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Determination of Structure and Polarity of SiC Single Crystal by X-Ray Diffraction Technique

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Abstract: Structure and polarity of the SiC single crystal have been analyzed with the four-circle X-ray diffraction method by a double-crystal diffractometer. The hexagonal $\{10\bar{1}5\}$ pole figure shows that this SiC sample has a 6H modification. The difference between the integrated intensities measured by ω scan in the triple-axis diffraction set-up finds some convincing evidence that the surface is either a Si-terminated face or C-terminated face. The experimental ratios of $|F(000L)|^2/|F(000\bar{L})|^2$ are in good agreement with the calculated ones after the dispersion corrections to the atomic scattering factors (L= 6, 12 and 18, respectively). Thus, this measurement technique is convenient for the application of the materials with remarkable surface polarity.

Key words: SiC single crystal; polarity; hexagonal 6H; scattering factor
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1 Introduction

Silicon carbide(SiC) is a promising candidate for the high-temperature, high-speed, high-frequency and high-power electronic devices owing to its excellent thermal and electrical properties[1—2]. Additionally, SiC is a potential excellent substrate for the GaN epitaxy due to its small lattice constant and the thermal expansion coefficient mismatched the later[3].

It is well-known that SiC can form numerous modifications[4](the so-called polytypes), including the common polytypes of hexagonal 4H, 6H and 8H, rhombohedral 15R, and cubic 3C. However, all 6H, 4H and 3C-SiC are assumed to be SiC bulk single crystals. so it is very crucial to identify the SiC polytype when the SiC bulk single crystal growth is performed.

In previous papers, a four-circle X-ray diffractometer has been applied to determine the SiC structure. The result shows that SiC has a 6H modification, which is noncentrosymmetric and contains a polar axis. Because of this axis, the bonding configuration of the surface atoms depends upon the crystallographic planes. Generally speaking, the various crystallographic planes are composed of the identical close-packed double layers. Based on a generally accepted assumption[5], an equivalent definition is obtained that for two opposite faces of a crystal, which are both perpendicular to the polar axis, one is Si-terminated [symbolically (0001)] and the other is C-terminated [symbolically (000-1)]. The [0001] vector points from the C face

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to the Si face. There might be many differences between the physical and chemical properties of the (0001) surface and the opposite ($000\bar{1}$) surface.

6H-SiC crystallographic structure consists of the stacking of Si-C bilayer in ABCACBA sequence along the c (or z) axis (0001) directions of the hexagonal structure, where each letter (A, B and C) corresponds to a Si-C bilayer. A bulk truncated atomic distribution of this structure in ($11\bar{2}0$) high-symmetry planes is shown in Fig. 1.

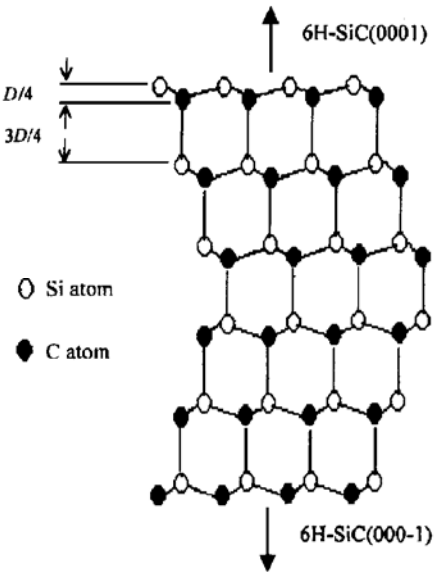

FIG. 1 Schematic View of SiC Single Crystallographic Atom Arrangement of ($11\bar{2}0$) Plane Perpendicular to {0001} Surfaces

From the close-packed structure, it is observed there exist two kinds of different interplanar distances between the neighbor (0001) planes. One is $D/4$, the other is $3/4D$. Here, D stands for the distance between two double layers. The effects of the polarity on the bond structure can be observed on the typical etched surfaces of an opposite face[6—7], by infrared attenuated total reflection spectroscopy[8], the piezoelectric effect[9] and X-ray photoelectron diffraction characterization[10]. In addition, such identifications can be made by observing the effects of the intensity of scattered X-ray[11—12]. It has been noted in previous papers, that the intensity measurements of the scattered X-ray were taken by a rocking curve on a double crystal X-ray spectrometer. Moreover, no more factors influencing the integrated intensity have been discussed. Besides, in order to reflect this rule better, the diffracted intensity was related to the structure factor for a mosaic crystal. A triple-axis X-ray setup was used in the intensity measurement. When the measurement of the diffracted intensity was carried out, a crystal analyzer had to be placed in front of the detector and some other factors taken into consideration. In this paper, the application of the X-ray dispersion effect in the determination of the polarity of SiC single crystal has been presented, using the triple-axis diffraction method with the purpose of our distinguishing between the Si-terminated face and C-terminated one prior to the subsequent treatment or deposit.

2 Experimental Procedure

The SiC single crystals were grown by Chemical Vapor Deposition (CVD), with the growth details being introduced elsewhere. SiC sample was analyzed by X-ray diffraction with a Rigaku diffractometer equipped with a Cu anode at 40kV and 100mA. Using a four-circle diffractometer, the pole figures are measured, whose geometry in real space is shown in Fig. 2 to identify the structure. The intensity measurement of scattered X-ray was taken according to the triple-axis diffraction method. The X-ray diffraction patterns of ω scan were obtained using a step of 0.002°. CuKα_1

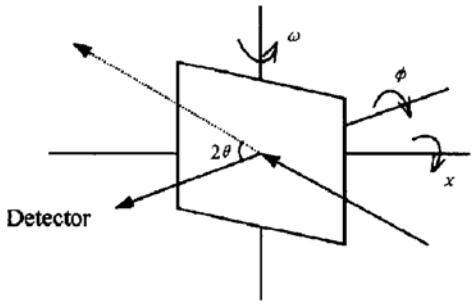

FIG. 2 Schematic Description for Configuration of Four-Circle Geometry

monochromatic radiation was selected by a single crystal Ge(004). Si(220) was used as an analyzer crystal.

3 Results and Discussion

According to crystallographic theory, it is easy for the close-packed or low Miller-index plane to appear on the surface of single crystal. As long as the hexagonal structure and the cubic structure are concerned, the distances between the crystallographic planes of cubic (111) and some hexagonal ($000L$) are almost identical. Therefore, X-ray diffraction information from crystal surface may result from either cubic 3C (111) plane or hexagonal ($000L$) plane(L is a positive integer). To distinguish these information, the diffraction of other plane is performed according to the interangle between two different plane in 3C unit cell and hexagonal unit cell (2H, 4H and 6H). The $\{10\bar{1}5\}$ pole figure shown in Fig. 3 was measured by fixing the detector at 2θ position of 45.28° and scanning the tilt angle χ from 0° to 60°, and the azimuth rotation angle Φ from 0° to 360°. Figure 3

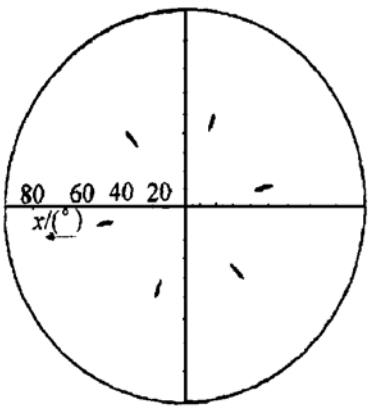

FIG. 3 XRD Pole Diagram Detector angle 2θ was set at 45.28° for $(10\bar{1}5)$ H-SiC. Angles χ and Φ were scanned.

shows that peaks appear when χ= 48.57°, which is equal to the interplanar angle between (0006) and $(10\bar{1}5)$ planes. It can be seen that the diffraction-geometry of the plane is in excellent agreement with that of a 6H structure. Additionally, the $\{10\bar{1}5\}$ diffracted peaks have the exactly symmetrical points of $\{10\bar{1}5\}$ of the hexagonal structure, i.e., the peak repeats itself by every 60°; This is consistent with the hexagonal symmetry of SiC material. therefore, a conclusion is drawn that the SiC single crystal has a 6H structure.

Initial measurements of the intensity of the scattered X-ray from opposite sides of a $\{000L\}$ single crystal were made with a triple-axis setup on a double crystal X-ray spectrometer. The intensity of a diffracted X-ray beam is proportional to $|F|^2$ for a mosaic crystal, measurements were taken with triple-axis crystal diffraction. Based on the extinction rule of X-ray diffraction, the (0006) diffraction should be the first order diffraction of the basal plane of a 6H structure. The (00012) and (00018) diffractions are the second and third ones, respectively. The X-ray beam was collimated by a 0.1mm aperture to obtain a beam size, which was small compared with the crystal area being analyzed. The intensities of the reflections by ω scanning mode were recorded and after each measurement, the crystal was displaced laterally with a small distance to obtain the reflection from some locations of each face. The results are shown in Fig. 4.

It is known that the integrated intensity is a function of the structure factor. Brack[11] deduced the ratios of the structure factor of ($000L$) to that of ($000\bar{L}$) as follows:

$$\frac{F(0006)}{F(000\bar{6})} = \frac{f_{Si} - if_C}{f_{Si} + if_C}$$

$$\frac{F(00012)}{F(000\,\bar{1}2)} = 1$$

$$\frac{F(00018)}{F(000\,\bar{1}8)} = \frac{f_{Si} + if_C}{f_{Si} - if_C}$$

where f_{Si} and f_C are atomic scattering factors of dispersion corrections, expressed as $f = f_0 + \Delta f' + i\Delta f''$, where f_0 is the normal scattering factor usually tabulated as a real function of $\sin\theta/\lambda$, and Δf

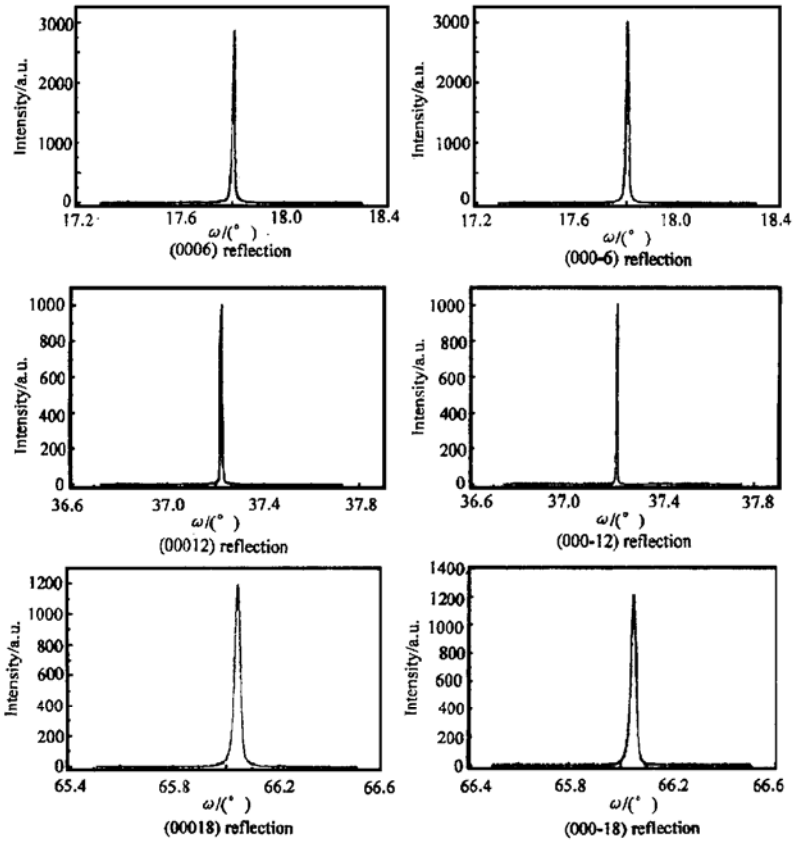

FIG. 4 Reflection Intensity with CuKα1 Radiation Showing a Series of (000L) Diffraction from Same Surface in Comparison with (000$\bar{L}$) Diffraction of Opposite Surface (L= 6, 12, 18, Respectively)

and $\Delta f''$ are the real and imaginary parts of a correction factor due to dispersion. f_0, Δf and $\Delta f''$ can be obtained from the related literature[13]. However, it should be noted that in Brack's work, some factors influencing the integrated intensities are not be mentioned. Based on the expression[14] of integrated intensity, the main factors are L_p, ΔV and $|F|^2$, where L_p is Lorentz-polarized factor, which is expressed as $(1+\cos^2 2\theta)/2\sin 2\theta$; ΔV is the crystal volume irradiated by X-ray; and F is the structure factor. Taking these factors into consideration, we obtain the experimental values of the integrated intensities among (0006), (00012) and (00018) reflections are in excellent agreement with the theoretical prediction. However, the factors L_p and ΔV are considered to be identical in the $\{000L\}$ and $\{000\bar{L}\}$ reflections. Here, the calculated values of $|F(000L)|^2/|F(000\bar{L})|^2$ and the measured ratios of integrated intensities from $\{000L\}$ reflections to those from $\{000\bar{L}\}$ reflections are listed in table 1.

Table 1 Calculated Ratios of Scattering Factors of $\{000L\}$ Planes to Opposite $\{000\bar{L}\}$ Planes in Comparision with Experimental Values

$\{000L\}$	Total Counts		$\lvert F(000L)\rvert^2/\lvert F(000\bar{L})\rvert^2$	$\lvert F(000L)\rvert^2/\lvert F(000\bar{L})\rvert^2$
	(000L)	(000$\bar{L}$)	Measure	Calculation
0006	14913	15764	0.946	0.949
00012	3314	3288	1.008	1.000
00018	4519	4135	1.093	1.076

From above, it is evident that the SiC single crystal, as a mosaic crystal, has the intensity ratios that are in good agreement with the calculated values. Therefore, according to the above values of $|F(000L)|^2/|F(000\bar{L}|^2$, i. e. 0.946, 1.008 and 1.093, we may conclude that the face, which is assumed to be the variation of the $|F(000L)|^2$ values, is Si-terminated face and the opposite is C-terminated face. Hence, it is also possible to determine the polarity of other SiC polytypes with stackings of Si-C double layers along the z-axis, such as hexagonal 2H-SiC{0001}, 4H-SiC{0001} and cubic 3C-SiC{111}.

4 Conclusion

The measurement of hexagonal SiC pole figure proves that the SiC single crystal has a 6H modification. The ratios of the experimental intensities of {0001} surface to the opposite $\{000\bar{1}\}$ surface are in excellent agreement with the calculated values of $|F(000L)|^2/|F(000\bar{L})|^2$. The result applies to determining which side is the Si-terminated (0001) face and which is the C-terminated $(000\bar{1})$ face.

References

[1] P. A. Ivanov and V. E. Chelnokov, Semiconductor Science and Technology, 1992, **7**: 863.

[2] LEI Tian-min, CHEN Zhi-ming, YU Ming-bin *et al.*, X-Ray Photoelectron Spectroscopy Study of 3C-SiC Thin Films Grown on Si Substrates, Chinese Journal of Semiconductors, 2000, **21**(2): 303—306.

[3] T. Gerge, W. Pine, M. Dhan, J. Kuznia and P. Chang-Chieu, J. Electron. Mater., 1995, **24**: 1241.

[4] G. R. Fisher and P. Barnes, Phil. Mag., 1990, **B61**: 217.

[5] W. Strock and V. A. Brophy, Am. Mineralogist., 1955, **40**: 94.

[6] R. E. Mariuger, J. Appl. Phys., 1958, **29**: 1261.

[7] H. A. Schell, Z. Metallk. 1957, **48**, 158.

[8] H. Tsuchida, I. Kamata and K. Izumi, J. Appl. Phys, 1999, **85**: 3569.

[9] J. L. Bischoff, D. Derttel and L. Kubler, Surf. Sci., 1998, **415**: 392.

[10] L. Muehlhoff, M. J. Bozack, W. J. Choyke and J. T. Yates, J. Appl. Phys., 1986, **60**: 2558.

[11] D. Coster, K. S. Knol and J. A. Prins, Z. Physik, 1930, **63**: 345.

[12] K. Brack, J. Appl. Phys., 1965, **16**: 3650.

[13] International Tables for X-Ray Crystallography, V. 3, Birmingham, Eng., Kgnoch Pr., 1974—1983.

[14] J. Z. Zhang, C. Z. Yang, Diffraction Foundation of Crystal, Nanjing University Publishing House, 1992.

使用 X 射线衍射技术判定 SiC 单晶体的结构和极性

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摘要: 使用四圆衍射仪和双晶衍射技术, 分析了 SiC 体单晶的结构和极性. SiC 单晶体由化学气相淀积法获得. 六方{10$\bar{1}$5}极图证明了该单晶结构为 6H 型. 三轴晶衍射中的 ω 模式衍射强度的差异判定了该单晶的 Si 终端面和 C 终端面, 即极性面。两个面的一、二、三级衍射强度的测量比值与经过散射因子修正后计算的结构振幅平方比值 $|F(000L)|^2/|F(000\bar{L})|^2$ 非常吻合. 因此, 利用极性面的衍射强度差异, 可以方便、严格地判断具有类似结构如 2H{0001}、4H{0001}及 3C-SiC{111}的极性.

关键词: SiC 单晶; 极性; 6H 结构; 散射因子

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