Poster Presentation

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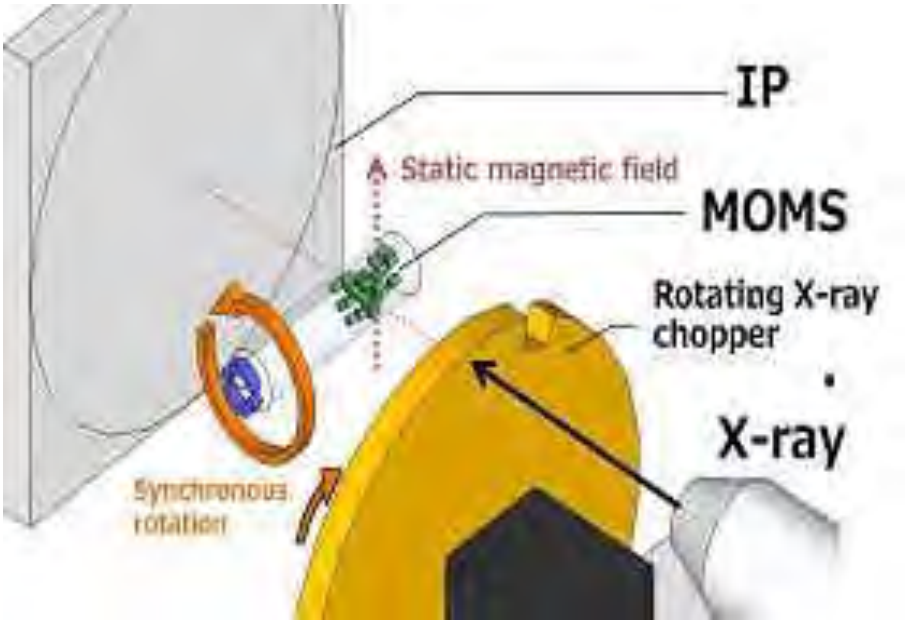
X-Ray Single Crystal Structural Analysis of Magnetically Oriented Microcrystals

C. Tsuboi[1], K. Aburaya[2], S. Higuchi[1], F. Kimura[1], M. Maeyama[2], T. Kimura[1]

[1]*Kyoto University, Graduate Schoole of Agriculture, Kyoto, Japan,* [2]*Rigaku Corporation, Tokyo, Japan*

We have developed magnetically oriented microcrystal array (MOMA) technique that enables single crystal X-ray diffraction analyses from microcrystalline powder. In this method, microcrystals suspended in a UV-curable monomer matrix are there-dimensionally aligned by special rotating magnetic field, followed by consolidation of the matrix by 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 problems as follows: 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 three-dimensional magnetically oriented microcrystal suspension (3D MOMS) of L-alanine. An experimental setting of the in-situ X-ray measurement of MOMS is schematically shown in the figure. 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 this chopper, it was possible to expose the X-ray only when the rotating MOMS makes a specific direction with respect to the impinging X-ray. This has the same effect as the omega oscillation in conventional single crystal measurement. A total of 22 XRD images of 10º increments from 0º to 220º were obtained. The data set was processed by using conventional software to obtain three-dimensional molecular structure of L-alanine. The structure is in good agreement with that reported for the single crystal. R1 and wR2 were 6.53 and 17.4 %, respectively. RMSD value between the determined molecular structure and the reported one was 0.0045 Å. From this result, we conclude that this method can be effective and practical to be used widely for crystal structure analyses.

[1] *T. Kimura, C. Chang, F. Kimura et al, J. Appl. Crystallogra, 2009, 42, 535,* ***[2]*** *F. Kimura, K. Mizutani, B. Mikami et al, Cryst. Growth. Des, 2011, 11, 12*



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