Convergent beam electron diffraction의 정량분석을 응용한 재료의 구조분석

Applications of quantitative convergent beam electron diffraction measurement for structural characterization

김규현[a*], 이민희, 정새은, 고세현
[a]한국생산기술연구원 융합공정신소재연구실용화그룹(E-mail:khkim1308@kitech.re.kr)

Abstract: The new algorithm was proposed to quantify symmetry recorded in convergent beam electron diffraction (CBED) patterns and symmetry mapping. The proposed algorithm is based on the normalized cross-correlation coefficient (γ) for quantifying the amount of symmetry in a CBED pattern. The quantification and mapping procedures are automatically controlled by the script implemented in Gatan Digital Micrograph©. We apply the quantitative CBED measurement to a strained Si sample to test the sensitivity to defects.

1. Introduction

Crystal symmetry often breaks down in real materials due to surface and interfacial stress and strain and the presence of defects. Thus, measurement of local symmetry can provide useful structural information that is difficult to obtain otherwise. While diffraction techniques, including convergent beam electron diffraction (CBED), are routinely used to determine the average crystal symmetry, determination of local crystal symmetry has received less attention. CBED using small electron probes is a powerful technique to extract symmetry information on the nanometer scale. The symmetry recorded in the CBED patterns is in general determined by direct visual inspection, which does not provide an objective, uniform, measurement. Furthermore, experimental CBED patterns are often noisy and deviate from the ideal symmetry because of the sample geometry and defects. The imperfection in experimental CBED patterns can lead to uncertainty in the symmetry determination. To overcome this limitation, we propose a symmetry quantification method for CBED patterns using the normalized cross-correlation coefficient (γ) [1]. The proposed technique was applied to a strained Si to show the sensitivity to defects in the specimen.

2. Experimental results

Figure 1 shows an experimental recorded CBED pattern from a unstrained Si at the zone axis of [110]. First, two diffraction disks are selected about the mirror plane (yellow line) as shown in Fig. 1(a). For the calculation, the selected CBED disks are named as template A and template A' (Figs. 1(b) and (f)), respectively. Each template is then rotated by an angle θ so that the mirror is aligned as shown in Figs. 1(c) and (g). The template A is used as the reference motif so that the symmetry element is calculated by comparing with template A'. For the mirror operation, the template A' is flipped horizontally to obtain a mirror image as shown in Fig. 1(h). The mirror-applied image will be referred to as A'_m. The circular mask shown in Figs. 1(d) and (i) is used to remove areas affected by CBED disk edge. Thus, the final templates are obtained by multiplying the mask image to the templates A and A'_m as shown in Figs. 1(e) and (j). The γ is then calculated from the final templates of A and A'_m [2]. The proposed symmetry quantification method can be then combined with a scanning electron diffraction technique. The series of CBED patterns are recorded for the scanning points and then simultaneously quantified using the implemented script. The final result is then represented as a symmetry map [3].

The symmetry mapping technique was then applied to a strained Si sample in order to test the sensitivity of the developed algorithm to defects. Figure 2(a) shows a typical bright field image for a stacking fault generated in the strained Si specimen. The mirror selected for quantification is along the yellow line as indicated in Fig. 2(b) in the CBED pattern. The measurement used the A/A', B/B' and C/C' disc pairs. From Fig. 2(a), the symmetry distribution was mapped on 20x10 grid points. A probe of 7.8 nm in FWHM (full-width half-maximum) was used for scanning CBED with a step length of 8 nm. Thus, the physical dimension of the scanned area is 152 x 72 nm^2. Figure 2(c) shows a magnified image of the investigated area, and Fig. 2(d) is the calculated symmetry maps for γ. The grid in the symmetry map becomes bright as the symmetry of the investigated grid matches the selected symmetry (i.e., mirror) while the dark contrast indicates symmetry breaking from the selected mirror symmetry. For example, the profile of γ values were selected along the line indicated in Fig. 2(c) and plotted in Figs. (e). In the area of stacking fault, the γ drops significantly from 0.98 to 0.18. Thus, the symmetry breaking is detected across the stacking fault and near the stacking fault.

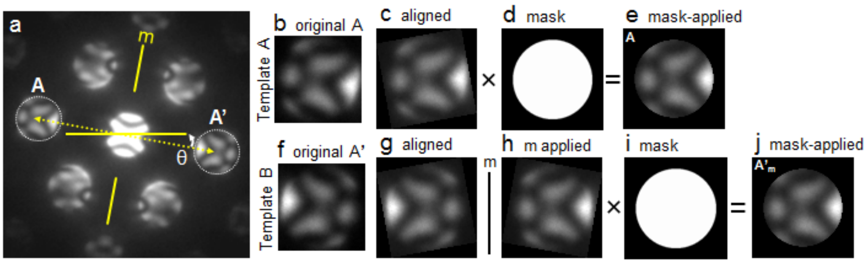

Fig. 1. Image processing procedures used for symmetry quantification. The example here is for the mirror symmetry. Two diffraction discs related by mirror are selected as indicated by the dotted circles A and A’ in the (a). Each disc is then processed to give two templates A and B as shown above.

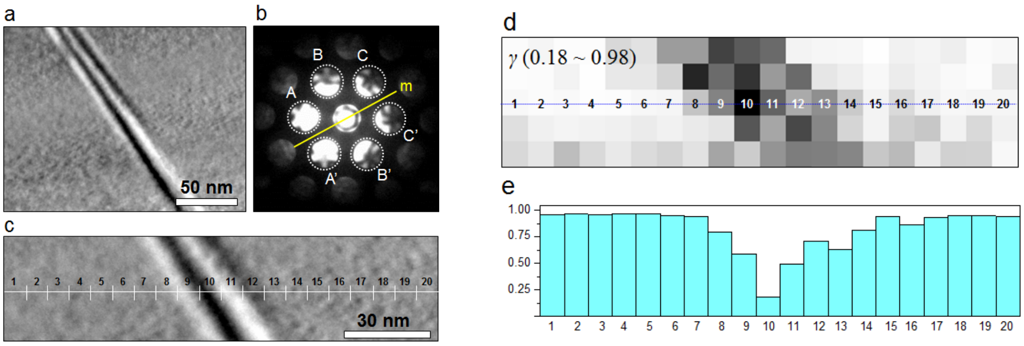

Fig. 2. (a) A medium magnification image of strained silicon showing a stacking fault, (b) the selected CBED pattern from the investigated area, and (c) a magnified image of the area investigated by scanning CBED. The symmetry map for γ is shown in Figs. 2(d). The (e) shows the γ profile across the stacking fault along the line indicated in the (c).

3. Conclusion

In this study, is was demonstrated that the quantitative CBED measurement is sensitive to study the defects in materials. We believe that the quantitative CBED measurement is possibly applied to other mateirials having the slight distortion or defects.

References

1. K.-H. Kim and J.-M. Zuo, Ultramicroscopy, **124**(2013) 71-76.
2. J.P. Lewis, Vision Interface, **95**(1995) 120-123.
3. K.-H. Kim, D. A. Payne and J.-M. Zuo, J. Appl. Cryst., **46**(2013) 1331-1337.

Acknowledgement

This work was supported by the research fund from Korea Institute of Industrial Technology (KITECH).