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1 MEV-ELECTRON IRRADIATION INDUCED DEFECTS  
IN EPITAXIALLY GROWN 3C-SiC

July 1989

Hisayoshi ITOH, Naohiro HAYAKAWA, Isamu NASHIYAMA\*  
and Eiichiro SAKUMA\*

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in Epitaxially Grown 3C-SiC

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Electron spin resonance (ESR) measurements have been performed for 1MeV-electron irradiated 3C-SiC crystals epitaxially grown by chemical vapor deposition method. The results indicate the presence of at least four paramagnetic defects (T1-T4 centers). The T1 center was found to consist of isotropic five lines equally spaced at about 1.5 G and to have a g-value of  $2.0029 \pm 0.0001$ . The anisotropic T2 center could be detected below about 100 K. The T3 and T4 centers were both anisotropic at room temperature. Isochronal annealing of electron irradiated 3C-SiC showed that the T1 center was annealed at three stages (150°C, 350°C, and 750°C) and that the T3 and T4 centers were annealed at 100°C and at 350°C respectively.

Keywords: Electron Spin Resonance, Electron Irradiation, 3C-SiC  
(Cubic Silicon Carbide), Chemical Vapor Deposition,  
Paramagnetic Defects, Annealing

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\* Electrotechnical Laboratory

エピタキシャル成長 3C-SiC における 1 MeV 電子線照射誘起欠陥

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伊藤 久義・早川 直宏

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(1989年6月29日受理)

化学気相成長 (Chemical Vapor Deposition) 法により, Si 上にエピタキシャル成長させて作製した立方晶シリコンカーバイド (3C-SiC) 結晶に対し, その 1 MeV 電子線照射効果を電子スピン共鳴法を用いて調べた。その結果, 電子線照射により 3C-SiC 中に 4 種類の常磁性欠陥 (T 1 ~ T 4 センター) が誘起されることが判明した。T 1 センターは, 等間隔で分離した (分離幅約 1.5 G) 等方的な 5 本線で構成され, その g 値は  $2.0029 \pm 0.0001$  であることが見いだされた。また, T 2 センターは 100 K 以下の低温で出現し異方性を持つこと, 並びに T 3 及び T 4 センターは室温において検出され異方性を示すことが解った。さらに, 電子線照射 3C-SiC の等時アニールの結果, T 1 センターのアニール過程において 3 種類のステージ (150 °C, 350 °C, 750 °C) が存在することが明らかになった。一方, T 3, T 4 センターについては, 各々 100 °C, 350 °C にアニールステージを持つことが見いだされた。

# Contents

1. Introduction .....	1
2. Experimental procedure .....	2
3. Results and discussion .....	2
4. Conclusions .....	6
Acknowledgments .....	6
References .....	6

# 目 次

1. 序 論 .....	1
2. 実 験 .....	2
3. 結果及び考察 .....	2
4. 結 論 .....	6
謝 辞 .....	6
参考文献 .....	6

## 1. Introduction

Cubic silicon carbide (3C-SiC) has extreme thermal and chemical stability. This material is a promising candidate for use in high-temperature and high-power electronic devices because it has a large band gap of 2.2eV, a high electron saturation velocity ( $2.7 \times 10^7$  cm/s), and a moderate electron mobility ( $\sim 10^3$  cm<sup>2</sup>/Vs).<sup>(1-3)</sup> Though only small single crystals of 3C-SiC could be grown by conventional growth techniques such as Lely method<sup>(4)</sup>, recent advance in heteroepitaxial growth techniques<sup>(5-7)</sup> of single-crystal 3C-SiC on Si makes it possible to grow a large-area epilayer. This leads to a great advantage in device fabrication and arises a growing interest in application of SiC for electronic devices, especially those used in hostile environments, e.g., aerospace and nuclear power plants.

When 3C-SiC epilayers are applied to electronic devices which work in radiation fields, it is important to know radiation damage in them. The knowledge is also required for an understanding of ion-implantation effects on 3C-SiC. There exists much literature on radiation effects on bulk SiC.<sup>(8)</sup> However, there has been only a little amount of information about structures and electronic-levels of radiation induced defects in 3C-SiC. Freitas et al.<sup>(9)</sup> reported photoluminescence (PL) bands for the D1 and D2 defects in ion-implanted epitaxial 3C-SiC. They showed that the intensity of these PL bands increased by thermal annealing up to 1600°C. It was suggested that the D1 and D2 defects were ascribed to some form of divacancy and the carbon di-interstitial, respectively.<sup>(8)</sup> Nagesh et al.<sup>(10)</sup> performed deep-level transient spectroscopy and resistivity measurements of neutron-irradiated 3C-SiC. They showed that most of the defects produced by neutron-irradiation had energy levels confined to the lower two-third of the band gap and that 90% of them could be removed by 350°C annealing.

The present paper describes paramagnetic defects newly observed in electron-irradiated 3C-SiC epitaxially grown on Si by chemical vapor deposition (CVD) method. A tentative model is discussed for the defects induced by electron-irradiation.

## 2. Experimental procedure

3C-SiC crystals were epitaxially grown on crystalline Si (100) substrates by CVD method using  $\text{SiH}_4\text{-C}_3\text{H}_8\text{-H}_2$  system at 1350°C. Si substrates were removed by  $\text{HF-HNO}_3$  etching after the growth. 3C-SiC samples used were undoped, showing n-type conduction with the electron mobility of about  $500\text{cm}^2/\text{Vs}$  at room temperature (RT) and their thicknesses were about  $20\mu\text{m}$ .

Electron-irradiations were made on the samples in air or in flowing He gas with fluences up to  $3\times 10^{18}/\text{cm}^2$ . Acceleration energy of electrons was 1MeV and their flux was about  $2.8\times 10^{13}\text{electrons}/\text{cm}^2\text{s}$  ( $4.4\mu\text{A}/\text{cm}^2$ ). The samples were placed on a water-cooled holder so as to avoid beam heating and then their temperature was kept below 50°C during irradiation. Electron-irradiated samples were annealed in pure  $\text{N}_2$  gas at temperatures up to 800°C in order to investigate annealing behavior of the defects.

Electron spin resonance (ESR) measurements were made over a temperature range from 4K to RT with an X-band (9GHz) microwave incident upon  $\text{TE}_{011}$  cylindrical cavity. The spin number of paramagnetic defects was determined to an accuracy of a factor of 3 by being compared with the known number of  $\text{Mn}^{2+}$  spin in MgO. Though, the relative accuracy was much better, less than  $\pm 15\%$ .

## 3. Results and discussion

Figure 1(a) shows a typical ESR spectrum at RT for the 3C-SiC irradiated with  $3\times 10^{18}/\text{cm}^2$ . This spectrum consists of five isotropic lines equally spaced at intervals of  $1.46\pm 0.05\text{G}$ , as indicated by solid arrows. A g-value for this ESR center was obtained to be  $2.0029\pm 0.0001$ . In addition, these five lines are symmetric around the central line. Intensity ratios of the inner and the outer sidelines to the central line were  $0.25\pm 0.02$  and  $0.03\pm 0.01$ , respectively. Figure 2 shows the dependence of the spin density per unit area of this center on electron fluence. The spin density increased proportionally with the fluence. Its spin density was about  $2\times 10^{14}/\text{cm}^2$  in the sample irradiated with  $3\times 10^{18}/\text{cm}^2$ . Assuming constant profile of the defect in the epilayer with

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20  $\mu$ m thickness, the concentration of the defect was estimated to be about  $1 \times 10^{17}/\text{cm}^3$ . From now on, this center is referred to as T1.

The T1 center can be explained by assuming that the sidelines are due to simultaneous hyperfine (hf) interaction between a paramagnetic electron and several  $^{29}\text{Si}$  nuclei at equivalent twelve Si sites.  $^{29}\text{Si}$  has a nuclear spin of  $1/2$  and a natural abundance of 4.7%. Then the probabilities that one  $^{29}\text{Si}$  and two  $^{29}\text{Si}$  exist at twelve Si sites are calculated to be 0.332 and 0.090, respectively. Using these probabilities and equivalence of the twelve Si sites, the intensity ratios of the inner and the outer sidelines to the central line are calculated to be 0.267 and 0.036, respectively. Since the probabilities that three and more  $^{29}\text{Si}$  nuclei exist are less than 0.02, their contribution to the line intensity can be neglected. The experimental intensity ratios of the inner and outer sidelines to the central line were  $0.25 \pm 0.02$  and  $0.03 \pm 0.01$ , respectively. These values are in good agreement with the calculated values. Therefore, the paramagnetic electron in the T1 center presumably interacts with several  $^{29}\text{Si}$  nuclei at twelve Si sites. This also explains why the five lines are equally spaced. Then an hf coupling constant  $|A|$  is calculated to be  $(2.73 \pm 0.01) \times 10^{-4} \text{cm}^{-1}$  from the interval of the T1 spectrum. This value is much smaller than that for Si dangling bond<sup>(11)</sup>. It suggests that the paramagnetic electron is distant from Si atoms. One possible model derived from our interpretation for the T1 center is a point defect at the Si site, which has the twelve second nearest neighbor Si atoms.

When ESR measurements of the epilayers irradiated in air were performed at temperatures lower than about 100K, additional ESR lines were apparently visible. An ESR spectrum at 60K for the sample irradiated with  $3 \times 10^{18}/\text{cm}^2$  is shown in Fig.1(b) as an example. This spectrum was observed under the condition that a magnetic field was applied parallel to the  $\langle 011 \rangle$  axis. Five lines ascribed to the T1 center are indicated by solid arrows, though the outer sidelines are not seen clearly because they are very weak and superposed on the additional lines and noises. These five lines were isotropic even at 50K. On the contrary, the additional lines were found to be anisotropic, and the dependence of their line intensities on a microwave-power was different from that of the T1 center. These results show that the additional lines

are not attributed to the T1 center but other defects. Then these defects are referred to as T2 here.

Figures 3(a), 3(b) and 3(c) show ESR spectra at RT for the sample irradiated with  $3 \times 10^{18}/\text{cm}^2$  in air when a magnetic field was applied parallel to the  $\langle 100 \rangle$ , the  $\langle 111 \rangle$ , and the  $\langle 011 \rangle$  axis, respectively. These spectra were observed under the condition that a magnetic field was swept 10 times wider and an amplitude was about 20 times larger than those for T1 spectrum in Fig.1(a). Several ESR lines can be seen around the T1 spectrum which is the most intense line in the central area. These lines were anisotropic at RT as shown in Figs.3(a), 3(b), 3(c), and 4(a) which shows the angular dependence of these lines in the irradiated 3C-SiC. In addition, the power dependence of their line intensities was a contrast to those of the T1 and T2 centers. These results indicate that their origin is different from the T1 and T2 centers. Here, we pay attention to apparent four lines indicated by arrows in Fig.3(c). Thermal annealing effects on these lines are shown in Figs.3(d) and 3(e). Outer ESR lines with a separation width of  $\sim 55\text{G}$  almost disappeared after  $150^\circ\text{C}$  annealing, whereas inner lines with a separation of  $\sim 25\text{G}$  did not change significantly after  $150^\circ\text{C}$  annealing and disappeared after  $450^\circ\text{C}$  annealing. The angular dependence of the inner lines in the sample annealed at  $200^\circ\text{C}$  is shown in Fig.4(b). The power dependence of ESR-line intensity for the outer lines differed from that for the inner lines. Therefore, these four lines are thought to originate from two different ESR centers: T3 (the outer lines) and T4 (the inner lines). Their spin densities were about  $1 \times 10^{13}/\text{cm}^2$  in the sample irradiated with  $3 \times 10^{18}/\text{cm}^2$ . The T2-T4 centers were not observed apparently in samples irradiated in He atmosphere, whereas the T1 center was visible. This indicates that the T2-T4 centers include some impurities such as H and O from air in their structures. The angular dependence of the T3 and T4 centers also suggests that some impurities which have a nuclear spin are introduced in the samples. In order to make clear origin of the T2-T4 centers, it is needed to examine impurity doping effect on radiation damage in 3C-SiC.

Electron-irradiated epilayers were annealed isochronally for 5 minutes and ESR measurements were successively done at RT in order to investigate annealing behavior of the T1 center. Figure 5 shows the

result of the isochronal annealing. It is clear that there exist three annealing stages at 150°C, 350°C, and 750°C. About 35% of the initial amount was annealed at stage I (150°C) and about 10% was annealed at stage II (350°C). The residual 55% disappeared at stage III (750°C). Isothermal annealing of the T1 center at around 750°C showed first-order reaction with an activation energy of about 2.2eV. Balona and Loubser<sup>(12)</sup> found an ESR spectrum (F center) with five lines in bulk 3C-SiC irradiated with electrons. They obtained a g-value of  $2.0032 \pm 0.0001$  and an hf coupling constant  $|A|$  of  $2.62 \times 10^{-4} \text{ cm}^{-1}$  for the F center. Since these values are almost the same as those for the T1, the T1 center observed in 3C-SiC epilayers is considered to be identical to the F center in bulk SiC. However, annealing behavior of the T1 center is a contrast to that of the F center which has only one annealing stage of 750°C and an activation energy of 3.1eV<sup>(12)</sup>. The discrepancy may be caused by difference in crystal-growth process, e.g., difference in impurities and/or defects introduced in the crystal during the growth.

Isochronal annealing shows that the T3 and T4 centers have annealing stages of 100°C and 350°C respectively, as shown in Fig.6. The annealing stages for the T3 and T4 centers are almost the same as the annealing stage I and stage II for the T1 center, respectively. It suggests that annealing behaviors of the T3 and T4 centers are related with that of the T1 center. It was shown by Nagesh et al.<sup>(10)</sup> that the resistivity of neutron-irradiated epitaxial 3C-SiC recovered at two stages of 150°C and 300°C. These recovery stages agree well with the annealing stages for the T1, T3 and T4 centers. The fact suggests that neutron-irradiation produces similar defects in 3C-SiC epilayers as electron-irradiation does. It also gives evidence that several defects which have annealing stages of less than 750°C are introduced by radiation in epitaxially grown 3C-SiC. Freitas et al.<sup>(9)</sup> reported from PL study of ion-implanted 3C-SiC that the intensities of the D1 and D2 luminescence bands increased with annealing temperature up to 1600°C. In the present study, increase in the spin density of radiation induced defects was not observed in thermal annealing of 3C-SiC epilayers irradiated with electrons and of those implanted with N-ions (200keV- $\text{N}_2^+$ ,  $1.7 \times 10^{14} / \text{cm}^2$ ). The result may suggest that the D1 and D2 defects are not paramagnetic.

#### 4. Conclusions

Electron-irradiation induced defects in 3C-SiC epitaxially grown by CVD method have been studied with ESR technique. Four ESR centers (T1-T4) were observed in electron-irradiated 3C-SiC. The T1 center ( $g=2.0029\pm0.0001$ ) consists of five isotropic lines with separations of about 1.5G. It can be interpreted by hf interaction between a paramagnetic electron and several  $^{29}\text{Si}$  nuclei, which have a nuclear spin of 1, at equivalent twelve Si sites. The T2 center was visible at temperatures below 100K and had an anisotropy. The T3 and T4 centers, which were anisotropic, were observed at RT. Some impurities may be incorporated in the structures of the T2-T4 centers. Isochronal annealing showed three stages for the T1 center, i.e., stage I, stage II and stage III were seen at around 150°C, 350°C and 750°C, respectively. This also indicated that the T3 and T4 centers annealed at stages of 100°C and 350°C respectively. A comparison of annealing data for the defects in 3C-SiC suggests that annealing behavior of the T1 center is related with those of the T3 and T4 centers. Further investigation is needed to clarify structures of these paramagnetic defects.

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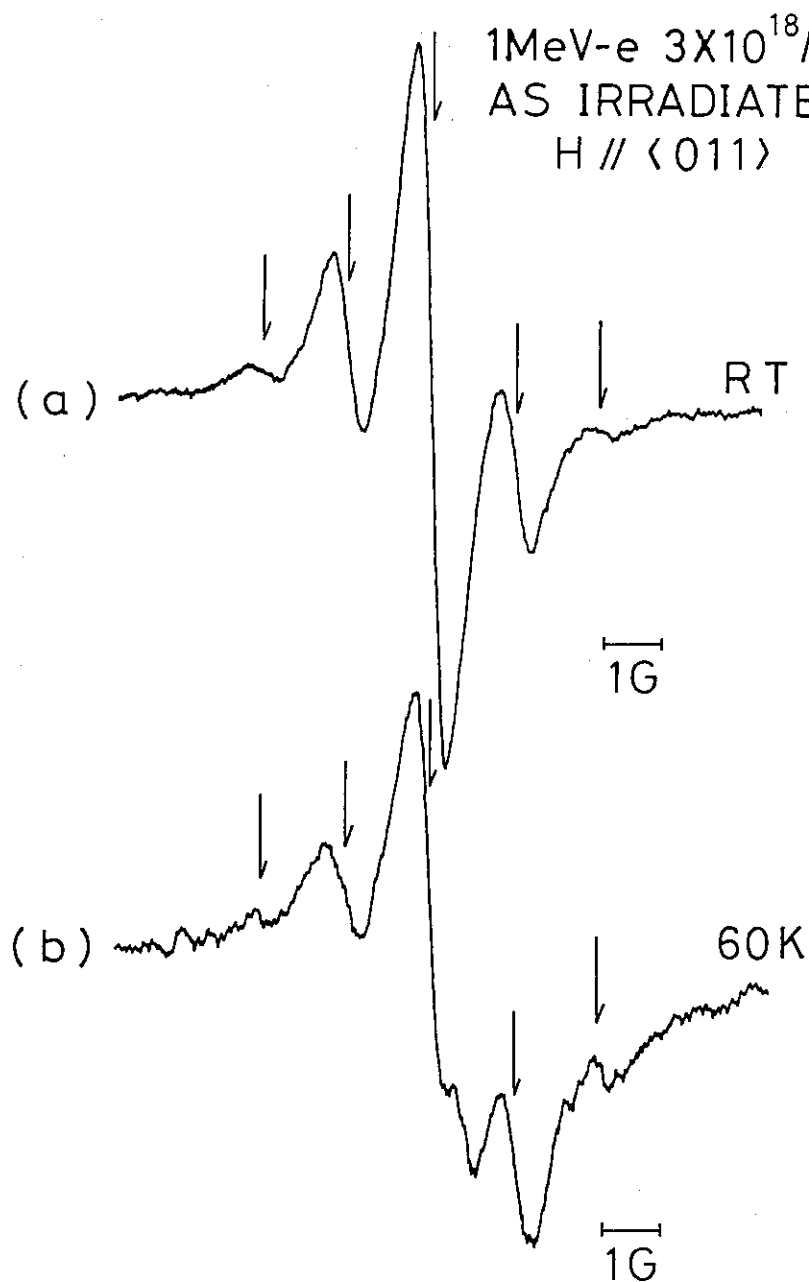


Fig.1 ESR spectra of 3C-SiC irradiated with 1MeV-electrons of  $3 \times 10^{18}/\text{cm}^2$ . The spectra were observed at room temperature (a) and at 60K (b) when a magnetic field was applied parallel to the  $\langle 011 \rangle$  axis. The arrows indicate five lines of T1 center.



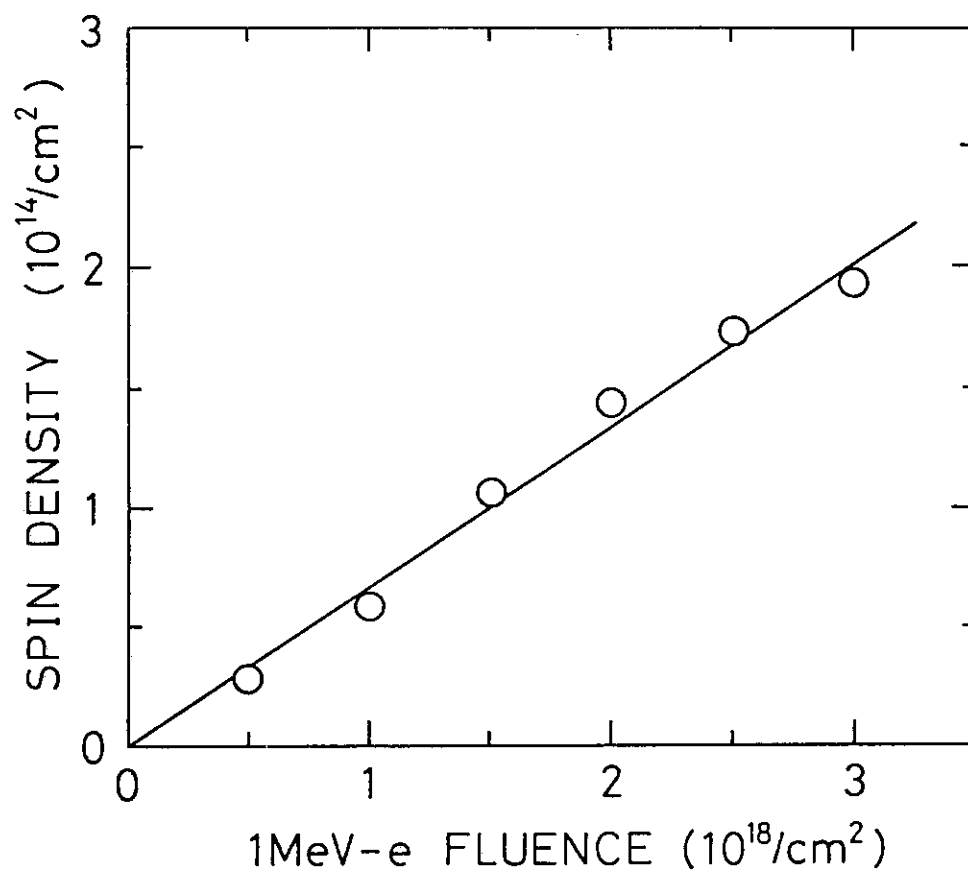


Fig.2 Electron-fluence dependence of the spin density of the five lines spectra (T1) observed in irradiated 3C-SiC.

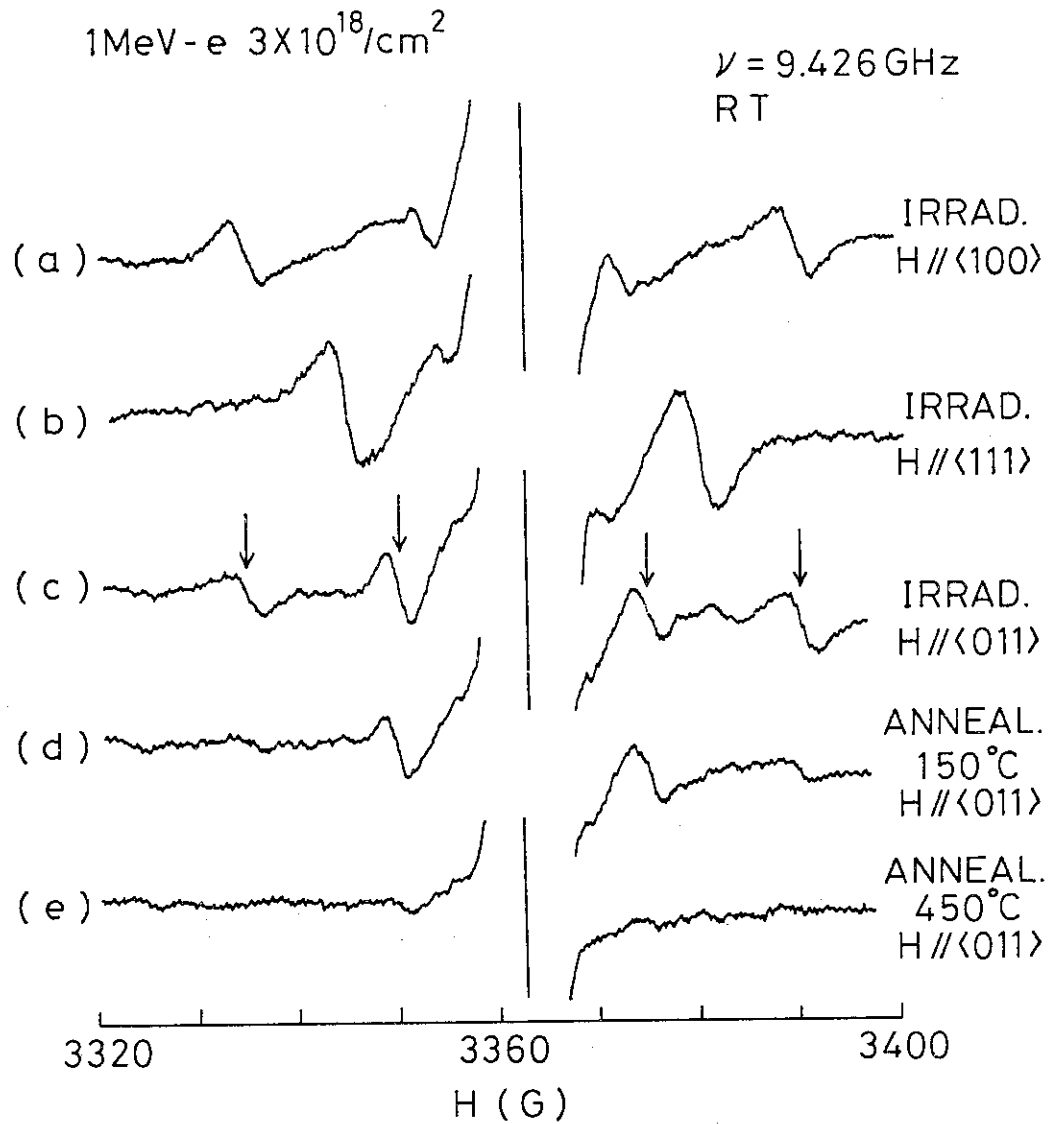


Fig.3 ESR spectra of electron-irradiated and subsequently annealed 3C-SiC. The spectra were observed at RT for 3C-SiC irradiated with  $3 \times 10^{18}/\text{cm}^2$ . Spectra for as irradiated sample are shown in (a), (b), and (c) in the case of H//<100>, <111>, and <011>, respectively. Then, (d) and (e) show spectra for the 3C-SiC after annealing at 150°C and 450°C, respectively, when H//<011>. The arrows indicate T3 (outer lines) and T4 (inner lines) centers.

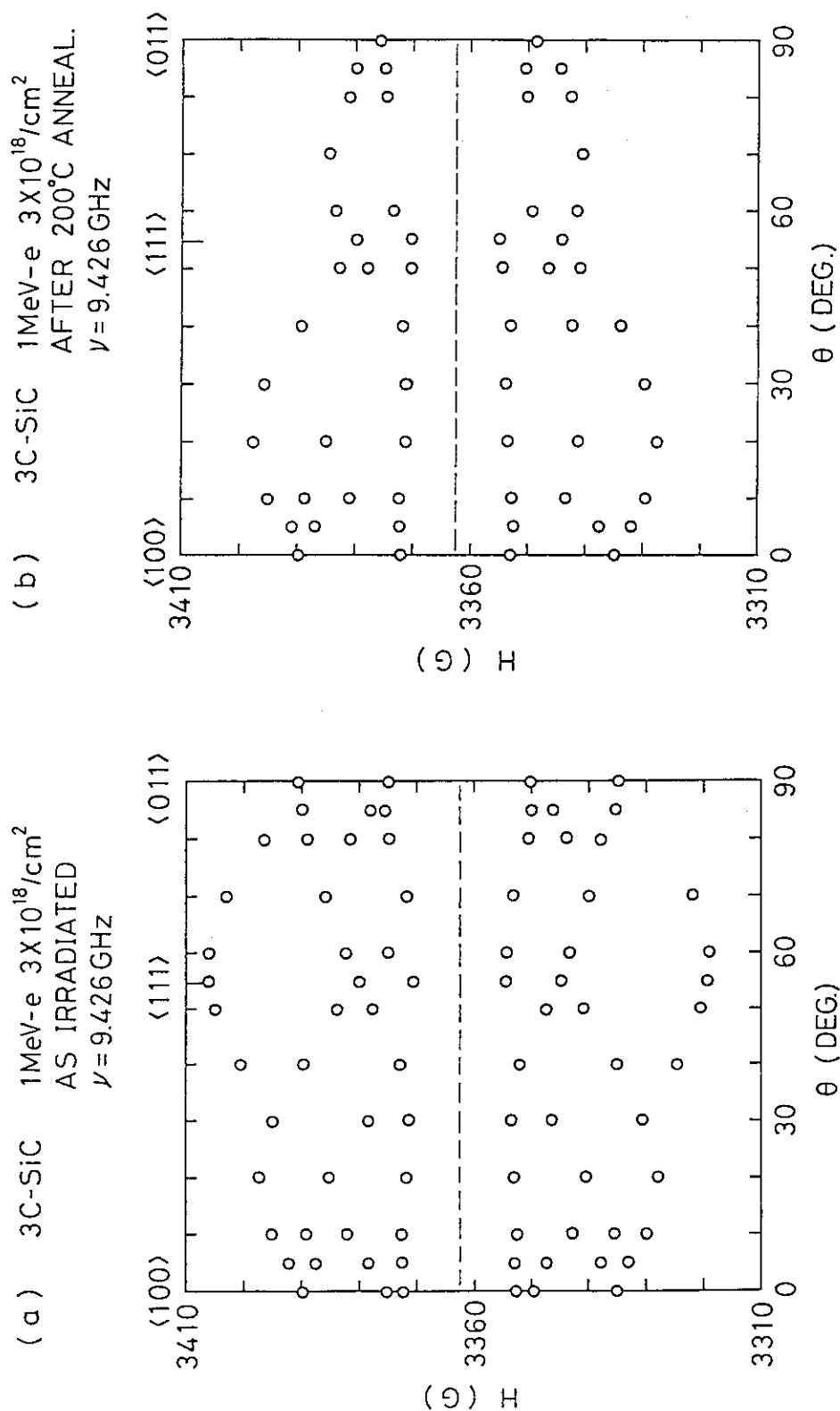


Fig.4 Angular dependence of anisotropic ESR spectra (T3 and T4 centers) in 3C-SiC irradiated (a) and subsequently annealed at 200°C (b). Magnetic field applied was rotated on the (011) plane. The abscissa indicates the angle between the magnetic field and the  $\langle 100 \rangle$  axis. The T3 center was not visible after 200°C annealing. Dashed lines indicate angular dependence of the T1 center (isotropic center).

ISOCHRONAL ANNEALING OF  
3C-SiC ( 1MeV-e  $3 \times 10^{18}/\text{cm}^2$  )

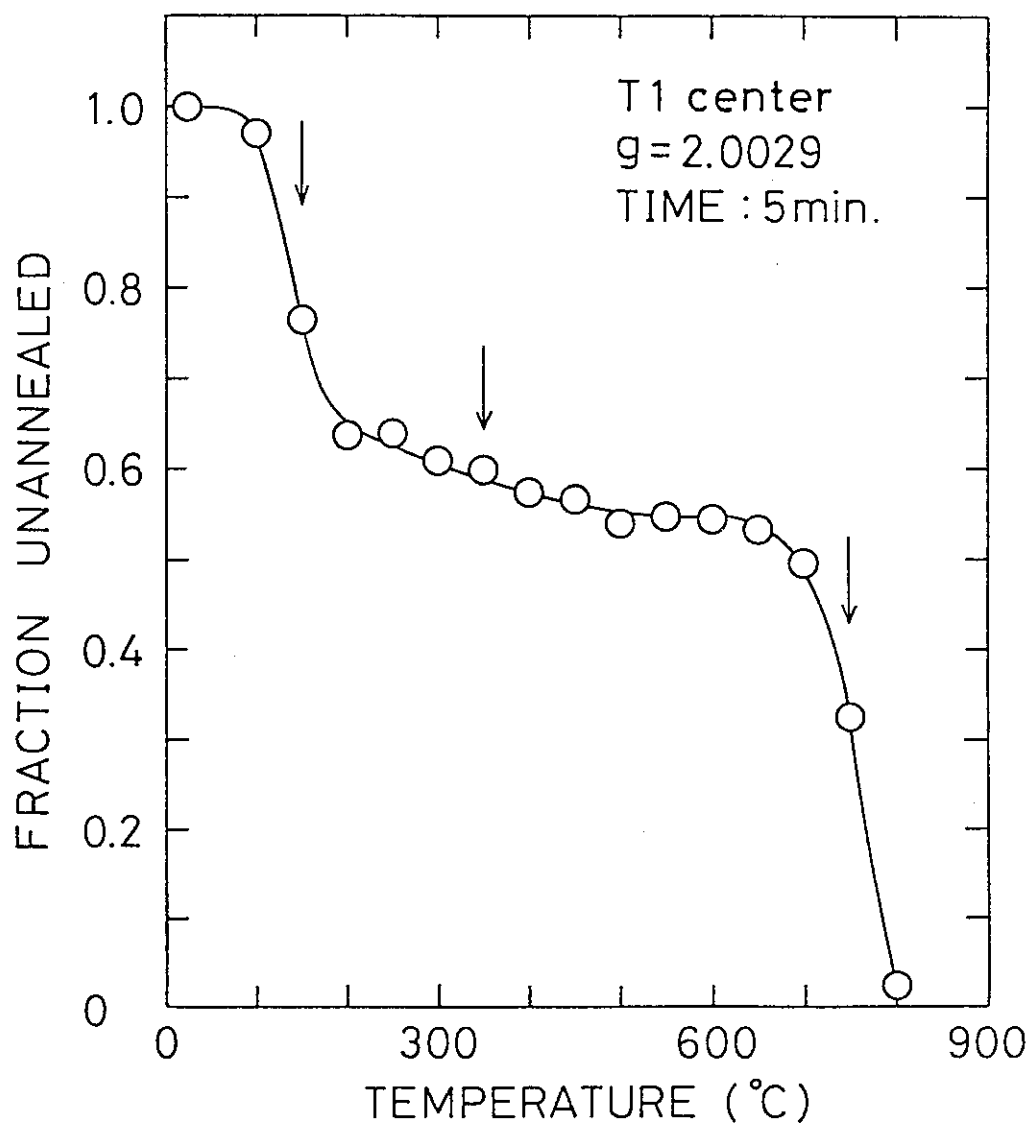


Fig.5 Isochronal (5min.) annealing of the T1 center in 3C-SiC irradiated with  $3 \times 10^{18}/\text{cm}^2$ . Three stages (stage I:150°C, stage II:350°C, and stage III:750°C) are indicated by arrows.

ISOCHRONAL ANNEALING OF 3C-SiC  
 1MeV-e  $3 \times 10^{18}/\text{cm}^2$   
 ANNEALING TIME : 5 min.

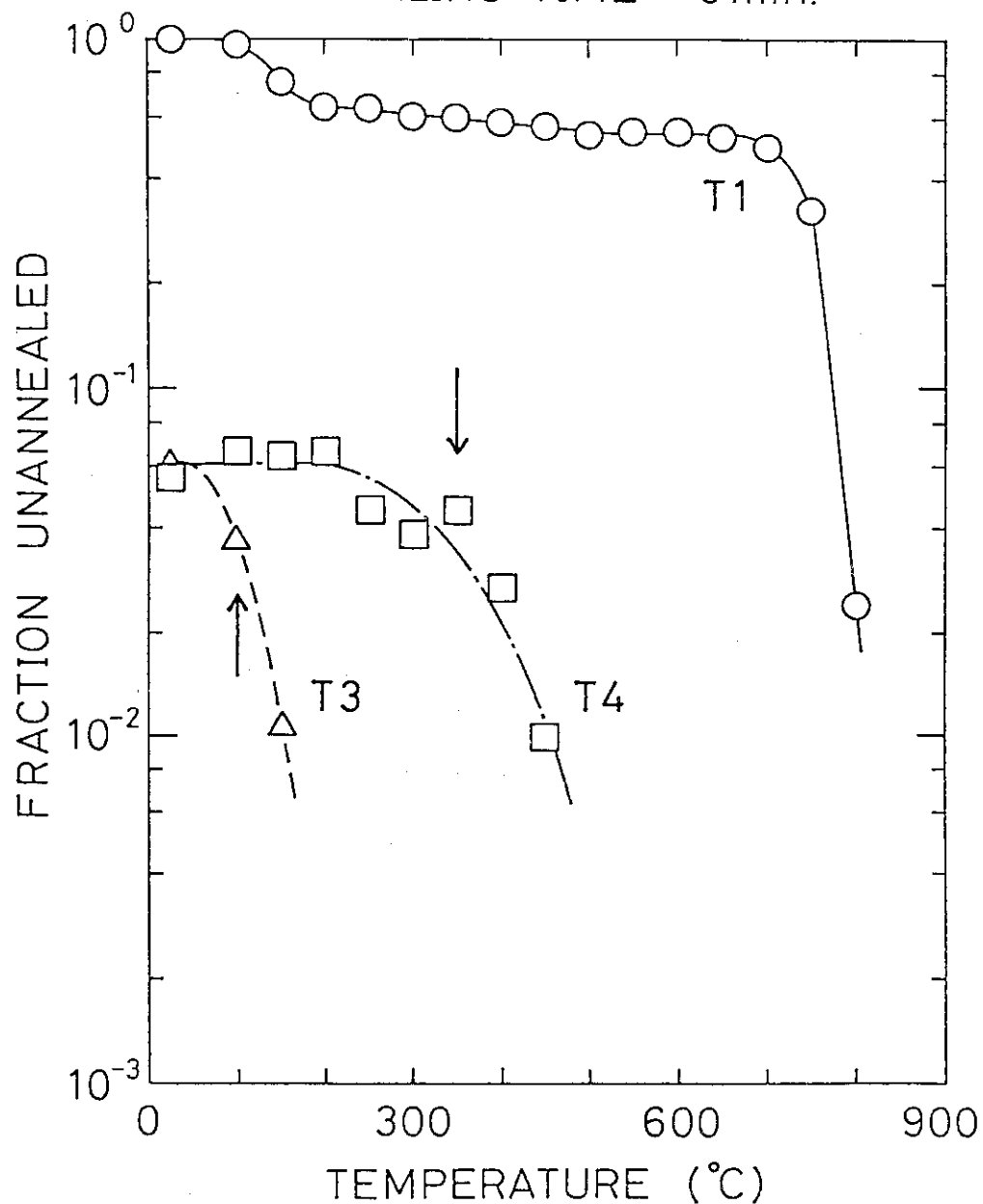


Fig.6 Isochronal annealing of the T3 and T4 centers in 3C-SiC irradiated with  $3 \times 10^{18}/\text{cm}^2$ . Data for the T1 center is also shown. The ordinate indicates the fraction unannealed which was normalized by the initial density of the T1. Stages for the T3 and T4 centers are indicated by arrows.