3, 4] or stress reduction in [111] oriented films [4-6]. From these studies, it seems that the optimal growth conditions could be specific to each orientation which makes the investigations trickier. In addition, the use of higher Miller's index substrate orientations were never reported so far for 3C-SiC heteroepitaxial growth. There is thus still room for purely exploratory work on the effect of Si substrate crystalline orientation on the resulting 3C-SiC layers. This is done in the present work.

Experimental

The layers were deposited by atmospheric pressure chemical vapour deposition (CVD) using SiH₄/C₃H₈/H₂ gas mixture. The Si substrates were pieces of ~1 cm² with various crystalline orientations, comprising (100), (111), (110), (211), (311), (331), (510), (553) and (995), as stated by the supplier. All orientations were on-axis except for (100) case for which 2°off-axis wafers were used additionally. They were ultrasonically degreased in methanol before loading in the CVD reactor. The growth procedure involved in-situ removal of the Si substrate native oxide under H₂ at 1000°C. Then, a standard two-step process was used including 10 min carbonization at 1165°C under 12 sccm propane followed by 60 min epitaxy at 1350°C under C/Si ratio of 4 using 1.5 sccm silane and 2 sccm propane. This is our optimal procedure for growing state-of-the-art 3C-SiC on

Si(100) substrate at a growth rate of $\sim 2 \mu\text{m/h}$. Each growth run was performed on four to five different Si substrates, including at least one piece of (100) oriented wafer for reference purpose.

Results and Discussion

The resulting 3C-SiC layers morphologies obtained on each kind of Si substrate are shown in Figure 1. They clearly display different surface features and roughnesses. By naked eyes, samples grown on Si(100), (211), (311), (553) and (995) look mirror like while the other ones are milky ((331), (510)) to dull ((110), (111)). The smoother surface (displaying the finest microstructure) seems to be the one grown on (100) 2°off and (995) substrates. Interestingly, none of these layers cracked upon cooling, even the 3C-SiC(111) layers which are known to undergo high tensile stress. The limited samples size probably explains the absence of crack. However, the bow of some samples, due to accumulated thermal stress, is detectable by optical microscopy at x1000 magnification via the impossibility to focus correctly on the full image area. These highly bowed layers (30 to 60 μm from center to the edge), grown on Si(111), (211) and (553) substrates, are all concave shaped and thus tensile strained. More experimental and theoretical work is required for correlating these orientations with the bow.

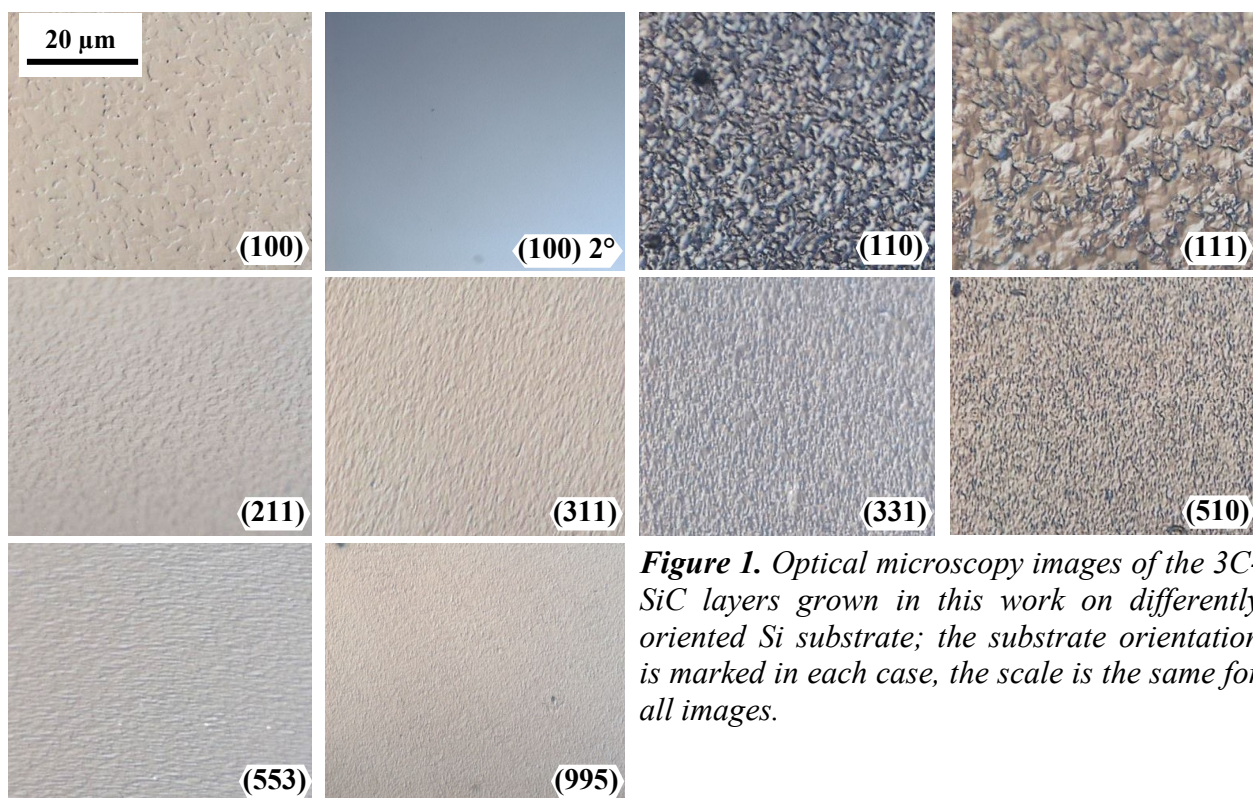


Figure 1. Optical microscopy images of the 3C-SiC layers grown in this work on differently oriented Si substrate; the substrate orientation is marked in each case, the scale is the same for all images.

Atomic force microscopy (AFM) images obtained on each of these samples (see Figure 2) follow the general tendencies found by optical microscopy. The corresponding RMS values (see Table 1) confirms that the smoothest surfaces are obtained on (100) 2°off and (995) orientations which display similar grain-like microstructure of 400 – 800 nm lateral size. Layer grown on (110) substrate is by far the roughest. Elongated and/or parallel features are commonly found on other orientations, with apparent boundaries between domains in some cases ((331), (510)).

X-Ray diffraction (XRD) characterizations (in θ -2 θ mode) were performed on all these samples in order to obtain information on the epitaxial relationships which may exist between each substrate/layer couple and to have. The results are summarized in Table 1. The layers with low h Miller index show the same orientation as the one of the substrate which means that the heteroepitaxial growth was successfully achieved. This was not the case for (110) orientation since both 3C-SiC(111) and (110) planes were detected. This layer is obviously polycrystalline.

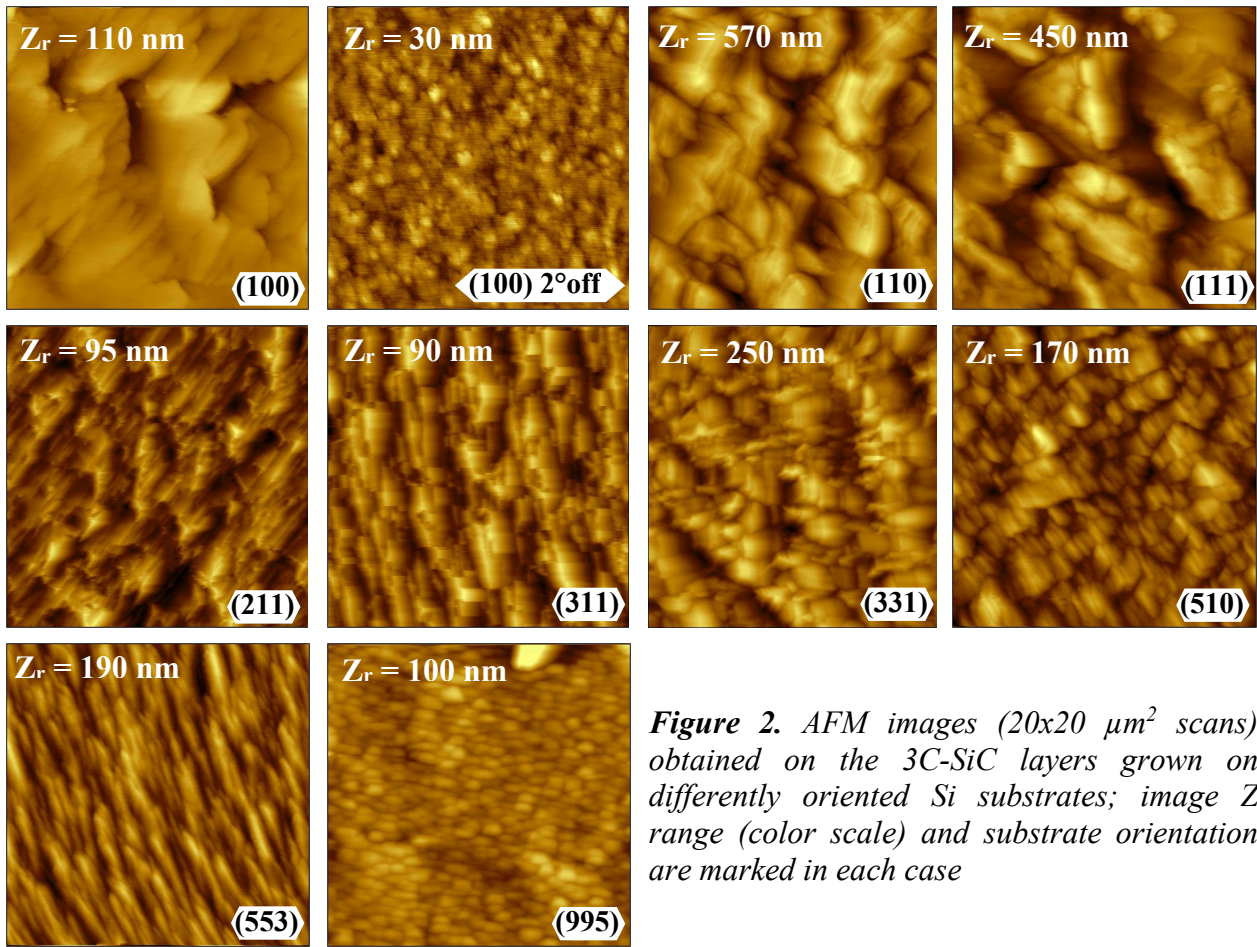


Figure 2. AFM images ($20 \times 20 \mu\text{m}^2$ scans) obtained on the 3C-SiC layers grown on differently oriented Si substrates; image Z range (color scale) and substrate orientation are marked in each case

Table 1. Summary of the XRD and AFM ($20 \times 20 \mu\text{m}^2$ scan) results obtained on the 3C-SiC layers grown on differently oriented Si substrate.

Si orientation	3C-SiC - XRD		AFM - RMS (nm) 3C-SiC layers
	peak	2θ (°)	
(100)	(200)	41.5	16
(100) 2°off	(200)	41.5	3.4
(110)	(111)	35.7	97.3
	(220)	60.02	
(111)	(111)	35.7	53
(211)	(422)	105.14	16
(311)	(311)	72.03	12,7
(331)	(331)	100.76	17,3
(510)	-	-	25,2
(553)	-	-	15,7
(995)	-	-	8,8

Such orientation mixing was already reported for the use of Si(110) substrate [4, 7] while other authors observed also the sole formation of (111) oriented 3C-SiC layers [2, 8]. It seems that this particular (110) substrate orientation requires adapting the growth conditions for optimizing either (111) or (110) full orientation of the 3C-SiC layer. For the layers grown on Si substrate with h Miller index ≥ 5 ((510), (553) and (995)), no XRD peak from the 3C-SiC layer or even the substrate could be detected. These high index planes are indeed not known as diffracting ones in XRD standard configuration. If the layers were polycrystalline, we would probably have detected some low index 3C-SiC peaks such as (111) or (200), so that their absence is a hint toward

monocrystallinity. If these layers are monocrystalline, their low-index planes could diffract by applying a proper tilt of the sample. Calculation of the crystallographic angles between these low and high index planes are reported in Table 2. One can see that the misorientation angle between these high and low index planes is high ($>10^\circ$) which explain

Table 2. Calculated angles between planes in CFC crystalline structure.

	(510)	(553)	(995)
(111)	47.21°	12.27°	13.81°
(100)	11.3°	49.39°	48.8°

their absence in the patterns recorded in standard θ -2 θ mode. Deeper XRD investigations, like using pole figures, are thus requested for confirming this point. This was performed on sample grown on (995) oriented substrate since this sample displays unusual grain-like surface morphology (which could be interpreted as resulting from columnar growth or twinning) and interesting low surface roughness. In Figure 3 are displayed the (111) pole figures recorded on the Si substrate and the 3C-SiC deposit. These figures are very similar, indicating that the (111) planes orientation of Si and 3C-SiC are identical. This means that the 3C-SiC layer is epitaxial with the Si substrate, without any twinning. Moreover, the central spot appears for a χ angle of about 15° , value which is close to the calculated angle between (111) and (995) planes (see Table 1). The three spots located at $\chi = 56.2^\circ / \varphi = 269.4^\circ$, $\chi = 79^\circ$ and $\varphi = 146.7^\circ$ and 33.6° are expected for a three-fold symmetry axis around the central [111] direction. Note that a weak spot appears also at about $\chi = 62^\circ / \varphi = 93.3^\circ$ for both substrate and layer. This spot is not predicted by the simulation and its presence remains under investigation. The crystal quality of the SiC-3C layer needs to be evaluated, for example by Rocking-Curve measurements, but the width of the spots observed along φ rotation on the (111) pole figure of 3C-SiC seems promising on this point. Pole figures remain to be done on the two other layers grown on Si(510) and (553) (which did not display any XRD peak in θ -2 θ mode) in order to confirm their heteroepitaxial nature (in which we strongly believe).

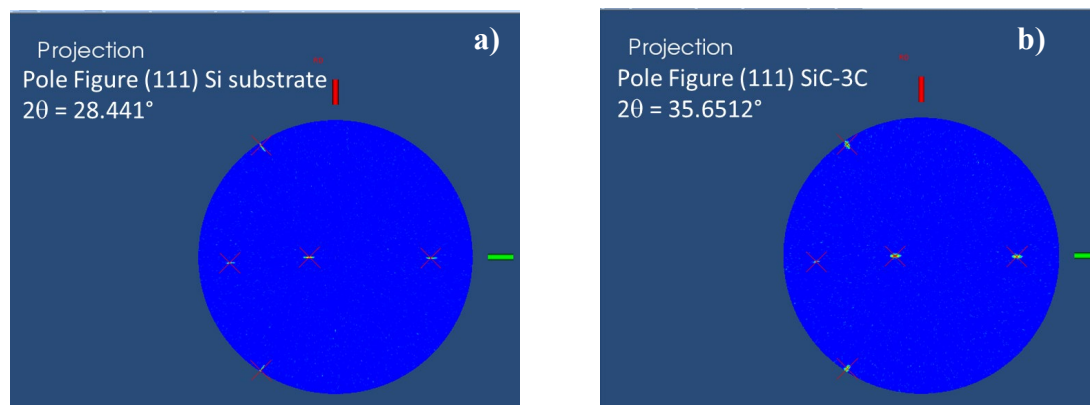


Figure 3. XRD pole figures recorded on the (995) oriented sample and showing the (111) planes of both the Si substrate (a) and the 3C-SiC layer (b).

Conclusion

The heteroepitaxial growth of 3C-SiC layers on Si substrate having various orientations was found to be successful for most of the orientations. Only (110) orientation led to obvious polycrystalline growth. High bowing was observed for Si(111), (211) and (553) substrates while low roughness was found for Si(100)2°off, and (995). Deeper XRD and Raman spectroscopy analyses are currently under investigation for completing this exploratory work.

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