Non-destructive defect imaging in the SEM

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Types of Defects

Intrinsic point defects Vacancy Self-interstitial

Extrinsic point defects

Extrinsic point defects

Substitutional
foreign atom

http://www.tf.uni-

kiel.de/matwis/amat/iss/kap_5/illustr/gang_of_four_lettered.gif



http://cmcintyre.com/blog/crystal-structure



https://en.wikipedia.org/wiki/Dislocation

Volumetric defects



https://commons.wikimedia.org/wiki/File:Metallography_of_iron_FeS_inclusions.PNG

Line defects: Dislocations



If you determine **b** and **u**, you have identified the dislocation

Dislocation creation, motion, annihilation, and properties (electrical, optical) are function of dislocation type and line direction

Line Defects – Current Issues

Dislocations continue to influence critical aspects related to device performance – solar cells, LEDS

Non-destructive methods for locating and identifying dislocations remain critical



K. Szot et al, Nature Materials. 5, 312 (2006).



K. Hartman et al, Appl. Phys. Lett. 93, 122108 (2008).



M. Yamaguchi et al, Solar Energy 82, 173 (2008).

Present Dislocation Imaging Approaches

Various techniques suffer certain limitations:

TEM – destructive, confined area

XRD – not local but global

SWBXT - requires a synchrotron x-ray source

AFM - contact, indirectly images dislocations

Chemical etching - destructive

Luminescence (CL, PL, EL) – little surface information, may require pre-processing, requires pre-existing knowledge of optical behavior

Dislocation Density → Average Dislocation Separation $10^{8}/cm^{2} \rightarrow 1 \ \mu m$ $10^{5}/cm^{2} \rightarrow 32 \ \mu m$



Y.N. Picard *et al*, Appl. Phys. Lett. **91**, 014101 (2007).

AFM of MBE GaN

B. Heying *et al*, J. Appl. Phys. **85**, 6470 (1999).



Electron Channeling Contrast Imaging (ECCI)

ECCI image of MOCVD GaN film



- Non-destructive
- Non-contact
- Commercial SEM
- Dislocation imaging
- Atomic morphology
- Orientation contrast
- Phase identification

Electron Channeling

η: backscattered electron yield

η is a strong function of the relative orientation between incident electron beam and crystal

incident incident. electron beam electron beam large η small η

Discovered by D.G. Coates, Phil. Mag. 16, 1179 (1967).

Fig. 1. Schematic representation of the variation in back scattered electron emission (η) as a function of the relative orientation between the incident electron beam and the crystal lattice (after Joy et al. 1982).

David C. Joy, Dale E. Newbury, and David L. Davidson. "Electron channeling patterns in the scanning electron microscope." *Journal of Applied Physics*53.8 (1982): R81-R122.

Diffraction in the SEM





Electron Channeling Pattern (ECP)



O. Engler and V. Randle, Introduction to Texture Analysis, 2nd. Edition, CRC Press 2009

Channeling in the SEM



Low Magnification BSE Image

Electron Channeling Contrast Imaging

"It should in principle be possible to use the <u>scanning electron microscope to detect dislocations</u> by the direct examination of unetched crystal surfaces. It is only necessary to orientate the crystal at the Bragg position and the <u>local bending</u> of these crystallographic planes should produce the necessary contrast. Such contrast should be directional and <u>could lead to Burgers vector determination</u>."



J. Ahmed et al, J. Microscopy 195, 197 (1999).

G.R. Booker, AMB Shaw, M.J. Whelan and P.B. Hirch, Philosophical Magazine **16**, 1185 (1967).

Intensity fluctuation due to local lattice bending by dislocation

This is the basis for nondestructive defect imaging by ECCI

Previous ECCI Work



More Recent ECCI Results





C. Trager-Cowan et al, Phys. Rev. B. 75, 085301 (2007).

Individual dislocations can be imaged in an SEM

Atomic steps visible

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Channeling vs. Conventional SEM

Backscatter Electron Detection – On Diffraction Condition

GaN (0001) 1/18/2010 WD mag mag spot GaN ECCI attemp 20 000

Secondary Electron Detection – Conventional SEM

Two Geometries



Example Experimental Approaches

Forescatter Geometry



Courtesy of Oxford Instruments

Backscatter Geometry



Forescatter diodes on EBSD 10-30 kV, 4-6 spot size Samples tilted 50-70°

nA's of current

Annulus BSE detector 10-30 kV, 5-6.5 spot size Samples tilted 5-20°

Modern ECCI Approach

1. BSE Image Single Crystal at Lowest Magnification



2. Use Resultant ECP to Orient Sample (Tilt/Rotate)



M. A. Crimp, "Scanning electron microscopy imaging of dislocations in bulk materials, using electron channeling contrast." **Microsc. Res. Tech. 69**, 374 (2006).

R.J. Kamaladasa, Y.N. Picard, "Basic principles and application of electron channeling in a scanning electron microscope for dislocation analysis" **Microscopy: Science, Technology, Education and Applications. Vol. 4**, 1583 (2011).

3. Magnify to Sample Surface





- 1. Channeling signal is much weaker than topographic and Z-contrast information in BSE image
- 2. Detector-sample positioning (working distance) needs to be optimized to maximize BSE collection
- 3. Detector gain settings (brightness/contrast/gamma) will need adjusting to maximize channeling BSE contrast

Single Screw Dislocation (4H-SiC)



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Single Screw Dislocation = Intensity Fluctuation



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19

Screw Dislocation: Contrast → Burgers Vector

4H-SiC (0001)



Reversing Burgers vector, **b**, reverses the dark-to-light contrast





D.B. Williams and C.B. Carter, from p.415 of *Transmission Electron Microscopy*, Plenum Press, New York (1996).

ECCI of GaN

Threading dislocations = dark/light spots

atomic steps = lines





Defect imaging by ECCI resolvable over areas approaching 50 x 50 µm

Screw vs. Edge Delineation by ECCI



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ECCI Validation – HVPE GaN Substrates



MgO:KOH etching confirms ECCI features are dislocations

Larger feature = Larger etch pit

TEM

Site-specific TEM confirms differentiation between edge and mixed dislocations

Larger etch pit = Mixed dislocation



SEM post-etching



R.J. Kamaladasa et. al. Journal of Microscopy, 244(3), 311-319 (2011)

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Edge Dislocations on Low-Angle Grain Boundaries



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2004.

Instruction

Manual, HKL

Edge Dislocations on Low-Angle Grain Boundaries





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Electron channelling contrast imaging for III-nitride thin film structures



G. Naresh-Kumar^{*}, D. Thomson, M. Nouf-Allehiani, J. Bruckbauer, P.R. Edwards, B. Hourahine, R.W. Martin, C. Trager-Cowan *Department of Physics, SUPA, University of Strathclyde, Glasgow G4 ONG, UK*

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ECCI technique sensitive to threading

edge dislocations

Simulating Threading Dislocation Contrast

GaN (0001)



Resolving dislocation type

M.E. Twigg and Y.N. Picard. J. Appl. Phys. 105, 093520 (2009).Y.N. Picard *et. al.* Scripta Mater. 61, 773 (2009).



Comparing ECCI Geometries



Y.N. Picard et. al. Scripta Mater. 61, 773 (2009).

Backscatter ECCI



Surface distortion: Screw dislocation > Edge dislocation

Contrast Dependent on Diffraction Vector

GaN (0001): Rotating diffraction vector, **g**, rotates dark-light contrast features



25 STRAIN FIELDS



W.J. Tunstall et. al. Phil. Mag. **9**, 99 (1964). (as taken from D.B. Williams and C.B. Carter, p.415 of *Transmission Electron Microscopy*, Plenum Press, New York (1996).)



Dislocation Channeling Contrast – Line Direction



J. Ahmed et al, J. Microscopy 195, 197 (1999).





Dark-Light Spot Feature





Surface Penetrating Vertical Dislocation

Y.N. Picard et al, J. Appl. Phys. 104, (2008) 124906.





Sub-Surface Horizontal Dislocation

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Dislocation Line Direction – Insights from Etching

SrTiO₃ (100): HF acid etching is defect selective



ECCI – Invisibility Criteria



Dislocation line: u = <100> Burgers vector: **b** Diffraction vector: **g**

Independent variable g vector

Dependent variable Dislocation feature contrast (strong, weak, invisible)

> <u>Feature shape</u> u (dislocation line)

Result: **b** identification (dislocation determination)

ECCI – Various Dislocation Interactions



Surface-penetrating half-loops

Sub-surface dislocation loops

Y.N. Picard et. al. Microscopy Today. 20 (2), 12-16 (2012).

5 µm

Dislocations along slip line



W. Jiang, R.J. Kamaladasa, Y. Lu, R. Berechman, P.A. Salvador, J.A. Bain, Y.N. Picard, M. Skowronski, "Local heating-induced plastic deformation in resistive switching devices" **Journal of Applied Physics**, **110**(5), 054514 (2011).

Accurate Simulation of ECCI features



GaSb (001)



Experimental Simulation

Experimental Simulation

Figure 4: (a) Experimental and (b) simulated ECP of GaSb (001) and (c) experimental and (d) simulated ECCI micrograph of surface penetrating dislocations in GaSb (001).

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Y.N. Picard, et. al., "Future prospects for defect and strain analysis in the SEM via electron channeling" **Microscopy Today**. **20** (2), 12-16 (2012).

Accurate Simulation of ECCI features



Non-destructive Mapping – Pre/Post Growth



F. Liu, et. al., Journal of Crystal Growth, 387, 16-22 (2014).

ECCI of In_{0.1}Ga_{0.9}N/GaN Multi-Quantum Wells



F. Liu, et. al., Journal of Crystal Growth, 387, 16-22 (2014).

channeling condition)

V-defects

ECCI of GaP on Si (100)



S. D. Carnevale, J. I. Deitz, T. J. Grassman, J. A. Carlin, Y.N. Picard, M. De Graef, S. A. Ringel, "Rapid Misfit Dislocation Characterization in Heteroepitaxial III-V/Si Thin Films by Electron Channeling Contrast Imaging" **Applied Physics Letters**, **104**, 232111 (2014). S.D. Carnevale, J. Deitz, J. Carlin, Y.N. Picard, D.W. McComb, M. De Graef, S. Ringel, T.J. Grasssman, "Applications of Electron Channeling Contrast Imaging for the Rapid Characterization of Extended Defects in III-V/Si Heterostructures" **IEEE Journal of Photovoltaics**, **5**(2) 676-682 (2015).



Anti-Phase Domain Boundaries



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39

Si-Ge Misfit Dislocations



SiGe (001) film on Si: High density of misfit dislocations on {110}



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Point Defect Clustering at Dislocations



Screw dislocations from substrate nucleate hillocks

Hillock apex corresponds to drop in 5.32-5.39 eV NBE intensity

FIG. 3. SE (a) and ECCI image (b) as well as the intensity (c) and energy (d) of the CL NBE peak of sample B.

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Spatial clustering of defect luminescence centers in Si-doped low resistivity AI0.82Ga0.18N

Gunnar Kusch, M. Nouf-Allehiani, Frank Mehnke, Christian Kuhn, Paul R. Edwards, Tim Wernicke, Arne Knauer, Viola Kueller, G. Naresh-Kumar, Markus Weyers, Michael Kneissl, Carol Trager-Cowan, and Robert W. Martin

Citation: Applied Physics Letters 107, 072103 (2015); doi: 10.1063/1.4928667

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5.265

5.260 2

5.255 aru

5.250 0

5.245

ECCI correlation to Optoelectronic Behavior



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G. Naresh-Kumar^{*}, D. Thomson, M. Nouf-Allehiani, J. Bruckbauer, P.R. Edwards, B. Hourahine, R.W. Martin, C. Trager-Cowan

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Single-electron transport in InAs nanowire quantum dots formed by crystal phase engineering

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42

ECCI of Slip Bands in Steel





30 wt% Mn Steel under high strain rate \rightarrow hardening behavior

Tensile axis is out-of-plane

a,b) planar dislocation glide {111} to slip band formation; c) slip bands have high stress between them



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ELSEVIER	journal homepage: www.elsevier.com/locate/actamat	
Full length article		

Strain hardening by dynamic slip band refinement in a high-Mn lightweight steel



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E. Welsch^a, D. Ponge^{a,*}, S.M. Hafez Haghighat^a, S. Sandlöbes^{a, b}, P. Choi^{a, c}, M. Herbig^a, S. Zaefferer^a, D. Raabe

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ECCI of Slip Bands and Dislocations Evolution in Steel



Fig. 4. Observation of features by ECCI: surface traces of different active slip planes (A, B), individual dislocations (C) (enlarged region) and dislocation structures such as veins (D).



Available online at www.sciencedirect.com ScienceDirect Acta Materialia 87 (2015) 86-99



Effects of strain amplitude, cycle number and orientation on low cycle fatigue microstructures in austenitic stainless steel studied by electron channelling contrast imaging

J. Nellessen, S. Sandlöbes* and D. Raabe



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Sub-grain boundary characterization



Fig. 1. (a) HR-SACP superimposed on a dynamical Kikuchi pattern simulated with the "Esprit DynamicS" software from Bruker. The red crosses in the HR-SACPs indicate the microscope optic axis. (b) ECC image of a sub-grain boundary showing HR-SACPs acquired from each side of the boundary reveals the direction of pattern shift (dotted red line) and the misorientation angle ($\approx 0.13^\circ$). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



Scripta Materialia 109 (2015) 76-79



Accurate electron channeling contrast analysis of a low angle sub-grain boundary

H. Mansour^{a,*}, M.A. Crimp^b, N. Gey^{a,c}, N. Maloufi^{a,c,*}

Inverted ECCI – High Resolution Bright-Field







Figure 8. Comparison between inverted ECCI (a) and convential TEM (b) micrograph of the dislocation arrangement in a globular γ -TiAl grain. [after 65]. Details see Figure 7.



Case studies on the application of high-resolution electron channelling contrast imaging – investigation of defects and defect arrangements in metallic materials

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Anja Weidner & Horst Biermann

ECCI of a High Entropy Alloy



Planar slip in a CoCrFeMnNi alloy

> Materials Science & Engineering A 648 (2015) 183–192 Contents lists available at ScienceDirect Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

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Fig. 8. Deformation substructures revealed by electron scanning contrast imaging for the representative (a) 21Mn and (b) 38Mn at different strain levels of 1%, 5% and 10%.

Dislocations progress to dislocation cells (DC) and high density dislocation walls (HDDW)

Non-equiatomic high entropy alloys: Approach towards rapid alloy screening and property-oriented design

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Summary

Defect imaging via SEM – highly accessible via electron channeling

- FEG SEM + BSE Detector
- Must consider crystallography \rightarrow **g** vector and defect geometry

Opportunities for progress

- Improved BSE detector sensitivity
- Coordination with in situ methods inside the SEM
- Coordination with SPM and other SEM modes \rightarrow defect-property correlations
- Automated high-speed imaging and mapping of millimeter areas
- Reliable defect identification \rightarrow accurate simulations
- Reliable g vector control \rightarrow informed by EBSD and/or SACP

I recommend this review article: Theory a

Theory and application of electron channelling contrast imaging under controlled diffraction conditions

Acta Materialia 75 (2014) 20-50

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A Pre-Meeting Congress hosted by the MSA Electron Crystallography and Automated Mapping Techniques -- Focused Interest Group

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The Electron Crystallography & Automated Mapping Techniques Focused Interest Group

(ECAMT-FIG) is a community of MSA members with a common interest in crystallography and advanced electron methods for materials characterization. Our goal is to provide a platform for the distribution and discussion of ECAMT-relevant information chosen by the membership. Our activities include the hosting of pre-meeting congresses and symposia during the annual M&M meeting. We also host a luncheon and business meeting during the annual M&M meeting.

ypicard@cmu.edu ECAMT-FIG Leader-Elect



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