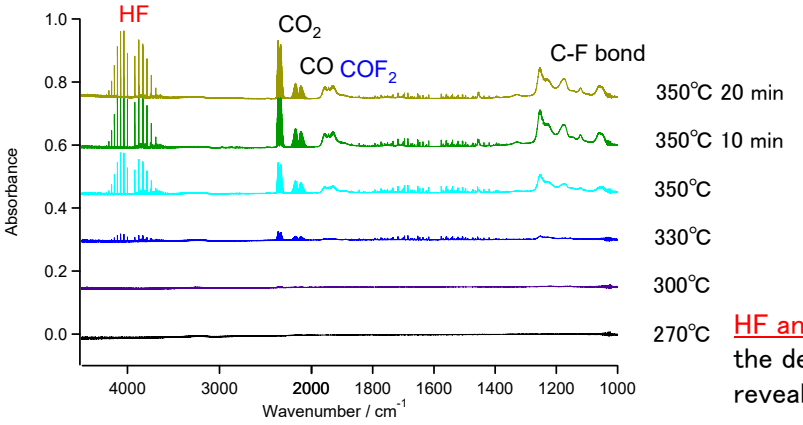
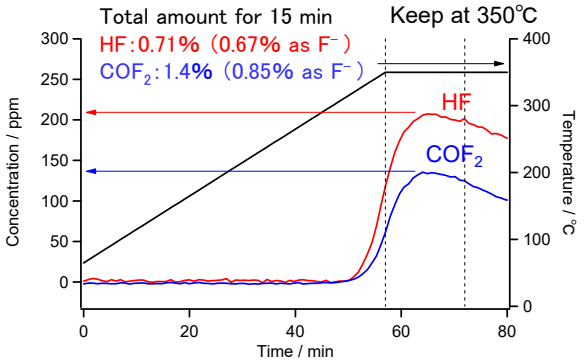
Spectroscopic analysis of thermal degradation and decomposition behavior of fluorine resins

Fluorine resins have attracted much attention as 5G materials because of their low dielectric loss. Their thermal degradation and decomposition behaviors can be evaluated by decomposed gas analysis, ESR, UV/Vis, FT-IR, and solid-state NMR measurements.

ETFE (ethylene tetrafluoroethylene) film was heated in simulated air at a heating rate of 5 ℃/min and kept at 350℃.

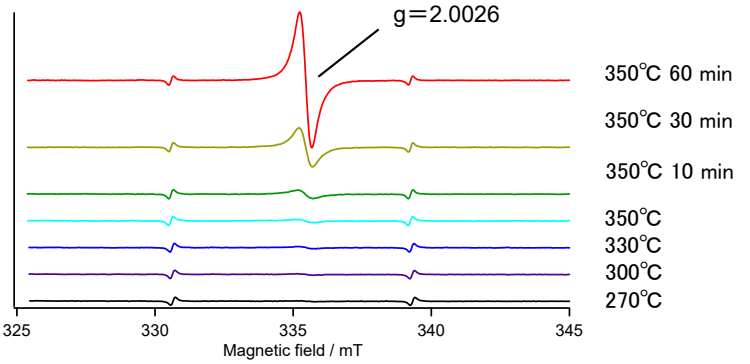
On-line decomposed gas analysis by FT-IR





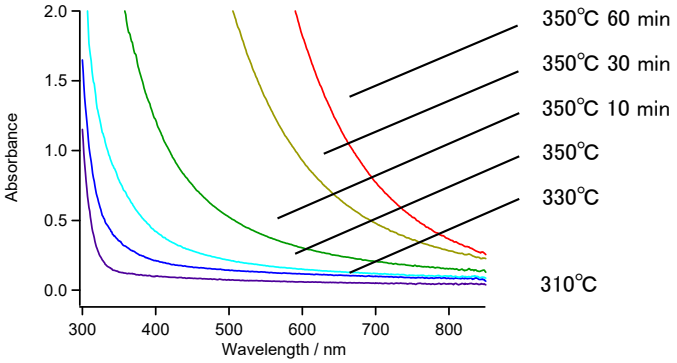
HF and COF_2 were detected separately, and the total amount of the decomposed gas can be estimated. This on-line analysis reveals that the decomposition of this sample begins at 320℃.

in situ ESR spectra with heating



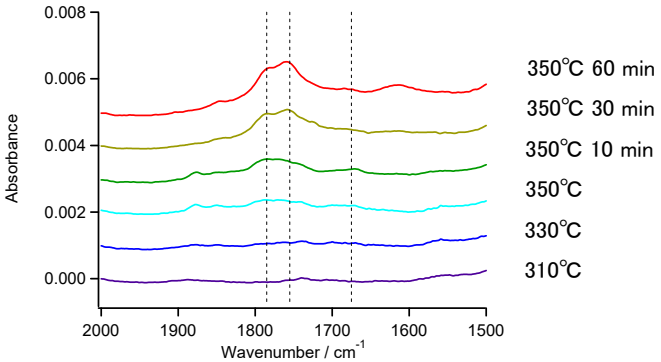
Radicals derived from C=C conjugated systems were generated beyond 300℃.

UV/Vis absorption spectra after heating



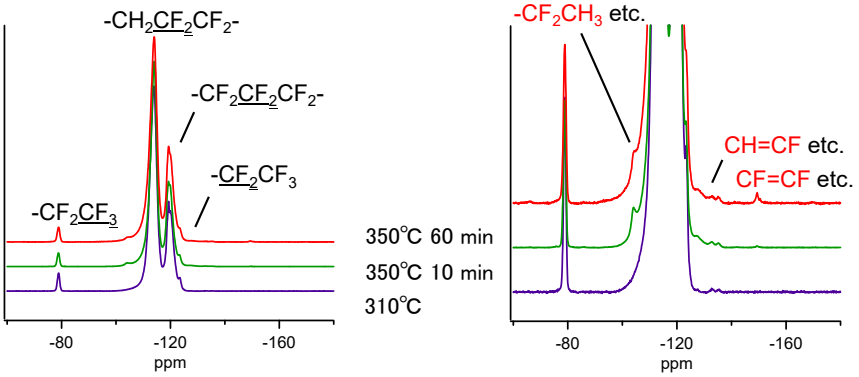
A broad absorption band suggests that conjugated systems with various lengths were generated.

FT-IR-ATR spectra after heating



C=O and C=C bonds adjacent to F atoms were formed.

^{19}F solid-state NMR spectra after heating



It is considered that C=C and $-CF_2CH_3$ are formed. Further detailed information such as the copolymerization ratio can be obtained by ^{13}C NMR.

(i) Formation of conjugated C=C due to elimination of HF by heating, (ii) generation of CO_2 and COF_2 due to molecular chain scission and oxidation, (iii) formation of C=O bonds, and (iv) formation of new terminals such as $-CF_2CH_3$ were observed.