Pulsed EPR and ENDOR study of SiC nanopowders

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The great potential of the micro- (mp) nano-particle (np) silicon carbide (SiC) for future applications in spintronics stimulates detailed studies of the effects of impurities and defects in their electronic properties. In particular, mp- and np-SiC doped with nitrogen (N) atoms is an important issue to be investigated, since N is a common contaminant in SiC bulk, acting as n-type dopant. Although N has been found to be present in the nanomaterials, there is no experimental evidence indicating for the formation of the N shallow donor state in np-SiC. The np-SiC samples with R < 50 nm were obtained by self-propagating synthesis of carbon (C) and highly active silicon (Si) at T = 1250^{0}C, while np-SiC with 50 nm <R < 100 nm were commercially produced. The green/black mp-SiC were produced in an electric resistance type furnace at T = 1750^{0}C with quartz sand and petroleum coke as its main raw materials. The X-band (ν = 9.7 GHz) two pulse field-sweep electron spin echo (FS ESE) spectra and pulsed electron nuclear double resonance (ENDOR) spectra were measured on Bruker ELEXSYS E580 spectrometer at T = 6 K (Fig. 1). All np-SiC samples revealed the FS ESE signal from carbon vacancy (V_C) located in 3C-SiC crystalline phase. Along with the V_C the triplet lines due to the N in 6H SiC crystalline phase was observed in the FS ESE spectrum of the np-SiC with the R > 50 nm which was identified by pulsed ENDOR as the N in quasi-cubic (N_{k2}) site. As the size of the np-SiC decrease, the signal from V_C in 6H SiC phase appeared in the FS ESE spectrum, which gives rise to the compensation of the N donors and as a result the FS ESE spectrum intensity of N decrease significantly. Only two signals due to the V_C located in 3C and 6H SiC phase were observed in the FS ESE spectrum of the np-SiC samples with the R < 50 nm. The fact that the ENDOR signal centered at the hydrogen (^{1}H) nuclear Larmor frequency (ν_n) was observed in the np-SiC with R < 100 nm indicates that H atoms are localized in the vicinity of the V_C and can form the H-related defect like (V_C+H). As a conclusion, two phases (α- and β-SiC crystalline phases) are presented in np-SiC.

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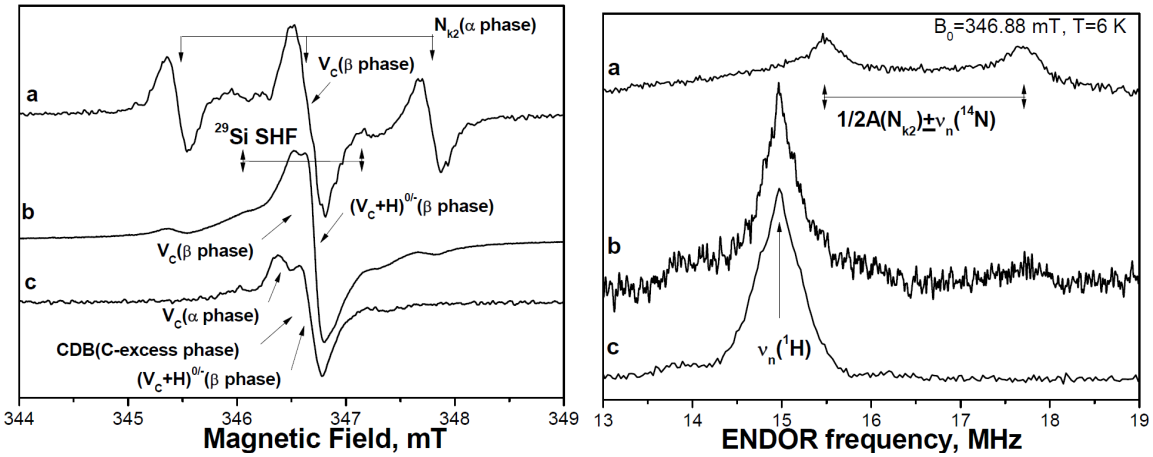

Fig. 1. The X-band first derivative of FS ESE (a) and pulsed ENDOR (b) spectra measured in np- and mp-SiC with R > 100 nm (a), 50 nm < R < 100 nm (b), R < 50 nm (c) at T = 6 K.