Surface Properties of SiC Layer Grown by Molecular Beam Epitaxy (MBE) with Helicon Sputtering Molecular Beam Source*

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Although there have been some attempts to produce a monocrystalline silicon carbide (SiC) flat surface, the surface properties, such as surface roughness, have not satisfied the required specifications. In this study, we apply a helicon sputtering device to molecular beam epitaxy (MBE) to improve those properties. The helicon sputtering device was used as a molecular beam source for generating a Si molecular beam, where the electric field caused by the helicon coil supplied energy to the sputtered Si molecules. The amount of energy was controlled by the electric power applied to the coil. High-purity acetylene gas was used as the carbon (C) molecular beam source. The substrate was a monocrystalline (111) Si wafer. With the increase of the electric power, that is, the supply of high energy to molecules, the roughness of the surface was improved. A uniform mirror surface of monocrystalline SiC was produced over the entire substrate with a roughness of 1 nm (Ra) order.

Key Words: Non-Traditional Machining, Ultra-Precision Machining, Surface Roughness, Molecular Beam Epitaxy, Helicon Sputtering, Silicon Carbide, Monocrystal

1. Introduction

Molecular beam epitaxy (MBE) is one of the crystal growth processes. The principle is based on the autonomous alignment and the crystal growth of molecules supplied from a molecular beam source. There have been some attempts to apply MBE to mechanical applications utilizing both high-purity monocrystalline material and the accuracy of molecular order^{(1),(2)}.

Silicon carbide (SiC) has excellent mechanical characteristics, but it is difficult not only to form high-purity monocrystalline SiC but also to achieve a smooth surface at the atomic level. There have been

some attempts to produce a flat monocrystalline SiC surface by nonmechanical processes, for example low-pressure CVD^{(3),(4)}. However, the surface properties, such as surface roughness, have not satisfied the required specifications. The use of MBE may make it possible to improve those properties, but this has not yet been achieved⁽⁵⁾. In current MBE, the energy that is necessary for epitaxial growth is supplied from the substrate as heat. The high temperature promotes the migration and desorption of supplied molecules on the substrate and the expansion of pits. Consequently, the roughness of the finished surface degrades. Furthermore, it is difficult to obtain a stable molecular beam of high-melting-point material such as silicon. One of the solutions to these problems is to supply energy in another form; for example, the helicon sputtering device can excite the molecules while maintaining a low temperature. We undertake the application of the helicon sputtering device to the MBE process to improve the surface properties.

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2. Experimental Setup and Conditions

In MBE, it is necessary to vaporize the growth material to obtain the molecular beam when that material is solid. The means of vaporization is usually melting and evaporation of the material by heating or electron irradiation. However, it is difficult to vaporize high-melting-point materials. Moreover, it is also difficult to control the internal energy and the kinetic energy of the vaporized molecules. However, these problems do not arise with a helicon sputtering molecular beam source because it vaporizes the growth material by sputtering. Figure 1 shows the schematic and a photograph of the helicon sputtering molecular beam source. This device is provided with a target of the growth material and a helicon coil installed in front of the target. Moreover, permanent magnets are equipped on the reverse side of the target. The high-frequency (RF) power supply of 13.56 MHz is applied to both the helicon coil and the target. When high-purity argon gas is introduced, the gas is excited by the RF power of target, and the target material is sputtered. Sputtering can stably vaporize even the high-melting-point material. The amount of sputtered molecules can be controlled by the RF power of target. When the sputtered molecules pass through the electromagnetic field generated by the RF power of coil, they are all aligned in the same direction and the energy is supplied to there. The amount of energy can be controlled by the RF power of coil. There is little influence on the substrate because this sputtering device operates independently of the substrate. We used the helicon sputtering molecular beam source made by ULVAC Incorporation as the supply source of silicon.

Fig. 1 Photograph and schematic of helicon sputtering molecular beam source

Figure 2 shows the schematic of the MBE apparatus. In this study, high-purity acetylene gas (6N) was used as the supply source of carbon. The pressure of this chamber can be lowered to 10^{-8} Pa or less. The substrate of 100 mm diameter can be heated to 1 473 K while rotating. The epitaxial growth of the silicon carbide can be carried out on the heated substrate onto which an appropriate amount of acetylene and silicon are injected.

Figure 3 shows the epitaxial growth process of the silicon carbide in this study. The process consists of two stages, that is, carbonization of the substrate and silicon carbide growth. In the carbonization process, the substrate is carbonized by heating at 1 273 K for 30 minutes and supplying only acetylene after the cleaned substrate is set in the chamber. This process is effective for the prevention of lattice mismatch at the interface between the substrate and the growth layer, because the lattice constants of silicon

Fig. 2 Schematic of MBE apparatus

Fig. 3 Process of epitaxial growth

Table 1 Experimental conditions

Carbonization	
C_2H_2 pressure Pa	$6.7E^{-2}$
Substrate temperature K	1273
Carbonization time min	30
SiC growth	
C_2H_2 pressure Pa	$3.3E^{-1}$
Substrate temperature K	1273
Si target power W	150
Si coil power W	0 - 150
SiC growth time min	15 - 60

Table 2 Specifications of substrate

Material	Single-crystal silicon
Surface orientation	(111)
Process	Czochralski(CZ) method
Type	P type (B doped)
Resistivity Ωcm	8~12
Diameter mm	100
Thickness μm	450

and silicon carbide are different. In addition, the carbonized layer is effective for preventing the evaporation of silicon from the substrate at high temperature. After the carbonization process, the epitaxial growth process forms the silicon carbide layer. This process can be carried out by supplying silicon and acetylene onto the carbonized layer at the same temperature, namely, 1273 K.

The experimental conditions are listed in Table 1. All the experiments can be performed under the same conditions by controlling, for example, the heating time and programming rate. Only the RF power of coil and epitaxial growth time were varied from 0 to 150 W and from 15 to 60 minutes, respectively. The other conditions were the same.

The specifications of the substrate are listed in Table 2. All the substrates were produced in the same lot. The substrates were cleaned according to

Fig. 4 Relationship between RF power of coil and grown thickness

the predetermined procedure, such as contaminant removal by ultrasonic cleansing with ethyl alcohol and natural oxidation film removal using hydrofluoric acid.

3. Basic Performance of Helicon Sputtering Molecular Beam Source

Figure 4 shows the thickness of the silicon layer deposited at room temperature when the RF power of coil was varied from 0 to 150 W. The thickness increases in proportion to the RF power of coil. It is thought that the amount and sticking probability of the molecules sputtered onto the substrate increased due to the effect of the molecules being aligned in the same direction and supplied energy from the RF power of coil.

Figure 5 shows an example of the epitaxially grown silicon carbide layer on the substrate of 100 mm diameter. The finished surface is specular with uniform thickness over the entire substrate. This means that epitaxial growth occurred uniformly on the substrate. The chemical composition of the epitaxially grown layer was analyzed by Auger elec-

Coil power 100 W
Target power 150 W
Temperature 1273 K
Time 60 min

Fig. 5 Example of epitaxially grown silicon carbide layer

tron spectroscopy and etching. Figure 6 shows an example of the results of analysis in the depth direction for the substrate in Fig. 5. The composition ratio

Fig. 6 Chemical composition analysis in depth direction

(a) Observation of geometric character of grown surface using AFM

(b) Observation of crystallinity of grown surface using RHEED from $[11\bar{2}]$ direction

Fig. 7 Surface properties of silicon carbide layer at various grown thicknesses (RF power of coil 150 W)

of silicon to carbon is uniformly 1:1 in the depth direction of the grown layer. There are no impurities in this layer. It is confirmed that a stable silicon carbide layer was generated.

4. Experimental Results and Discussion

To clarify the epitaxial growth process, the relationship between the growth time, namely, the grown thickness, and the surface properties was investigated. The grown thickness was varied from 50 to 220 nm, which is the equivalent of the growth time of 15–60 minutes, while other conditions were kept the same. The surface geometry and crystallinity, which were observed by atomic force microscopy (AFM) and reflection high-energy electron diffraction (RHEED), respectively, were the investigated surface properties. Figure 7 shows the change of the surface properties of the silicon carbide layer with grown thickness. All layers are monocrystalline regardless of the thickness. However, the crystallinity deteriorated slightly at thicknesses of 170 and 220 nm. The surface consists of three-dimensional nuclei of nanometer order. There are not very many changes of the shape of the nucleus with thickness. In the former experiments performed without using the helicon sputtering molecular beam source, many triangular pits with side lengths of about 100 micrometers were observed on the surface⁽⁶⁾. However, no pits are observed in the present experiments with using the helicon sputtering molecular beam source. The surface roughness improves with increasing thickness. The reason for these changes is not clear. It is shown that the grown thickness should be selected appropriately to obtain the desired surface properties.

To clarify the effect of the RF power of coil on the epitaxial growth process, the relationship between the RF power of coil and the surface properties was investigated. The RF power of coil was varied from 0 to 150 W while the other conditions were kept the same. The results are shown in Fig. 8. The layer in the case of the RF power of 0 W at the coil is polycrystalline. However, it is monocrystalline at all other RF powers. Moreover, the crystallinity improves with increasing RF power of coil. This means that the energy at the RF power of 0 W was insufficient for epitaxial growth, and the RF power contributed to the energy for epitaxial growth. The RF power of coil also influenced the surface geometry. The best roughness was obtained in the case of 50 W. The size of the nucleus on the surface depends on the RF power, as shown in Fig. 9. Although the radius of the nucleus is not greatly influenced by the RF power, the height of the nucleus increases with RF power. This means that the roughness degrades when the energy exceeds a

(a) Observation of geometric character of grown surface using AFM

(b) Observation of crystallinity of grown surface using RHEED from [112] direction

Fig. 8 Surface properties of silicon carbide layer at various RF powers of coil

Fig. 9 Relationship between RF power of coil and size of the nucleus

certain level. The reason for this is not yet clear and is being investigated now. It is shown that the RF power of coil should also be selected appropriately to obtain the desired surface properties.

5. Conclusions

We produced monocrystalline silicon carbide by MBE with a helicon sputtering molecular beam source

for silicon. This method can yield stable and uniform monocrystalline extrasmooth silicon carbide of 100 mm diameter stably and uniformly. The surface properties of the grown layer can be controlled by adjusting the RF power of coil in the helicon device. The best results were obtained in the case of the RF power of 50 W, where the roughness was 1.5 nm (Ra).

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