MLF Experimental Report	提出日 Date of Report 21 July, 2017
課題番号 Project No. 2017A0235 実験課題名 Title of experiment Mechanical milling dependences of short- and medium-range structure in AgI-doped As_2Se_3 system 実験責任者名 Name of principal investigator Takeshi Usuki 所属 Affiliation Yamagata University	装置責任者 Name of responsible person Toshiya Otomo 装置名 Name of Instrument/（BL No.） BL-21 (NOVA) 実施日 Date of Experiment 21 - 22 May, 2017 (27hr)

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.

$(AgI)_x(As_2Se_3)_{1-x}$ (x = 0.4, 0.6) with different milling periods (2, 10, 40h)

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.

Time of flight measurements were made at room temperature using a standard vanadium container for $(AgI)_x(As_2Se_3)_{1-x}$ (x = 0.4, 0.6) samples (prepared by a mechanical milling method). Figure 1 shows structure factors $S(Q)$ for 60 mol% AgI-doped samples with different milling periods (2, 10, 40h). Although Bragg peaks of starting crystalline materials remain in the early stage of the milling, diffraction patterns at high Q region change little. On the other hand, it can be seen that there are large differences at low Q region especially at Q < 3 $Å^{-1}$. A Bragg peak located at 1.75 $Å^{-1}$ decreases with the milling, whereas a new peak seems to be appeared at around 1.25 $Å^{-1}$, which is considered to be an indication of intermediate-range ordering (IRO) constructed by $As(Se_{1/2})_3$ pyramidal units.

Figure 2 gives pair distribution functions $g(r)$ for the present samples with different milling periods. A first peak located at 2.4 Å becomes larger with increasing milling time, indicating the formation of As-Se covalent bonds. A hump at around 2.8 Å is related to the Ag-I bonds. It is of interest that curves of g(r) at the second

shell region between 4 and 5 Å largely change. One of dominant contributions of these region is I-I correlation of AgI. The result therefore indicates that the deformation of I-I sub-lattice is seriously enhanced with increasing milling time. According to a previous report, the present system shows a large increase in their ionic conductivity at the early stage of the milling [1]. Then, it can be predicted that disorderness around Ag ions would be strongly related to the enhancement of the ionic conductivity in the present system. The three-dimensional structure modelling using the reverse Monte Carlo method is now progressing for discussing deeply the ionic diffusion mechanism in the present system.

[1] M. Sekine, Y. Suzuki, H. Ueno, Y. Onodera, T. Usuki, T. Nasu, S. Wei, J. Non-Crystalline Solids, 353 (2007) 2069-2073.

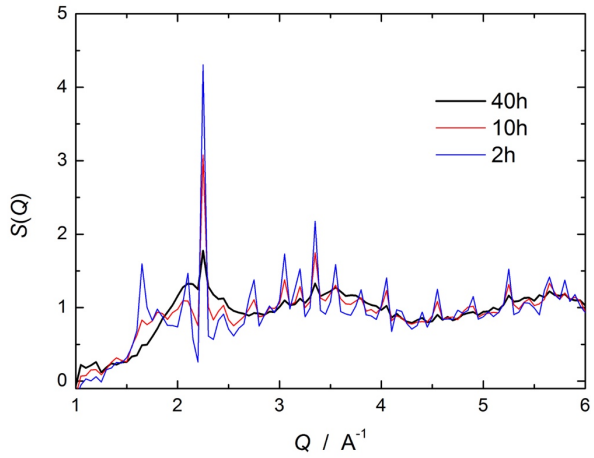

Figure 1 Total structure factors for AgI-doped glasses with different milling periods.

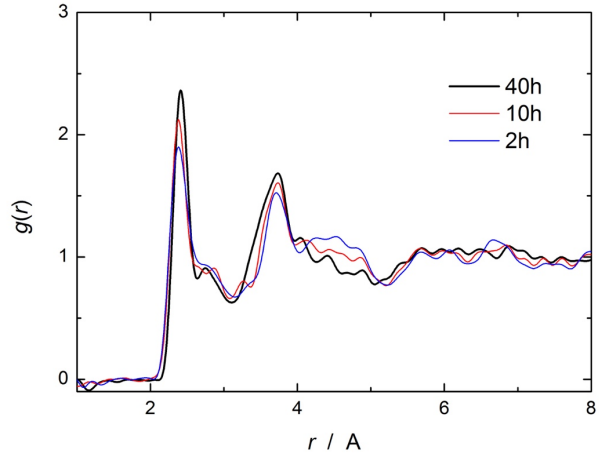

Figure 2 Total pair distribution functions for parent and AgI-doped glasses with different milling periods.