Oxygen in PVT Growth of Bulk AlN: Influence on Growth Process and Crystal Properties.

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Oxygen is the most common impurity in semiconductor nitrides. It is known to influence electrical, optical and thermal properties. However, the role of oxygen during sublimation-recondensation (PVT) growth of bulk AlN crystals has been paid little attention by now. Our current study was performed to evaluate the influence of oxygen contamination on the growth process and the crystal properties.

Sublimation experiments were performed with AlN powders containing up to 1 % wt of Al_2O_3 in tungsten crucibles at temperatures in the range from 2100-2400°C. It was found that at growth temperatures exceeding 2000°C oxygen, even at high concentrations, acts as an effective transporting agent, but does not produce any oxynitrides and does not alter the AlN crystal structure. However, during heating stage at temperatures of 1300…1500°C, oxynitride formation on the seed was observed. The temperature reversal method was used to resublime the oxynitride layer after bringing it to working temperature and to improve seeding conditions. Additionally, the AlN powder was purified significantly by resublimation prior to growth. Applying these techniques, dense polycrystalline boules up to 2 inch in diameter containing crystallites up to 5 mm in size have been grown.

We were able to fabricate AlN crystals with the lowest oxygen content reported up to date. Chemical analysis carried out by glow discharge mass spectroscopy (GDMS) showed an oxygen content of lower than 80 ppm wt, equaling a concentration of 1 x 10^{19} cm^{-3} in the crystal. Optical absorption measurements in the 200…3000 nm wavelength range reveal distinct absorption bands at about 300 nm and about 450 nm. Following Slack and co-workers, they may be attributed to oxygen content and nitrogen vacancies, respectively. The low absorption coefficient of α = 30…60 cm^{-1} obtained in this range confirms the low oxygen content of our crystals. Using the laser flash method, a thermal conductivity of λ = 186 W / m K at room temperature and a T^{-1} dependence in the temperature range between 300 K and 1500 K were found. Additionally, Raman scattering and FTIR measurements confirm high quality and low oxygen content of the grown crystals.