Title:

Single crystal SiC thinfilm produced by epitaxial growth and its application to micro-mechanical devices

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Abstract:

This paper deals with the fabrication process of single-crystal silicon-carbide (SiC) thin-films and its application to microdevice. SiC thin film was synthesized using molecular beam epitaxy, where single crystal SiC layer was grown on single crystal silicon (Si) substrate. Using lithography and etching process, microscopic cantilevers were fabricated. Typical dimensions of the cantilevers were 10-60μm in length, 10-30μm in width, typically 180nm in thickness. Young's modulus estimated from bending test was almost same with that of bulk material. Finally, an application is demonstrated where nickel was deposited on the cantilever and biomorphic actuation was carried out. The displacement at the tip was about 2μm when the temperature change was 40K. The time constant of the step response was about 0.07s.

Key words: silicon carbide, single crystal, cantilever, actuator

1. Introduction

Silicon carbide (SiC) has excellent mechanical properties and has been applied for micro-sensors for high-temperature environments [1]. However, no absolute processes for the production of SiC structure have been established. Material defects such as grain boundaries or dislocations should be eliminated in the application in microscopic scale because they trigger the fracture of material. Thus, single crystal material is ideal rather than the poly-crystal.

So far, chemical vapor deposition (CVD) process is applied to produce single crystal SiC on Si substrate [2]. In many cases, more than two gas-sources are necessary and the control of the process is not easy. To overcome this problem, another process that uses one gas-source, 1,3-Disilabutane, has been proposed [4], and its mechanical properties have been evaluated [5]. However, it is not easy to produce single-crystal layer without defects. Epitaxial growth process can afford high-quality single-crystal thin film though the growth rate is limited. Hexagonal lattice SiC, 6H-SiC, is often applied because the difference of lattice constant from that of Si is smaller than other lattice types [7]. Multi-layered structures [8] or new type of process is proposed [9] though the evaluations of properties are not completed. Hetero-structure often causes unnecessary residual stresses. Various studies [10-11] have made clear how to reduce the stress.

On the other hand, profiling process after the SiC layer deposition is also important. So far, reactive ion etching (RIE) has been applied in various applications [12-13]. However total process from the patterning to the final stage is not necessarily discussed, though this kind of study will accelerate the application of SiC microstructures.

The authors have been studying the production process of single crystal 3C-SiC on Si substrate aiming to obtain atomically smooth surfaces utilizing the property of epitaxial growth [14]. In this paper, we propose the profiling process of SiC layers and examine the mechanical property, Young’s modulus, of micro-cantilever beams. Then, an application to bimorph micro-actuator is demonstrated.

2. Fabrication of SiC micro-structure

2.1 Epitaxial growth [14]

Figure 1 shows the schematic diagram that shows the problems in hetero-epitaxial process of SiC on Si substrate. The figure shows the crystal lattice along [110] direction on (111) substrate. The lattice constant of Si is 0.543nm and that of SiC is 0.436nm. This difference causes unnecessary distortion at the interface between SiC layer and Si substrate and induces defect such as grain boundary or anti-phase boundary where the lattice orientation is aligned but the phase is not consistent. Other problems are the composition ratio of silicon and carbon and conformity at the boundary. In this study, the nominal thickness of SiC layer was typically 180nm.

Figure 2 shows the apparatus for experiments. A four-inch silicon wafer is held upside down in a vacuum chamber and heated from its backside. The molecular beams of source materials are supplied from the bottom in the chamber, where the silicon is supplied by melting the silicon ingot using an electron beam evaporator while the carbon is supplied by dissociating the acetylene gas. The crystal structure of the substrate can be examined by the reflection high-energy electron diffraction (RHEED) patterns. The main specifications are also shown in tabular form.

Figure 3 shows a typical process procedure. The process is divided into two stages, namely, the carbonization and the epitaxial growth process. In the carbonization stage, only the acetylene gas is emitted into the chamber. The acetylene molecules approached to the substrate are dissociated by the heat radiated from the wafer, and the carbon molecular beams reach to the substrate. This process is necessary for inhibiting the migration and dissociation of the silicon atoms from the surface. After the carbonization, the silicon molecular beams are supplied to synthesize SiC layer together with the acetylene gas.

Figure 4 shows the transmission electron microscope (TEM) image of the interface between SiC layer and Si substrate after the epitaxial growth. The lattice is disordered near the interface or carbonized layer while it is ordered in Si substrate. Crossing the disordered interfacial layer, ordered lattice of SiC is clearly observed. Its lattice orientation is just aligned with that of the Si substrate and the interval is smaller than that of Si substrate. Thus, epitaxial growth was confirmed. However, the lattice of SiC layer is not perfectly ordered and anti-phase boundaries are observed. This defect may affect the mechanical properties, but the effect is smaller than that of grain boundaries that means poly-crystalline.

2.2 Patterning of SiC layer

Figure 5 shows the patterning process of SiC layer. Patterned photoresist (MicroChem SU-8, 30μm thick) was patterned with lithography and used as etching mask for dry etching process (RIE). After the etching, the Si substrate was selectively etched with isotropic etchant to undercut the part beneath the cantilever. Finally, the mask was removed with so-called piranha solution (H_2SO_4:H_2O_2=3:1). The conditions for SiC etching and Si etching are shown in Table 1 and 2 respectively.

Figure 6 shows the results of scanning electron microscope (SEM) observation. The structure is so thin that SiC layer looks white and transparent with electron microscopy. The surface profile beneath the beam can be seen. The substrate profile at the fixed end is not ideal straight line but has specific profile that the edge profile of the beam is thinned to inner direction. The effect of this problem will be discussed later.

It can be seen that the beam is bending upward in the figure. The residual stress during the process may cause this deflection because the direction of deflection was always same. The reason is considered as the difference of lattice constants between Si and SiC.

The intension of the deltaic cantilever is the reduction of so-called sticking force during wet process and the subsequent drying process. In addition, unnecessary torsion deformation in the following experiment will be minimized. Typical width of the cantilever is 10-30μm while other dimensions vary with the purpose of the experiments.

3. Mechanical property and discussions

3.1 Mechanical property

To evaluate the property quantitatively, samples that have different specifications were prepared and tested. In this experiment only, the thickness of SiC layer was set at 511nm because of the easiness of for the convenience of handling. Bending tests were carried out on a nano-indenter as shown in Fig.7. Instead of measuring hardness, we measured the relationship between the applied force and the deflection of the beam. In the experiments, constant force of 150mN was applied at the point located a μm from the free-end. The location of this point was determined using the microscope installed on the indenter. The deflection was measured with the displacement gage on the indenter.

The theoretical relationship between the force and deflection is derived as follows using the beam theory:

$$E = \frac{12Wl}{vb_0h^3}\left[\frac{x^2}{2} - ax\log x + \{-l + a + a\log l\}x + \left\{-\frac{l^2}{2} + al\log l - l(-l + a + a\log l)\right\}\right]$$

where, E is the Young's modulus [GPa], W is the applied force [N], l is the length of the beam [m], v is the deflection[m], x is arbitrary position from the beam end [m], b_0 is the width the fixed end of the beam, and h is the thickness of the beam [μm]. Substituting a for x together with other values, the Young's modulus E was estimated. In this estimation, the boundary condition at the fixed-end was assumed simple such as the beam is supported along the linear edge support.

Figure 8 shows the relationships between the force and the deflection for three different cantilevers. The estimated Young's moduli are also shown in the figure based on the former equation. Typical Young's modulus of bulk material varies from 400 to 450GPa. It is found that the estimated value is almost corresponds with the value of the bulk. Table 3 summarizes both of the dimensions of the cantilevers and the calculated Young's moduli.

3.2 Discussion

Various types of errors are included in the estimation of Young's modulus. The measurement of the dimension is very difficult especially in the thickness because its value is in sub-micron order while others are tens of micron meters. In addition, the effect of thickness on the deflection is not proportional to first power as other dimensions as width but third power. The thickness is estimated from the epitaxial growth rate and time. However, the subsequent etching process may also decrease the thickness. Thus, strict quantitative discussion is difficult.

There is a tendency that the Young's modulus decreases with the decrease in the length of cantilever. This tendency might result from the difference of boundary conditions. As mentioned above, the cantilever is not supported at linear edge but both sides of the cantilever are free to deflect because of no support. This means that the deflection tends to large compared with the ideal model, thus the Young's modulus tends to be estimated small.

4. Fabrication of biomorphic actuator and its evaluation

4.1 Principle and design of the actuator

Figure 9 shows the schematics of bimorph actuator. Nickel was deposited on the SiC cantilever produced as described above. The reason why the nickel was chosen is that the coefficient of thermal expansion much differs from that of SiC. Two layers expand differently at the same temperature change and induce deflection at the free end of the cantilever. The principle of this actuator is old-fashioned but the dimension is rather small and thin as tabulated in the figure. The thickness of the nickel layer was determined by the conventional bimetal design theory to maximize the deflection. It is preferable that the total Young's modulus is larger because the thickness of the structure is thin and not necessarily easy to generate large actuation force. This is another reason to choose nickel. The mechanical properties are also tabulated in the figure. In this experiment, the rectangular cantilever was used instead of the deltaic cantilever. The increase in the surface area is preferable to improve the response time in this type of actuator. In addition, torsion deformation causes no problem as in the indentation test.

The nickel thin film was also used as an electric resister. Contact electrode was fixed at two points and the current through this film was converted into heat. The resistance was about 500Ω. The current path is considered broad over the film depending on the electric filed, thus, wide area near fixed-end must be the heat source. The conducted heat to the cantilever causes the temperature change and thus the thermal deformation. In the experiments, both the voltage and current were measured and the input energy was calculated. The temperature near the fixed end of the cantilever was measured with thermocouple of which diameter is 20μm and thinned to 10μm in thickness at the tip.

4.2 Evaluation of the performance

Figure 10 shows the transient time response after applying step input of constant current. The dimension of the cantilever is 20.5μm in length and 20.3μm in width, thus the appearance is different from that of figure. The deflection of the beam was about 2μm when the input power was 0.37W and the saturated temperature was 333K. Periodic fluctuation can be observed in the result of displacement. This is considered as the effect of deflection of the other structures because of its low natural frequency. The response could be modeled as a first-order system and its time constant was 0.073s. This time is rather long considering the small dimension of the cantilever. However, the thermal input of this case is not efficient because the heat dissipates into other part of the structure such as the substrate.

Further improvement in the time response as well as the increase in the deflection is necessary for actual applications. Smallness has advantage in thermal actuator since the heat capacity decrease in proportional to the third power of dimension, while the surface area decreases in proportional to the second power of dimension. Thus, quick motion is easy to obtain though the generated force decreased. The application of such materials that have high elasticity as SiC and nickel is preferable. The intension of the application of single crystal material is the improvement of properties such as strength and durability. The evaluation of these properties is one of the future problems.

5. Conclusions

This paper aimed the application of single-crystal silicon carbide epitaxially-grown on silicon substrate to micro-mechanical devices. The results as summarized as follows:

- Single-crystal silicon carbide (3C-SiC) thin film was grown on silicon substrate
- SiC cantilevers, tens of μm in width/length 180nm in thickness, were produced using lithography and etching
- The Young's modulus of the thin film was almost same with that of bulk material
- Bimorph thermal actuators were prototyped. The displacement at the tip was about 2μm when the temperature change was 40K. The time constant of the step response

was about 0.07s.

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7. References

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Table 1 Conditions for SiC dry etching

Etching gas	CF_4(80%), O_2(20%)
Flow rate	25sccm(total)
RF power	50W
Pressure	6.4Pa (0.048Torr)
Etching time	15min.

Table 2 Conditions for Si wet etching

Etching solution	HF(9%), HNO_3(68%), CH_3COOH(23%)
Etching rate	2-6μm/min [16]

Table.3 Summary of dimensions and calculated Young's modulus

	α[deg]	l[μm]	b_0[μm]	h[nm]	a[μm]	W[mN]	v[nm]	E[GPa]
Cantilever A	30.5	66	36	511	30	150	1999	305
Cantilever B	45.0	41	34	511	10	150	1551	290
Cantilever C	54.5	35	36	511	10	150	897.2	244