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# Temperature effect on the chemical composition of a polytetrafluoroethylene surface under the irradiation of synchrotron radiation by means of the X-ray photoelectron spectroscopy

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**Abstract**

The chemical composition and components of a polytetrafluoroethylene (PTFE) surface was investigated as a function of the temperature under the irradiation of synchrotron radiation (SR) by the X-ray photoelectron spectroscopy (XPS). When the temperature of PTFE under the SR irradiation was less than 100 °C, the C-rich surface appeared. With increasing the temperature more than 150 °C, the relative intensity of the F 1*s* peak to the C 1*s* peak increased markedly. At the temperatures of 150–180 °C, the C–C component became small and the $CF_2$ component was dominant. With further increasing the temperature more than 200 °C, $CF_3$, CF and C–CF components grew in addition to $CF_2$ component. Based on these XPS results, the temperature effect on the chemical composition and components is discussed.


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*Keywords:* Synchrotron radiation; X-ray photoelectron spectroscopy; XPS; Polytetrafluoroethylene; PTFE; Substrate temperature

## 1. Introduction

Since it is well known that a polytetrafluoroethylene (PTFE) is easily decomposed by the irradiation of synchrotron radiation (SR), the micromachining and the film formation of PTFE have been extensively attempted in recent years (Inayoshi et al., 1995; Zhang et al., 1995; Zhang and Katoh, 1996; Katoh and Zhang, 1996; Yoshida et al., 2003). Katoh and Zhang demonstrated that the deposition rate of PTFE thin films increased with increasing the temperature of the PTFE target and that the excellent PTFE thin films were produced at the target temperature of 200 °C (Katoh and Zhang, 1996). In the case of the micromachining using the SR irradiation, the microstructures with a higher aspect ratio were produced at the temperature of 200–250 °C (Zhang et al., 1995; Zhang and Katoh, 1996). Thus, the

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temperature of PTFE under the SR irradiation is very critical. Very recently, the SR irradiation experiments on the surface modification of PTFE indicated that the surface morphology and the wettability were controlled, depending on the temperature of PTFE under the SR irradiation (Kanda et al., 2003). As for the chemical composition of the PTFE surface, the depletion of F atoms was observed at room temperature (RT) after the SR irradiation (Haruyama et al., 2003). The depletion of F atoms is understood by the mechanism that the F-rich species like saturated fluorocarbon gas are desorbed from the PTFE surface (Fisher and Corelli, 1981; Wheeler and Pepper, 1982; Simons et al., 1994; Katoh and Zhang, 1996; Haruyama et al., 2003). However, the effect of the temperature under the SR irradiation on PTFE has not been fully understood. It is important to clarify the temperature dependence of the chemical composition and components on the PTFE surface under the SR irradiation.

In this study, we investigated the temperature effect of the PTFE surface under the SR irradiation by means of X-ray photoelectron spectroscopy (XPS). The chemical composition, as well as the chemical components and their intensity ratios were determined. Based on the XPS results, the temperature effect of the PTFE surface under the SR irradiation is discussed.

## 2. Experimental

The SR irradiation on the PTFE sheet was performed on a beamline (BL) 6 at the NewSUBARU facility, University of Hyogo. The electron energy of the storage ring was 1 GeV and the typical beam current of the storage ring was ~100 mA. At BL 6, the photon energy ($h\nu$) of SR ranged from 50 to ~1000 eV. The photon flux curve of BL 6 was calculated by Taniguchi et al. (2002). The size of the SR was ~10 mm$\phi$ on the PTFE sample. The typical size of the PTFE samples was $10 \times 10 \times 1\,mm^3$. After PTFE sheets were attached to the Cu sample holder, the irradiation chamber of BL 6 was evacuated up to $\sim 1 \times 10^{-5}$ Pa. The temperature of each PTFE sheet was set from RT up to 240 °C and the same SR dose was irradiated to each PTFE sheet. The SR dose was represented by the product of the storage ring current and the irradiation time. In this experiment, the SR dose was 80 mA h. PTFE sheets were heated with the resistive heater and the temperature of the PTFE sheet was measured with the Cr–Al thermocouple. After the SR irradiation, the temperature of the PTFE sheet was returned to RT. Then, PTFE sheets were once exposed in air and led to the photoelectron analysis chamber at BL 7B for XPS measurements.

XPS measurements were carried out on the BL 7B end station by using the conventional photoelectron spectroscopy apparatus, which is mounted with a CL150 (VSW Ltd.) hemispherical electron energy analyzer. The Mg K$\alpha$ line ($h\nu = 1253.6$ eV) was used as an X-ray source and was incident at 45° with respect to the surface normal of PTFE. All XPS spectra were recorded at the emission angle of 45° to the surface normal. The total energy resolution was about 0.7 eV. The base pressure in the photoelectron analysis chamber was $2 \times 10^{-8}$ Pa. All XPS measurements were preformed at RT.

## 3. Results and discussion

Fig. 1 shows the XPS spectra of the SR irradiated PTFE sheets as a function of the temperature under the SR irradiation. The temperatures of PTFE under the SR irradiation are denoted beside each spectrum. There are several peaks originating from the C and F atoms in each photoelectron spectrum. Three peaks at ~690, ~290, ~40 eV are assigned to the F 1*s*, C 1*s* and F 2*s* levels, respectively while a peak at ~600 eV is assigned to Auger electrons caused by F KLL decay process (Wagner et al., 1978). In addition to these peaks, the O 1*s* peak appeared at ~530 eV when the temperature of PTFE under the SR irradiation was less than 100 °C. On the other hand, above 150 °C, the O 1*s* peak did not appear clearly. The appearance of the O 1*s* peak indicates that only the PTFE surface irradiated at less than 100 °C becomes active for adsorption since all

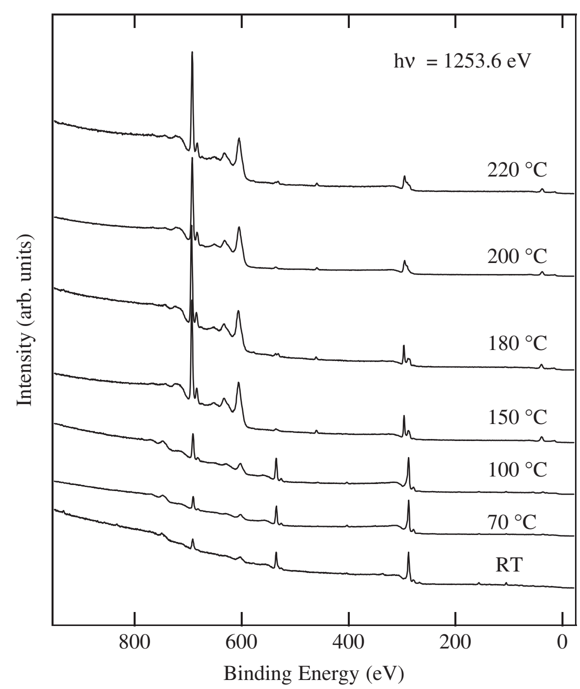


Fig. 1. Wide scan of the XPS spectra of the SR irradiated PTFE sheets as a function of the temperature under the SR irradiation.