

 <b>MLF Experimental Report</b>	提出日 Date of Report 2017. 4. 10
課題番号 Project No. 2104B0249 実験課題名 Title of experiment Neutron-diffraction study of effects of the reduction annealing on the electronic state in electron-doped high- $T_c$ $T'$ -superconductors 実験責任者名 Name of principal investigator Tadashi Adachi 所属 Affiliation Department of Engineering and Applied Sciences, Sophia University	装置責任者 Name of responsible person Takashi Kamiyama 装置名 Name of Instrument/(BL No.) BL08 (SuperHRPD) 実施日 Date of Experiment 2016. 6. 16 - 17

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.  Electron-doped high- $T_c$ $T'$ -superconductor $\text{Pr}_{1.7-x}\text{La}_{0.7}\text{Ce}_x\text{CuO}_{4+\delta}$ ( $x = 0.10$ ) Single crystals
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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.  Neutron diffraction measurements were performed at temperatures of 11 K, 150 K and 300 K for 6 hours in the electron-doped high- $T_c$ $T'$ -superconductor $\text{Pr}_{1.7-x}\text{La}_{0.7}\text{Ce}_x\text{CuO}_{4+\delta}$ ( $x = 0.10$ ) single crystals. The single crystals were reduced in vacuum by four steps; the protect annealing at 800°C for 24 hours, the low-temperature annealing at 400°C for 24 hours, the dynamic annealing at 500°C for $4 \times 6$ hours, the dynamic annealing at 750°C for $4 \times 5$ hours. The single crystals of the weight of 1 gram were crushed into powders and put into a vanadium sample cell.
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## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

At 11 K, the preliminary Rietveld analysis of the data using the Z-Rietveld software revealed that the occupancy of the oxygen in the  $\text{CuO}_2$  plane is lower than 100% in the present reduced single crystals of  $\text{Pr}_{1.7-x}\text{La}_{0.7}\text{Ce}_x\text{CuO}_{4+\delta}$  ( $x = 0.10$ ). This suggests that the four-step reduction annealing brings about the oxygen defects in the  $\text{CuO}_2$  plane of  $\text{Pr}_{1.7-x}\text{La}_{0.7}\text{Ce}_x\text{CuO}_{4+\delta}$  ( $x = 0.10$ ). On the other hand, whether or not the excess oxygen at the apical site of the  $\text{CuO}_2$  plane exists, which is in the heart of the present study, has not yet clarified. We continues to analyze the data at three temperatures in order to clarify effects of the reduction annealing on the oxygen at the apical site and in the  $\text{CuO}_2$  plane.