実験報告書様式（一般利用課題・成果公開利用）

（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。）

J-PARC **MLF Experimental Report**	提出日 Date of Report
課題番号 Project No. **2014B0001** 実験課題名 Title of experiment **Neutron Reflectivity for Revealing the In-depth Phase Separation of an Organic Photovoltaic Film and Migration of PC71BM into the PEDOT** 実験責任者名 Name of principal investigator **Dr. JENG U-Ser** 所属 Affiliation **National Synchrotron Radiation Research Center, Taiwan**	装置責任者 Name of responsible person **Norifumi YAMADA** 装置名 Name of Instrument/（BL No.） **BL 16** 実施日 Date of Experiment **14 th Apr 2015 – 17th Apr 2015**

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.
PTB7 - thieno[3,4-b]thiophene-*alt*-benzodithiophene copolymer (C41H53FO4S4) PTB7-Th - thieno[3,4-b]thiophene-*alt*-benzodithiophene copolymer (C49H59FO2S6) 60-PCBM - [6,6]-phenyl C61 butyric acid methyl ester (C72H14O2) 70-PCBM - [6,6]-phenyl C71 butyric acid methyl ester (C82H14O2) PEDOT:PSS - poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (C6H6O2S:C8H8O3S) ZnO - sublimed zinc oxide thin films

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.

The purpose of this experiment is to reveal the vertical volume fraction profiling of components in the active layer spin-cast on PEDOT:PSS substrates. Some differences between PTB7:PC71BM blend films processed with DIO additive or without additive can be observed by AFM and device efficiency. That is, the DIO-added cases reach higher PCE value than non-DIO cases. In terms of the nano-structural arrangement, both horizontal and vertical phase segregation are critical to PCE. To investigate the in-depth profiling, by using the BL16 SOFIA neutron reflectometer, NR data covering λ from 2 to 8.8 nm with time-of-flight mode can span a q_z-range of 0.007−0.15 Å^{-1} with three incident angles θ = 0.3°, 0.7°, and 1.6°. The first step is to measure the SLD profile of PEDOT:PSS/Si substrate. Both the analysis and its corresponding in-depth profiling are shown in Figure 1 and the inset. And then we need to suppose the conservation of neutron SLD of PEDOT:PSS in other complex polymer blend cases spin-cast on PEDOT:PSS/Si.

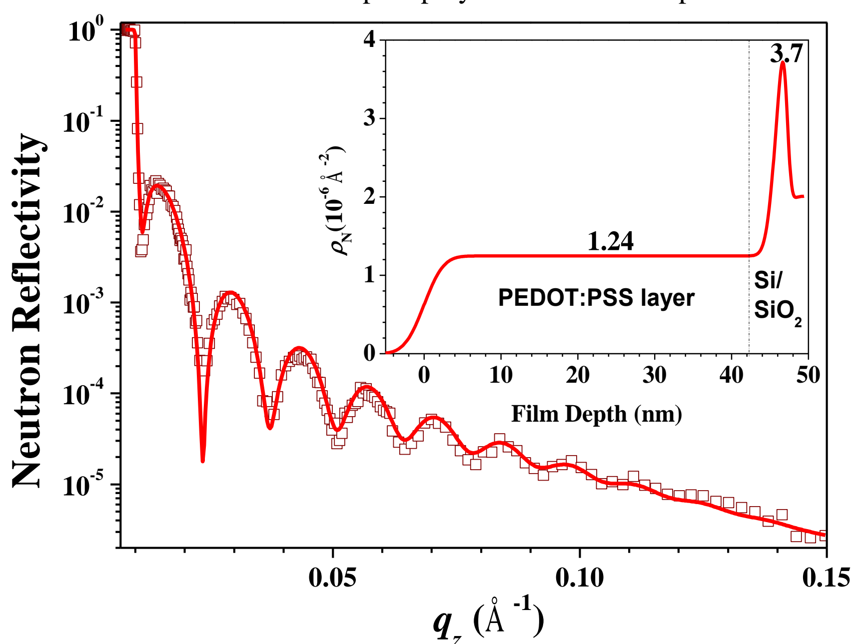

Figure 1. The NR data for the PEPOT:PSS film spin-cast on Si wafer. The data are fitted by double-layer model. The vertical SLD profiling and the averaged SLD are shown in the inset. Note that the thin SiO_2 layer can be detected by NR, due to its higher contrast in neutron.

The second step is to measure the polymer blend spin-cast on PEDOT:PSS/Si. Single-layer model and triple-layer model was used for analyzed the experimental data of PTB7:PC71BM (1:1.5 w/w) processed with CB and DIO/CB, as shown in **Figure 2**, implying that DIO might facilitate the vertical component distribution to be more uniform, while the non-DIO cases show that three zones of different SLD distribution. Note that the PEDOT:PSS/Si substrates hold the same SLD profiling as the pristine film. Therefore, the vertical component concentration profiles can be acquired.

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

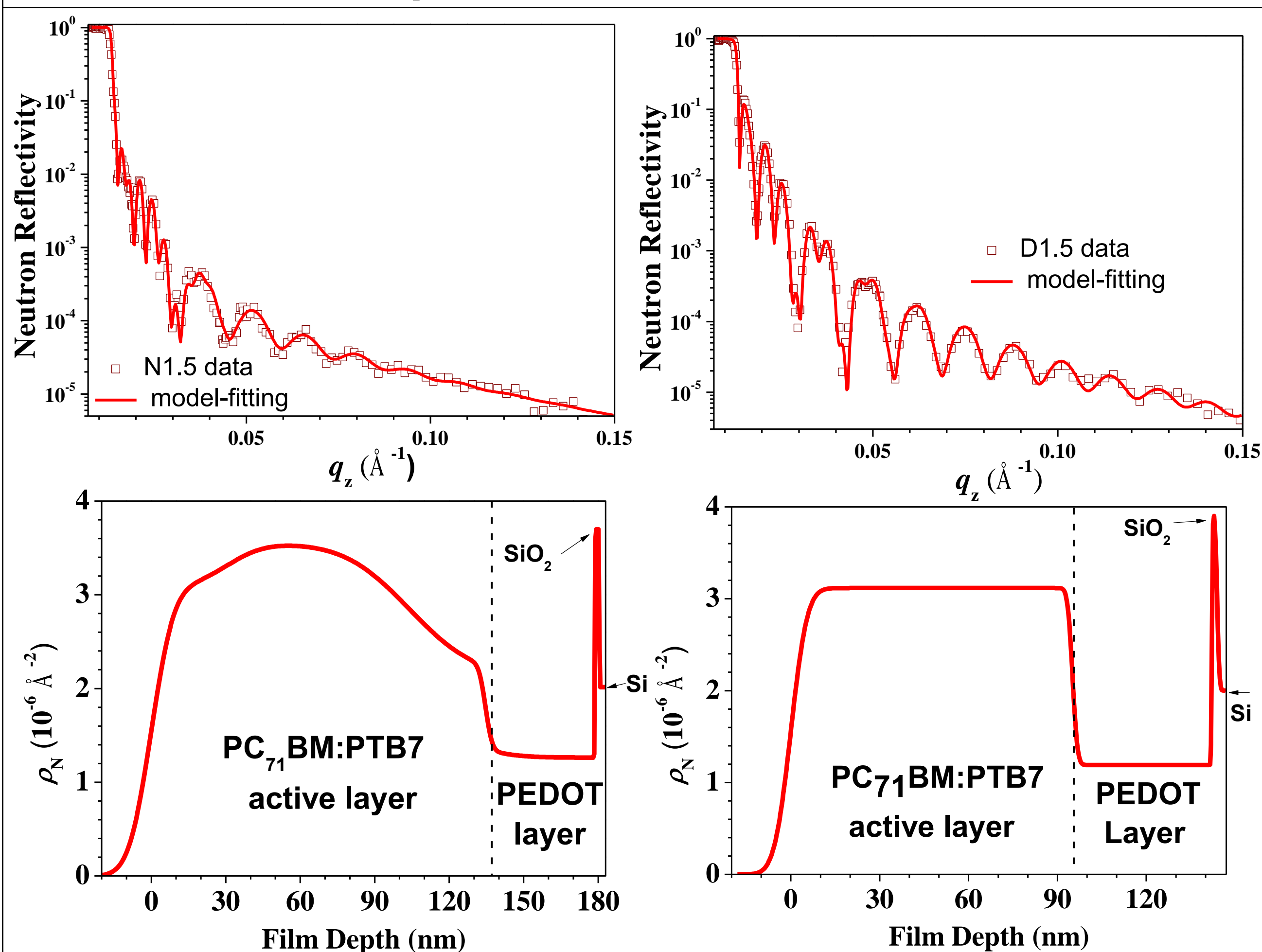

Figure 2. NR data and its corresponding in-depth SLD profiling for the N1.5 (left-hand side of four graphs) and D1.5 (right-hand side of four graphs) film. The similar SLD for PEDOT:PSS layer is supposed the same as the pristine film.

The final step is to compare neutron SLD with X-ray SLD. By the known SLD of components determined by previous experiments, the component volume fraction profiles can be obtained by scattering contrast variation. The revealed vertical component profiles suggest the relationship between distribution and PCE. Moreover, the effect of DIO can modify both in-plane and in-depth morphology, hence the improved device performance. Our results provide a methodology to insight the OPV films by nondestructive measurements of reflectometry.

Not all samples brought to J-PARC were tested as planned, because of the unsteady neutron light source and unexpected low neutron scattering intensity of ZnO films. Except for that, both the data quality and the service in BL 16 of MLF are magnificent. We are grateful for the kind help by Dr. YAMADA and MLF.