Electrically Detected Magnetic Resonance (EDMR) Measurements of Bulk Silicon Carbide (SiC) Crystals

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Electrically detected magnetic resonance (EDMR) measurements of bulk silicon carbide (SiC) crystals, which have wide band-gaps, were conducted. A high-power ultraviolet light-emitting diode was used as a low-noise light source to excite excessive carriers. Under light irradiation, EDMR spectra of a 4H–SiC wafer (8-mm wide, 8-mm long) at an ESR frequency of 900 MHz was obtained with a g-value of 2.000 ± 0.003, indicating that the major recombination center was a vacancy. EDMR spectra were observed at all detection phases of magnetic field modulation. The spectrum observed at in-phase (0 degrees) was a single line; at out-of-phase (90 degrees), the different spectrum was observed.

Electrically detected magnetic resonance (EDMR) spectroscopy is a method for analyzing semiconductor materials by detecting the changes in conductivity of a sample under ESR conditions.[1,2] This makes it possible to perform a highly sensitive and selective detection of paramagnetic recombination centers in the sample.

Recently, silicon carbide (SiC) has attracted attention because it is an excellent semiconductor material for next-generation, high-voltage, high-speed, and low-loss devices. However, the process for producing a SiC crystal of high purity is not well established. Furthermore, methods for analyzing the various properties of the SiC crystal have not yet been completely established. There have been attempts to observe EDMR spectrum of SiC used in MOSFET applications.[3,4] However, the spectrum of the bulk SiC has not been observed, where only the characteristic of SiC crystal is selectively observed. Since SiC has a wide band gap (2 to 3 eV), an ultraviolet (UV) light is needed to excite the excessive carriers to increase recombination for EDMR observations. Therefore, a sufficient sample space is needed in the EDMR instrument for placing both the sample and the irradiation light source. We have succeeded in establishing a method to observe EDMR at 900 MHz,[5–7] in which a large sample space is available without loss of sensitivity. Using our 900-MHz EDMR system, we succeeded in observing the EDMR spectrum of bulk SiC.

In our EDMR instrument, a resistive magnet (modified RE3X, JEOL, Japan) was used as the main magnet. The magnetic field was modulated at 70 to 770 Hz by a pair of modulation coils for lock-in detection using a lock-in amplifier (Model 5210, PARC, U.S.A.). The modulation signal was generated by an oscillator of the lock-in amplifier. The modulation coils were driven by a power amplifier (4020, NF, Japan; gain, 46 dB). A bridged shield loop-gap resonator (BLGR) was used so that the microwave field (B_1) would be efficiently and uniformly applied to the sample.[8,9] The resonant frequency of the BLGR was 890 MHz, and the unloaded Q of the BLGR was 510. The inner diameter of the resonator loop was 43 mm, and the axial length of the loop was 10 mm.

The sample used in our study was a nondoped 4H–SiC wafer. The sample was cut into a square plate (8-mm wide, 8-mm long). Two gold electrodes were formed on the surface of the wafer to provide ohmic contacts (8-mm wide, 3-mm long) for the sample. An n-type silicon (Si) wafer was also used as a reference material, and the data obtained from the Si wafer were compared with those obtained from the SiC sample. The Si wafer was also cut into a rectangular plate (8-mm wide, 19-mm long) and electrodes were formed.

Two Teflon holders (33.4-mm in diameter, 5-mm thick) were placed in a quartz glass tube (33.5-mm inner diameter; 38-mm outer diameter) to sandwich the sample. A 16×9 mm window was located at the center of one holder for illumination. A pair of gold-plated copper contacts was attached to the other holder for the electrical detection.

The excessive carriers were excited by a DC-drive high-pressure mercury lamp (ES-USH05, Ushio Inc., Japan; power, 500 W) and a high-power UV-light emitting diode (LED) (NCCU001, Nichia Co., Japan; center wave length, 380 nm; typical UV power, 100 mW). The light from the mercury lamp was condensed by a concave reflector, collimated by a UV convex lens, and irradiated onto the sample. The light from the power UV-LED was condensed through two UV convex lenses on the sample. Using these optical settings, the light source was separated far enough from the sample to avoid the influences of heating and disturbance of B_1.

EDMR measurements were performed during light irradiation using a high-pressure mercury lamp and UV-LED. During the UV-LED light excitation, we successfully observed EDMR spectra. However, the EDMR spectra were not observed during the mercury lamp excitation owing to the high noise level, 20 times larger than that for UV-LED irradiation. The noise during mercury-lamp irradiation may have been caused by flickering of light amplitude originating from discharges in the mercury lamp.

In this study, the detection phases of the magnetic field modulation were defined as 0 degrees (in-phase) when the maximum signal intensity of the EDMR spectrum of the Si wafer was obtained. We also defined another detection phase of 90 degrees (out of phase) when the minimum signal intensity of the EDMR spectrum was obtained. At 90 degrees, no signal was detected in Si. The EDMR spectra of the SiC were observed at all detection phases of the magnetic field modulation at 770 Hz. While the spectrum of SiC obtained at 0 degrees was a single line

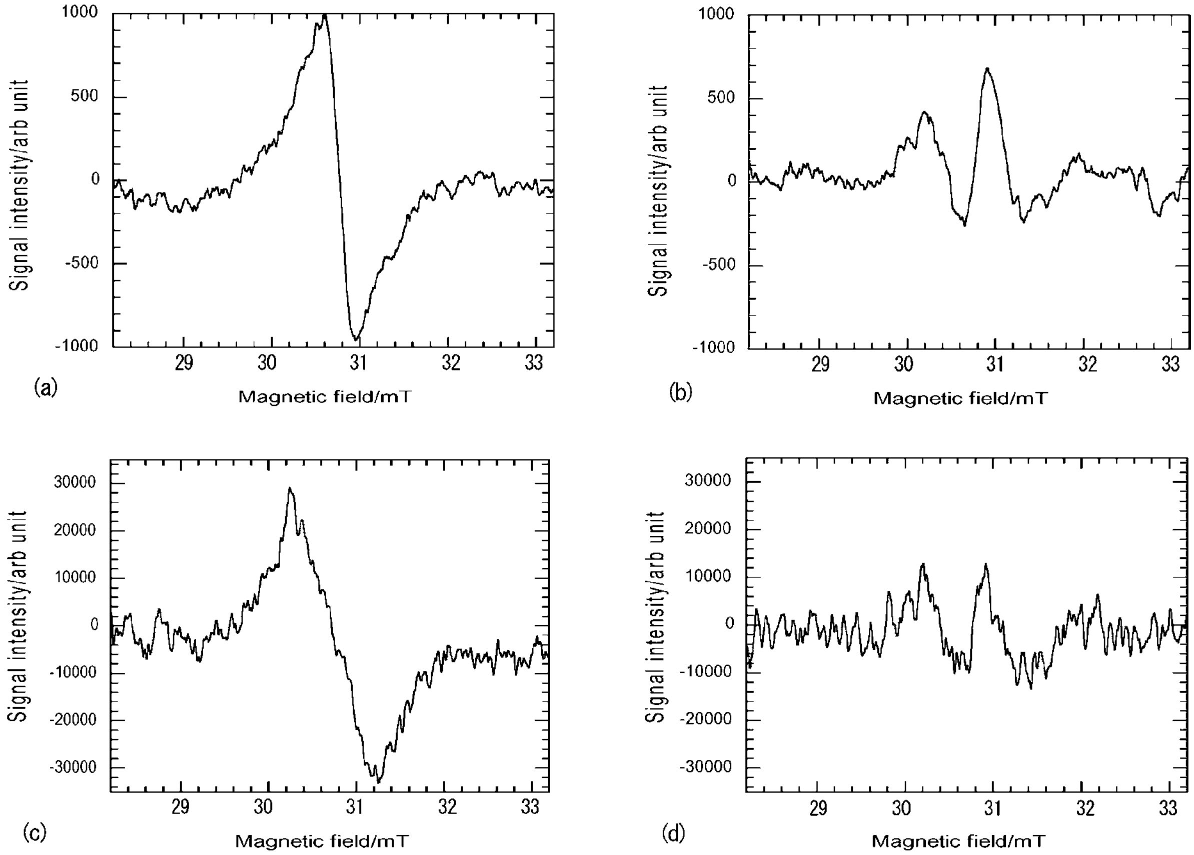

Figure 1. The EDMR spectrum of SiC wafer at magnetic field modulation frequency and detection phase of 770 Hz and 0 degree (a), 770 Hz and 90 degree (b), 70 Hz and 0 degree (c), or 70 Hz and 90 degree, respectively. Instrumental conditions: static magnetic field, 31 mT; sweep width, 5 mT; modulation width, 0.15 mT; sweep time, 30 s; RF power, 1 W.

(Figure 1a), a different spectrum was observed at 90 degrees (Figure 1b). The signal intensity at 90 degrees was about 40% of that obtained at 0 degrees. Similar phenomena were observed at modulation frequencies ranging from 70 to 770 Hz. For example, EDMR spectrum obtained at 0 degrees at 70 Hz is exhibited in Figure 1c, and EDMR spectrum obtained at 90 degrees at 70 Hz is exhibited in Figure 1d. Because the recombination time is needed for EDMR detection, the EDMR signal intensity was bigger at lower modulation frequency. The differences between linewidths of the spectra obtained at the lower and higher modulation frequencies were observed at the in-phase detection (Figures 1a and 1c), suggesting that two sites with the longer and shorter recombination times might be observed at the lower frequency. Because the phenomena that signal appears at 90 degree were observed at wide range of modulation frequencies, they might not concern with the recombination time, which were underway.

To measure the g-value of the SiC, EDMR measurements of SiC and Si were alternatively performed under the same conditions. Because the g-value of Si was already known ($g = 2.005$),[6,11] the g-value of the SiC could be determined by measuring the difference in values at the zero-crossing point of the spectra for both SiC and Si in the magnetic field. The magnetic field sweep width was calibrated by using a longitudinally detected ESR magnetometer.[10] The g-value of the SiC obtained was 2.000 ± 0.003, which was closer to the free electron than that of the Si, indicating that the major recombination center of SiC is a vacancy.[3,4]

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