

Residual Stress Measurement by Raman on Surface-Micromachined Monocrystalline 3C-SiC on Silicon on Insulator

F. La Via^{1,a*}, L. Belsito^{2,b}, M. Ferri^{2,c}, S. Sapienza^{2,d}, A. Roncaglia^{2,e},
M. Zielinski^{3,f}, V. Scuderi^{1,g}

¹CNR-IMM Catania Stradale Primosole Catania, Italy

²CNR-IMM Bologna Via Gobetti 101, 40129 Bologna, Italy

³NOVASiC, Savoie Technolac, Arche Bat 4, BP267, 73375 Le Bourget du Lac, France

^afrancesco.lavia@imm.cnr.it, ^bbelsito@bo.imm.cnr.it, ^cferri@bo.imm.cnr.it,
^dsapienza@bo.imm.cnr.it, ^eroncaglia@bo.imm.cnr.it, ^fmzielinski@novasic.com,
^gviviana.scuderi@imm.cnr.it

*Corresponding author email: francesco.lavia@imm.cnr.it

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Abstract. In this work, we investigate, by μ -Raman spectroscopy the distribution of stress field on a micro-machined structures. They were realized on a 3C-SiC substrate, grown on a Silicon On Insulator (SOI) wafer, after lithography and etching processes. Various structures, such as strain gauge, single and double clamped beams, were analyzed, showing different stress distributions. All the structures show an intense variation of stress close to the undercut region.

Introduction

3C-SiC resonators have been investigated for sensing applications [1,2], thanks to the high Quality (Q)-factor that they can achieve at resonance [3], to the higher frequency with respect to other materials for the same geometrical dimensions [4] and to the high Young Modulus [5,6]. In a previous paper [7] has been observed that for crystalline layers the Young modulus increases increasing the thickness thanks to the reduction of the crystal defects (essentially SFs) and that at least a thickness of 3 μm is needed to obtain the theoretical Young modulus value. In a more recent paper [8], improving the material quality, it has been observed that high values of the Young modulus can be achieved even at lower thickness around 1 μm for both (100) and (111) 3C-SiC layers. This is a large improvement of the material because it has been observed that the Q-factor depends strongly on the thickness of the layers and high Q-factors can be observed in very thin 3C-SiC layers (250 nm)[9]. Q-factor in flexural 3C-SiC resonators critically depends on the presence of stress within the layer, particularly the one appearing on the anchors of the resonator [3] and on the defects present at the interface with the silicon substrate. This effect can be studied with a high resolution spatial technique (μ -Raman) that show the stress effect close to the anchors of the resonator [10]. In the previous paper the study has been performed on thick films (2.5 μm) while in the present paper μ -Raman measurements on 3C-SiC micromachined structures of less than 1 μm thickness are utilized to derive the residual stress distribution on resonators.

MEMS Realization

The films prepared for this study followed typical characteristics of as-grown 3C-SiC/Si material presented in previous papers (see details in [11]). 3C-SiC thickness dispersion was below 5%. The RMS roughness ($5 \times 5 \mu\text{m}^2$) was in the 5–10 nm range for (100) epilayers and the 2–5 nm range for (111) orientation. The 3C-SiC layers were grown on the SOI wafer using classic two-step growth process with silane/propane chemistry. No structural or morphological degradation of 3C-SiC was observed with respect to films grown on bare Si substrate. The process illustrated in Fig. 1 was adopted to realize surface micro-machined structures on SOI substrates with 10 μm SOL and 0.5 μm

BOX. After 3C-SiC growth, a double layer of SiO₂ and polycrystalline silicon was deposited on the SiC and patterned by Reactive Ion Etching (RIE) to create the geometry of the structures. Afterwards, the SiC layer was etched by RIE as well, up to the device layer of the SOI underneath and the structures released by etching the device layer by an isotropic SF₆ plasma. The residual SiO₂ mask after release was removed by HF vapor etching. Empirical calculations [5] on the entire matrix containing several micro-machined structures show a residual stress of about 160 MPa tensile. The stress measure with these structures is essentially connected to the thermal stress due to the presence of the two different materials (3C-SiC and Si) that have two different thermal expansion coefficients.

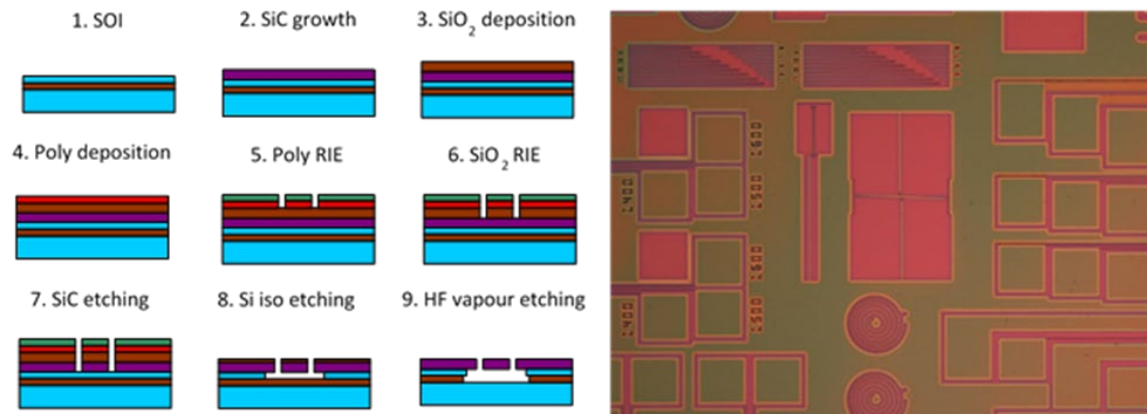


Fig. 1. Fabrication process flow (left panel) and picture of micromachined 3C-SiC test structures (right panel).

Raman Measurements

In order to study the distribution of stress field we performed micro-Raman maps (using a continuous He-Ne laser (632.8 nm)) on micro-machined structures. They generally consist of freestanding structures having one or two anchor points with the unreleased region (called undercut region). μ -Raman spectroscopy is a non-destructive technique particularly useful for the local stress study in micro structures due to the diameter of the laser spot $\sim 1 - 1.5 \mu\text{m}$. Transverse optical (TO) Raman mode analysis reveals the stress relaxation on the free standing structure (796.5 cm^{-1}) with respect to the stressed unreleased region. Different structures, such as strain gauge, single and double clamped beams, cantilever, bridge were analysed.

Due to the length of the structure, to reduce the noise present in the experimental acquisition Fig. 2 shows the smoothing of the stress profile extracted from the Raman map acquired on a strain gauge. It is essentially a cantilever with an anchor on a suspended structure. Applying the equation proposed by Olego et al. [12, 13], which links the shift of Raman phonon modes to stress relaxation within a 3C-SiC epitaxial layer, we obtained a particular trend of the stress. Close to the anchor point the stress is compressive, along the cantilever the stress decreases and at the end is tensile. This behavior was observed on several strain gauges analysed. However, the cause of this behavior is not yet clear.

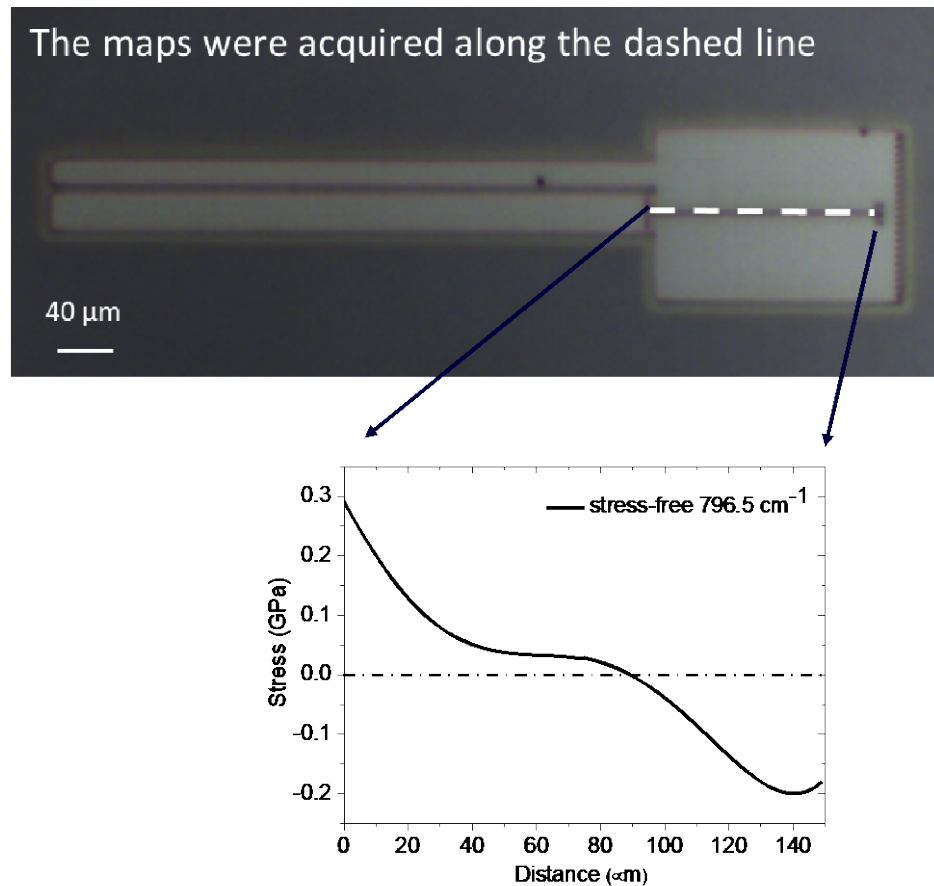


Fig. 2: Optical image of the strain gauge (top panel); stress profile extracted from the map (down panel).

Fig. 3 shows the TO Raman shift map (Fig. 3(a) and 3(c)) and the stress profile (Fig. 3(b) and 3(d)) extracted from the maps, for a cantilever and a bridge. Both microstructures are 4 μm wide and 30 μm and 25 μm long, respectively. For the cantilever, Fig. 3(b), the stress is quite constant along the structure, with an average value of 160 MPa compressive. Instead, a stress-free value close to the anchorage point of the structures (spectrum between the two dashed lines) was detected. In this case the stress measured by Raman is essentially the intrinsic stress of the film due to the defects present in the layer. Similarly, for the bridge (Fig. 3(d)) the stress is quite constant along the structure, with an average value of 150 MPa tensile, and it reaches a value of 500 MPa tensile close the two anchor points (spectrum between the dashed lines). It is not possible to acquire Raman spectra of SiC correctly, moving in the "bulk" region of the system (beyond the dashed lines in Fig. 3 (a-d), non-released areas). In fact, due to the thickness of the SiC (less than 1 μm), in the non-released areas, the Raman signal comes mainly from the SOI substrate.

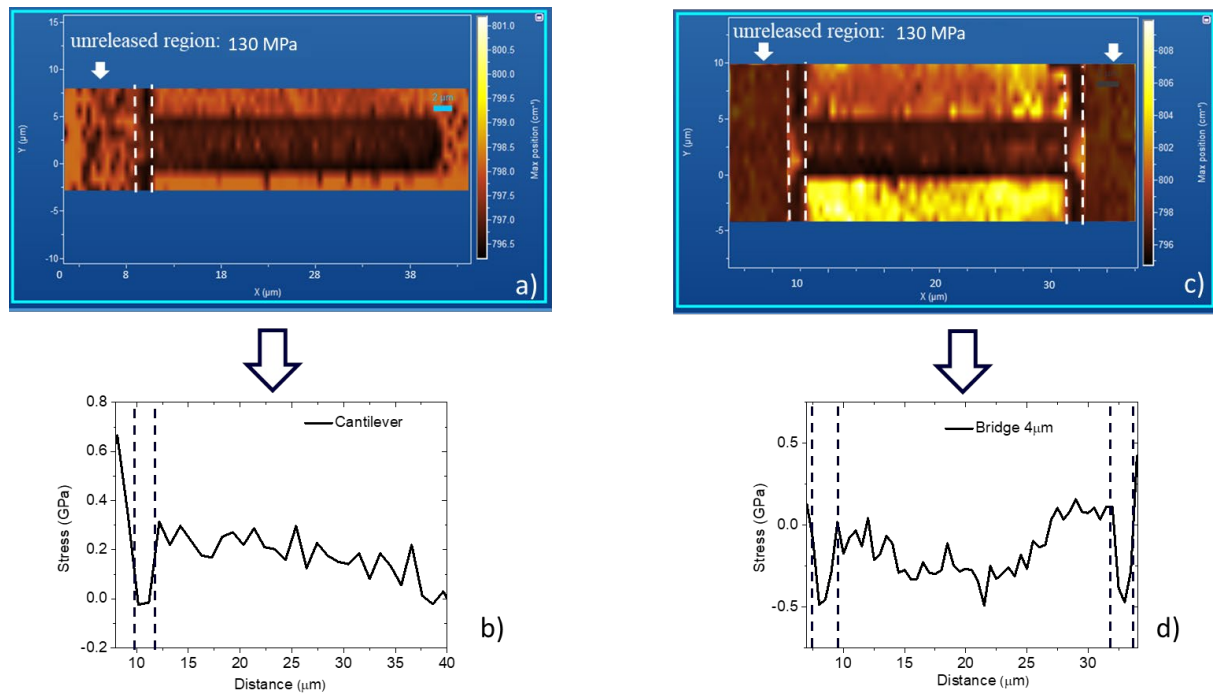


Fig. 3: (a) TO Raman shift map of a cantilever; (b) stress profile extracted from the map; (c) TO Raman shift map of a bridge; (d) stress profile extracted from the map. In the panel a) and c), scale bar is 2 μm . The dashed lines delimit the undercut region. The unreleased region shows a stress of 130 MPa.

For all microstructures the TO Raman shift in the undercut region evidences a different stress within the film. This difference is due to the processes carried out to realize the microstructure. The plasma etching is an anisotropic process. During the silicon etching, the process also affects the 3C-SiC/Si interface, removing a thin layer of 3C-SiC. This leads to a small variation in thickness between the center and the edges of the structures which induces a reorganization of the stress fields. The intense variation of the stress near the undercut region can be attributed to a deep modification of the stress tensor where the role of the shear component cannot be neglected [10].

Summary

In this work, we investigate, by μ -Raman spectroscopy the distribution of stress field on a micro-machined structures. In particular, strain gauge, cantilever and bridge were analyzed. For all microstructures the TO Raman shift in the undercut region evidences a different stress within the film. This is associated to the removal of the silicon substrate due to the processing required to fabricate the microstructure.

So, with this technique it is possible to understand the behaviour of the stress inside the microstructures and to optimize the design of the structure and of the anchor points from the point of view of the stress. Future works will be dedicated to analysing systems properly fabricated in order to minimize the generated stress field and defect density within the epitaxial layer.

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References

- [1] L. Belsito, M. Bosi, F. Mancarella, et al. J. Microelectromech. Syst. 29, 117-128 (2020).
- [2] A. R. Kermany, G. Brawley, N. Mishra, et al. Appl. Phys. Lett. 104, 81901 (2014).
- [3] E. Romero, V. M. Valenzuela, A. R. Kermany, et al. Phys. Rev. Appl. 13, e44007D (2020).
- [4] Y. T. Yang, K. L. Ekinici, X. M. H. Huang, L. M. Schiavone, M. L. Roukes, C. A. Zorman and M. Mehregany, Appl. Phys. Lett., 78(2), 162 (2001)
- [5] Lijun Tong, Mehran Mehregany, and Lawrence G. Matus, Appl. Phys. Lett. 60 (24), 2992(1992)
- [6] V Cimalla, J Pezoldt and O Ambacher , J. Phys. D: Appl. Phys. **40**, 6386 (2007)
- [7] R. Anzalone, M. Camarda, A. Canino, N. Piluso, F. La Via, and G. D'Arrigo, Electrochemical and Solid-State Letters, 14 (4) H161-H162 (2011)
- [8] S. Sapienza, M. Ferri, L. Belsito, D. Marini, M. Zielinski, F. La Via and A. Roncaglia, *Micromachines* 12, 1072 (2021).
- [9] Atieh R. Kermany, George Brawley, Neeraj Mishra, Eoin Sheridan, Warwick P. Bowen, and Francesca Iacopi, Appl. Phys. Lett. 104, 081901 (2014)
- [10] N. Piluso, R. Anzalone, M. Camarda, A. Severino, A. La Magna, G. D'Arrigo and F. La Via, J. Raman Spectrosc. 44, 299 (2013)
- [11] M. Zielinski, S. Monnoye, H. Mank, C. Moisson, T. Chassagne, A. Michon, M. Portail, Mater. Sci. Forum, 924, 306 (2018)
- [12] D. Olego, M. Cardona, Phys Rev B 25: 1151 (1982).
- [13] D. Olego, M. Cardona, P. Vogl Phys Rev B 25: 3878, (1982).