Abstract: With the development of ultralarge scale integrated circuit, new interlayer dielectrics with low dielectric constant for multilevel interconnections are required, instead of conventional SiO₂ films. For the sake of seeking perfect dielectrics, amorphous fluoropolymer (AF) thin film with a thickness of about 0.9μm has been prepared by spin-coating method, following the principle of phase separation. By capacitance-voltage (*C-V*) measurements the dielectric constant of the thin film is equal to 1.57 at 1 MHz, which is attributed to numerous pores contained in the film matrix. X-ray photoelectron spectroscopy (XPS) spectra show that after annealing, about 71% CF₃ groups in the AF film have decomposed into CF₂, CF, etc. This leads to the increase of CF₂ groups by three times and CF groups by 8% in the AF film. In a word, compared with the film without being annealed, about 25% carbon, 7% fluorine and 12% oxygen atoms will be lost after annealing at 400°C for 30min.