

4H-SiC Crystal Growth Using Recycled SiC Powder Source

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Abstract. A new method for reducing the cost and the fabrication time of source material required for SiC crystal growth has been proposed through a heat treatment of recycled powder bulk in this study. The actual crystal growth with using a conventional powder and a recycled powder bulk source has been performed under identical growth condition and then systematically compared in terms of the crystal quality. With applying the recycled powder bulk for SiC crystal growth, similar growth results were obtained as a result grown by conventional high-purity powder source. In terms of crystal defects, slight improvement was observed when high purity recycled powder bulk source was applied.

Introduction

Commercially available SiC-power-devices of MOSFETs and SBDs are recently fabricated on n-type 4H-SiC substrates with 6-inch in diameter. For improving device performance and device yield, the quality of large diameter SiC wafer is important [1,2], and high purity SiC source materials are indispensable for growing high quality SiC crystal. However, various difficulties follow in the fabrication process of SiC powder sources used in the PVT method to grow SiC crystal. In order to form a high-purity SiC powder source, a high-purification treatment through chemical reaction has to be involved, but the cost required for this process is basically very high. In addition, problems relating with the environmental contamination due to various chemical reactions and the by-products occurrence during the high-purification process may occur, and even after the high-purification process, unstable reproducibility and crystal quality degradation due to residual impurities could occur [3,4]. In this study, a reproducible growth method of high-quality 150mm SiC ingots through the cost reduction by the source powder recycling was proposed with reducing impurities in source materials.

Experimental

The heat treatment process consisting of three steps was carried out for the recycled powder bulk, as shown in Fig. 1. In the first step for removal of the carbon ash content, carbon ash on the surface of the powder bulk that could cause carbon inclusion in the crystal can be removed by heating the powder bulk at a temperature of 600°C for 12 hours using a furnace in air atmosphere [5]. In the second step for the removal of oxide layer, HF treatment is performed at room temperature for 6 hours to remove the oxidation layer on the surface of the powder bulk. The final drying step is performed at 600°C for 12 hours under an inert Ar atmosphere using a high-temperature furnace. Photograph of heat-treated recycled powder bulk after the heat treatment and a schematic diagram of crucible showing loaded recycled powder bulk were shown in Fig. 2. The heat-treated recycled powder bulk placed in the center of the crucible and powders with small sintered particle filled in remaining part of graphite crucible were used as source materials in a physical vapor transport (PVT) process. Fig. 3 exhibited the growth condition including temperature and pressure profile of each growth step for SiC single crystal. The SiC crystal were grown at the temperature of 2000~2400°C and with argon inert gas of 1~40 torr including 5~10% nitrogen. The axial thermal gradient of the SiC crystal during the growth

is estimated at the range of 15~20°C/cm. The seeds and the source materials of high purity SiC are placed on opposite side in a sealed graphite crucible which is surrounded by graphite insulator. Two SiC single crystals grown with using the conventional powder and the recycled powder bulk sources under the identical growth condition were systematically compared. UVF and etch pit density (EPD) measurements were used to investigate the crystal quality of grown crystals. The resistivity of crystal wafer was measured to check the change of the electrical properties caused by nitrogen incorporation into the recycled powder bulk prepared through the high-purification treatment.

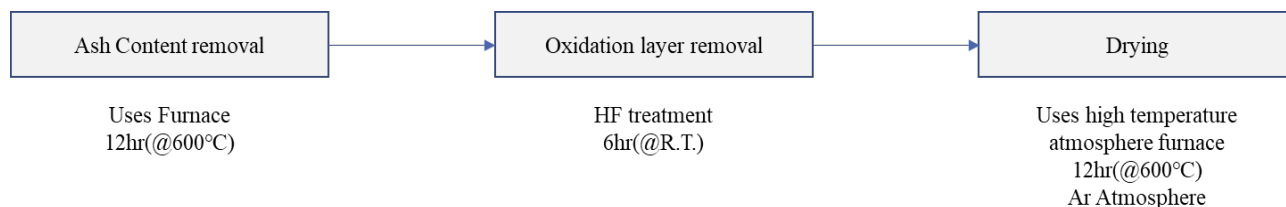


Fig. 1. The flow chart for heat treatment process and heat treatment conditions for each step.



Fig. 2. Photograph of heat-treated recycled powder bulk after the heat treatment and schematic diagram of crucible showing loaded recycled powder bulk.

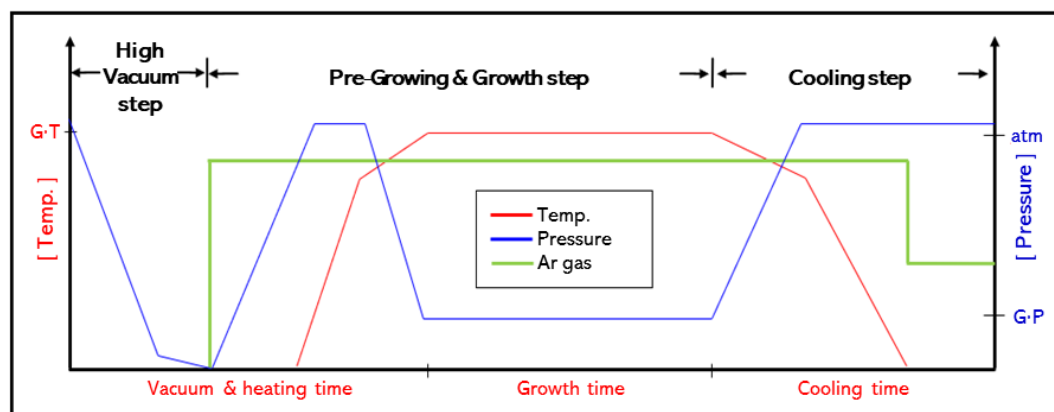


Fig. 3. The growth condition for SiC single crystal. Temperature and pressure profile for growth step (G.T., G.P.) were indicated on the graph.

Result and Discussions

Growth rate and reproducibility. The growth rate, polytype inclusion and crystal ingot shape of the SiC crystal ingots grown using conventional high-purity powder source and the recycled powder source were compared. Table 1 shows the shape and growth thickness of both crystals and the growth thickness for two crystals were observed to be almost similar level. The surface shape of the growth ingots exhibited a convex shape on both crystals and the crystal shape grown using recycled powder source had relatively more convex. Fig. 4 exhibited the height distribution table from the center and edge of the SiC ingots grown using each powder source used in PVT process. While SiC crystal grown with the conventional high purity source showed a sharp crown convex shape, a uniform convex shape was obtained in SiC crystal grown with recycled powder source. The temperature

gradient to determine the crystal front shape is well known to affect crystal quality, such as polytype inclusion and dislocation formation.

As a result of UVF analysis to check polytype inclusion in Fig. 5, it was found that polytype inclusion was suppressed even in grown crystal grown with recycled powder source. In summary, the recycled powder source resulted in similar growth results as the conventional high purity powder, in terms of growth rate and reproducible polytype stability.

Table 1. Shape and growth thickness of crystals grown using high purity powder and recycled powder sources.

	High purity powder	Recycled powder
Shape ($\Delta C-E$)	2.83mm	2.85mm
Thickness	20mm	19mm

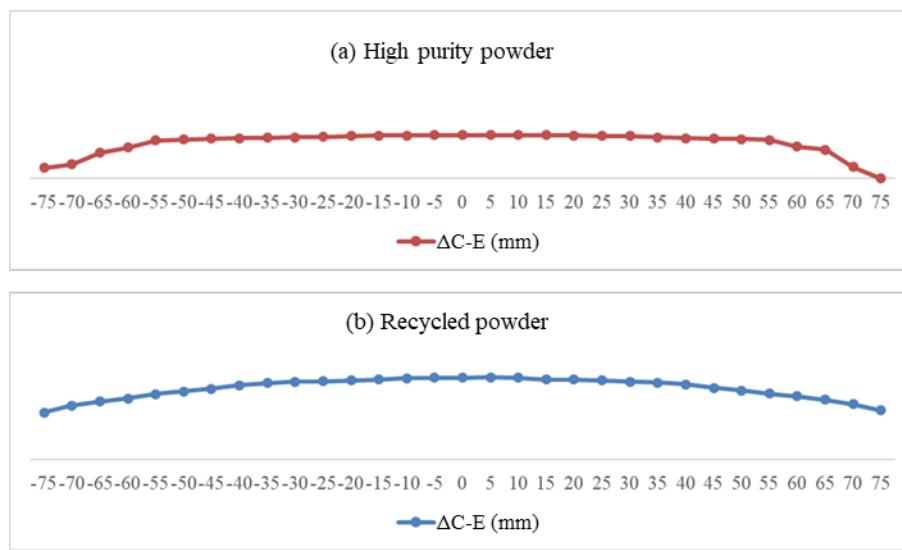


Fig. 4. Height distribution from edge to center of SiC ingots with using (a) conventional high purity powder and (b) recycled powder sources.

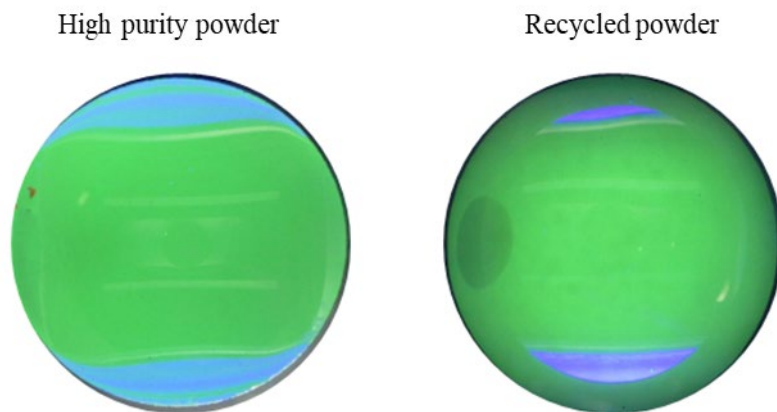


Fig. 5. UVF images of crystal ingots grown using conventional high purity powder and recycled powder sources.

Suppressing defect formation. Table 2 and Fig. 6 exhibited EPD analysis results of SiC crystals grown with grown conventional high-purity powder and recycled powder sources. No distinct difference in EPD values except for threading screw dislocation (TSD) density between two grown crystals was observed. TSD value of SiC crystal grown with recycled powders was definitely lower than that of conventional powder. It is presumed that the formation of defects could be affected by

different source materials, the sublimation of smaller-sized high purity powder and the sintered body composed of small-sized particles that were highly purified and treated with hydrofluoric acid at high temperature [6,7].

Table 2. EPD of SiC crystals grown with grown conventional high-purity powder and recycled powder sources.

	High purity powder	Recycled powder
BPD (ea/cm ²)	4,200	1,500
TED (ea/cm ²)	400	520
TSD (ea/cm ²)	200	10

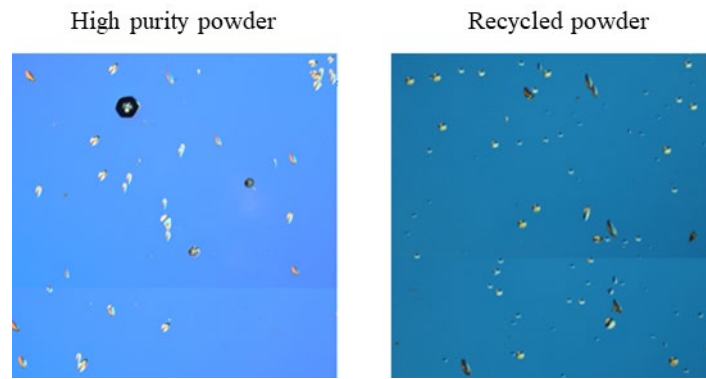


Fig. 6. EPD images of SiC crystals grown with grown conventional high-purity powder and recycled powder sources.

Effects on electrical properties. Impurities in the powder source were removed through the high purification process. To apply this powder source to the growth of semi-insulating-SiC crystal, much attempt to reduce nitrogen concentration in the powder through high purification treatment has to be required. Therefore, the resistivity of the wafer to investigate the effect of the high-purification treatment proposed in this study on residual nitrogen concentration. Fig. 7 exhibited the resistivity measurement value of wafers grown with conventional high purity powder and recycled powder source. The average value of wafers grown with conventional high purity powder and recycled powder source were 21.06mΩ·cm, and 20.97mΩ·cm, respectively, indicating no difference between two crystals having similar effect in suppressing nitrogen incorporation.

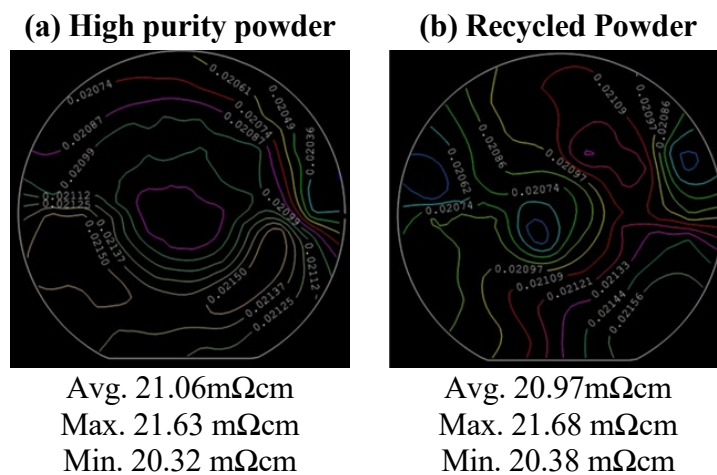


Fig. 7. Resistivity mapping and resistivity values of SiC crystal wafers prepared with using (a) conventional high purity powder and (b) recycled powder sources.

Summary

In this study, heat-treated recycled powders was used as source materials of PVT process for the growth of high-quality SiC ingots. The actual crystal growth with using a conventional powder and a recycled powder bulk source has been performed under identical growth condition and then systematically compared in terms of the crystal quality. The recycled powder source resulted in similar growth results as the conventional high purity powder, in terms of growth rate and reproducible polytype stability. No distinct difference in EPD values except for threading screw dislocation (TSD) density between two grown crystals was observed. TSD value of SiC crystal grown with recycled powders was definitely lower than that of conventional powder. In terms of crystal defects, slight improvement was observed when high purity recycled powder bulk source was applied.

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