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Electron Crystallographic Study of Incommensurate Modulated Structures

Fan Hai-fu, Li Yang, Wan Zheng-hua, Fu Zheng-qing, Mo You-de, Cheng Ting-zhu & Li Fang-hua

Institute of Physics, Chinese Academy of Sciences, Beijing 100080, China

As a tool of structure analysis, electron crystallography has well-known advantages in comparison with X-ray techniques. For example, it enables the combination of microscopic imaging and diffraction analysis making the phase problem much easier to solve. Apart from this and some others, electron crystallography has additional advantages in studying incommensurate modulated structures. First, crystals having incommensurate modulation are often such imperfect that they are too poor and too small to carry out an X-ray single-crystal analysis. However those crystals are suitable for electron microscopic observation. Secondly, diffraction patterns of incommensurate modulated structures consist of main reflections and satellites. The latter is the main carrier of modulation information. Electron diffraction shows much stronger satellites enabling observation of weaker structural modulation in smaller area. A big problem of electron crystallography is the strong dynamical-diffraction effect. However, this effect is considerably weakened by the imperfection of periodicity due to incommensurate structural modulation.

Strictly speaking, incommensurate modulated structures do not have 3-dimensional periodicity. However, they can be considered as a 4- or higher-dimensional periodic object cut with the 3-dimensional physical space. Direct methods have been extended to use in multi-dimensional space enabling *ab-initio* solution of incommensurate modulated structures [1]. The main points are as follows. (i) The basic/average structure is solved by conventional direct methods in 3-dimensional space using only the main reflections. (ii) Phases of satellite reflections are derived by multi-dimensional direct methods based on the known phases of main reflections. (iii) A multi-dimensional Fourier map is calculated with experimental structure-factor magnitudes and direct-method phases derived from the previous steps. The actual incommensurate modulated structure will then revealed objectively on the 3-dimensional hyper-section of the multi-dimensional Fourier map. (iv) The modulated structure model is constructed according to the multi-dimensional Fourier map without any preliminary assumptions on the property of modulation.

The above method has been applied to study incommensurate modulation in bismuth based high Tc superconductors [2 - 4]. Results are shown in Figures 1 - 3. Influence of the dynamical effect of electron diffraction to the results has been extensively examined by a series of simulating calculation. Some of the results are given in Figure 4. It turns out that the method may be applied to samples as thick as 300Å. An MS Windows program *VEC* (Visual computing in Electron Crystallography) [5] has been written and includes the direct method for solving incommensurate modulated structures. The program is freely available for academy use and has the following features: (i) searching defocus value from a single electron microscopy image; (ii) resolution enhancement of electron microscopy images using direct methods; (iii) simulation of dynamical/kinematical electron diffraction patterns and electron microscopy images for conventional and incommensurate modulated crystals; (iv) 2-, 3- and 4-dimensional FFT; (v) 2-dimensional half-tone-graph display of 2-, 3- and 4-dimensional Fourier maps; (vi) direct-method solution of incommensurate one-dimensionally modulated structures and composite structures of two subsystems.

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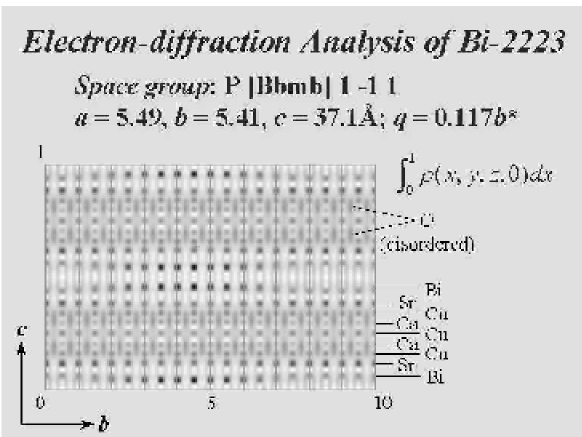

Figure 1. Potential distribution of the high Tc superconductor Bi-2223 projected down the ***a*** axis

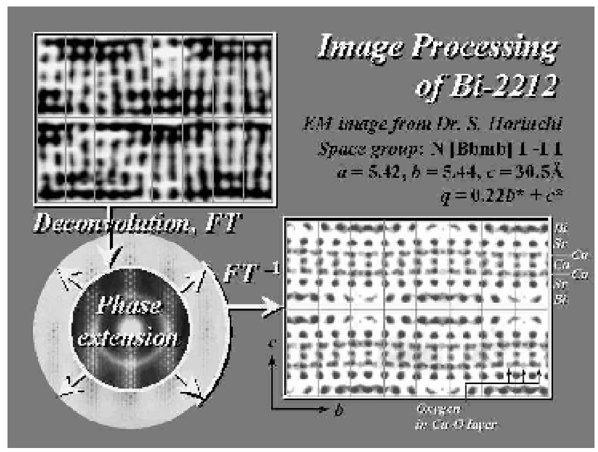

Figure 2. Electron micrograph (upper left), electron diffraction pattern (lower left) and potential distribution (right) of Bi-2212

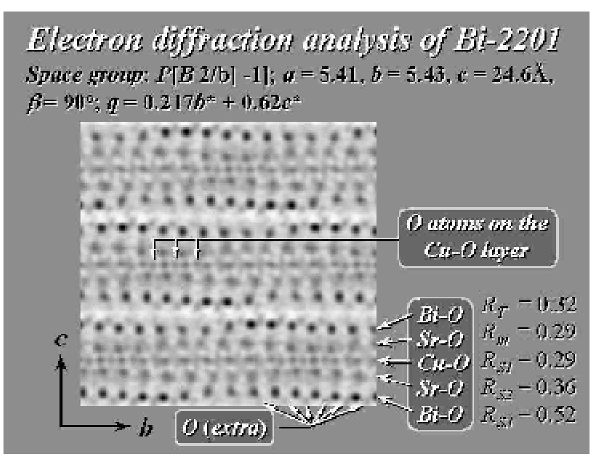

Figure 3. Potential distribution of Bi-2201 projected down the ***a*** axis

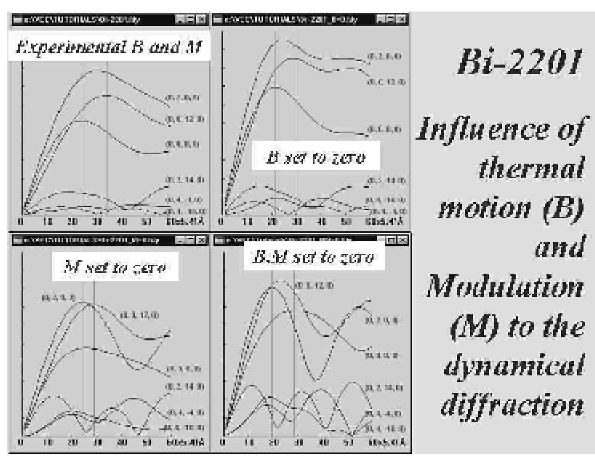

Figure 4. Curves of dynamical electron-diffraction intensity versus sample thickness

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