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2002 J. Phys.: Condens. Matter 14 12777

(http://iopscience.iop.org/0953-8984/14/48/316)

INSTITUTE OF PHYSICS PUBLISHING JOURNAL OF PHYSICS: CONDENSED MATTER
J. Phys.: Condens. Matter **14** (2002) 12777–12782 PII: S0953-8984(02)54080-7

# Analysis of dislocation loops by means of large-angle convergent beam electron diffraction

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Received 27 September 2002
Published 22 November 2002
Online at stacks.iop.org/JPhysCM/14/12777

**Abstract**

Diffusion-induced dislocation loops in GaP and GaAs were analysed by means of large-angle convergent beam electron diffraction (LACBED) and conventional contrast methods of transmission electron microscopy. It is demonstrated that LACBED is perfectly suited for use in analysing dislocation loops. The method combines analyses of the dislocation-induced splitting of Bragg lines in a LACBED pattern for the determination of the Burgers vector with analyses of the loop contrast behaviour in transmission electron microscopy bright-field images during tilt experiments, from which the habit plane of the dislocation loop is determined. Perfect dislocation loops formed by condensation of interstitial atoms or vacancies were found, depending on the diffusion conditions. The loops possess $\{110\}$-habit planes and Burgers vectors parallel to $\langle 110\rangle$. The LACBED method findings are compared with results of contrast analyses based on the so-called 'inside–outside' contrast of dislocation loops. Advantages of the LACBED method consist in the possibility of determining the complete Burgers vector of the dislocation loops and of an unambiguous and fast loop type analysis.

## 1. Introduction

Dislocation loops in semiconducting materials may form during ion implantation, during plastic deformation, during crystal growth or as result of dopant diffusion. Analyses of the vacancy or interstitial type of loops formed by condensation of point defects during dopant diffusion in III–V semiconductors, for instance, may be used to clarify defect formation mechanisms and their relation with basic diffusion mechanisms [1, 2]. The crystallographic nature of a dislocation loop is characterized by the normal $\boldsymbol{n}$ of the loop habit plane and the Burgers vector $\boldsymbol{b}$ of the loop. For the type determination of larger loops, analysis of the so called inside–outside contrast may be used, for which different methods have been published in the literature. These methods are based on differences between the projected loop image contrast lying either inside or outside the true loop position, depending on the image conditions. The contrast behaviour depends on the vacancy or interstitial type of the loop, its habit plane orientation with respect to the electron beam, the operating diffraction vector $\boldsymbol{g}$, and the excitation error $\boldsymbol{s}$. Dislocation loops in many materials have been analysed successfully using these methods [3–10].

Diffraction contours from high-order reflections visible in large-angle convergent beam electron diffraction (LACBED) patterns under two-beam conditions split on crossing an isolated perfect dislocation. The analysis of the contrast splitting which results from the effects of the local strain field of the dislocation allows one to determine the Burgers vector of the dislocation by applying rules which describe the relation between the dislocation line direction, the Burgers vector, and the sign of the excitation condition used for imaging, as has been shown by Cherns and Preston [11, 12]. By comparing simulated splittings of diffraction contours with experimental images, Tanaka has analysed the diffraction contour splittings to determine the Burgers vector of isolated small dislocation loops in Si [13].

This paper summarizes a method based on LACBED which may be used for the determination of the vacancy or interstitial type of larger dislocation loops. The method has been developed and tested using the example of diffusion-induced dislocation loops in GaAs and GaP. The approach combines the determination of the Burgers vector by application of the rules for Bragg line splittings which were formulated by Cherns and Preston [11, 12] with the determination of the loop habit plane from the projected diffraction contrast of the loops in large-angle tilt experiments.

## 2. Method

The effects of splitting and twisting of Bragg lines at crossings with a dislocation line in LACBED images taken under different imaging conditions are related to the Burgers vector of a dislocation which may be derived applying simple rules published first by Cherns and Preston [11, 12]. These rules are based on the so-called FS/RH (finish–start/right-hand) convention for the definition of the Burgers vector $\boldsymbol{b}$. The splitting and twisting of the Bragg lines in LACBED images and their relation to the sign of the deviation parameter $s$ are schematically illustrated in figure 1(a) and may be summarized as follows:

(1) The number of subsidiary maxima $n$ in bright-field LACBED patterns is given by

$$\boldsymbol{g} \cdot \boldsymbol{b} = n, \tag{1}$$

where $\boldsymbol{g}$ is the diffraction vector of the Bragg line.

(2) The sign of $\boldsymbol{g} \cdot \boldsymbol{b}$ describing the characteristic twisting of the Bragg line and the sign of the deviation vector $\boldsymbol{s}$ are coupled with each other: if $\boldsymbol{g} \cdot \boldsymbol{b} > 0$, the contour is bent towards $s > 0$; on the other hand, if $\boldsymbol{g} \cdot \boldsymbol{b} < 0$, then the contour is bent in the opposite direction. Figure 1(a) illustrates this for $n = \pm 3$; all possibilities of the twisting and the resulting values for $\boldsymbol{g} \cdot \boldsymbol{b} = n$ are included.

The application of the rules to the analysis of dislocation loops is illustrated schematically by figures 1 and 2. For larger dislocation loops ($\geqslant$100 nm), two splittings of the Bragg line are observed at the two separated intersections between the dislocation loop and the Bragg line (figure 1(b)). In general, the splittings of three linear independent Bragg lines have to be analysed for a determination of the Burgers vector. Application of equation (1) leads then to a set of three linear equations from which the complete Burgers vector can be determined. In principle, more than three intersections of Bragg lines with the dislocation loop may be used for an analysis, so independent control of the results is possible.

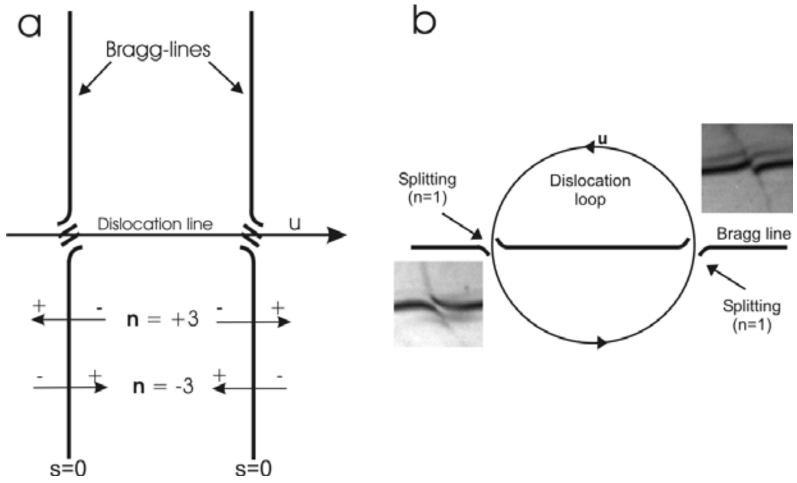


**Figure 1.** Bragg line splittings with subsidiary maxima and the relation to the sign of the deviation parameter $s$. (b) An illustration of the splitting and crossing of a Bragg line which intersects a large dislocation loop.

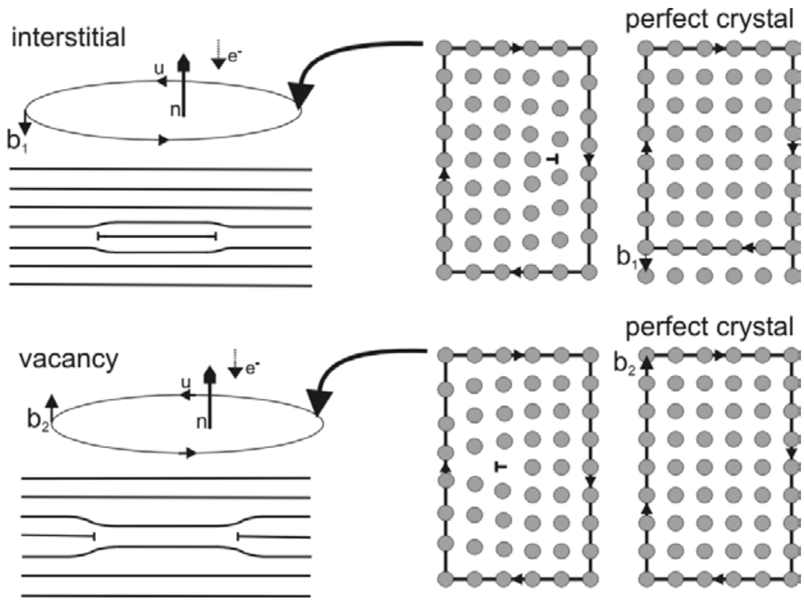


**Figure 2.** The relationship between the loop normal $\boldsymbol{n}$, Burgers vector $\boldsymbol{b}$, and dislocation line direction $\boldsymbol{u}$ for dislocation loops of interstitial type and of vacancy type. Using the so-called FS/RH rule, the Burgers circuit results in a positive sign of $\boldsymbol{b} \cdot \boldsymbol{n}$ for vacancy loops and in a negative sign of $\boldsymbol{b} \cdot \boldsymbol{n}$ for interstitial loops.

The determination of the loop type also requires the knowledge of the habit plane of the dislocation loop. The loop normal is chosen as pointing towards the electron source by definition. The vacancy or interstitial type of the dislocation loop results then from the sign of $\boldsymbol{n} \cdot \boldsymbol{b}$, requiring that the analysis of the Burgers vector $\boldsymbol{b}$ using the FS/RH rule be performed first in the disturbed crystal and then in the perfect crystal.

Figure 2 illustrates schematically the Burgers circuit for a dislocation loop of interstitial type and for a loop of vacancy type. Using this convention leads to the nature of the loop:

$\boldsymbol{n} \cdot \boldsymbol{b} > 0$ loop of vacancy type

$\boldsymbol{n} \cdot \boldsymbol{b} < 0$ loop of interstitial type.

The application of the dislocation loop analysis using the LACBED technique will be demonstrated in the following for the example of a dislocation loop in GaAs. Independently, the result has been confirmed by applying a conventional method based on the so-called 'inside–outside' contrast [14].

## 3. Example: a diffusion-induced dislocation loop in GaAs

Figure 3 shows the steps of the analysis of the Burgers vector of a dislocation loop in GaAs (diameter ~250 nm). The superposition of the diffraction information and the shadow image of the dislocation loop (figure 3(a)) as well as the splittings at the intersection between the two Bragg lines and the dislocation loop are clearly visible in the LACBED image. The indexing of the Bragg lines is performed using kinematical simulations with the program Electron Diffraction [15]. The indices of the two Bragg lines are determined to be $\bar{8}6\bar{2}$ and $6\bar{4}\bar{6}$. The small dashes perpendicular to the simulated Bragg lines indicate the positive direction of the deviation vector $\boldsymbol{s}$ ($s > 0$). Assuming that the dislocation line direction of the loop runs anticlockwise, the values $\boldsymbol{g} \cdot \boldsymbol{b}$ are determined, resulting in $\boldsymbol{g} \cdot \boldsymbol{b} = 1$ and $-1$. Figure 3(b) shows another example for the splitting of a Bragg line with the Miller indices $\bar{7}5\bar{1}$. From the analysis of the experimental LACBED image, a value of $\boldsymbol{g} \cdot \boldsymbol{b} = -1$ is determined. Finally, from the solution of the set of linear equations

$$-8b_1 + 6b_2 - 2b_3 = -1$$
$$+6b_1 - 4b_2 - 6b_3 = +1$$
$$-7b_1 + 5b_2 - b_3 = -1$$

the Burgers vector $b = +(a/2)[110]$ is obtained.

The dislocation loop normal $\boldsymbol{n}$ is determined from the analysis of the projected loop contrast in images taken in different zone axis orientations (figure 3(c)). In the chosen example, the loop normal is nearly parallel to the TEM sample foil normal. Therefore a trace analysis of the loop normal from an edge-on image is not possible. Figure 3(c) shows the dislocation loop projected along the three different zone axes [100], [110], and [010]. The projected loop area reaches a maximum near the [110] zone axis, resulting in $\boldsymbol{n} = [110]$ for the loop normal. Combining the results for the dislocation loop normal $\boldsymbol{n}$ and for the Burgers vector $\boldsymbol{b}$, the dislocation loop is identified as a loop of vacancy type.

## 4. Discussion

The analysis of Bragg line splittings from LACBED images taken under different imaging conditions within a small range of the sample tilt allows the determination of the complete Burgers vector, i.e. its absolute value and sign. As compared to conventional methods, such as the inside–outside contrast analysis, which necessitates the analysis of images of the projected loop contrast taken for different well-defined imaging conditions, the procedure described is fast and generally leads to unambiguous results. Using the inside–outside contrast analysis techniques, the imaging along different zone axes requires consecutive switching between the imaging mode and the diffraction mode and thus may easily lead to a loss of the area

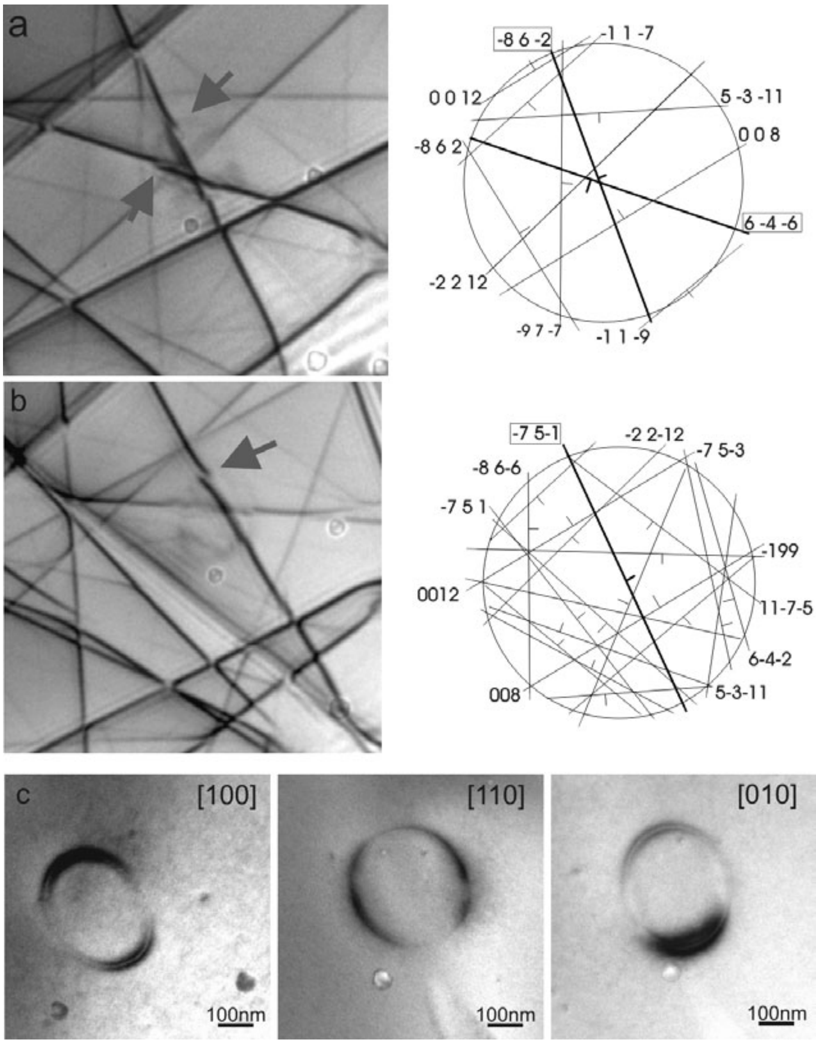


**Figure 3.** Determination of the Burgers vector $\boldsymbol{b}$ of a dislocation loop of vacancy type in GaAs by means of LACBED. (a) Crossing of the $\bar{8}6\bar{2}$ and $6\bar{4}\bar{6}$ Bragg lines with the dislocation and (right) the corresponding simulation of the Bragg lines finding the Miller indices of the experimental LACBED image. (b) Splitting of the $\bar{7}5\bar{1}$ Bragg line with the dislocation loop and (right) the corresponding simulation of the Bragg lines finding the Miller indices. The small lines perpendicular to the simulated Bragg lines show the direction of $\boldsymbol{s}$ ($s > 0$). (c) Large-angle tilt experiments for determining the loop normal $\boldsymbol{n}$. If the sample is tilted near the [110] zone axis, the dislocation loop has its major axis dimensions. In this case the loop normal $\boldsymbol{n}$ is nearly parallel to the [100] direction.

under investigation during the various tilt experiments. This problem is excluded in LACBED experiments due to the superposition of the diffraction pattern and the shadow image.

The inside–outside contrast analysis allows one to determine only the sign of the Burgers vector. However, in the cubic point groups the absolute value of the Burgers vector is well known. The energetically favourable habit planes are the $\{111\}$ and $\{110\}$ planes. Possible Burgers vectors are $(a/3)\langle 111\rangle$ and $(a/2)\langle 110\rangle$. In principle, dislocation loops with more complicated Burgers vectors can be formed. For such cases, the determination of the complete

Burgers vector is expected to be a major advantage of the LACBED method as compared to the inside–outside contrast analysis.


## Acknowledgments

The authors would like to thank J Pöpping and N Stolwijk (University of Münster) for providing the GaP crystals. The Kiel group acknowledges financial support by the Deutsche Forschungsgemeinschaft (German Science Foundation, contract DFG JA 908/2-1).


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