MLF Experimental Report	提出日 Date of Report 2012.9.1
課題番号 Project No. 2012A0035 実験課題名 Title of experiment Molecular Dynamics of Polymer Chains in the Crystal and Amorphous Regions of Overall Fluorinated Polymers (Teflon-like Polymers). 実験責任者名 Name of principal investigator Go Matsuba 所属 Affiliation Yamagata University	装置責任者 Name of responsible person Kenji Nakajima 装置名 Name of Instrument/(BL No.) BL14 実施日 Date of Experiment 2012.5.13-18

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Sample

In this proposal, we use crystalline “overall fluorinated polymer” (Teflon-like polymer), which compose of poly(tetra fluoro-ethylene) (PTFE) and poly(perfluoroethylvinylether) in Figure 1. This overall fluorinated polymer consists only of carbon, oxygen and fluorine and no hydrogen. The incoherent cross-section of C, O and F are under 0.001. The crystallinity X_c is 30 % and 25 % controlled by cooling rate (0.5 °C/min and 10 °C/min) from molten state, respectively.

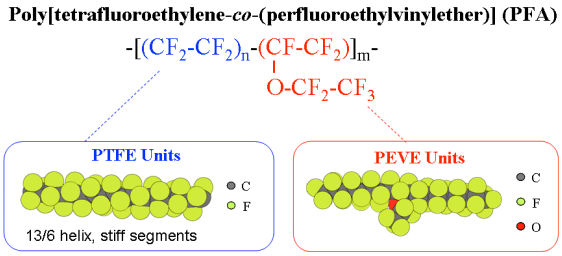

Figure 1. Schematic drawing/chemical struture of PFA polymer.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Experimental Method:

The incident energy conditions were 3.131meV, 7.732meV and 40.2meV, because of observation of high resolution and wide spatial resolution modes. The temperature was 50K, 200K, 230K, 270K, 300K and 323K controlled by 4He-closed-refrigerator.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Results

We carried out inelastic/quasi-elastic neutron scattering measurements with the AMATERAS spectrometer. Two crystallinity samples (X_c = 30 % and 25 %) were measured. Especially, we focused on crystallinity dependence on the polymer chain dynamics especially in the range of sub-nm (Å) scale. The sub-nm scale structure and chain dynamics have strong correlation to gas permeability.

Figures 2 shows Q-E map for E_i = 7.732 meV with X_c = 30 % and 25 % samples at 50K. The signals around Q = 1.0 Å^{-1} and 2.5 Å^{-1} are caused by phonon dispersion. The intensity at 50 K decreases with crystallinity.

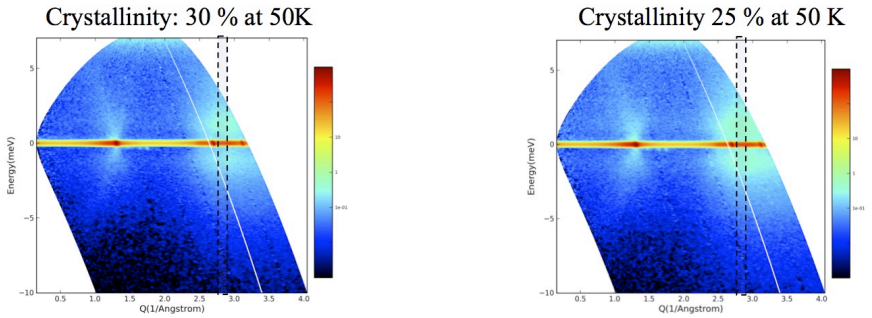

Figure 2 Q-E_i map for two crystallinity samples.

Figure 3 shows dynamic scattering laws $S(Q, \omega)$ of two crystallinity samples (X_c = 30 % and 25 %) in Q = 2.8 ~ 2.9 Å^{-1} (within a square with dotted lines in Figure 2) measured with AMATEAS spectrometer. In 50 K, we could find the so-called Boson-peak in Energy = 1.2 meV. The inelastic scattering peaks were observed in 0.8 meV (230 K) ~ 0.5 meV (250 K) and appropriate Energy = 0 meV in increasing temperature. This suggests that the transition of side chains might be below 230 K. Above 270 K, we could find very strong quasi-elastic scattering profiles and increases with temperature.

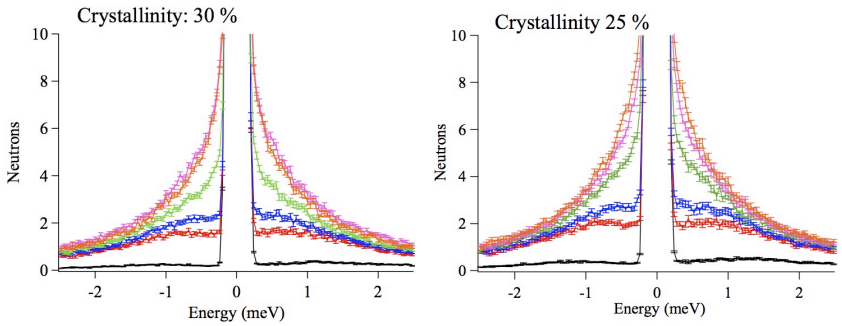

Figure 3 Dynamic scattering laws $S(Q,\omega)$ of Teflon-like polymers for two crystallinity samples.

Now, we analyze that these data are deconvoluted into elastic (vanadium scattering profile) and quasielastic (lorentzian) and linear background scattering contribution with UTSUSEMI software. After this analysis, we can find that mean square displacement $\langle u^2 \rangle$ in two crystallinty samples.