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SiC structural characterization by non destructive near-field microscopy techniques

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Abstract—SiC sputtered and e-beam evaporated layers have been deposited on 4H-SiC substrates. Recrystallization has been tried by high temperature annealing with two plateaus at 1400°C and 1700°C. The crystallinity was systematically investigated by non destructive near-field microscopy techniques, particularly adapted to the thin thickness of our layers. Good recrystallization is confirmed only on the e-beam evaporated layers and after the higher annealing plateau by preserving 4H substrate polytype.

Keywords—4H, a-SiC, PiFM, s-SNOM, μ Raman

1. Introduction

Silicon carbide (SiC) is today a familiar semiconductor material for power electronics, with discrete SiC devices such as Schottky diodes and transistors (BJT, JFET, MOSFET) being already commercialized. Due to its superior material properties when compared with other semiconductors, SiC is also a promising material for fabricating sensors operating in harsh environments and scalable integrated quantum photonic systems emitting from the UV to the IR telecom wavelengths [1].

Engineering and recovery of defects created in SiC by irradiation, as during ion implantation doping steps, have a critical impact on the operational performances of these fabricated devices. High defect densities generating amorphous layers occur after room temperature (RT) ion implantation. High temperature annealing treatments are necessary to recover the initial crystallinity, which for the today's SiC technology is 4H-SiC. However, the cubic 3C-SiC polytype market is now reconsidered because its color

center emission better fits telecommunication wavelengths.

Structural characterizations, down to nanoscale range, as we present in this paper, are necessary to monitor the crystallinity of the SiC active layers during or post-fabrication technological process.

2. Experimental part

A. Sample description

Commercial n-type 4H-SiC off-axis substrates have been utilized for this study. Amorphous SiC layers have been created in 4H-SiC sample surfaces by Al RT ion implantation with a total dose of 1.75×10^{15} cm⁻² [2], a classic technological step utilized to obtain highly doped p-type layers for emitters in 4H-SiC bipolar diodes and transistors. SiC amorphous layers have also been formed on 4H-SiC substrate surfaces by Physical Vapour Deposition (PVD) techniques: electron-beam evaporation with an Elettrorava machine and sputtering with MP400 Plassys equipment. In order to (re)crystallize the SiC amorphous layers, annealing have been performed with an AET RTP furnace in several steps with two plateaus of 1400°C and 1700°C of 30 min each.

B. PiFM and SNOM characterization

Photo-induced force microscopy (PiFM) and scattering scanning near field optical microscopy (s-SNOM) have been performed,

as a comparative and complementary study, in order to identify and monitor the generation and recrystallization of the amorphous layers.

Our PiFM and s-SNOM are based on a VistaScope system (MolecularVista). Figure 1 shows a schematic diagram of the experiment. Based on an atomic force microscope (AFM) in non-contact mode, a tunable mid-infrared quantum cascade laser (QCL), with the spectral range 770-1860 cm^{-1} , is focused to the tip apex. The illuminated gold coated tip creates a strong near-field nano-focus at its apex, probing the properties of the sample underneath the tip. The near-field scattered lights are extracted from the background for s-SNOM images and spectra. On the other hand, PiFM detects the photo induced forces between the tip and sample, mechanically by the motion of the oscillating AFM tip. Both techniques can provide relatively strong contrast near the LO phonon frequency, notably because the surface phonon-polariton (SPhP) are strong in crystalline SiC.

These two techniques are particularly adapted to analyze thin layers [3], few 100 nm thick in our case, to decorrelate the layer response from the substrate one or other nearby functional layers, which are in SiC also.

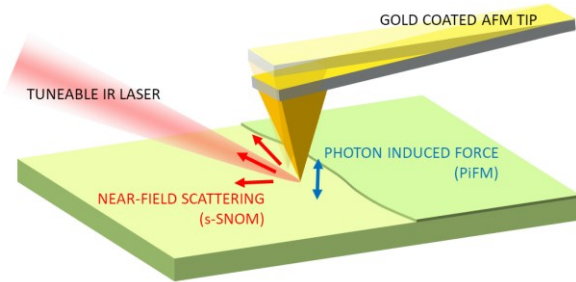


Fig. 1 Schematic diagram of the PiFM and s-SNOM experiments

C. μ Raman and other structural characterisation methods

The different SiC layers were examined using a LabRAM spectrometer of Horiba Scientific with several laser lines from UV ($\lambda_{\text{ex}}=325$ nm) to IR ($\lambda_{\text{ex}}=785$ nm). The μ Raman measurements have been performed

under backscattered geometric configuration excited at RT. A $40\times$ long-focus objective lens and a $100\ \mu\text{m}$ confocal pinhole were used for the measures in UV light. The grating was 300g/mm.

The penetration depth in SiC for Raman laser wavelengths strongly varies from few nm for the amorphous SiC to more than several μm in single crystal SiC [4] even by using the UV 325nm line that we favored in this study. Therefore, the near-field techniques, although less common, have a real potential for probing SiC surface with a much smaller penetration depth [5-6], mostly ruled by the tip diameter.

To confirm the presence of the SiC PVD layers, thicknesses were measured with Dektak Bruker stylus profilometer.

3. Results and discussion

A. Recognition of amorphous layers

Figure 2 presents PiFM spectra measured on amorphous a-SiC layers obtained by high doses RT ion implantation and PVD compared to bulk 4H-SiC. On both amorphous layers we remark the absence of the surface phonon-polariton (SPhP) peak at $\sim 922\ \text{cm}^{-1}$ which is a signature of the layer crystallinity as we will confirm in the next paragraphs. An increasing of the peak at $\sim 1090\ \text{cm}^{-1}$ is also observed, a peak related to the presence of native SiO_2 [7] in surface with usual higher thickness on the a-SiC layers.

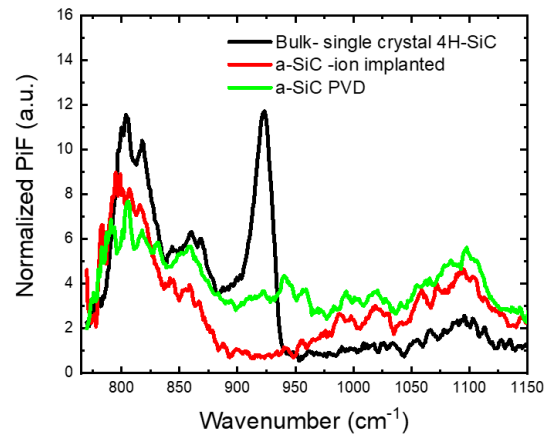


Fig. 2 PiFM spectra on amorphous SiC layers compared to 4H-SiC bulk.

Typical spectra from amorphous layers we obtained by μ Raman are presented in Figure 3. We remark the absence of the SiC LO and TO peaks.

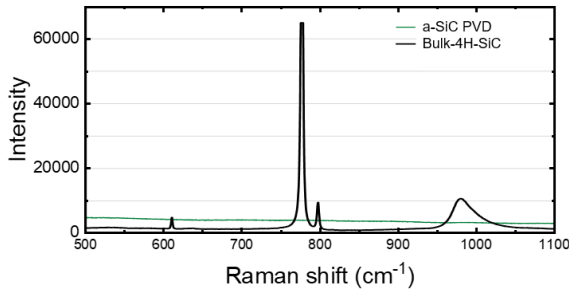


Fig. 3 Raman spectra on amorphous SiC layer compared to 4H-SiC bulk.

B. Recrystallization of amorphous layers

We tried to crystallize by high temperature annealing the amorphous SiC layers deposited by PVD, e-beam evaporation and sputtering. The annealing has been performed in two steps at 1400°C and 1700°C. PiFM and μ Raman analyses have been performed to check the crystallinity grade and if the same polytype as the substrate (4H-SiC) is reproduced. The PiFM spectra presented in Figure 4 indicate a good recrystallization of the layers deposited by e-beam evaporation according to the intensity of the SPhP peak at 922 cm^{-1} . The intensity of the peak increases from 1400°C to 1700°C annealing and becomes similar to the PiFM spectrum of the bulk 4H-SiC. However, for the SiC layers deposited by sputtering, we observe a less intense SPhP peak at 922 cm^{-1} after the last

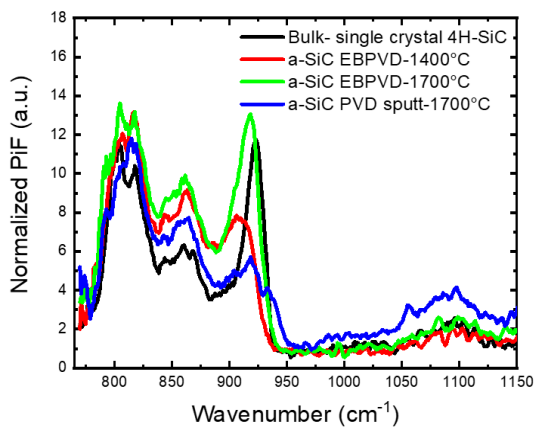


Fig. 4 PiFM spectra on amorphous SiC layers, fully or partially crystallized by high temperature annealing compared to 4H-SiC bulk.

1700°C annealing which denotes a partial recovery of the 4H-SiC lattice.

A better recrystallization for the e-beam evaporated a-SiC layers compared to the sputtered ones was also put in evidence by Raman analyses (Figure 5) but in this case, even by utilizing the UV 325nm laser line, the depth penetration into SiC was beyond the 150nm deposited layer.

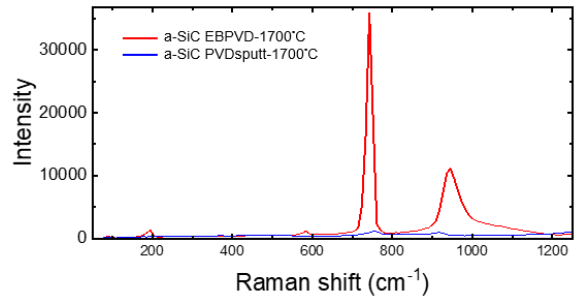


Fig. 5 Raman spectra on a-SiC layers e-beam evaporated and deposited by sputtering after 1700°C annealing.

Figure 6 compares normalized PiFM and s-SNOM spectra of the e-beam evaporated SiC layer after the second annealing at 1700°C. As for the PiFM spectrum, the SPhP peak at 922 cm^{-1} , indicating the crystallinity of the sample, appears also in our s-SNOM spectrum. Another peak is also present at $\sim 885 \text{ cm}^{-1}$.

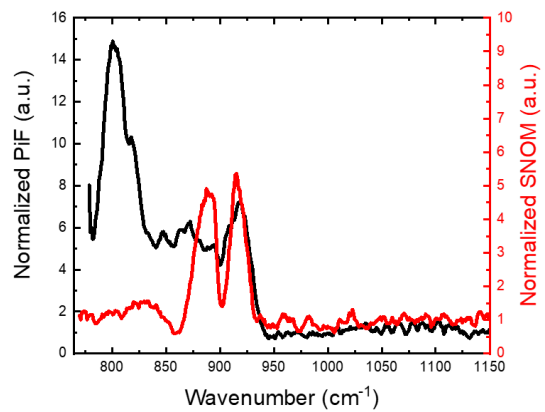


Fig. 6 Comparison of PiFM and s-SNOM spectra on SiC e-beam evaporated and annealed up to 1700°C

We made mapping at the substrate-evaporated SiC boundaries obtaining images with the SPhP 922 cm^{-1} peak PiFM and s-SNOM distribution. This allows us to confirm the crystallinity and the 4H polytype. The AFM topography is firstly presented (Figure 7), the higher area (yellow) is the evaporated SiC, and the lower is the SiC substrate (in blue). We confirm the thickness

of the evaporated layer, slightly overestimated by stylus profilometer. After 1700°C we recover higher PiFM and SNOM signals and even with a higher PiFM signal measured on the evaporated part. Single 4H-SiC crystal is thus confirmed. However, after 1400°C annealing uniform color distribution is obtained (as AFM topography, not shown here). Thus, to recrystallize a-SiC layers higher temperatures are needed and thus it seems to be impossible to obtain 3C-SiC on silicon substrates by this method, the needed temperatures being above Si melting temperature.

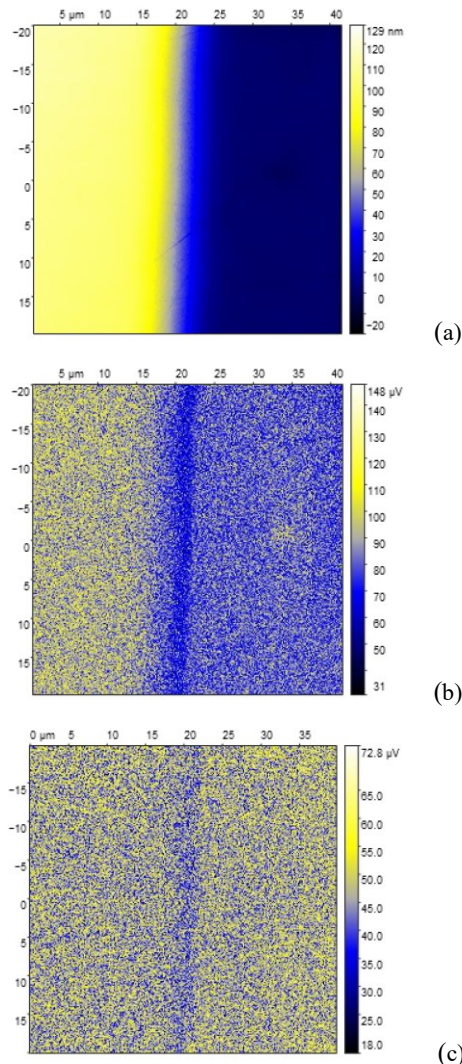


Fig. 7 AFM Topography (a) PiFM (b) and SNOM (c) images obtained from the substrate-evaporated SiC boundary area after 1700°C annealing, scanned with laser at 921cm^{-1} .

4. Conclusion

The nondestructive near-field microscopy techniques PiFM and s-SNOM allowed us to check the crystallinity of thin SiC layers after

different technological steps: PVD, annealing, decorelating them from the substrate signal response. This allows us to highlight that when the PVD process is done by e-beam evaporation we reproduce the polytype of the substrate, 4H, obtaining a “low-cost” homoepitaxial process. However, this process is not compatible with 3C-SiC heteroepitaxial growth on Si substrates.

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References

- [1] Stefania Castelletto and Alberto Boretti "Silicon carbide color centers for quantum applications", 2020 J. Phys. Photonics, **2**(2). 022001, 2020.
- [2] M. Lazar, S. Sejil, L. Lalouat, C. Raynaud, D. Carole, D. Planson, G. Ferro, F. Laariedh, C. Brylinski, H. Morel, "P-Type Doping of 4H-SiC for Integrated Bipolar and Unipolar Devices" Rom. J. Inf. Sci. Technol., **18**(4), pp. 329–342, 2015.
- [3] J. Li, J. Jahng, J. Pang, W. Morrison, J. Li, E. S. Lee, J-J. Xu, H-Y. Chen and X-H. Xia, "Tip-Enhanced Infrared Imaging with Sub-10 nm Resolution and Hypersensitivity", J. Phys. Chem. Lett., **11**(5), pp. 1697-1701, 2020.
- [4] Z. Xu, Z. He, Y. Song, X. Fu, M. Rommel, X. Luo, A. Hartmaier, J. Zhang, F. Fang, "Topic Review: Application of Raman Spectroscopy Characterization in Micro/Nano-Machining". Micromachines, **9**, 361, 2018.
- [5] B. Hauer, C. E. Marvinney, M. Lewin, N. A. Mahadik, J. K. Hite, N. Bassim, A. J. Giles, R. E. Stahlbush, J. D. Caldwell, T. Taubner, "Exploiting Phonon-Resonant Near-Field Interaction for the Nanoscale Investigation of Extended Defects", Adv. Funct. Mater., **30**, 1907357, 2020.
- [6] A. M. Gigler, A. J. Huber, M. Bauer, A. Ziegler, R. Hillenbrand, and R. W. Stark, "Nanoscale residual stress-field mapping around nanoindentations in SiC by IR s-SNOM and confocal Raman microscopy", Opt. Express, **17**, pp. 22351-22357, 2009.
- [7] T. Oh and C. K. Choi, "Comparison between SiOC Thin Films Fabricated by Using Plasma Enhance Chemical Vapor Deposition and SiO₂ Thin Films by Using Fourier Transform Infrared Spectroscopy" J. Korean Phys. Soc., **56**(4), pp. 1150-1155, April 2010.