X-ray Diffraction Laboratory

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About Lab.

X-ray diffraction laboratory is engaged in structural analysis, identification of the powder, and single crystal characterization for solid state physics. We support the ***Weissenberg type imaging plate, CCD area detector, 4-circle diffractometer, Laue camera, powder diffraction, and imaging plate scanner***. Some facilities are daily used for researchers.

Main facilities

We use Weissenberg type imaging plate equipped with the helium closed-cycle refrigerator for the exposure of rather low-noise oscillation photograph and single-crystal structure analysis at low temperature to investigate the origin of the phase transition on the interesting materials (Fig. 1). The x-ray beam is 21 kW high power. And, the high-pressure and low-temperature single crystal structure analysis can be also performed with the easily centering system of the diamond anvil cell.

CCD area detector is useful for the single-crystal, structure analysis with the very small sample, the highly anisotropic absorption sample, the twin crystal and the air unstable sample in the temperature range of 90-300K (Fig. 2). This system has the user friendly interface for many reasearcher.

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X-ray Structure Analysis

Low Temperature

High Pressure

Organic conductor

Fig.1 Weissenberg type imaging plate equipped with the helium closed-cycle refrigerator. High-pressure and low-temperature single crystal structure analysis can be performed.

Fig.2 CCD area detector for single-crystal structure analysis with high sensitivity and high accuracy in the temperature range of 90-300K.

Activities

Many organic and inorganic compounds have been studied for the origin of the interesting phase transition and structuralchange at low temperature and under high pressure mainly using Weissenberg type imaing plate and CCD area detector.

β'-$Pd(dmit)_2$ anion radical salt is characterized by a solid-crossing columns composed by strongly dimerized $Pd(dmit)_2$ units. This is a unique two-band system associated with two-dimensional HOMO-based band and one-dimensional LUMO-based band. Several compounds show metallic and superconducting states under pressure which are followed by a non-metallic state under higher pressure region. X-ray single crystal structure analysis was carried out for the high-pressure superconductor β'-$Et_2Me_2P[Pd(dmit)_2]_2$ (Tc = 4 K at 7 kbar) under pressure at room temperature and low temperature to clarify the nature of the electronic states. The diffraction data under pressure were collected using an imaging plate type Weissenberg camera equipped with a diamond anvil cell. We calculated the band parameters based on the extended Hückel method from the refined crystal structure under pressure, indicating an increase of bandwidth and a decrease of the dimensionality of a HOMO-based band. Figure 3 shows the calculated Fermi surface of the HOMO band, which means the decrease of dimensionality under pressure. The above findings are related to the pressure-induced metallic state and the high-pressure non-metallic state.

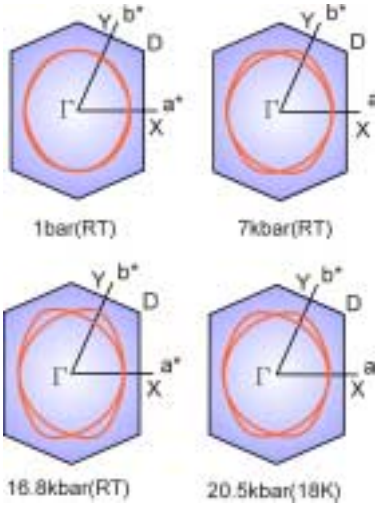

Fig. 3 Fermi surface of the HOMO band under pressure at room and low temperatures for $Et_2Me_2P[Pd(dmit)_2]_2$. They are multiple ones due to solid-crossing columns.

R1,R2-$DCNQI_2Cu$ salts have a 1D column structure of DCNQI molecules, where the column networks are bridged by Cu ions. They exhibit various unique physical properties due to the hybridization between 1D organic DCNQI p π -band and Cu dxy-orbitals near the Fermi level. Among them, we are interested in the R1=R2=I (DI) and R1=R2=Br (DBr) salts having the complicated pressure-temperature phase diagram, that is, the ground state changes successively under pressure as metal(MI) → insulator(II) → metal(MII) → insulator(III) → metal(MIII). To identify the origin of the complicated phase diagram, we measured the x-ray diffraction of the powder samples under pressure for DI and DBr salts. We found the different pressure dependence in the lattice parameter (Fig. 4), estimated by the Rietvelt method, for each salt. This suggests that the electronic state under pressure is different between them although they have similar conductive properties under pressure.

Vanadium oxide bronzes β(β')-$Na_{0.33}V_2O_5$ having one-dimensional chain and ladder along the b-axis was studied by x-ray diffraction measurements. We observed the **q**=1/2**b*** and **q**=1/6**b*** satellite reflections at low temperatures. From the superstructure analyses, those origins are the cation ordering and the charge ordering on the vanadium ladder, respectively. Moreover, the high-pressure x-ray diffraction indicates the rapidly disappearing the **q**=1/6**b*** satellite with applying pressure, which is relevant to the high-pressure superconducting state appearing.

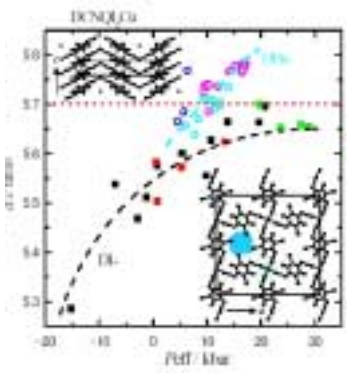

Fig.4 Pressure dependence of the lattice parameter ratio of a/c for DBr and DI salts with the crystal structure of R1,R2-$DCNQI_2Cu$. The pressure value is normalized by the critical pressure for each salt.

Representative Publications

'High Pressure X-ray Study of $Pd(dmit)_2$ System', J. Yamaura and R. Kato, *Mol. Cryst. Liq. Cryst.*, **379** (2002) 47.
'Low Temperature X-ray Study of $A_{0.33}V_2O_5$', J. Yamaura, M. Isobe, H. Yamada, T. Yamauchi and Y. Ueda, *J. Phys. Chem. Solids,* **63** (2002) 957.