# Highly Accurate Lattice-Strain mapping near Interfaces of Hetero-Structures by Convergent-Beam and Nano-Beam Electron Diffraction

Koh Saitoh, Hirotaka Nakahara, Yoshiharu Daikyo, Nobuo Tanaka

Nagoya Univ. Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan Phone: +81-3-5549-6909 E-mail: ssdm secretariat@intergroup.co.jp

## 1. Introduction

Accurate measurement of lattice strains in local areas has been growing its importance especially in the modern semiconductor industry because of its strong correlation to the carrier mobility. Convergent-beam electron diffraction (CBED) has been used to determine lattice parameters because of its high spatial resolution and high accuracy. However, higher-order Laue-zone (HOLZ) line patterns, which are observed in the transmitted beam of CBED pattern, are often split into two lines at heavily strained region such as the vicinity of the interfaces of hetero-structures and even disappear.

In the present study, a method to determine lattice parameters and parameters characterizing bending strain of the lattice, the direction and magnitude of the displacement field of the bending strain, by using split HOLZ line patterns is proposed. In this method, all of the parameters are determined simultaneously by a fit of two Hough transforms of experimental and kinematically simulated HOLZ lines patterns [1]. Furthermore, the nano-beam electron diffraction (NBD) technique is applied to determine lattice parameters at around the interface regions, where no clear HOLZ lines can be observed.

## 2. Determination of the lattice bending

In the present study, we assume that bent lattice can be represented by a stack of unstrained 2D lattices which are mutually displaced in the direction perpendicular to the beam direction, as shown in Figure 1. It was found from kinematical simulation that the single major peak of the rocking curve profile for an unstrained lattice splits into two major peaks whose distance is proportional to  $\mathbf{g} \cdot \mathbf{R}$ , where  $\mathbf{R}$  indicates the vector whose direction is parallel to the lattice displacement. The linear relation between the split width of the two peaks and  $R_{\text{max}}$ , the maximum value of the displacement, was observed for various displacement models. Figures 2(a) and 2(b) show a bright field disc of a Si [230] pattern simulated by the Howie-Whelan method and by the present method, respectively. An excellent agreement of the positions of the HOLZ lines indicates the validity of the present method.



Fig. 1 (a) Schematic diagram of a bent lattice whose displacement varies along the beam direction, z. (b) Si [230] HOLZ line patterns simulated by the Howie-Whelan method and the present kinematical simulation (blue lines) with bending vectors R parallel to  $[3\bar{2}9]$  with  $R_{\text{max}} = 1.0$  and lattice parameter a = 0.550 nm.

## 3. Experimental

A Si<sub>0.7</sub>Ge<sub>0.3</sub> crystalline film was grown on a Si substrate by a molecular beam epitaxy (MBE) technique. Cross sections of the SiGe layers were prepared by mechanical thinning followed by Ar-ion thinning. CBED patterns were taken at an incidence along the [320] orientation from the Si substrate region near the interface, indicated by the rectangle in Fig. 2(a). The probe size of the convergent electron beam was approximately 5 nm in diameter. 10 × 20 CBED patterns were taken from the rectangle area by scanning the probe in x and y directions with a pitch of 15 nm.

A Fe<sub>2</sub>VSi crystalline film was epitaxially grown on the (001) surface of an  $Mg_2AlO_4$  substrate by a sputtering technique. Cross sections of the specimens were prepared by mechanical thinning followed by Ar-ion thinning. A series of NBD patterns were taken at an incidence along the [from a line perpendicular to the interface with a pitch of 2 nm.

## 4. Results and Discussion

## Convergent-Beam Electron Diffraction

Figures 3(a), 3(b) and 3(c) show maps of the direction of **R**,  $R_{\text{max}}$  and lattice parameter a in the rectangle region in Fig. 3(a), respectively. It is clearly visualized that the directions of R are perpendicular to the interface with a slight fluctuation of the direction, and that  $R_{\text{max}}$  and a show a rather steep increase towards the interface from the area deep in the substrate where the lattice is completely relaxed.



Fig. 2 Maps of the direction (a) and magnitude (b) of bending vector  $\mathbf{R}$  and lattice parameter a (c).

## Nano-Beam Electron Diffraction

□ Figure 3(a) shows a TEM image of the Fe<sub>2</sub>VSi/Mg<sub>2</sub>Al<sub>4</sub>O specimen. A collimated parallel beam of about 10 nm in diameter was scanned from the surface to the interface as are indicated by the white circles. In order to achieve the highly precise measurement of the lattice parameters, we use HOLZ reflections observed at higher scattering angles. Because such high scattering region suffers from distortion of the lens, the spot positions must be calibrated. The present study we introduced the radial, spiral and elliptical distortions up to the third order. More than 20 spots including HOLZ spots are used for the determination at each probe position. Figure 3(b) shows  $\Delta a/a$ ,  $\Delta c/c$  and c/a as a function of the distance from the surface. The errors of the lattice parameters are estimated to be in order of 10<sup>4</sup> nm, which is comparable to the method using HOLZ line

patterns.



Fig. 3 (a) A cross-section TEM image of  $Fe_2VSi/Mg_2AlO_4$ . A series of NBD patterns were taken at positions indicated by white circles. (b)  $\Delta a/a$ ,  $\Delta c/c$  and c/a as a function of the distance from the surface.

## 3. Conclusions

CBED and NBD were applied for the highly accurate determination of lattice parameters in local areas. In the CBED method, not only the lattice parameters but also parameters characterizing bending strain of the lattice can be determined by using split HOLZ line patterns. The high accuracy of the lattice parameter determination comparable to the CBED analysis is obtained by NBD by using HOLZ spots.

#### Acknowledgements

The authors are grateful to Prof. Zaima and Dr. Nakatsuka for the preparation of the SiGe/Si specimens, and to Prof. Asano for the preparation of the  $Fe_2VSi/Mg_2AlO_4$  specimens. This work was supported by the Ministry of Education, Science, Sports and Culture, Grant-in-Aid for Scientific Research (20360007).

#### References

- K. Saitoh, Y. Yasuda and N. Tanaka, International Journal of Advanced Microscopy and Theoretical Calculations 1 (2008) 90.
- [2] K. Saitoh, Y. Yasuda, M. Hamabe and N. Tanaka, J. Electron Microsc. 59 (2010) 397.
- [3] K. Saitoh, Y. Yasuda and N. Tanaka, International Journal of Advanced Microscopy and Theoretical Calculations 2 (2010) 38.