A method of studying dislocation core structures by high-resolution electron microscopy

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Abstract

A method to study the crystal defects at atomic level by high-resolution electron microscopy (HREM) is introduced. The image taken with a field-emission high-resolution electron microscope and not directly reflecting the examined crystal structure can be transformed into the structure image by means of image deconvolution in combination with dynamical scattering effect correction. The principle of image deconvolution and the procedure of technique are briefly introduced. The results of applications on the epilayer of Si$_{0.76}$Ge$_{0.24}$/Si are given. It is shown that atoms in the dislocation core structures have been distinguished individually in the deconvoluted images and the point resolution of images can be improved up to the information limit of the field-emission high-resolution electron microscope.

Keywords: High-resolution electron microscopy; Crystal defect; Image deconvolution

1. Introduction

The development of high-resolution electron microscopy (HREM) has afforded a powerful technique to directly observe the projected structures of crystals [1,2], though the conditions for directly observing the crystal structures in images are sever. Fig. 1 gives the schematic diagram of image formation process in HREM. Passing through an object the electron wave is modulated to form the exit wave $q(r)$ on the bottom surface of the object. The exit wave carries the information of object structure and becomes the object wave for the objective lens. The diffracted and image waves are formed on the back focal plane and image plane of objective lens, respectively. The diffracted wave function $Q(H)$ is the Fourier transform (FT) of object wave and the image wave $\psi(r)$ is the inverse FT of diffracted wave modulated by the transfer function $T(H)$ of objective lens. The transfer function depends on various electron-optical parameters acting in the process of image formation. Only images taken near the Scherzer focus [3] can directly reflect the projected object structure when the sample is sufficiently thin, while an arbitrary image may not directly reflect the structure. In addition, owing to the insufficient microscope resolution atoms may not be resolved individually even in images taken at the Scherzer focus except those taken with a high-voltage electron microscope.

It has attracted attention to solve the inverse problem in HREM, i.e. to go back from the image to the object structure with a resolution higher than the point resolution of electron microscope by posterior image processing. For instance, to obtained the exit wave on the bottom surface of the object from a series of images taken under different defocus condition [4–12], or to derive the structure directly from the image [13–16]. In the last two decades two kinds of electron crystallographic image processing techniques have been developed and applied to the derivation of crystal structures and crystal defects, respectively, in the Institute of Physics, Chinese Academy of Sciences [17]. In the present paper, the technique for crystal defect study at atomic level is reviewed.

2. Image deconvolution in combination with dynamical scattering effect correction

The present technique is developed in the light of the high coherence of field-emission (FE) high-resolution...
electron microscopes. Though the point resolution of FE microscopes is the same as that of non-FE microscopes (Fig. 2), the information limit of FE microscopes is much higher than the point resolution. However, images including those taken close to the Scherzer focus condition can hardly show the crystal structure faithfully owing to the strong oscillation of CTF in the high spatial frequency region. The purpose of image deconvolution is to deducting the distortion of image contrast due to the modulation by CTF and to enhance the image resolution up to the information limit of the microscope. Besides, to improve the quality of deconvoluted image, a method was proposed to reduce the dynamical scattering effect.

2.1. Principle of image deconvolution

It is well known that the weak-phase-object approximation (WPOA) clearly and simply interprets the one-to-one correspondence between the Scherzer focus image and the projected crystal structure. Under the WPOA the image intensity is expressed as

\[ I(r) = 1 + 2\sigma \phi(r) * \mathcal{F}^{-1}[T(H)], \]  

where \( \sigma = \pi \lambda U \), \( \lambda \) denotes the electron wavelength and \( U \) the accelerating voltage, \( \phi(r) \) and \( T(H) \) represent the projected potential distribution function which represents the projected structure and CTF, respectively, \( * \) and \( \mathcal{F}^{-1} \) are operators of convolution and inverse FT, respectively. The CTF depends on various electron-optical parameters, such as spherical aberration coefficient of objective lens, defocus amount, defocus spread due to the chromatic aberration and so on. At the Scherzer focus condition function \( T(H) \) is approximate to a constant, and hence the image is almost linear to \( \phi(r) \).

Since, most of crystals observed in electron microscopes are thicker than the weak-phase objects, an alternate image contrast theory named the pseudo-weak-phase-object approximation (PWPOA) was derived by Li and Tang [18] in 1985 to meet the applicable crystal thickness range. Fortunately, a formula very similar to Eq. (1) was obtained for the image intensity in the PWPOA as follows:

\[ I(r) = 1 + 2\sigma \phi'(r) * \mathcal{F}^{-1}[T(H)]. \]  

where \( \phi'(r) \) represents the potential of an artificial crystal which has the same structure as the examined real structure but different constituent atoms such that the heavy atoms in the artificial crystal is lighter than those in the real crystal and vice versa. Hereafter, \( \phi'(r) \) is named the pseudo projected potential distribution function or in short pseudo potential. The FT of Eq. (2) yields the diffractogram

\[ i(H) = \delta(H) + 2\sigma F'(H)T(H), \]  

where \( F'(H) \) is the FT of \( \phi'(r) \) and named pseudo structure factor. Eq. (3) is rewritten into

\[ F'(H) = \frac{i(H)}{2\sigma T(H)} \]  

with the transmitted beam omitted. Eq. (4) indicates that it is easy to deduct the modulation due to the CTF in the reciprocal space to obtain the pseudo structure factor \( F'(H) \), once \( T(H) \) is known. Then the image deconvolution is fulfilled by inversely Fourier transforming \( F'(H) \) to yield the pseudo potential \( \phi'(r) \).

Eq. (2) is available when the thickness of observed crystal is below a critical value, which depends on the electron wavelength and weights of constituent atoms in crystals. The critical crystal thickness is generally less than 10 nm for 200 and 300 kV microscopes, but may equal or larger than 10 nm for light atoms.

2.2. Procedure of technique

The technique mainly contains three parts, the defocus determination, image deconvolution and dynamical scattering effect correction. Fig. 3 gives the flow chart of the procedure.

All parameters included in \( T(H) \) are usually known except the defocus amount \( \Delta f \). In the case of structure determination for perfect crystals a method was proposed to determine the defocus amount in the process of image deconvolution [19]. In the present case a rough defocus amount is firstly determined by means of the Thon diffractogram [20] obtained from an amorphous region nearby the examined crystalline area. Then the defocus refinement is performed in the process of dynamical
scattering effect correction [21]. For this several trial defocus values close to the roughly determined one with a small focus step, for instance 0.5 nm, are assigned. Then for each trial defocus value the modulation due to the CTF is deducted from the diffractogram of the image containing the examined defect. An artificial unit cell corresponding to the image is constructed, and a set of $F_{\text{trial}}(H)$ is calculated by means of Eq. (4). Accordingly, several $F_{\text{trial}}(H)$ sets are obtained. To reduce the dynamical scattering effect, all $F_{\text{trial}}(H)$ sets are corrected by forcing the integrated amplitudes of reflections to be equal to the amplitudes of corresponding structure factors for perfect crystal $F(H)$ [21]. The corrected $F_{\text{trial}}^\text{corr}(H)$ for the $i$-th pixel is

$$|F_{\text{trial}}(H)|_i^\text{corr} = K_H |F_{\text{trial}}(H)|_i$$  \hfill (5)

and

$$K_H = \frac{|F(H)|}{\sum |F_{\text{trial}}(H)|_i},$$  \hfill (6)

where $F(H)$ is the structure factor for the perfect crystal, $K_H$ is the correction coefficient that is constant for all pixels of the same reflection and different for different reflections. Thus, inversely Fourier transforming all corrected $F_{\text{trial}}(H)$ sets yields the trial potential maps of the defected crystals. Finally, the best map is selected as the correct deconvoluted image from among all trial maps. In this map, atoms in both the perfect and defect regions should be resolved the best.

3. Recognizing dislocation types and twin boundary in Si

The image processing technique was tested with simulated images for two structure models of Si crystal containing 60° dislocations of Shuffle and glide types, respectively [22]. Fig. 4(a) shows the [110] projected structure with the dislocation of Shuffle type and Fig. 4(b) the [110] projected structure with the dislocation of glide type. In both cases a half (111) plane is inserted as pointed by arrows. The distance between two adjacent atoms is 0.136 nm. The difference between the two types of 60° dislocations is in the atomic configuration at the end of inserted half (111) plane. For the 60° dislocation of Shuffle type there is a pair of atoms at the end, while for that of glide type there is a single atom as shown inside the circles in Fig. 4(a) and (b). The image simulation for the two models of Si indicates that the image contrast difference is too small to distinguish the two types of 60° dislocations (see the contrast inside circles in Fig. 4 (c) and (d)). After the image deconvolution the two types of dislocations are seen clearly in the deconvoluted images as shown in Fig. 4(e) and (f) [22].

The technique was also tested with the simulated images of Si containing the twin boundary, and the validity exists up to the crystal thickness about 11.5 nm [23].

4. Atomic configuration restored from [110] images of epilayer $\text{Si}_{0.76}\text{Ge}_{0.24}/\text{Si}$

4.1. Lomer dislocation [24]

The crystal structure of $\text{Si}_{0.76}\text{Ge}_{0.24}$ is isomorphic to that of Si. In the projected structure of $\text{Si}_{0.76}\text{Ge}_{0.24}$ the distance between two adjacent atoms is about 0.14 nm, which is close to the information resolution limit of 200 kV FEG high-resolution electron microscope. Several images are taken under different defocus conditions.
The FT is performed for all images to obtain the diffractograms. To reveal the atoms individually after the image processing, the structure information with the spatial frequency up to \((0.14 \text{ nm})^{-1}\) should be contained in the original image. This implies that the reflection 004 should be seen with an obvious contrast in the diffractogram of the image. In addition, since for perfect crystals there are four independent reflections with the indexes 111, 220, 113 and 004 up to spatial frequency \((0.14 \text{ nm})^{-1}\), to avoid a big part of information loss, all the four independent reflections should appear in the diffractogram. The image of the best quality is selected from among all images.

Fig. 5(a) is the [110] projected high-resolution image taken with a JEM-2010F electron microscope with the accelerating voltage 200 kV and spherical aberration coefficient 0.5 mm. A dislocation is seen in the framed area, of which the magnified picture is shown in Fig. 5(b). Although it can be recognized that the dislocation is of the Lomer type, the atomic configuration does not reveal directly from the image. Several models have been proposed for the Lomer dislocation. Bourret, Desseaux and Renault proposed two symmetric and two asymmetric core models in Si and Ge. McGibbon, Pennycook and Angelo claimed to have observed the Hornstra-like and an unexpected core structure in polar compounds CdTe/GaAs [26]. In the case of SiGe/Si, no sufficient evidence has been found yet to support any model mentioned above or a new one. Fig. 5(c) is the diffractogram obtained from a circular area of diameter about 17.57 nm, with the dislocation at the center. All reflections with the spatial frequencies for resolving the two adjacent atoms appear with rather dominant intensity. This implies that no reflection falls in the vicinity of zero cross of CTF. The FT of image area including both the amorphous and crystalline regions near the dislocation yields the picture showing the superposition of Thon diffractogram and diffractogram of crystal image. The defocus value is roughly determined to be \(-39 \text{ nm}\) by matching the intensity profile of Thon diffractogram with several CTF curves. The accelerating voltage is 200 kV, spherical aberration coefficient 0.5 mm and defocus spread due to the chromatic aberration 3.8 nm for all curves, but the focus values are different from one another.

Seven trial \(F'({\mathbf{H}})\) sets were obtained from the corresponding trial defocus values from \(-40\) to \(-37.5 \text{ nm}\) and the defocus step 0.5 nm, and then the corresponding corrected trial \(F'({\mathbf{H}})\) sets were calculated. The inverse FT of the corrected trial \(F'({\mathbf{H}})\) sets yields seven potential maps. The best map (Fig. 5(d)), in which all atoms are resolved the most clearly was selected from among the seven maps. The atomic arrangement in the dislocation core is seen more clearly by linking the adjacent atoms to show the bonding situation. It can be seen that atoms forming the core structure of Lomer dislocation are resolved individually. Each black dot represents the position of an atomic column projected in the [110] direction. A five-membered ring and a seven-membered ring form the symmetric undisassociated core without dangling bond. This is in agreement with the Hornstra model. The present result confirms that the Lomer dislocation with Hornstra structure can be formed in the region of epilayers close to the interface boundary.

4.2. 60° dislocation complex [27]

Fig. 6(a) shows another [110] image of Si_{0.76}Ge_{0.24} with a dislocation in the framed area. The deconvoluted image corresponding to the framed area in Fig. 6(a) is
shown in Fig. 6(b). The magnified picture given in Fig. 6(c) corresponds to the framed rectangular area in Fig. 6(b), where the detailed geometry of the dislocation complex can be seen clearly by linking the adjacent atoms to show the bond situation. It is seen that the dislocation complex is composed of a perfect 60° dislocation existing in the left part and an extended 60° dislocation in the right part. The seven-membered rings and five-membered rings are seen in both core regions of perfect and expanded 60° dislocations. The extended 60° dislocation dissociates into a 90° partial dislocation and a 30° partial dislocation with a short stacking fault (only three lattice spacing) between them.

5. Conclusion

An image processing technique has been developed to reveal crystal defects at atomic level by HREM. It consists of image deconvolution based on the PWPOA and dynamical scattering effect correction. The initial image is selected from among several images of different defocus values such that all necessary reflections should appear in the diffractionogram. The modulation due to the CTF is deducted so that the final image directly reflect the structure and its resolution is improved up to the information limit of the microscope. The image processing for simulated image of Si indicates that it is possible to distinguish the glide type of 60° dislocation from that of Schuffle type, though the difference is in a single atom. The applications of the technique on SiGe epilayer yield the dislocation core structure maps of a Lomer dislocation and a complex dislocation. The latter consists of a perfect 60° dislocation and an extended 60° dislocation which dissociates into a 30 and a 90° partial dislocations. All atoms are resolved individually in the structure maps.

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