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実験課題名 Title of experiment
Project research on structure and dynamics of proton, superionic and amorphous functional materials

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1. 研究成果概要(試料の名称、組成、物理的・化学的性状を明記するとともに、実験方法、利用の結果得られた主なデータ、考察、結論、図表等を記述してください。
Outline of experimental results (experimental method and results should be reported including sample information such as composition, physical and/or chemical characteristics.

1) Structure analysis of drag related small molecular organic material

Sample : Glycylglycine($C_4H_8N_2O_3$)

Size : 1.5 x 1.0 x 0.3 mm^3

Purpose : Biochemical materials are one of the best example in which light atoms, especially hydrogen atoms, play crucial roles in realization of functionalities of materials. The understanding of the mechanism of the functionalities is essential for developing and designing new medicines and drags. Since the functionalities and molecular structures are, in general, closely related with each other, determination of the precise crystal/molecular structure is considered to be the indispensable first step in the course of research. Here, we conducted structural measurement of a simple bio-molecular material, glycylglycine, so as to assess the feasibility of the structural analyses of such bio-material on the newly built single crystal neutron diffractometer, SENJU.

Experimental : The experiment was carried out at room temperature. The sample was mounted on the room temperature goniometer at BL18, SENJU. Quasi-Laue diffraction patterns were collected in five different crystal orientations spending seven hours for each orientation.

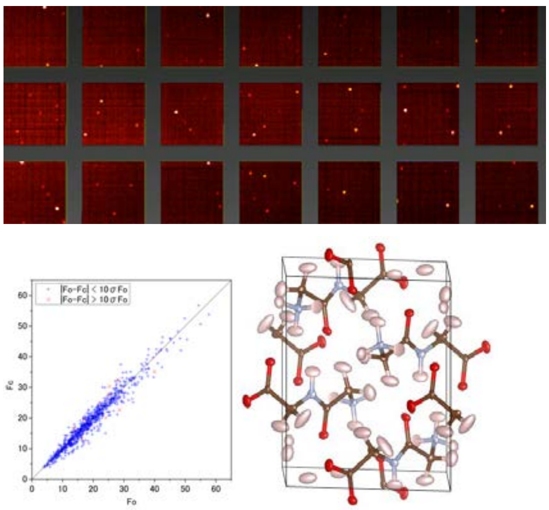

Fig.1 (upper) Observed TOF-Laue pattern. (lower left) Results of structure analysis. (lower right) obtained crystal structure of glycileglycine.

１. 研究成果概要（つづき） Outline of experimental results (continued).

Results : One of the obtained quasi-Laue diffraction pattern is shown in Fig. 1 (upper). Many Bragg reflections can be clearly observed even from such a small organic sample. Lattice parameters were calculated from the positions of the Bragg reflections and determined at a = 8.159 Å, b = 9.589 Å, c = 7.799, β=107.62, that are compatible with the previously reported values. The crystal structure was analyzed using the obtained Bragg intensities. Several thousands Bragg reflections were detected, and among them 812 reflections satisfying I/sigI > 4 were actually used for the structure analysis. Structural parameters including anisotropic thermal factors of all atoms were refined and converged into physically reasonable values. The result is shown in Fig. 1 (lower). The left figure shows the comparison between the observed and calculated structure factors, and they are well coincide with each other. The right figure shows the obtained structure of the molecule. The anisotropy of the thermal motion of hydrogen atoms reflecting the bonding direction is well described. In this study, the feasibility of the structure analyses of bio-chemical materials on SENJU was confirmed, thus actual materials with scientific interests will be studied in the next period.

2） Structural analyses of protonic conductor under high pressure

Sample : $Rb_3H(SeO_4)_2$

Size : 1.5 x 1.0 x 0.8 mm^3

Purpose : The materials represented as $M_3H(XO_4)_2$ (M = alkaline metals, X=Se, S) are known to exhibit high protonic conductivities at moderately high temperature. The high protonic conductivity emerges upon a structural phase transition. The conductivity and the phase transition temperature vary depending on the species of M and X. Recent study showed that the application of pressure increases the conductivity and lowers the phase transition temperature. In order to clarify the relation between the internal structure and the conductivity and the phase transition, structural studies under high pressure are indispensable. To achieve this purpose, as a first step, a structure analysis of $Rb_3H(SeO_4)_2$ in a pressure cell was performed on SENJU (ambient pressure).

Result : The sample measured was 1.5 x 1.0 x 0.8 mm^3 and sealed in a piston-type pressure cell made of Cu-Be alloy. In the measurement, a pinhole was installed in front of the cell in order to avoid the incident neutron from hitting the cell as much as possible. With this pinhole mask, even though the sample was completely surrounded by the cell, Bragg reflections from the sample were clearly observed. From these reflections, the lattice constants were calculated at a = 15.47(4) Å, b = 6.091(5) Å, c = 10.56(2) Å, β = 103.3(2), that are well coincide with previously obtained values. An orientation matrix was also calculated to extract integrated intensity data. The problem is the fact that the powder diffraction from the cell are still strong and make it difficult to extract accurate integrated intensities out of the obtained 3D data. As a next step, a more effective pinhole will be made for the next experiment, and more sophisticated data reduction system will be developed.

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