

Optimised EBSD Analysis with the SUPRA® FE-SEM

Introduction

Electron Backscattered Diffraction (EBSD), also known as EBSD, BKD or OIM™, is a SEM technique applicable to crystalline materials. It provides absolute crystal orientation information down to the nano scale level when a dedicated Field Emission SEM is used. EBSD allows identification of phases and discrimination between phases in materials. The EBSD patterns are produced when an electron beam strikes a specimen surface. The electron scattering in the material causes electrons to travel in all directions. Electrons that satisfy the Bragg condition, i.e. $n\lambda=2d \sin\theta$, for a crystal plane are channelled and show the Kikuchi bands, see fig.1.

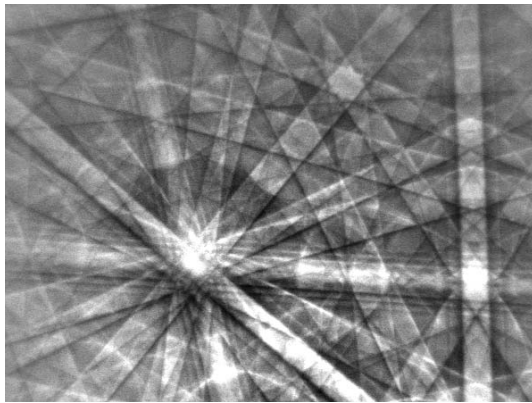


Fig. 1: Example of EBSD pattern.

EBSD patterns were first observed by Kikuchi in a TEM. For the EBSD detection in a SEM a fluorescent phosphor screen in front of a highly sensitive CCD camera is used. The EBSD detector system is mounted on one of the side ports of the specimen chamber and should bring the fluorescent screen close to the specimen. For clear EBSD patterns the specimen surface has to be distortion free. This can only be achieved with proper specimen preparation techniques like electro-polishing or using a focussed ion beam system like the CrossBeam®.

Instrumentation requirements

In order to enable EBSD in a FE-SEM, the EBSD detector has to be mounted on the side port of the specimen chamber. The specimen has to be tilted to 70 degrees to project the pattern on the EBSD detector. Forward scattered BSE detectors are mounted on the EBSD detector to enable phase and Z-contrast imaging of the specimen surface. In order to combine chemical and phase identification an EDS detector needs to be installed facing towards the specimen at exactly the same coincidence point as the EBSD detector.

The ZEISS SUPRA® 55 and 55VP FE-SEMs both have integrated design features for superior EBSD analysis:

- GEMINI® FE-SEM column supplies a highly stable electron beam for fully automated analysis of large areas
- High Current, Depth of Field module with the virtual FE source moved to infinity (fig. 2) supplies a highly parallel beam with a high current density enabling improved lateral resolution (sub 10 nm), high angular resolution (0.25°) and fast detection times (100 points/sec).

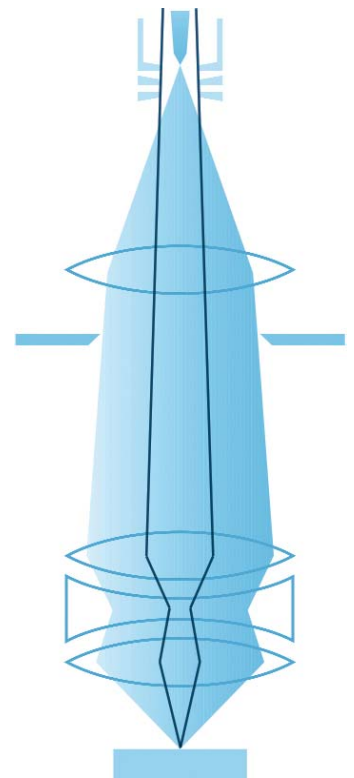


Fig. 2: GEMINI® High Current mode with virtual parallel beam: optimised EBSD.



We make it visible.

- Precise scan control and calculated compensation for tilt correction combined with dynamic focus gives distortion free imaging, see fig.3
- Chamber geometry supplies an excellent platform for coplanar EBSD and EDS analysis at 8.5mm WD and specimen tilt of 70°, see fig. 4
- Eucentric specimen stage provides accurate tilt to 70° and stability for large area automated phase detection
- Variable pressure capabilities to analyse non conducting specimens like ceramics and semiconductors (for the SUPRA® 55VP only)
- No magnetic field outside the GEMINI® end lens eliminates distortions when analysing magnetic or para-magnetic specimens

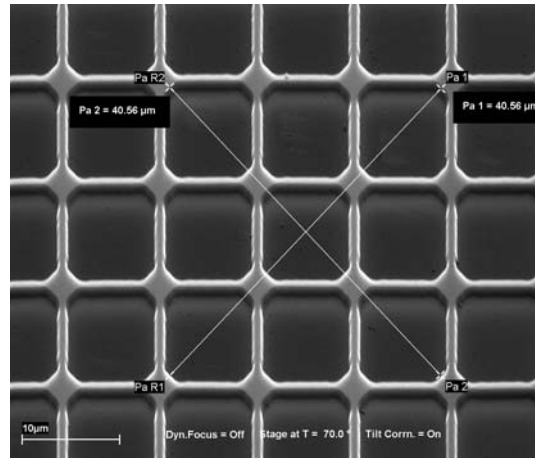


Fig. 3: Undistorted geometry at 70° tilt. Tilt correction on, dynamic focus not used!

Applications (courtesy of HKL Technology)

EBSD analysis is an extremely useful technique for crystal orientation, phase identification (combined with EDS analysis), phase verification (like in duplex steels), texture measurement, boundary characterisation and deformation measurements.

The following application examples have been provided by HTL Technology A/S, Hobro, Denmark.

The instrumentation used for the EBSD analysis was the HKL Nordlys II EBSD Detector, Channel 5 software and the ZEISS SUPRA® 55VP FE-SEM.

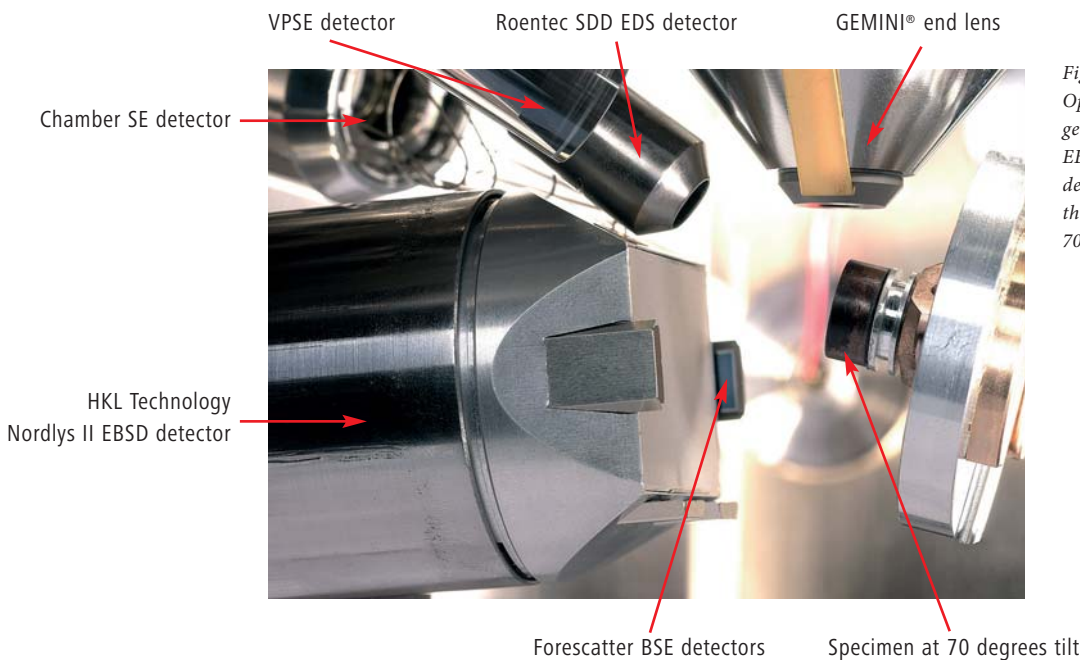


Fig. 4: Optimised EBSD geometry: EBSD and EDS detectors close to the specimen at 70 degrees tilt.

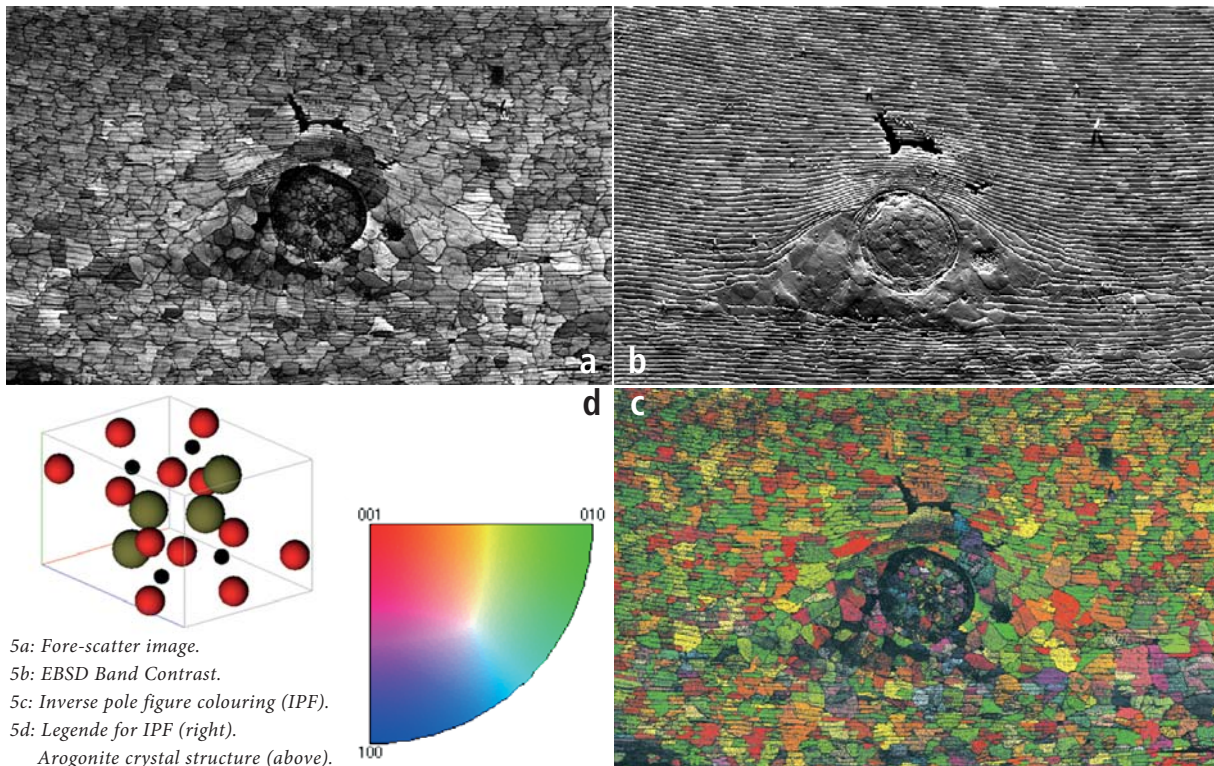


Fig. 5: Example of EBSD Imaging of Angaria Delphinus shell which has an unstable CaCO_3 (aragonite) structure under the electron beam. EBSD pattern where taken with a speed of 100 patterns/sec to minimise beam damage.

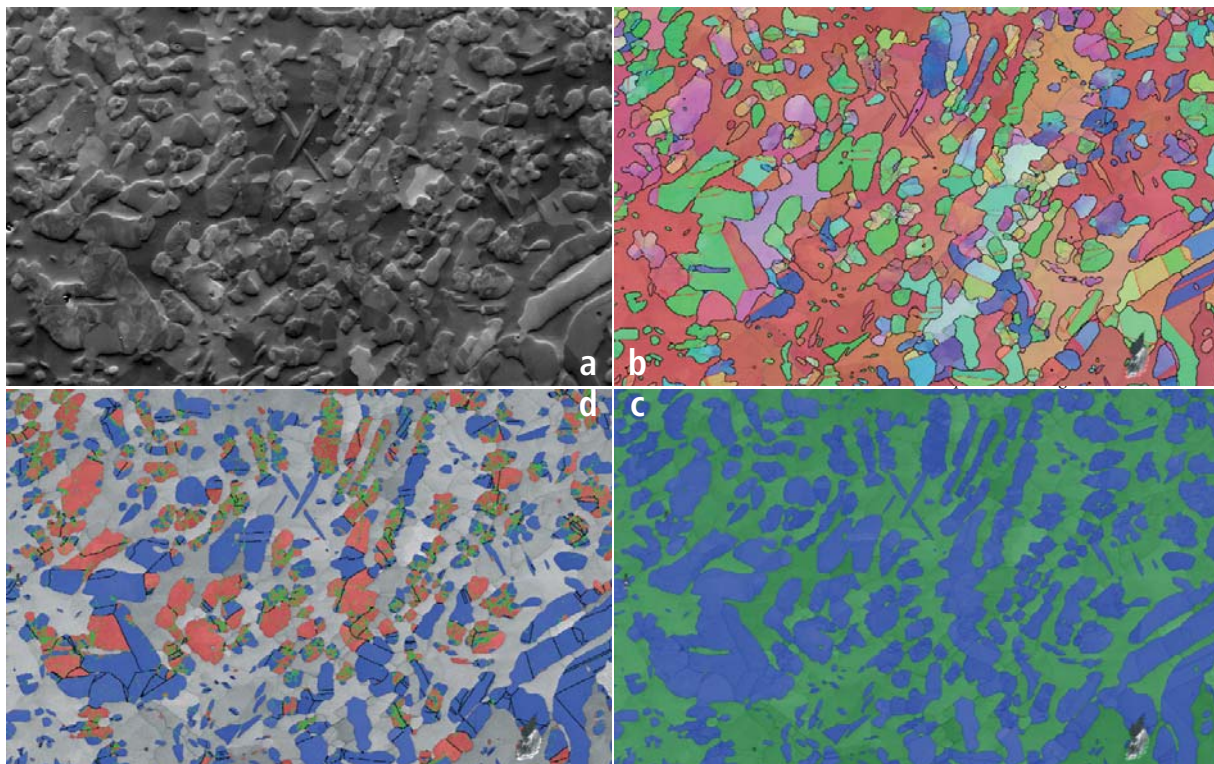


Fig. 6: Example of EBSD on duplex stainless steel:
 6a: Fore-scatter image;
 6b: EBSD Map;
 6c: Phase map: ferrite green/austenite blue;
 6d: Recrystallised blue, deformed red.



Fig. 7: SUPRA® 55VP FE-SEM with the HKL Technology Nordlys EBSD detector.

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Carl Zeiss NTS GmbH

Carl-Zeiss-Str. 56
73447 Oberkochen
Germany
Tel. +49 73 64 / 20 44 88
Fax +49 73 64 / 20 43 43
info@nts.zeiss.com

Carl Zeiss NTS, LLC

One Corporation Way
Peabody, MA 01960
USA
Tel. +1 978 / 826 1500
Fax +1 978 / 532 5696
info-usa@nts.zeiss.com

Carl Zeiss NTS Pte. Ltd.

50 Kaki Bukit Place #04-01
Singapore 415926
Singapore
Tel. +65 65 67 / 30 11
Fax +65 65 67 / 51 31
info.sea@nts.zeiss.com

Carl Zeiss NTS Ltd.

511 Coldhams Lane
Cambridge CB1 3JS
UK
Tel. +44 12 23 41 41 66
Fax +44 12 23 41 27 76
info-uk@nts.zeiss.com

Carl Zeiss NTS S.a.s.

Zone d'Activité des Peupliers
27, rue des Peupliers -
Bâtiment A
92000 Nanterre
France
Tel. +33 1 41 39 92 10
Fax +33 1 41 39 92 29
info-fr@nts.zeiss.com

www.zeiss.com/nts



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