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Pressure-Induced Changes in Crystal Structures of Highly Crystalline Polyimide Particles and Monomer Salt Single Crystals

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Introduction

Pressure-induced variations in the crystal structures of polyimides (PIs) have been investigated to clarify the relationship between the molecular aggregation structures and the physical properties of PIs.[1] However, it is difficult to determine the atomic arrangement in the crystal lattice due to their lower crystallinity, which leads to insufficient number of diffraction spots. In this study, pressure-induced variations in the crystal structure of PMDA/PPD monomer salts single crystal (MC) and PMDA/PPD poly-crystalline particles (PP) were investigated by synchrotron wide-angle X-ray diffraction (SR-WAXD) measurements.

Experiment

Highly crystalline PMDA/PPD-PP were prepared by solid-state polymerization PMDA/PPD-MC (**Fig. 1**).[2] To generate pressure, the PI samples were loaded into a diamond anvil cell (DAC, Syntech Co. Ltd.). Variable-pressure SR-WAXD measurements were conducted at the BL-10C at the Photon Factory (KEK, Tsukuba, Japan).

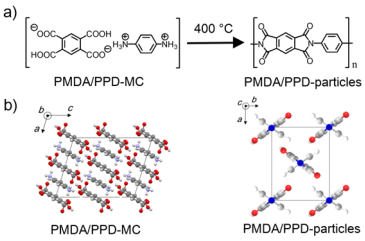

Fig. 1 a) Chemical and b) crystal structures of PMDA/PPD -MC and -PP.

Results and Discussion

The pressure-induced strains of the crystal lattice in PMDA/PPD-PP and -MC are shown in **Fig. 2**. These samples showed significant compression by ca. 7.0 % along cofacial stacking direction (*a*-axis) upon applying pressure up to 3 GPa. In addition, the *c*-axis of PMDA/PPD-MC, which is close to the direction of strong ionic bonds in the MC and PI chains generated after solid phase polycondensation, exhibited smaller compression compared to the other crystal axes as with the polymer chain axis (*c*-axis) of PMDA/PPD-PP. Furthermore, PMDA/PPD-PP having relatively high density (d =1.76 g/cm^3) showed a volume compression by ca.16 % as with PMDA/PPD-MC (d =1.58 g/cm^3). In summary, it was clarified that the compression behavior of PMDA/PPD-MC is similar to that of PMDA/PPD-PP in spite of the remarkable difference in density.

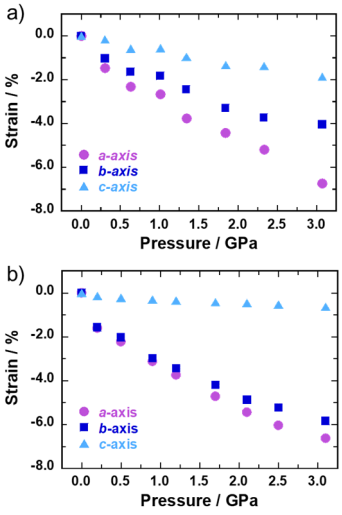

Fig. 2 Pressure-induced variations in the strain (ε) of a) PMDA/PPD-MC and b) -PP.

References

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