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Pulsed EPR and ENDOR study of SiO2:C nanopowders

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The carbonized silica (SiO₂:C) nanopowders prepared by chemical modification of fumed silica (aerosil) by $C_9H_{14}O_3Si$ in the N_2 flow were studied by field-sweep electron spin echo (FS ESE) and pulsed electron nuclear double resonance (ENDOR) spectroscopy at T=20 K. Just after the synthesis no FS ESE spectra were observed in the samples while after annealing at Tann 2700°C the samples revealed a strong FS ESE and ENDOR signals. The first derivative of the FS ESE spectrum measured in the annealed SiO2:C (the inset on Figure) revealed the single line with Gaussian shape and linewidth of 0.56 mT at $g_{150}=2.0030(3)$ that was attributed to the carbon-related radical (CRR). The ENDOR spectrum shown in the Figure was measured at the CRR FS ESE line position and it consists of superhyperfine (shf) lines due to the interaction of CRR with surrounding 29 Si, 13 C and 1 H nuclei. It was found that the main contribution to the FS ESE linewidth comes from the shf interaction with ${}^{1}\text{H}$ nuclei with the largest splitting A_6=0.52 mT. We suggest that after annealing at Tann²700°C the hydrogen from the broken methyl groups becomes available to saturate the C dangling bonds forming the (C-H) radicals with the parameters close to those observed for hydrogen-related defects labeled H1 and H2 in polycrystalline diamond.