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Growth rate and growth steps of 6H-SiC single crystals in the sublimation process

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Abstract 6H-SiC bulk crystals were grown by sublimation method with different conditions in term of gaseous pressures and source temperatures. In order to optimize the growth rate, pressure at growth period and source and substrate temperatures were investigated as experimental variables. The results were compared with each other and finally the optimum growth conditions were discussed. Furthermore the relation of the growth steps and defects formation was evaluated in the point of reducing the micropipes. Subsequently the growth steps were observed leading to the lower step height with the lower growth rate.

1. Introduction

Silicon carbide is well-known wafer materials for high temperature and/or power devices from the starting of blue LED's wafers as well as abrasives. In order to apply the materials, it is required to reduce the defects such as micropipes and internal planar defects. Recently, 6H- and 4H-SiC were commercialized by a diameter of up to 3 inches. The number of micropipes with a few per square centimeter could be essential for the semiconductor uses [1, 2]. However 6H-SiC single crystals are originally grown along c-axis with spiral growth pattern, so the micropipes that may have the origin as growing spiral defects center can be grown together with the step growth. For semiconductor grade of SiC wafers, the defects concentration should be decreased down to the value of almost free and the growth rate have to be controlled to make the flat growth planes having more large area parallel to hexagonal basal plane at growing surface.

In this work, in order to optimize the growth rate, pressure at growth period and seed substrate temperatures were investigated as experimental variables. The results were compared with each other and finally the optimum growth conditions were discussed. Furthermore the relation of the growth steps and defects formation was evaluated in the point of reducing the micropipes. Subsequently the growth steps were observed leading to the lower step height with the lower growth rate.

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2. Experimental

SiC single crystals were grown by sublimation process (modified Lely method). Using the 5 kHz radio frequency induction heating generator system, the seed and SiC powder source temperature were controlled in the range of 2000~2100°C and 2100~2300°C. The ambient pressure was controlled also in the range of 400~600 torr. The seeds used in this experiment were grown crystals by Acheson process, which was attached to a lid of graphite crucible, so that the seed was put on the upper side of the crucible. Temperature difference between the source surface and seed substrate in the crucible was referred to the temperature gradient and then the temperature gradient was about 22~35°C/cm.

The growth rate for grown crystals was evaluated by calculated values divided the length of the crystals over crystal growing time. We observed the surface morphology and growth steps of as-grown crystals using an optical microscope.

3. Results and Discussion

3.1. Kinematic theory

The relation between crystal surface and step mor-

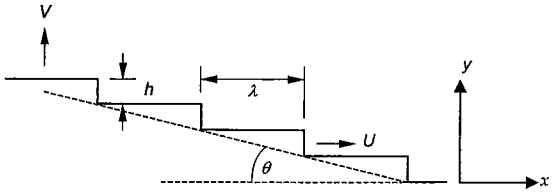


Fig. 1. Two-dimensional diagrammatic representation of steps on a crystal face

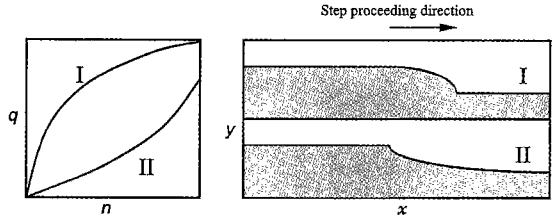


Fig. 2. (a) Step flux density curves: type I, $d^2q/dn^2 < 0$; type II, $d^2q/dn^2 > 0$. (b) Surface profiles arising from bunches with type I and type II kinetics, respectively

phology and the growth rate was discussed on the basis of kinematic theory for the growth step formation. Two simultaneous process are involved in the layer growth of crystals, at first, the generation of steps at some source on the crystal face followed by the movement of layers across the face. The characteristics of step motion and step bunching have been considered and developed to a kinematic theory of crystal growth [3].

Growth, and the reverse process of dissolution, may be considered in terms of the progression of steps across a crystal face (Fig. 1). The step velocity, u, depends on the proximity of the other steps since all steps are competing for growth units. Thus

$$u = q/n \tag{1}$$

where q is the step flux (the number of steps passing a given point per unit time) and n is the step density (the number of steps per unit length in a given region). The distance between steps, $\lambda = n^{-1}$. The slope of the surface, p, with reference to the close packed surfaces, i.e. the flat ledges, is given by

$$p = \tan\theta = hn \tag{2}$$

and the face growth rate, v, normal to the reference surface by

$$v = hq = hnu \tag{3}$$

where h is the step height.

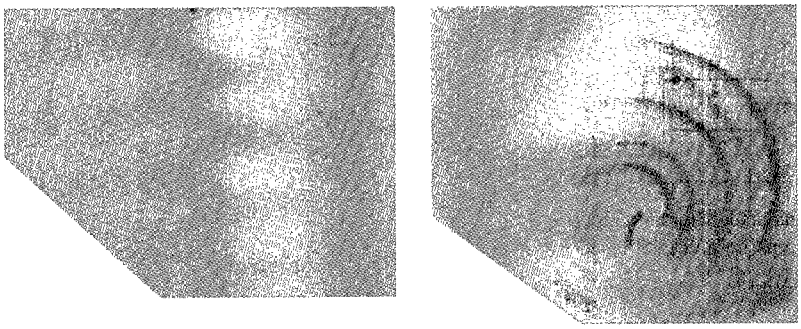
If the steps are far apart ($\theta \rightarrow 0$), and diffusion fields do not interface with one another, the velocity of each step, u, will be a maximum. As the step spacing decrease and the slope increases, u decrease to a minimum at $hn = 1$ ($\theta = 45$). As the slope q increases, the face growth velocity v ($= u \tan \theta$) increases, approaches a flat maximum and then decreases to zero. The shape of this v (p) curve, which is affected by the presence of impurities, is an important characteristic of the growth process.

For the two-dimensional case depicted in Fig. 1, another velocity, $c = dx/dt$, may be defined which represents the motion of kinematic waves (regions on the crystal surface with a constant slope p and velocity v). These waves do not contain the same monomolecular steps all the time, as the step velocity $u = v/p$ can be greater or less than c. When two kinematic waves of different slope of different slope meet, a discontinuity in slope occurs, giving rise to shock waves across the surface.

The kinematic theory also can be adapted to the step bunching. The steps that flow across a face are usually randomly spaced and of different height and velocity. Consequently they pile-up or bunch, Growth, and dissolution can be characterized by the relationship between the step flux, q, and step density, n. Two general forms of this relationship can be considered depending upon whether $d^2q/dn^2 < 0$ (Type I) or $d^2q/dn^2 > 0$ (Type II). The former is analogous to the flow of traffic along a straight road and the latter to flood water on a river (see Fig. 2).

3.2. Growth rate and steps

In the case of 6H-SiC, the crystal grew along the seed direction, <0001> and the large loops of growth steps were formed on the grown surface. These steps are appeared by two kinds of loops. The one was the smooth wave shape boundary of large flat terrace planes and another was a spiral growth step due to the screw dislocation (Fig. 3). There was micropipes in the center of spiral steps in Fig. 3(b), so it was recognized that the origin of micropipes might related to the screw dislocation. Impurities on the growth surface, mainly carbon particles or aggregates, could give rise to form a micropipes by the way of substitution to the growth kink in the lattice [4]. Theoretically when the super saturation concentration was low, the spiral growth steps were formed and step spacing was kept constantly.



(a) (b)

Fig. 3. Typical growth steps pattern in the grown SiC single crystal surface. (a) smooth loop pattern, (b) spiral pattern.

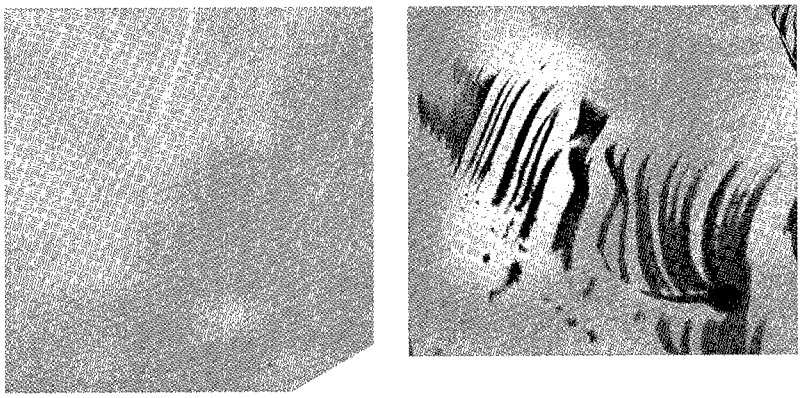
Table 1
The calculated growth rate according to the growth conditions with the variables of the source temperature and gas pressure (mm/hr)

Gas pressure	Growth temperature	2200°C (Source side)	2300°C (Source side)
(a) 400~500 torr		0.3~0.5	0.1~0.2
(b) 500~600 torr		0.7~1.2	0.5~0.7
Temperature gradient		22~35°C/cm	

Since SiC had the habit of spiral growth, we could obtain an important point in the SiC crystal growth. That was if large terrace plane was formed from the growth start, the grown surface could have more large flat area and the lower growth step height [5, 6]. The various growth conditions are presented in Table 1 with the variables of source temperature and growth pressure in the growth chamber.

Considering the relationships between each growth rate condition and resultant growth steps, at first, the case (a), the steps propagation pattern looked like a stable sequential arrangement of paper sheets. Micropipes were not formed in this area and any spiral step pattern also was not observed. The reason of this kind of pattern formation was because the vertical face growth velocity (v) was less than the ledge propagation velocity (u) in the growth equilibrium state. This kind of step pattern was due to the small temperature gradient in the horizontal direction on the growth plane. Step bunching was observed in the steps that were formed in central region in the crystal [Fig. 4, (a)]. Growth steps bunched in a certain region and the pattern was type II in Fig. 2.

In Fig. 4(b), it was recognized that the micropipes could prohibit the propagation of the growth steps forming a circular shape hole vacancy with the step bunching. This condition of the growth was the case of (b). Growth rates were faster than the former conditions even on the basis of temperature. This meant



(a) (b)

Fig. 4. Micrograph of growth steps (a) in the central region and (b) in the peripheral region.

that the sublimed atoms (Si and C) and molecules (Si_2C, SiC_2 and etc) could not have enough time to move to and locate at the sites which were made by BCF theory in the crystal lattice. The rearrangement of the atoms on the growing surface was become unstable, so the step flux, q, became to have higher value. The type I was due to this reason.

The growth rate in the optimum condition of 6H-SiC was (a) condition in Table 1. The lower the growth rate, the larger the ledge plane. This meant that the horizontal growth velocity became to higher value, and then the terrace area in the growing surface could be larger. The growth steps also could propagate from center region to the peripheral region with the shape of floodwater on a river.

4. Conclusion

6H-SiC single crystal was grown by sublimation process (modified Lely method) in the optimum growth condition as Table 1. The grown crystal surface was observed by optical microscope and obtained the morphology of the growth steps and micropipes defects. In the region, which the stable growth steps were formed, the horizontal temperature gradient was small. However, the closer to the peripheral region, the larger the temperature gradient, and then growth steps were bunched each other (type II).

It was recognized that the step bunching near the micropipes was due to the prohibition of the step propagation according to the micropipes. Also, since the growth steps morphology was not affected by the growth rate directly, the super saturation concentration of the atoms near the growth plane even though the growth rate and temperature were optimum values. As the super saturation concentration was a function of the temperature gradient, the growth steps behavior could be independent on the growth rate.

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