

# Close Space PVT Growth of N- and P-Type Quasi-Bulk SiC in a Classic PVT Setup and a Newly Developed TableTopCS™ Growth Machine

S. Strüber<sup>1,a</sup>, C. Grazzi<sup>1,b</sup>, L. Tana<sup>1,c</sup>, O. Schneider<sup>1,d</sup>, T. Wagner<sup>1,e</sup>,  
J. Wiedemann<sup>1,f</sup>, P. Wunder<sup>2,g</sup>, P. J. Wellmann<sup>h\*</sup>

<sup>1</sup>Crystal Growth Lab, Materials Department 6, <sup>2</sup>Engineering Workshop, Faculty of Engineering, Friedrich-Alexander-Universität Erlangen-Nürnberg, 91058 Erlangen, Germany

<sup>a</sup>sv.en.strueber@fau.de, <sup>b</sup>cristina.grazzi@fau.de, <sup>c</sup>lucrezia.tana@fau.de, <sup>d</sup>ole.schneider@fau.de,  
<sup>e</sup>wato.t.wagner@fau.de, <sup>f</sup>jakob.wiedemann@fau.de, <sup>g</sup>philipp.wunder@fau.de,  
<sup>h</sup>\*peter.wellmann@fau.de

**Keywords:** close space PVT, CS-PVT growth, 4H-SiC, bulk growth, thick epitaxy, doping.

**Abstract.** Close Space PVT (CS-PVT) is a modification of standard PVT exhibiting a short source-to-seed-distance and enabling a large variety of growth process variations to meet the specific requirements of the SiC material (i.e. special polytype and/or doping) to be grown. In this work, we study the growth of 4H-SiC p-i-n structures exhibiting thick SiC layers to be used as SiC photovoltaic cells for remote power transfer in space. Nevertheless, the found results are also applicable (i) to the SiC thick layer growth of power electronic devices and (ii) SiC pucks with a thickness of up to 10mm. In addition, we present the new type of growth machine TableTopCS™ in its design being dedicated for the special crucible configuration of CS-PVT.

## Introduction

Recently silicon carbide (SiC) has proven to offer physical properties which are not only advantageous for power electronics, but also for photonic applications like photocatalytic water splitting, fluorescent SiC, and waveguide applications. In this field of application, special SiC bulk materials exhibiting intrinsic properties and p-type doping are needed. In addition to 4H-SiC, also 3C-SiC and 6H-SiC are of importance. Close Space PVT (CS-PVT), which is a modification of standard PVT exhibiting a short source-to-seed-distance, enables a large variety of growth process variations to meet the specific requirements of the SiC material (i.e. special polytype and/or doping) to be grown. This for example enabled the bulk growth of 3C-SiC [1] which profits from a high supersaturation and a Si-rich gas phase composition. Indication for the high crystalline quality of CS-PVT grown hexagonal SiC was already proven in the early work of Syväjärvi and Yakimova [2, 3].

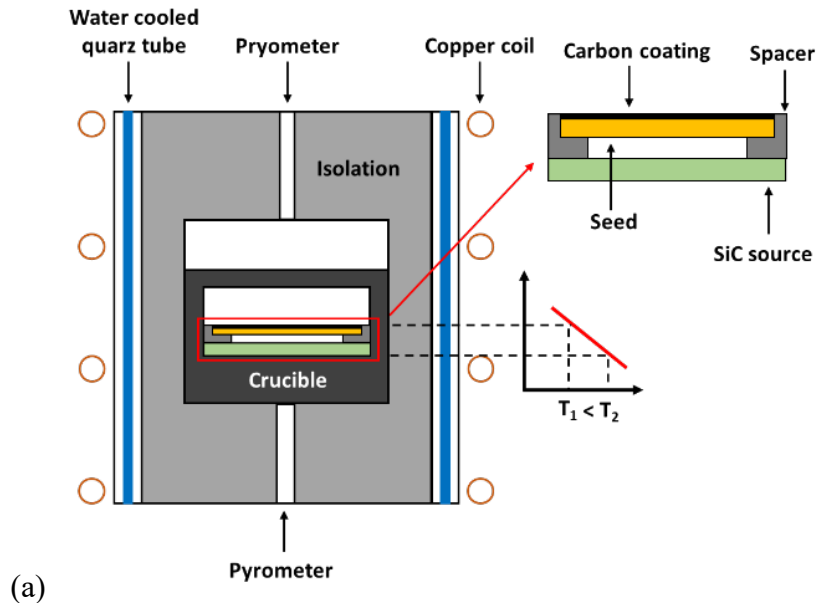
In this work, we study the growth of 4H-SiC p-i-n structures to be used as SiC solar cells for remote power transfer in space using laser power converter (LPC) devices. Note: The presented growth method is also applicable for the fabrication of large area SiC wafers and p-i-n-layer stacks as used in power electronics. We used the CS-PVT method [4], which bridges the gap between the state-of-the-art bulk growth of SiC using the PVT method (boule thickness of 1-50 mm) and the chemical vapor deposition (CVD) of thin SiC films in the thickness range of ca. 1 to 100 μm. In addition to the SiC growth study, we will also introduce the newly developed TableTopCS™ growth machine which meets special requirements for advanced CS-PVT growth. While this work mainly shows results of 100mm 4H-SiC layer deposition, growth cells of 150mm CS-PVT are already applied in the lab.

In the following we are addressing in this study (i) the achieved 4H-SiC polytype stability and (ii) new aspects of advanced p-type doping by aluminum which are possible in the ballistic mass transport regime of CS-PVT.

## Growth Experimental Procedure

### CS\_PVT process.

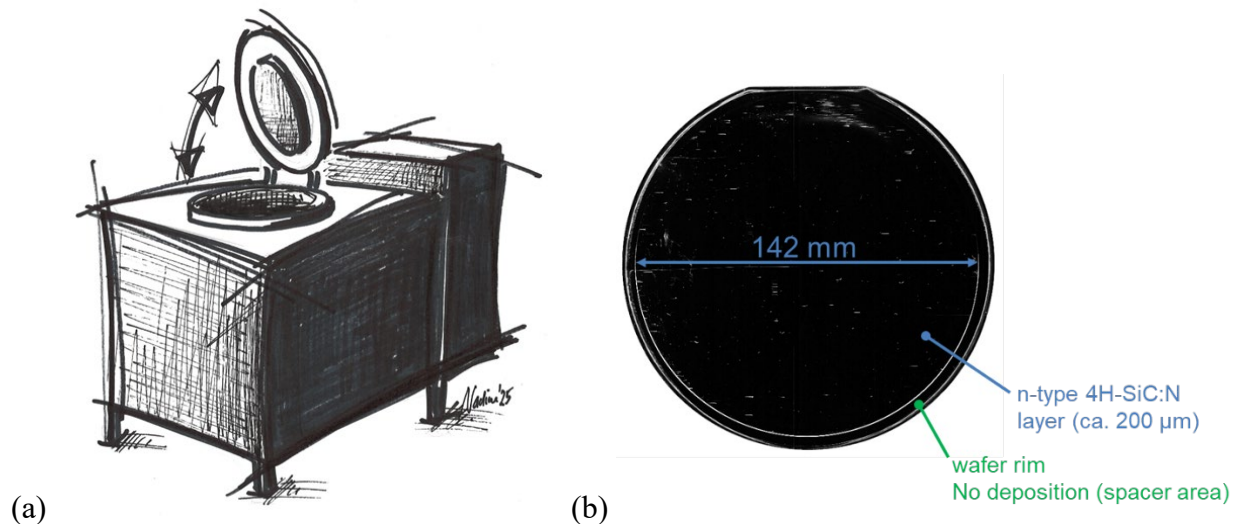
The CS-PVT growth configuration makes use of a solid SiC (single-)crystalline source and a single-crystalline seed which are separated by a spacer in the range of 0.5 to 2 mm. Mass transport from source to seed is established by establishment of a T-difference between source and seed. Typical axial T-gradients can be as low as a few °C/cm but may reach above 100°C/cm. One technical solution to realize CS-PVT growth is to place basic CS-PVT stack out of SiC source, spacer and SiC seed in a standard PVT growth crucible as sketched in Fig. 1.



**Fig. 1.** (a) Sketch of the CS.PVT growth cell [1]. (b) Examples of a standard PVT growth reactor hosting the CS-PVT growth cell.

### TableTopCS™ Growth Machine.

In order to have more control of the growth parameters in CS-PVT, a new growth machine was designed and produced which heating system and technical implementation match the needs of the close space sublimation process. As indicated in the name “TableTopCS™” the new system is a compact setup with small footprint (Fig.2a) and which is designed for optional usage in a cleanroom for electronic device fabrication.



**Fig. 2.** (a) Sketch of the outer view of the new CS-PVT growth tool TableTopCS™. (b) Greyscale optical image of one of the first 150 mm 4H-SiC samples prepared in the new growth tool.

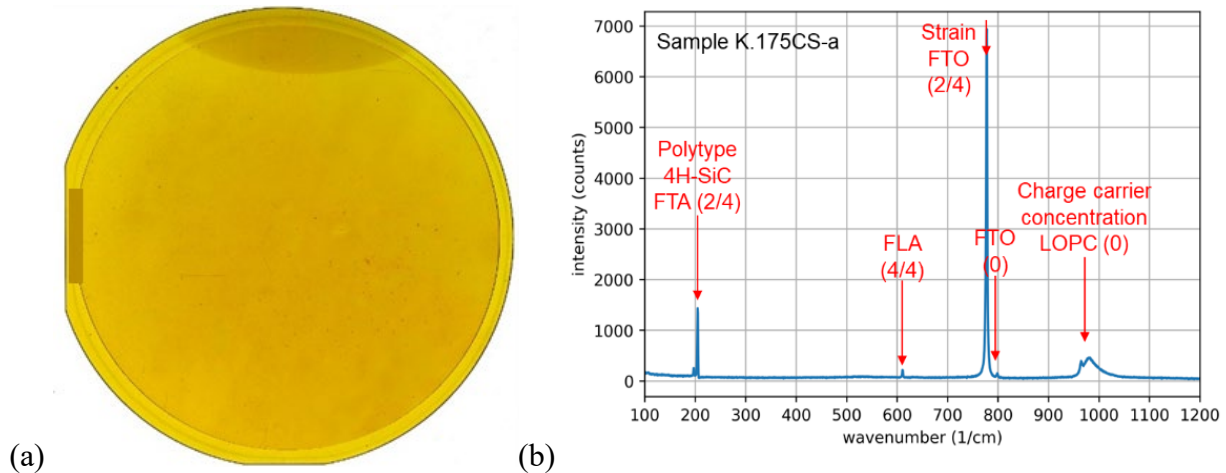
### CS-PVT Growth Process Parameter Setting.

The p-i-n structures were fabricated in two consecutive CS-PVT growth runs. As substrate we used 100 mm 4H-SiC wafers ( $4^\circ$  off-axis) either C-face or Si-face oriented to carry out homoepitaxial nucleation and growth of 4H-SiC (figure 1), respectively. The growth temperature measured at the top of the CS-PVT growth cell was  $1900^\circ\text{C}$  (growth rate ca.  $50\ \mu\text{m/h}$ ). The background gas pressure was  $< 0.1\ \text{mbar}$ . In the first step nitrogen doped n-type or nominally undoped, quasi-intrinsic layers (either residual n-type or residual p-type) with a thickness of ca.  $100\ \mu\text{m}$  layer were deposited (growth time = 2 h). Intentional p-type doping of the second thin top layer (growth time = 10 min to 2 h) was performed by using aluminum doped solid 4H-SiC:Al and 6H-SiC:Al sources which were prepared by the M-PVT method [5].

## Results and Discussion

### Properties of 4H-SiC layer stacks on C-face seeds.

In standard PVT growth above  $2000^\circ\text{C}$  it is standard to grow a 4H-SiC crystal on c-face seeds. However, Si-face seeding fails, because of a heteroepitaxial transition of the polytype to 6H-SiC and 15-SiC.

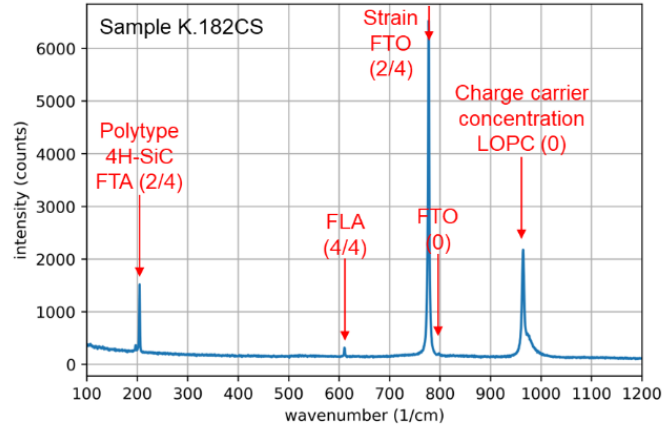


**Fig. 3.** (a) Examples of a high quality 4H-SiC layer (ca.  $100\ \mu\text{m}$ ) deposited on 100 mm 4H-SiC Substrate (c-face). (b) Raman spectra of two CS-PVT grown  $100\ \mu\text{m}$  thick 4H-SiC layer on a Si-face 4H-SiC ( $4^\circ$  off-axis) substrate.

Intentional p-type doping of the second thin top layer (growth time = 10 min) was performed by using aluminum doped solid 4H-SiC:Al and 6H-SiC:Al sources which were prepared by the M-PVT method [5] (see section “Doping during CS-PVT growth”). Also in this case, the 4H-SiC polytype prevailed.

### Polytype stability of 4H-SiC layers on Si-face seeds.

As mentioned above, in standard PVT at temperatures above  $2000^\circ\text{C}$  growth of bulk 4H-SiC fails, a polytype switching to 6H-SiC and 15R-SiC is observed. However, chemical vapor deposition (CVD) growth of 4H-SiC performed at  $1600$  to  $1700^\circ\text{C}$  is routinely performed on Si-face SiC seed wafers. One important finding of this work is that 4H-SiC CS-PVT growth at  $1900^\circ\text{C}$  is possible on Si-face substrates making CS-PVT growth compatible with the SiC device processing routine (on Si-face substrates) exhibiting comparably high growth rates of  $50$  to  $200\ \mu\text{m/h}$ . The Raman spectrum in Fig. 4 proves the high 4H-SiC polytype stability when growth is carried out on the Si-face. This finding was not expected because often we find reports in literature indicating CS-PVT on Si-face (0001) oriented SiC wafers being favorable to switch to the cubic polytype 3C-SiC [6, 7].



**Fig. 4.** Raman spectra of a CS-PVT grown 100  $\mu\text{m}$  thick 4H-SiC layer on a Si-face 4H-SiC ( $4^\circ$  off-axis) substrate.

The result suggests further exploration of the applicability of CS-PVT for the growth of intrinsic, thick 4H-SiC layers for power electronic applications. One of the challenges will be to limit the residual charge carrier concentration to values in the  $10^{14} - 10^{15} \text{ cm}^{-3}$  range.

#### Doping during CS-PVT growth.

In sublimation growth doping of SiC by aluminum is rather challenging because of the much greater partial pressure of the Al gas species compared to Si- and C-related gas species (i.e. Si, Si<sub>2</sub>C and SiC<sub>2</sub>). Without special emphasis like in the M-PVT growth process, it is basically impossible to obtain homogeneous dopant incorporation. CS-PVT, however, enables a new growth mode for advanced doping which makes use of ballistic mass transport. At a low background gas pressure of  $<0.1$  mbar we showed earlier [8] that ballistic mass transport dominates the mass transport between the closely spaced SiC source and SiC seed (distance  $< 1\text{mm}$ ). In such ballistic growth mode, the phenomenon of the partial pressure of atomic or molecular species does not exist and the species are directly transported from source to seed.

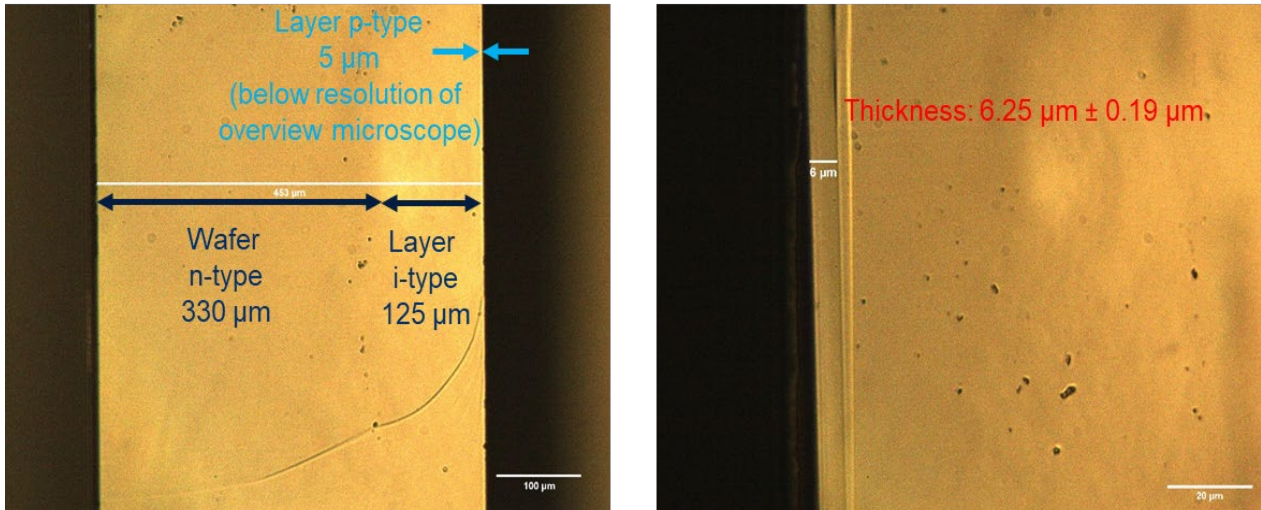
In this study, we demonstrate dopant transfer from the source to the seed using nitrogen. Unlike standard PVT, where nitrogen is added as a gaseous source, CS-PVT uses intentionally nitrogen-doped SiC sources.

**Table 1.** Overview of the transfer of N dopants from the SiC source to the SiC seed/crystal in the CS-PVT growth system under ballistic growth conditions. Note that the dopant concentration was determined using Raman spectroscopy by analyzing the energetic position and shape of the LOPC peak.

Sample	SiC Source	T-growth	Ambient	Charge carrier concentration [ $\text{cm}^{-3}$ ]	
				SiC source	SiC layer
K.170CS	n-type (N)	1900°C	Vacuum	$1.7(\pm 0.2)10^{18}$	$1.1(\pm 0.2)10^{18}$
K.187CS	n-type (N)	1900°C	Vacuum	$2.8(\pm 0.3)10^{17}$	$7.7(\pm 0.3)10^{17}$

### 4H-SiC p-i-n-layer stacks.

Using the above describe deposition, p-i-n layer stacks were prepared comprising two subsequent growth runs for the nominally first intrinsic or better to say slightly n-type 4H-SiC layer and a second highly p-type doped 4H-SiC:Al layer. The growth time and layer thicknesses were 2 hours and 125  $\mu\text{m}$  for the n-type layer and 10 minutes and 6  $\mu\text{m}$  for the p-type layer (see sample cross section in Fig. 5), respectively.



**Fig. 5.** Cross-sectional optical images of 4H-SiC p-i-n structure.

### Summary

We have shown that CS-PVT has been successfully applied to grow thick C-face 4H-SiC p-i-n-layer stacks and with high polytype stability for application in photonic LPC devices.

We have demonstrated that even growth of 4H-SiC on Si-face 4H-SiC is possible with high polytype stability suggesting the usage of CS-PVT for the growth of thick intrinsic 4H-SiC layers for power electronic applications.

The establishment of a ballistic mass transport regime in CS-PVT enabled the efficient direct aluminum dopant transfer for fabrication of p-type SiC.

While the presented results are mainly shown for layers below 150 $\mu\text{m}$ , in a previous study we already emphasized that CS-PVT is applicable to grow bulk SiC pucks of 3-5 mm in height. Technologically, to keep constant growth conditions a crystal height of ca. 10mm should be achievable.

### Acknowledgement

This work is funded by the European Union, Horizon Europe contract #101160868 (RePowerSiC).

### References

- [1] F. La Via, M. Zimbone, C. Bongiorno, A. La Magna, G. Fisicaro, I. Deretzis, V. Scuderi, C. Calabretta, F. Giannazzo, M. Zielinski, R. Anzalone, M. Mauceri, D. Crippa, E. Scalise, A. Marzegalli, A. Sarikov, L. Miglio, V. Jokubavicius, M. Syväjärvi, R. Yakimova, P. Schuh, M. Schöler, M. Kollmuss, P. Wellmann, *Materials*, 14, 18, 5348 (2021).
- [2] M. Syväjärvi, R. Yakimova, P.-A. Glans, A. Henry, M. F. MacMillan, L. I. Johansson, E. Janzen, *Journal of Crystal Growth*, 198-199, 1-4, 1019 (1999).
- [3] R. Yakimova, N. Vouroutzis, M. Syväjärvi, J. Stoemenos, *Journal of Applied Physics*, 98, 3, 034905 (2005).

- [4] M. Kollmuss, M. Schöler, R. Anzalone, M. Mauceri, F. La Via, P. J. Wellmann, *Materials Science Forum*, 1062, 74 (2022).
- [5] P. Wellmann, P. Desperrier, R. Mueller, T. Straubinger, A. Winnacker, F. Baillet, E. Blanquet, J. M. Dedulle, M. Pons, *Journal of Crystal Growth*, 275, 1-2, e555 (2005).
- [6] V. Jokubavicius, G. R. Yazdi, R. Liljedahl, I. G. Ivanov, R. Yakimova, M. Syväjärvi, *Crystal Growth and Design*, 14, 12, 6514 (2014).
- [7] V. Jokubavicius, G. R. Yazdi, R. Liljedahl, I. G. Ivanov, J. Sun, X. Liu, P. Schuh, M. Wilhelm, P. Wellmann, R. Yakimova, M. Syväjärvi, *Crystal Growth & Design*, 15, 6, 2940 (2015).
- [8] T. Hupfer, P. Hens, M. Kaiser, V. Jokubavicius, M. Syväjärvi, P. J. Wellmann, *Materials Science Forum*, 740-742, 52 (2013).