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Effect of Anisotropic Lattice Vibration in CBED Intensities and Detection of Local Change in Oxygen Deficiency of $YBa_2Cu_3O_{7-x}$

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Improvement of the critical current density, *Jc* is one of the important subjects for practical application of high *Tc* superconductor $YBa_2Cu_3O_{7-x}$ (YBCO). In particular the peak-effect in *Jc-H* curve has been of interest in relation to pinning centers under higher magnetic fields. The remarkable peak-effect does not appear in a detwinned YBCO but in a twinned one [1]. The peak-effect sensitively depends on heat-treatments. Oxygen concentration of YBCO easily changes depending on atmosphere and local change in oxygen deficiency may occur. Thus it is very important to measure a local change in oxygen concentration to understand a mechanism of the peak-effect and to improve the *Jc* characteristic of YBCO. Convergent beam electron diffraction has been used to extract information on charge distribution and/or oxygen concentration [2-4]. The accuracy of the measured oxygen concentration depends on the reliability of temperature factors (B-factors) used in the analysis. In the present paper, the effect of anisotropic temperature factor is examined and the actual temperature of a specimen under electron beam irradiation is estimated. Then local changes in oxygen concentration are measured by analyzing large angle CBED patterns observed with a FE-TEM: JEM-2010FEF equipped with an energy filter of omega-type(Fig.1). In the calculation based on the dynamical theory we used the positional parameters of all ions reported by Cava et al. [5] and isotropic B-factors by Izumi et al. [6] or anisotropic B-factors by Beno et al.[7]. Changes in charge distribution on CuO_2 and CuO planes are considered in the calculation.

Figure 2 shows diffraction intensities calculated as a function of 7-x in YBCO. Indices of 00l series are preferable for the analysis of oxygen deficiency whereas h00 or hh0 is useful for estimation of specimen temperature since intensities are less sensitive to the change in 7-x as seen in Fig.2. Intensities calculated with isotropic and anisotropic B-factors are compared in Fig.3 where intensity ratio I_{300K}/I_{100K} is plotted. The effect of the anisotropy differently appears depending on an index of diffraction. Values of B-factors at 100 K are calculated from those at room temperature using a Debye model of thermal vibration. A constant value of B-factor for each ion is assigned regardless of its occupation site in the isotropic model. An anisotropic B-factor is more realistic because YBCO is a layered structure associated with oxygen defects. In particular an amplitude of thermal vibration of oxygen may vary depending on its occupation site. Furthermore fitting was unsatisfactory when isotropic B-factors were used [4]. Consequently we adopt anisotropic B-factors hereafter.
In the present experiment a specimen is cooled with liquid nitrogen to reduce the effect of thermal diffuse scattering. A temperature of the cooled specimen was estimated to be 90 ± 15 K by comparing intensities of [001] diffraction patterns as shown in Fig.4 where an effect of diffraction on the first Laue zone was taken into account. Oxygen concentration was determined by analyzing intensities of a 00l systematic diffraction pattern obtained with a probe size of 3~4 nm. One of examples is shown in Fig.5. Figure 6 demonstrates a local change in oxygen concentration in YBCO sample prepared with the QMG method. No super lattice spots were observed in [001] diffraction patterns indicating that oxygen deficiency occurs with random depopulation of oxygen along a Cu-O chain. Fluctuation of oxygen deficiency is the most probable reason for the peak-effect in YBCO.

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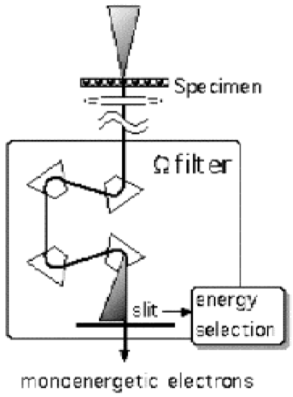

Fig.1 Shematic drawing of energy filtering CBED.

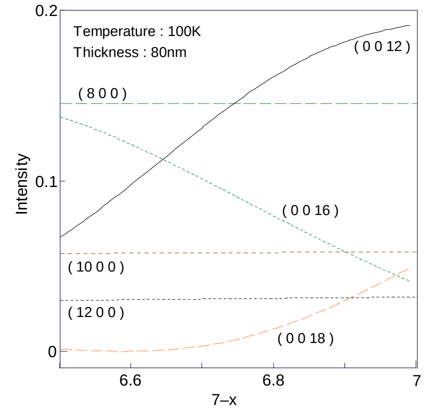

Fig.2 Intensity of diffraction calculated as a function of oxygen concentration in YBCO.

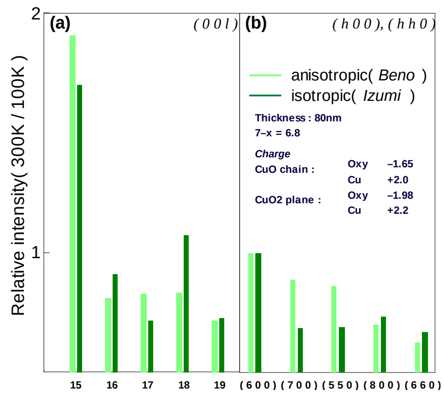

Fig.3 Comparison of intensity ratio I_{300K}/I_{100K} between isotropic and anisotropic B-factors.

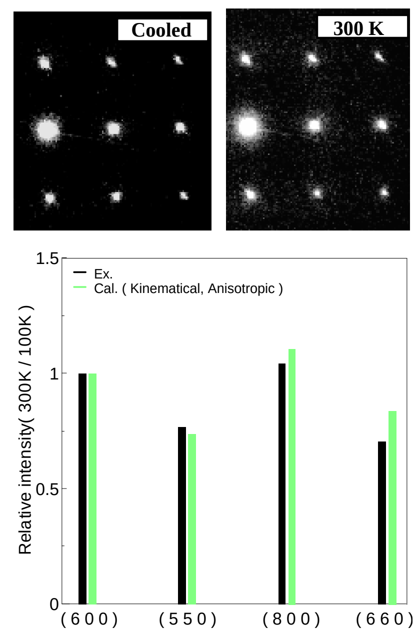

Fig.4 Observed [001] diffraction patterns and fitting of intensities.

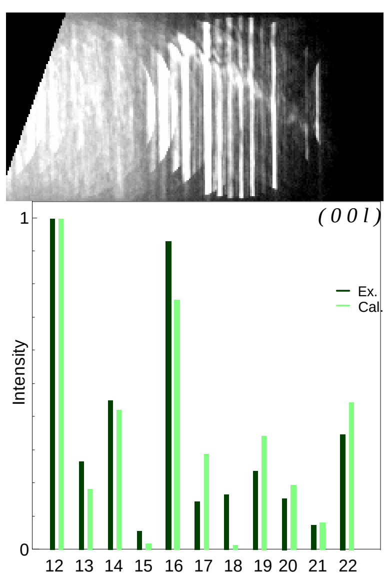

Fig.5 Observed large angel CBED pattern and fitting of intensities; determination of oxygen concentration.

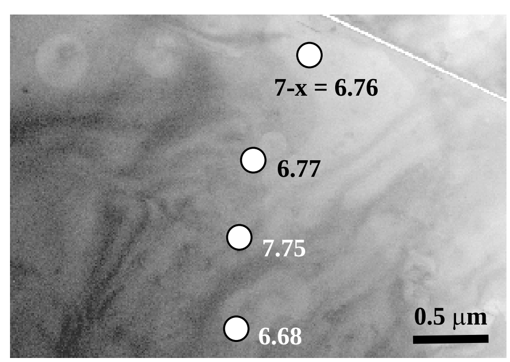

Fig.6 Bright-field image showing local changes in oxygen concentration in YBCO.

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