# On the Preparation of Vanadium-Doped Semi-Insulating SiC Bulk Crystals

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High resistivity, semi-insulating SiC single crystals are gaining more and more importance as substrates for high frequency electronic devices based on both SiC and GaN. Vanadium can act in SiC as a deep level for the electrical compensation of residual impurities. As nitrogen is the predominant impurity in nominally undoped crystals, V doping leads to the activation of the V acceptor level, resulting in a specific resistivity of about $10^{11}$ Ωcm at room temperature. Co-doping of V and a p-type dopant like Al or B is required to activate the almost mid-gap V donor level leading to specific resistivities up to $10^{15}$ Ωcm. In any case, the relatively low solubility limit of V in SiC must not be exceeded. For obtaining bulk crystals of semi-insulating SiC, doping homogeneity is crucial as will be discussed in this paper.

More than 30 bulk 6H-SiC crystals with 35…40 mm in diameter were grown by the modified Lely technique using on-axis seeds. The crystals were doped by boron, vanadium or B/V co-doped by adding solid sources to the SiC starting material. Results from the growth of nominally undoped crystals were taken as a reference. Here, nitrogen was found to be the residual impurity on a very low level, with charge carrier concentration n decreasing exponentially with growth time. Wafers with n as low as $8\times10^{15}$ $cm^{-3}$ were obtained. The dependence of seed polarity on nitrogen incorporation will be addressed. Assuming the residual impurity incorporation is the same for all of our growth experiments, the impact of impurity incorporation on electrical properties during doped SiC growth can be determined.

Tab. 1: Chemical analysis of the B concentration in the sublimation source and in the grown crystals for two experiments, measured both at the beginning and at the end of growth, respectively.

| Boron [ppm wt.] | | SiC powder | SiC crystal |
|---|---|---|---|
| B-doped #1 | Start | 2,7 | 2,0 |
| | End | 5,5 | 2,0 |
| B-doped #2 | Start | 26,7 | 7,3 |
| | End | 5,4 | 4,6 |

The homogeneity of boron incorporation was measured by temperature-dependent Hall effect, absorption mapping and specific resistivity mapping. B incorporation was found to depend on seed polarity (growth on C or Si face). The hole concentration increases with growth time, whereas boron losses during growth lead to a decrease in the boron content of the source material (see Tab. 1). Compensation of boron by nitrogen impurity incorporation can explain this behavior only in the beginning of growth. A mechanism for the increase of hole concentration will be discussed. Also, the influence of dopant incorporation on crystal quality and defect nucleation was investigated with optical microscopy. Lateral homogeneity of a B doped wafer as measured with absorption mapping was as low as $\Delta p/p \approx 15\%$.

V incorporation was found to be related to partial pressure of the V species during growth. Additionally, V is incorporated in higher concentrations when growth on the Si face is

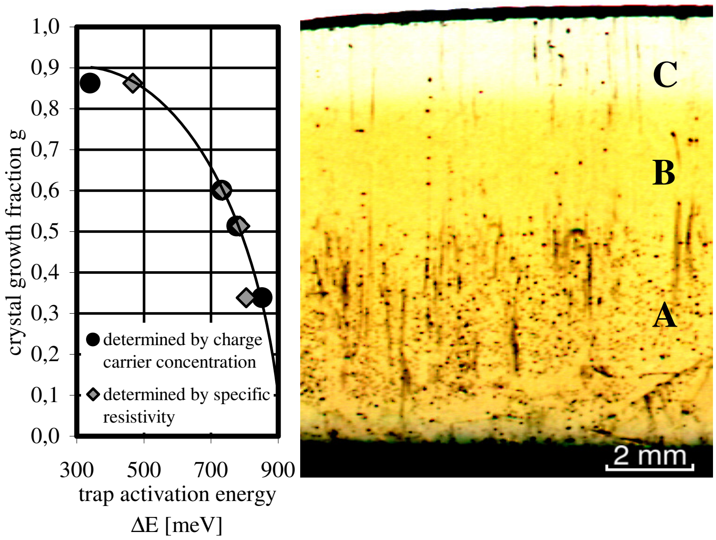


Fig. 1: Cross-sectional cut of a V doped SiC wafer (growth direction is upwards). On the left side, trap activation energies of the respective positions in the crystal are shown. For description of the regions A, B, C see text.

performed. As $p_{VC} > p_{SiC}$ at growth temperature, the V source depletes during growth. The obtained crystals exhibit axial and lateral inhomogeneities (see Fig. 1). When the V solubility limit of $3 \ldots 5 \times 10^{17}$ $cm^{-3}$ is exceeded, vanadium-rich precipitates form (Fig. 1, A), which were identified by EDX measurements. On the other hand, when the V source depletes, residual nitrogen becomes predominant leading to n-type conducting behavior (Fig. 1, C). By reducing V species evaporation rate, bulk SiC crystals exhibiting precipitate-free, semi-insulating behavior (Fig. 1, B) were obtained.

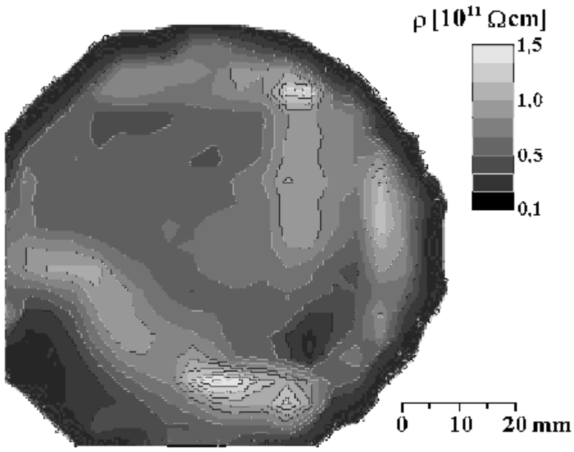


Fig. 2: Specific resistivity mapping on a V doped SiC wafer, obtained by the scanning capacitance method.

Characterisation of doping inhomogeneities and doping-related defects were carried out by optical microscopy and scanning electron microscopy. The electrical properties of V doped SiC crystals were measured by temperature-dependent Hall effect and by the scanning capacitance method. As a result, specific resistivities extrapolated to room temperature are about $2 \ldots 8 \times 10^{10}$ Ωcm, while resistivity mappings reveal doping imhomogeneities (Fig. 2). The influence of V concentration and compensation ratio on the electrical behavior was analyzed. Optical absorption peak structures in the near infrared, which are attributed to inner shell transitions of $V^{4+}$, and electron spin resonance showing $^{51}V^{3+}$ were used to verify the compensation mechanism in the investigated crystals.

A combination of boron and vanadium doping allows growth of bulk SiC with a specific resistivity of $\rho_{293K} \approx 10^{15}$ Ωcm. First results on B/V co-doped SiC crystals show that boron and vanadium incorporation do not interfere with each other. Using the elaborated transfer coefficients for the dopants and their evolution of incorporation during growth time, semi-insulating bulk crystals of B/V co-doped SiC were obtained. Compensation behavior was studied using optical absorption. Specific resistivity mapping as well as temperature-dependent Hall effect measurements were used to determine electrical behavior and doping inhomogeneities.