Single Crystal Structure Analysis of Magnetically Oriented powder Crystal

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Introduction

We have been developed Magnetically Oriented Microcrystal Array (MOMA) method to make it possible to carry out single crystal X-ray diffraction analysis from microcrystalline powder. In the method, microcrystals suspended to the UV-curable monomer are there-dimensionally aligned by frequency-modulated rotating magnetic field. Then the obtained alignment is consolidated by the photopolymerization. From thus achieved MOMAs, we have been succeeded in crystal structure analysis for some substances [1] [2]. Though MOMA method is an effective technique, it has some following problems; in a MOMA, the alignment is deteriorated during the consolidation process. In addition, the sample microcrystals cannot be recovered from a MOMA. To overcome these problems, we performed an *in-situ* X-ray diffraction measurement using a 3D Magnetically Oriented Microcrystal Suspension (MOMS) of L-alanine.

Experiments

A measurement setting of the MOMS technique is schematically shown in **Fig 1**. L-alanine microcrystal suspension was poured into a glass capillary and placed on the rotating unit equipped with a pair of neodymium magnets. Rotating X-ray chopper with 10º-slits was placed between the collimator and the suspension. By using the chopper, it was possible to make specific direction of the rotating MOMS be exposed to the X-ray, realizing the same measurement situation as the 10 degree oscillation angle measurement for the usual single crystal measurement. A total of 22 XRD images of 10º increments from 0º to 220º were achieved.

Results & Discussion

The data set was processed in the same way as the single crystal measurement and the crystal and 3D Molecular structure of L-alanine was determined. They showed well agreements with the reported one determined from the single crystal (Fig. 2). *R*1 and *wR*2 were 6.53 and 17.4 %, respectively. RMSD value between the achieved 3D molecular structure and the reported one was 0.0042 Å. From the result, we concluded that this method can be effective and practical way to perform crystal structure analysis.

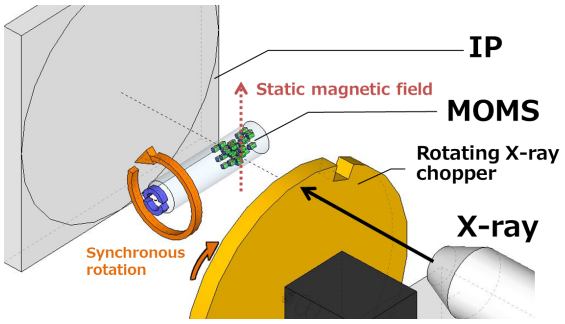

Fig. 1 Schematical image of the measurement setting.

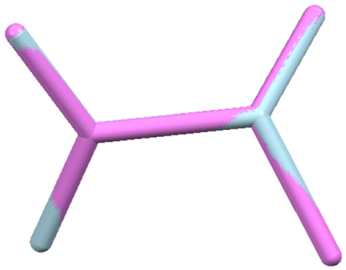

Fig. 2 Comparison of the structure determined in this study (blue) and reported one (pink).

Reference

1) T. Kimura, C. Chang, F. Kimura and M. Maeyama: J. Appl. Crystallogr., 42, 535 (2009).
2) F. Kimura, K. Mizutani, B. Mikami and T. Kimura: Cryst. Growth. Des., 11, 12 (2011).