Preparation of ferrous metals for Electron Backscatter Diffraction (EBSD) analysis

Struers
Ensuring Certainty

Application Notes

This Application Note focuses on the preparation of ferrous metals for the EBSD analysis and includes practical recommendations for the preparation of specimens and EBSD set-up. Various types of ferrous metals have been selected and the complete preparation processes used as well as an evaluation of the microstructure, microtextures and grains boundaries are described.

As EBSD is a surface-sensitive technique, with the diffraction signal coming from the top few nanometres (5-50 nm) of the crystal lattice, it is essential that this top layer remains free from damage and also free from contamination or oxidation layers – and this makes specimen preparation absolutely critical for collecting good EBSD data. The fact that the specimen must be tilted up to high angles (typically 70°), means that the surface topography/relief must be kept to an absolute minimum to avoid shadowing problems.

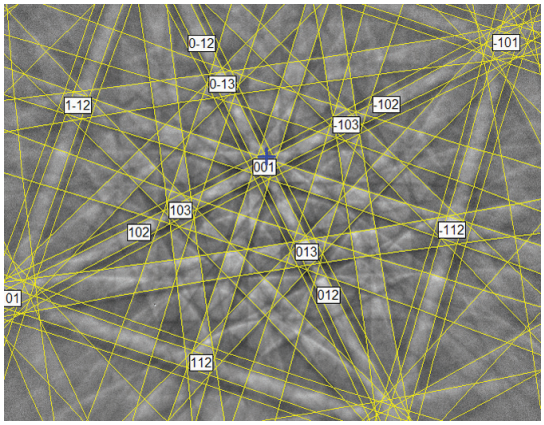
There are a number of techniques for preparing specimens for EBSD, and choosing the correct technique is dependent on the composition and structure of the specimen. Unfortunately there is no single technique that works with all materials, and an experimental approach is often required to achieve a suitable result.

EBSD is the technique by which an SEM can be used to evaluate the microstructure of a specimen based on crystallographic analysis.

A scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. Usually scanning electron microscopes also have EDS[1] system which allow combined EDS/EBSD mapping in order to compare chemical and crystallographic information in one map.

EBSD provides absolute crystal orientation information and allows identification of phases and discrimination between phases in materials, texture measurements, boundary characterisation and deformation measurements. EBSD therefore is becoming a more and more popular examination and measurement technique.

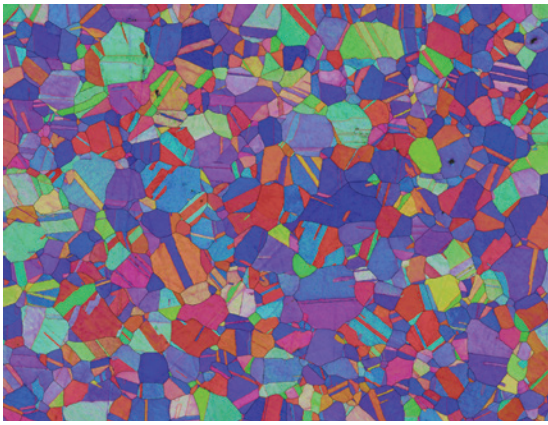
The EBSD patterns are produced when the electron beam strikes the specimen surface. The electron scattering in the material causes electrons to travel in all directions. Electrons that satisfy the Bragg[2] condition for a crystal plane are channelled and show the Kikuchi bands[3].



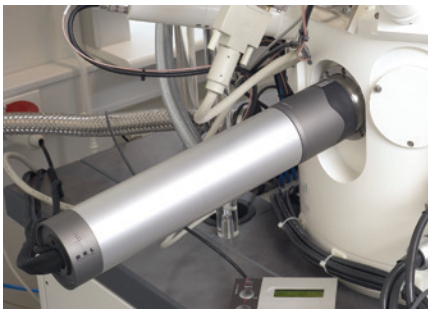
Kikuchi bands of face cubic centered (fcc) crystal structure. Electron Backscattered Pattern (EBSP) of austenite with indexing.

Electron Backscatter-like patterns EBSP were first observed by Kikuchi (1928).
EBSD as it exists today was developed during the 1980's initially by Dingley and later in the early 1990's by Wright. Concurrently and independently, Niels Schmidt in Denmark developed a general solution to the computer-aided indexing problem that was the first to accurately index all seven crystal systems. Application of the Hough transform[4] allowed EBSP orientation measurements to be automated.

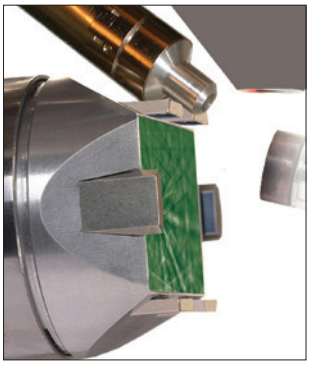
Today, Electron Backscatter Diffraction (EBSD) is applied across a wide variety of research fields: in the metals processing industry, the aerospace-, nuclear-, automotive- and microelectronics industry, earth sciences etc. An EBSD system therefore may be used at one laboratory to characterise the texture in an aluminium alloy, and at another laboratory to identify the different phases in a lead-free solder.



Electron Backscattered Diffraction (EBSD) map of stainless steel X5CrNi18-10. For examination of grain size and grain orientation. Original magnification 200x.



The Nordlys EBSD detector mounted on a SEM. Courtesy of Oxford Instruments plc.



The Nordlys EBSD detector inside the SEM chamber. Courtesy of Oxford Instruments plc.

Preparation difficulties

Specific to the use of SEM:

Size of specimens can vary and is limited due to the capacity of the SEM chamber. Generally the size should not exceed 2 x 1 x 0.5 - 1.2 cm (w x d x h).

Electrical contact is not optimum.

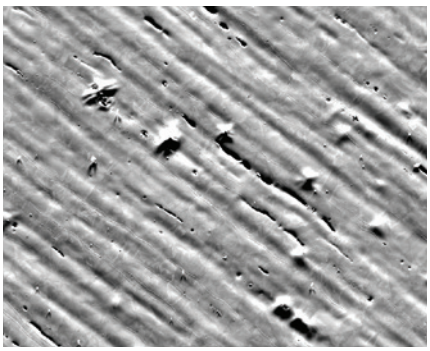
Charging of the mount.

Specific to the metallographic preparation process:

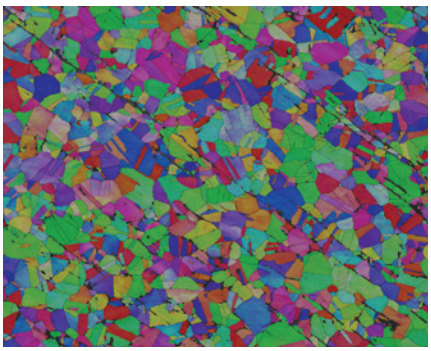
Deformation and excessive relief on the surface of the specimen.

Cleaning prior to examination. Insufficient cleaning will not allow an EBSD map of a good quality.

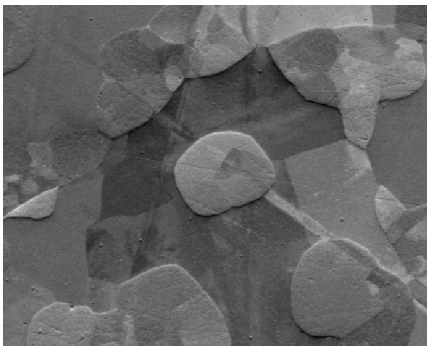
Protection of prepared specimens from scratching and the environment.



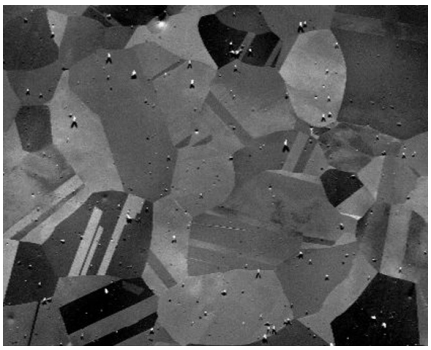
Deep scratches in stainless steel X5CrNi18-10. SEM, original magnification 300x.



EBSD map of insufficient grinding prior to electrolytical polishing, showing IPF[5] colouring (Inverse Pole Figure map component). The black lines are remaining deformations on the specimen's surface. Original magnification 300x.



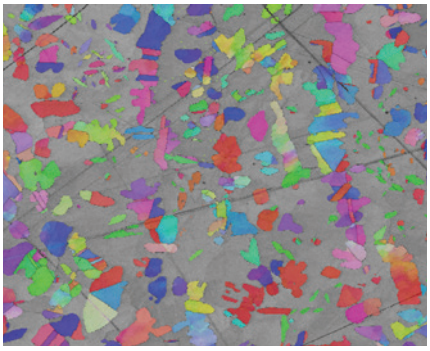
Scratches and excessive deformations in duplex stainless steel. SEM, original magnification 1000x.



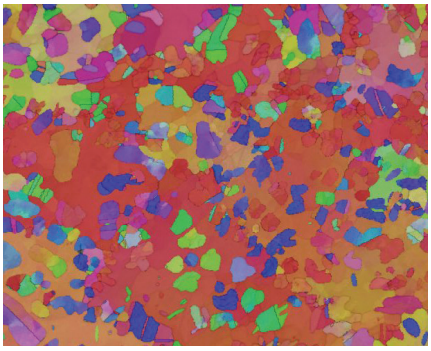
The visible white spots are stains on the prepared surface. An EBSD map of a good quality of these areas is not possible. SEM, original magnification 1000x.

Solution

- Select the correct cut-off wheel to minimize introduction of structural damages.
- Ensure appropriate mounting or clamping procedure.
- Select the proper mechanical preparation process (using low resilience preparation surfaces and short polishing times).
- Use of electrolytic polishing.
- Ensure a thorough cleaning and protection of prepared surfaces.

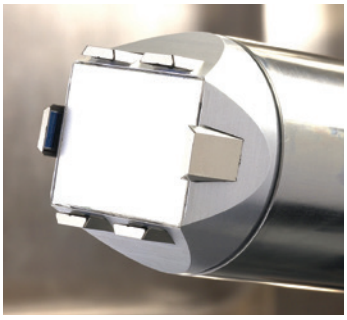


EBSD Map of a insufficiently polished duplex stainless steel, IPF colouring. Original magnification 500x.



EBSD Map of the same duplex stainless steel, properly prepared, IPF colouring. Original magnification 500x.

Description of EBSD working principles



The nose (phosphor screen) of the Nordlys EBSD detector. Courtesy of Oxford Instruments plc.

EBSD - Electron Backscatter Diffraction is a SEM based technique to measure crystal orientations and is applicable to any crystalline material (in theory). EBSD provides the absolute crystal orientation with sub-micron resolution and is also a useful tool for discriminating between phases.

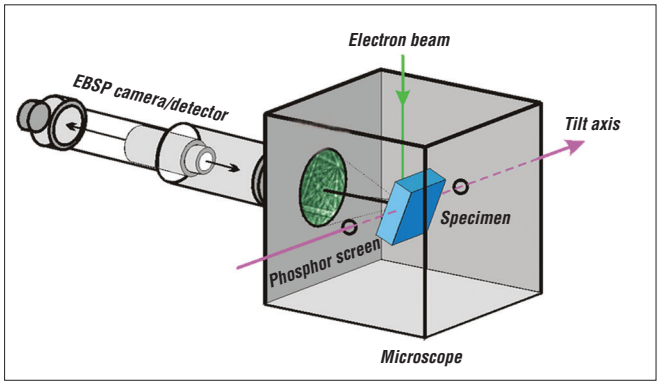
EBSD operates by arranging a highly polished specimen tilted at a high angle (usually 70º) to the incident electron beam. With moderate to high electron beam acceleration voltages (10 to 30 kV) and incident beam currents from 1 to 15 nA, the electron beam is diffracted by the crystal lattice of the specimen at the incident beam point on the specimen surface. An Electron Backscatter Diffraction pattern (EBSP) emanates spherically from this point in all directions.

The EBSP detector will intersect a portion of this diffraction pattern. The detector is a digital camera contained within a vacuum compatible and retractable body. The camera's CCD chip is illuminated by a phosphor screen that intersects the spherical diffraction pattern. The phosphor converts the diffracted electron into light suitable for the CCD to record. The EBSP is analyzed and is uniquely defined by the lattice parameters of the particular crystal under the beam, and by its orientation in space. Possible identities and orientations of the crystal are matched to the EBSP until the best fit is found. The pattern is then considered indexed.

As the speed of pattern analysis has increased, it has become practical to scan the beam over multiple points on the specimen to create an orientation map. This is now the most common method for a microstructural investigation with EBSD.

Practical recommendations during EBSD set up:

Many of the problems encountered when acquiring EBSD data are due to difficulties in generating the EBSPs. Sometimes these problems will be unavoidable - for example due to the state of the specimen (poor preparation, highly deformed etc.). Often the results can be improved by careful setting up of the EBSP detector and the image enhancement.



Schematic diagram of the typical set up for EBSD. Courtesy of Oxford Instruments plc.

Although the size and shape of the SEM chamber and stage is often limited, small changes in the specimen to detector geometry can result in big improvements in the EBSP:

- Move the phosphor screen as close to the specimen as possible but still in a safe and comfortable limit – this gives a stronger signal and a larger capture angle.

- Adjust the working distance so that the strongest signal in the raw (unprocessed) EBSP is in the centre of the phosphor screen.

The typical EBSD operating conditions are 1-15 nA probe current with 15-30 kV accelerating voltage. However often the EBSPs can be improved by moving away from these beam conditions. Generally increasing the probe current will give a stronger signal, and therefore will give better EBSPs.
• For very deformed or very fine grained specimens, the pattern may get improved by decreasing the size of the beam spot size on the surface – i.e. decrease the probe current.
• When working on a coated specimen, it sometimes helps to increase the accelerating voltage to 25-30 kV to penetrate through the coating.

Forescatter System or Orientation Contrast Imaging:

The Oxford Instrument Forescatter Detector (FSD) system is designed to produce orientation contrast images from a wide range of materials. It uses multiple silicon diodes placed around the EBSD phosphor screen to produce microstructural images. For some materials, the top diodes will also produce an atomic number contrast signal. All SEM pictures in this Application Note are acquired using the forescatter system.

Phosphor screen

The shape of the phosphor screen on the Nordlys detector exactly matches the shape of the CCD chip in the camera. This means that the whole diffraction pattern imaged on the phosphor screen is used in the CHANNEL indexing software: this increases both the reliability and the accuracy of the EBSD data.

Charging of the mount

During the examination in a SEM, a charge will tend to accumulate unless the specimen is earthed. This can be avoided using a conductive mounting resin suitable for SEM like PolyFast. For specimens mounted in other mounting materials a conductive trace from the specimen to the stub/holder is prepared, by coating the surface with gold or carbon, using sputtering equipment or conductive paint or tape.

Fixing of specimen

It should be ensured that the specimen is fixed properly to the stub/holder using for instance silver cement in order to avoid drift caused by the specimen moving.

Preparation difficulties

Fig.1: The versatile cutting machine Secotom-50

The wide variation in the size, shape and physical properties of ferrous metal specimens to be examined using EBSD may require a different approach to the preparation processes e.g. mechanical and/or mechanical/electrolytical.
Consequently, it is important, prior to the preparation, to decide how that process should be carried out.
Does the specimen need to be cut? The specimen must be cut at the area of interest, in the proper orientation and the correct cut-off wheel must be selected.
Does the specimen need to be mounted or not? A correct mounting eases handling and the mechanical preparation process; on the other hand it is easier to carry out electrolytical polishing on an unmounted specimen.
Then a suitable mechanical or mechanical/electrolytical preparation method must be selected to ensure nearly zero deformation and minimum relief. Mechanical preparation is usually satisfactory but time consuming. Electrolytic polishing subsequent to a short mechanical process will give a fast result and higher contrast, and is very useful for homogeneous materials.
Furthermore, to be able to create an EBSD map of an optimal quality, it is important that the prepared surfaces are thoroughly cleaned and also well protected until the analysis is started.

Preparation recommendations

Cutting

The size of a specimen often needs to be reduced to a size suitable to the vacuum chamber of the SEM in use.
The most commonly used specimen size is 2 x 1 x 0.5 - 1.2 cm (w x d x h).

The microstructure over an entire component may not be the same. It is therefore important to decide beforehand the best location of a cut and at the same time ensure that the specimen orientation is preserved e.g. rolling direction, transverse direction.

An abrasive wet cutting machine is recommended to avoid a deep deformation level and heat damages, e.g. Secotom-50 (Fig.1). Correct cutting parameters must be selected, the main one being the cut-off wheel. An Al_2O_3 cut-off wheel selected according to the hardness of the ferrous metals is suitable for sectioning. This is usually a medium hard to soft cut-off wheel. For ferrous metals containing a large amount of carbides a CBN cut-off wheel is recommended.

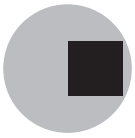
After cutting, the cut piece is rinsed with water, ethanol and dried to avoid corrosion.

Mounting

When mounting is required to facilitate handling during the mechanical preparation a conductive resin suitable for SEM (no charging) is selected, for instance the hot mounting material PolyFast containing graphite powder. In order to ensure good adhesion of the mounting resin to the specimen material it is essential to degrease the sample with alcohol and to dry it thoroughly and carefully with a blow dryer. The final mount should be high enough so that it can be used in the automatic grinding and polishing equipment, but at the same time not too high so that it still can fit into the SEM chamber. A recommended size is 25 or 30 mm in diameter and a height between 5-12 mm.

Practical Tips:

• Contrary to normal recommendations for mounting (risk of mount cracking), the specimen should be placed close to the edge of the mount to minimize the distance between the specimen and the electron beam in the SEM chamber.



• Specimens higher than 5-12 mm can be prepared and then cut down after the metallographic preparation is completed. The prepared surface must be protected during cutting (Fig. 2).

• If the mounted specimen is not high enough for the mechanical automatic preparation polishing equipment, a dummy specimen can be glued on the back of it using double adhesive tape which can easily be removed later on (Fig. 3).

• If non-conductive mounts need to be prepared and examined in EBSD, it is necessary to cover them with conductive adhesive tape, or better conductive cement, or to coat them with a conductive medium by a sputter coater or an evaporator. The specimen area should then be masked as the best results are obtained on a bare surface. As many adhesive metal tapes have non-conductive adhesive, the use of carbon/silver conductive cement may be required at seams.

• If a specimen mounted, in PolyFast or in non-conductive mounting materials, needs to be electrolytically prepared, a good electrical connection between the prepared surface and the anode of the polishing unit is necessary.

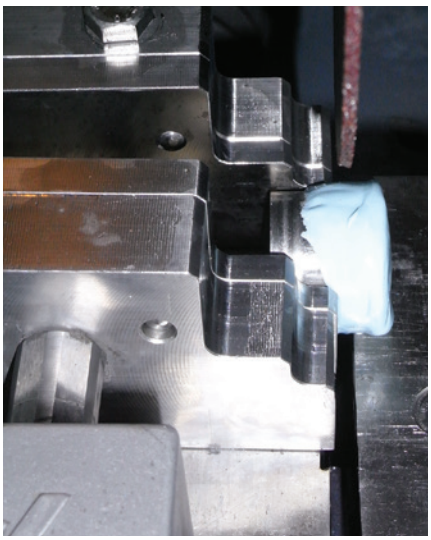


Fig. 2: Prepared surface of a specimen protected with RepliFix prior to cutting.

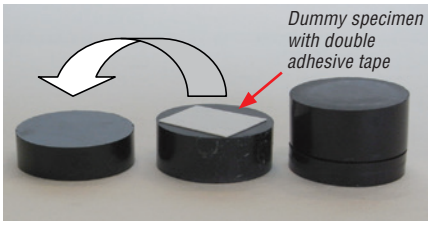
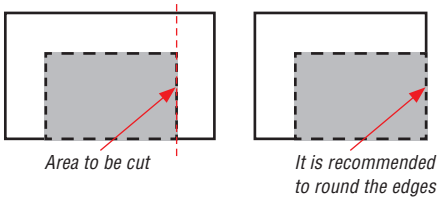


Fig. 3: A dummy specimen is glued with double adhesive tape onto the back of a specimen that is not high enough to be prepared automatically using a specimen mover plate for single specimens.



This can be achieved in different ways:
- By breaking the mount. This solution is not optimal as it can damage the prepared surface.
- Using silver glue. This solution is not optimal as heat is generated on the area of the silver glue.
- Cutting a part of the mount in such way that the specimen is accessible. Silver glue, conductive tape or foil of a conductive material can be used to ensure the electrical connection. Such specimen can be prepared on automatic preparation equipment prior to the electrolytic polishing. It is then recommended to round the cut edges to avoid excessive wear of the polishing cloths (see above drawings).

Handling of unmounted specimens
Round unmounted specimens of 25 or 30 mm in diam. are easy to handle. If the height is not sufficient, a dummy specimen can be glued on the specimen as mentioned earlier (Fig. 3).

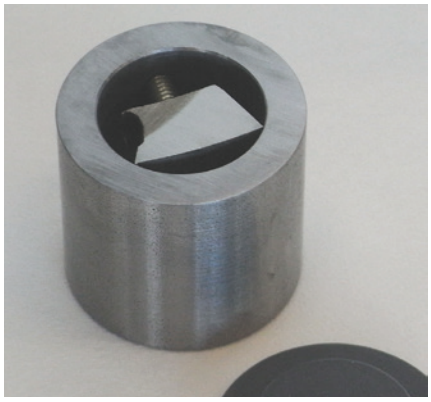


Fig. 4: Small, irregularly shaped specimen clamped in a holder.

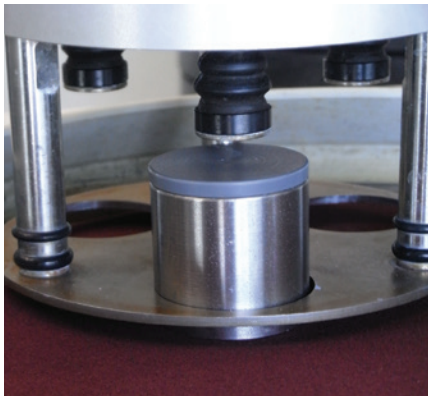
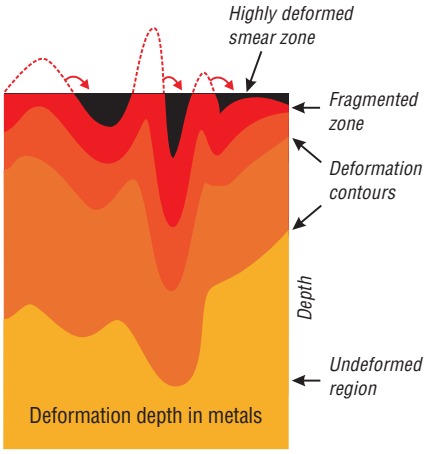


Fig. 5: Holder with specimen ready for polishing.



For small and irregular specimens the custom holder (Fig. 4) is very useful. The specimen can be clamped and prepared together with the holder. The pressure foot of the grinding and polishing machine will apply the necessary force through the top part (Fig. 5). Unmounted specimens and specimens clamped in such holder are easier to polish electrolytically as the electrical connection is best when it is in direct contact with the ferrous metal.

Grinding and polishing
Different metallographic processes can be selected for the preparation of ferrous metals for EBSD analysis, mechanical or mechanical/electrolytical. Satisfactory results for ferrous metals are obtained using conventional preparation procedures either mechanical or electrolytical. However a better contrast of the steel matrix is achieved using electrolytical polishing.

The deformation depth from a cut surface of ferrous metals is in the micrometer range. Depending on the hardness of the metal the range is around 100 - 300 µm. Enough material needs to be removed during grinding and polishing without introducing new deformation. A mechanical preparation followed by electrolytical polishing is the most efficient preparation process for homogeneous materials.

Mechanical preparation
For EBSD analysis of ferrous metals, it is recommended to use SiC Foil/Paper for plane grin-ding whatever the hardness of the specimen is. Furthermore very coarse SiC Foil/Paper grits and high pressures should be avoided as this introduces deep deformation. As a rule, the finest possible abrasive grain size should be used for plane grinding, in relation to the specimen area. Fine grinding is carried out with diamond on a rigid disc for maximum planeness, followed by a thorough diamond polish on a medium soft cloth with low force.

The final polish is carried out with colloidal silica (OP-S / OP-U) or Alumina (OP-A). The time of the final polishing step should be extended until a satisfactory result is obtained. It is pointed out that any deformation from the first grinding step which is not removed by fine grinding will leave its traces and cannot be removed by final polishing.

All the different ferrous metals presented in this paper were inspected using EBSD directly after mechanical or electrolytic polishing:

C45 steel
Stainless steel (X5CrNi18-10)
Tool steel (9CrWMn)
Nodular cast iron (GGG60)
Duplex stainless steel

Usually ferrous metals prepared for a materialographic purpose require contrasting of the structure for observation in the optical microscope. This contrast is mainly obtained by creating a relief between different structural elements of the specimen using chemical etching or by the formation of surface layers of different thicknesses. Sometimes etching is also used for the EBSD analysis, but very often the resulting contrast is too high and causes problems with shadowing, and oxide

Fig. 6: Medium size automatic grinding and polishing System Tegramin-25

Step		PG	FG	DP 1		DP 2	OP
Surface		Foil/Paper 320	MD-Largo	MD-Dur		MD-Nap	MD-Chem
Abra-sive	Type	SiC	Diamond	Diamond		Diamond	Collodial Silica
	Size	#320	9 µm	3 µm		1 µm	0.04 µm
Suspension / Lubricant		Water	DiaPro Allegro/Largo 9	DiaPro Dur 3		DiaPro Nap B 1	OP-S NonDry
rpm		300	150	150		150	150
Force (N)/ specimen		30	30	30	15	10	10
Time (min)		1	5-10	5	5	5	1-5

Basic mechanical preparation method for ferrous metals

This preparation method is the basis for mechanical preparation of ferrous metals for EBSD analysis.

layers can completely suppress diffraction. Therefore these contrasting techniques are not necessary for EBSD analysis of ferrous metals.

Only few and small pieces of a material are usually available for EBSD analyses. Therefore a small to medium size automatic preparation system is sufficient as shown in fig. 6.

The mechanical preparation methods described in this Application Note are for a single specimen of 30 mm in dia. prepared on an automatic preparation system with grinding and polishing discs of 200 mm in dia.

Note:
- As an alternative to DiaPro, polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with blue lubricant.
- DP 1 is run twice for 5 min with different forces.

Steels consisting of ferrite and cementite

The full process is suitable for steels consisting of ferrite and cementite in various proportions. The result obtained for a medium carbon steel (C45 steel) is illustrated in figs. 7-10.

Fig.7: C45 steel etched with 3 % Nital. The white phase is ferrite and the rest is pearlite. Optical microscope. Bright Field (BF), original magnification 200x.

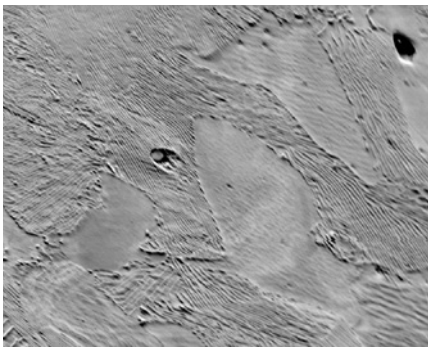


Fig. 8: Forescatter diffraction image of C45 steel. SEM, original magnification 2000x.

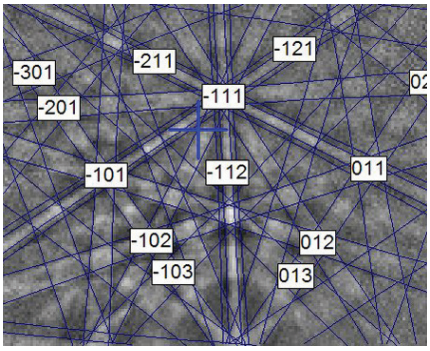


Fig. 9: EBSP of ferrite body cubic centered (bcc) in C45 steel, indexed.

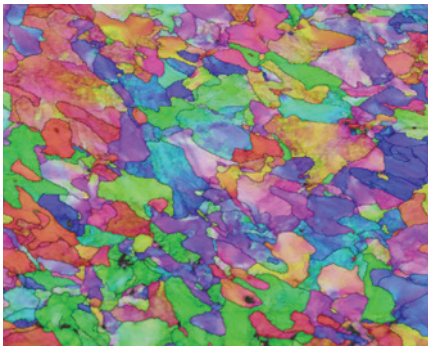


Fig.10: EBSD map of medium C45 steel using IPF colouring. For examination of grain size and grain orientation. Original magnification 500x.

Fig.16: Storage of prepared specimens in an desiccator

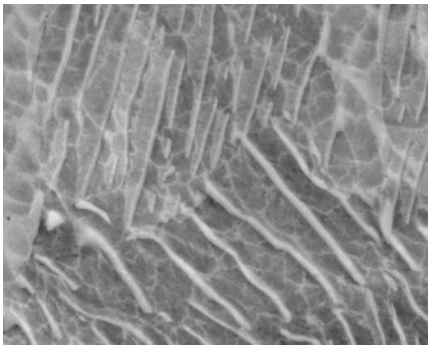


Fig.11: Pearlite (ferrite and cementite) in C45 steel, SEM, original magnification 20.000x.

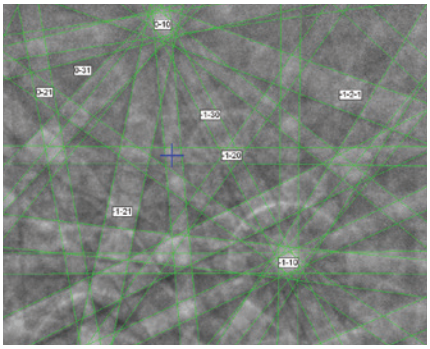


Fig.12: EBSP of cementite (Fe_3C) in C45 steel, indexed.

The constituents of pearlite (ferrite and cementite) can also be visualised on an EBSD using much higher magnification e.g. 20.000x. See figures 11 and 12.

High alloy steels

The basic mechanical preparation, with exception of the 1 µm polishing step, is also suitable for the preparation of high alloy steels having an austenitic structure only.

See the result obtained with a stainless steel X5CrNi18-10 (Figs.13-15) or of a combination of a ferritic and austenitic structure, see the result obtained with duplex stainless steel (Figs. 25-30).

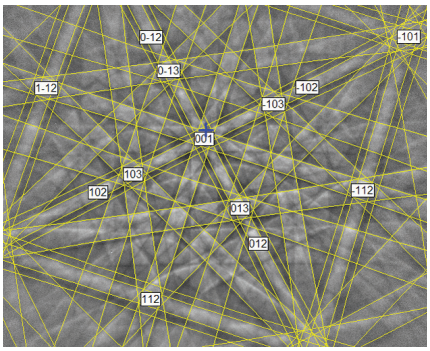


Fig.14: EBSP of austenite (fcc) in X5CrNi18-10, indexed.

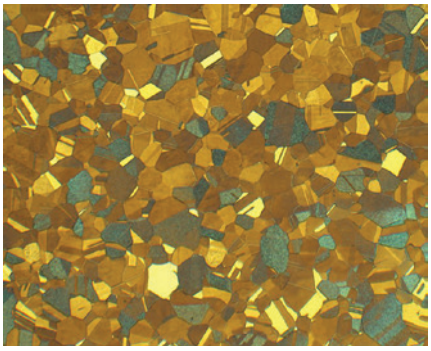


Fig.13: Austenite in a stainless steel X5CrNi18-10, colour etched with Beraha II. Optical microscope. BF, original magnification 200x.

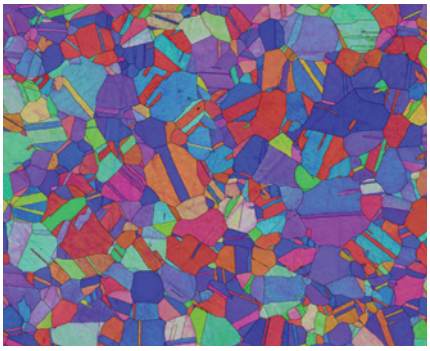


Fig.15: EBSD map of stainless steel X5CrNi18-10 (IPF colouring). For examination of grain size and grain orientation. Original magnification 200x.

Cleaning

Good cleaning practices are essential to get an optimal preparation result. Cleaning between individual preparation steps keeps the preparation surfaces free of unwanted cross-contamination with larger abrasive particles from previous preparation steps.

After the plane grinding step on SiC-Paper, the individual specimens and specimen mover plate are rinsed with water and dried with clean compressed air.

During the fine grinding and diamond polishing steps, diamonds are added to the grinding/polishing surfaces which leave a black deposit of abraded material on the individual specimens and the specimen mover plate. For cleaning, the individual specimens are sprayed with a neutral detergent, rinsed with plenty of water, then with ethanol and dried with clean compressed air.

After the final polish with colloidal silica or alumina, water is flushed onto the polishing cloth for 10-20 seconds, rinsing specimens and cloth.
Then the individual specimens are immediately washed with detergent rubbing the prepared surface very carefully with a thumb, rinsed thoroughly with water, sprayed with ethanol and dried with a strong stream of warm air. After each preparation step, the specimen mover plate is removed from the machine and cleaned as well.

Specimens should, under no circumstances, be left unattended with water remaining on the surface, especially not after the last polishing step. It causes stains, discolouring and corrosion that can affect the quality of the following analysis.

If the EBSD inspection cannot be carried out right after the preparation of a specimen is completed, the prepared surface must be protected to avoid an alteration of the surface (dust or dirt on the surface, scratching, formation of oxide layers, moisture). This is accomplished by spraying a protecting layer (Struers Protecting lacquer) removable with acetone or by storing the prepared specimen in a desiccator (Fig.16).

Other ferrous metals

The three first preparation steps of the basic preparation method described earlier are identical for all ferrous metals. Small changes introduced in the fine polishing steps improve the quality of the EBSD mapping of some specific applications.

Hardened steels

The distorted lattice of martensite makes it difficult to get a good quality EBSD. Good results are obtained on hardened plain carbon and low alloy steels containing various amounts of martensite using a long fine diamond polishing process. Alumina (OP-A) is prefered for final polishing to obtain a proper final result with a minimum amount of relief. The result obtained for a tool steel (9CrWMn) is illustrated in Figs.17-20.

Note:
Depending on their size and nature, second phase and inclusions in steel can also be recognized by EBSD. Some examples of constituents found in steels by EBSD include iron carbides, boron carbides, silicon carbides, chromium carbides, titanium carbo-nitrides, manganese sulphide, iron oxides.

Hardened steel								
Step		**PG**	**FG**	**DP 1**		**DP 2**	**DP 3**	**OP**
Surface		Foil/Paper	MD-Largo	MD-Dur		MD-Nap	MD-Nap	MD-Chem
Abra-sive	**Type**	SiC	Diamond	Diamond		Diamond	Diamond	Alumina
	Size	#320	9 µm	3 µm		1 µm	¼ µm	
Suspension / Lubricant		Water	DiaPro Allegro/Largo 9	DiaPro Dur 3		DiaPro Nap B 1	DiaPro Nap ¼	OP-A
rpm		300	150	150		150	150	150
Force (N)/ specimen		30	30	30	15	10	10	10
Time (min)		1	5-10	5	5	5	5	5

Note: - As an alternative to DiaPro, polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with blue lubricant.
- DP 1 is run twice 5 min with different forces.

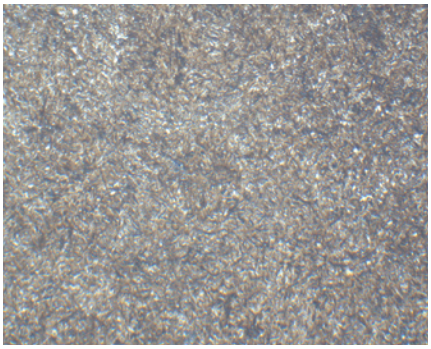


Fig.17: Martensite in a tool steel (9CrWMn) etched with 3% Nital. Optical microscope. BF, original magnification 500x.

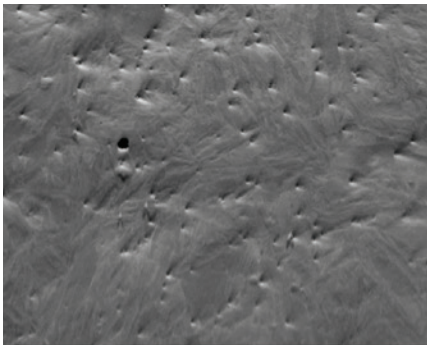


Fig.18: Forescatter diffraction image of martensite in a tool steel (9CrWMn) SEM, original magnification 1500x.

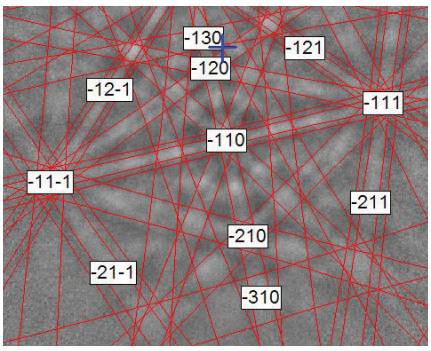


Fig.19: EBSP of martensite (bcc) in a tool steel (9CrWMn), indexed.

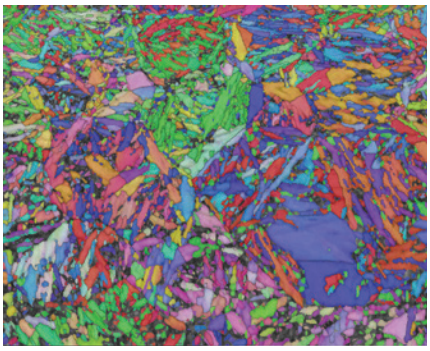


Fig. 20: EBSD map for examination of crystalline structure in a tool steel (9CrWMn). IPF colouring. Original magnification 1500x.

Grey and nodular cast irons

The main difficulty when preparing grey and nodular cast irons is to retain the soft graphite in the form of flakes or nodules in their true shape and size. The fine diamond polishing is therefore carried out using a low resilience polishing cloth MD-Dur and OP-U NonDry for final polishing to ensure an optimal planeness of the graphite (this process is also useful when preparing steels with inclusions and carbides). The result obtained for a nodular/spheroidal graphite cast iron GGG60 is illustrated in Figs. 21-24.

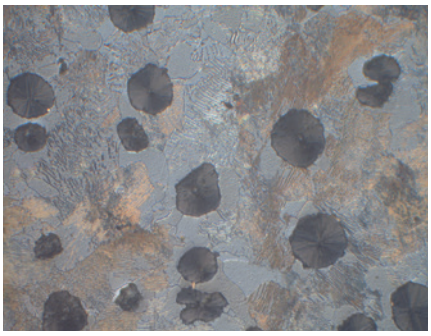


Fig. 21: Nodular cast iron, GGG60 etched with 3% Nital. Ferrite is light grey, the graphite nodules very dark grey, and the rest is pearlite (dark grey/brownish). Optical microscope, DIC, original magnification 500x.

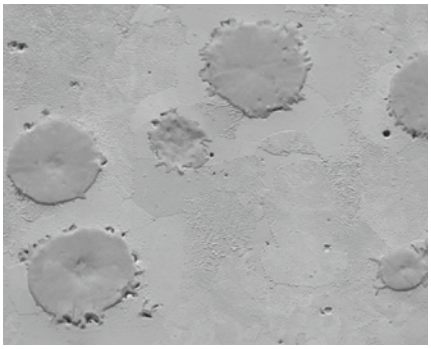


Fig. 22: Forescatter diffraction image of nodular cast iron, GGG60. Note the preserved graphite nodules. SEM, original magnification 750x.

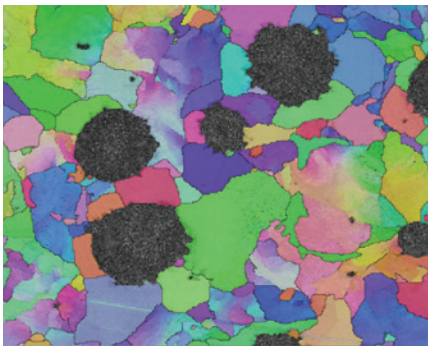


Fig. 23: EBSD map of cast iron GGG60 (IPF colouring). For examination of grain size and grain orientation. Original magnification 750x.

Grey cast irons							
Step		**PG**	**FG**	**DP 1**		**DP 2**	**OP**
Surface		Foil/Paper	MD-Largo	MD-Dur		MD-Nap	MD-Chem
Abra-sive	**Type**	SiC	Diamond	Diamond		Diamond	Collodial Silica
	Size	#320	9 µm	3 µm		1 µm	0.04 µm
Suspension / Lubricant		Water	DiaPro Allegro/Largo 9	DiaPro Dur 3		DiaPro Nap B 1	OP-U NonDry
rpm		300	150	150		150	150
Force (N)/ specimen		30	30	30	15	10	10
Time (min)		1	5-10	5	5	5	1-2

Note:
- As an alternative to DiaPro, polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with blue lubricant.
- DP 1 is run twice for 5 min. with different forces.

This preparation process allows an EBSD inspection of the specimen's matrix and shows well retained graphite without excessive relief. However, the nature of graphite, even after a long final polishing, does not permit EBSD to set up an automatic run of the nodules.
The EBSPs of graphite are of poor quality. Nevertheless Fig. 24 shows that it is possible to index graphite.

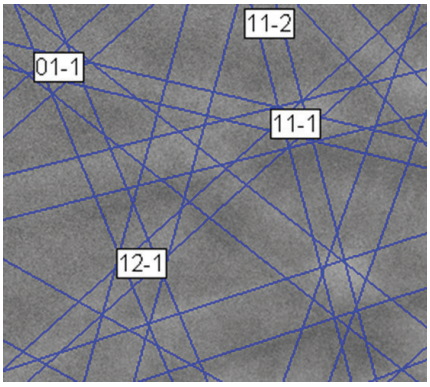


Fig. 24: EBSP of hexagonal close packed (hcp) graphite in cast iron GGG60, Indexed.

Electrolytical polishing

Instead of mechanical preparation, electrolytic polishing is often a good alternative preparation technique for ferrous metals as it is fast (few seconds) and does not leave any mechanical deformation.

Electrolytic polishing involves the removal of material (anodic dissolution) of the specimen surface in an electrolytic cell, the specimen being the anode.

Electrolytic polishing requires that the structure of the specimen is relatively homogenous. For ferrous metals having several different phases (meaning different electrochemical potentials), one of the phases will be preferentially polished compared to another and therefore result in excessive relief or damage of this phase.

The automatic polishing equipment LectroPol-5 controls most of the parameters involved in an electrolytic process for a specific metal, and the software keeps records of the parameters: voltage, size of polishing area, electrolyte, temperature of the electrolyte, flow rate and the process time.

Prior to the electrolytic polishing a pretreatment of the surface to be polished is required. The specimen is first ground on SiC Foil/Paper e.g. #320, #500 and #1000 for a minute on each paper. Less relief and better flatness can be obtained when the specimen is mechanically polished down to a 3 µm step following the basic preparation method.

Electrolytic preparation for most steels

EBSD patterns of good quality can be obtained by polishing plain carbon steel and low alloy steels electrolytically using the data shown in Table 1.

This process can also be used for revealing the matrix of grey irons and steels containing inclusions or carbides. Because of their different electrochemical potentials these constituents will not be polished but washed out and the places they occupied will appear enlarged.

However, for the EBSD analysis of martensite, electrolytical polishing is not suitable. It can not be avoided that during the polish the structure is also etched by the electrolyte. The resulting contrast is too pronounced so that a good EBSD pattern cannot be obtained because of shadowing.

Electrolytic preparation method for high alloy steels

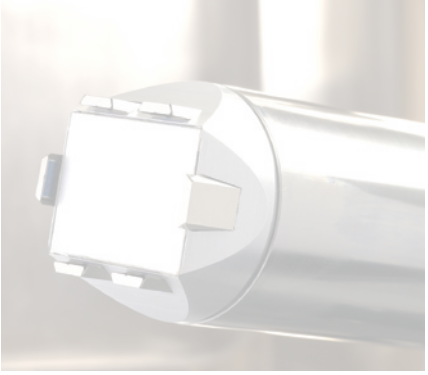
EBSD patterns of a very good quality can be obtained by polishing high alloy steels electrolytically, using the data shown in Table 2.

LectroPol-5, automatic electrolytic polishing and etching equipment.

Table 1 Plain carbon steel, low alloy steel	
Electrolyte:	A2*
Area:	1 cm2
Voltage:	40 V
Flowrate:	14
Time:	12 s

Table 2 High alloy steels	
Electrolyte:	A3*
Area:	1 cm2
Voltage:	35 V
Flowrate:	13
Time:	25 s

*For the electrolyte formula, please contact your Struers representative.



Mechanical preparation compared to electrolytical preparation

Even though the surface of the specimen looks very different in the SEM after the same material is prepared electrolytically or mechanically, EBSD maps of high quality are obtained with both surface finishes. Figs. 26 and 27 of a duplex stainless steel specimen illustrate the differences.

A result with nearly zero deformation and a minimum of relief can be obtained using a suitable mechanical preparation for almost all ferrous steels. Structural elements as graphite, inclusions and carbides can then be analysed as well using EBSD. However it is a time consuming process of 25-35 min. On the other hand, electrolytic polishing is fast and gives a higher contrast, showing the specimen's matrix after a total time of about 5 min.

Duplex stainless steel

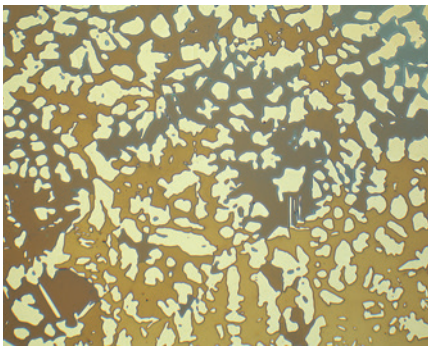


Fig. 25: Duplex stainless steel etched electrolytically with 40% aqueous sodium hydroxide solution, showing white austenite (fcc), and yellowish and bluish ferrite (bcc). Optical microscope, BF, original magnification 200x.

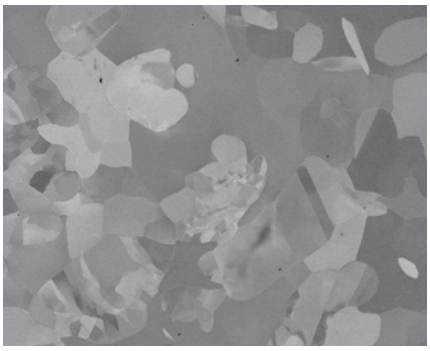


Fig. 26: Duplex stainless steel ground with SiC-Paper up to 1000# followed by an electrolytic polishing. SEM, original magnification 800x.

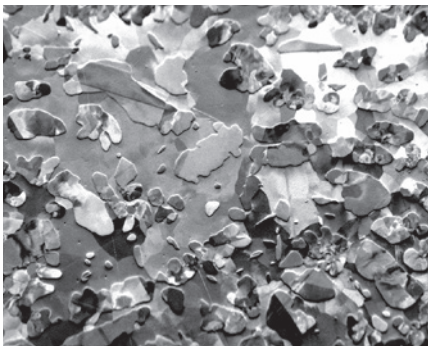


Fig. 27: Duplex stainless steel after mechanical polishing down to OP-S. SEM, original magnification 800x.

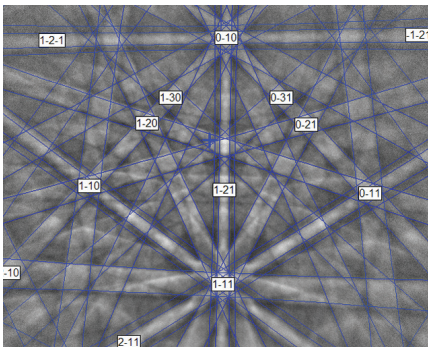


Fig. 28: Indexed EBSP, bcc crystal structure, ferrite in duplex stainless steel.

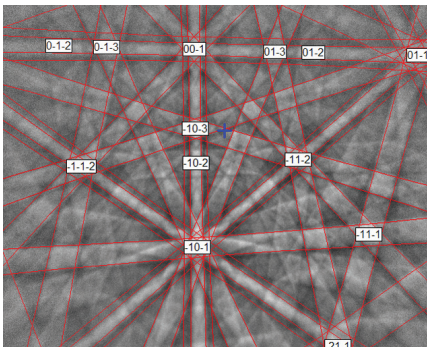


Fig. 29: Indexed EBSP, fcc crystal structure, austenite in duplex stainless steel.

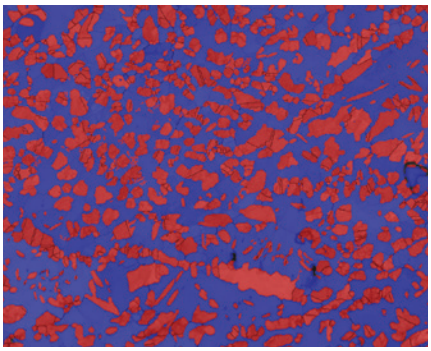


Fig. 30: Phase map of duplex stainless steel showing the fcc phase in red and the bcc phase in blue. Original magnification 800x.

Summary

The variety of information gained using EBSD analysis is vast: evaluation of grain size; analysis of the shape, distribution, size, boundary, deformation, orientation and identification of phases, inclusions, carbides and precipitates.

The described examples in this Application Note illustrate some of the applications of EBSD analysis of ferrous metals. The EBSD technique is applied for many other specific analyses of specimens from this material group, for instance: evaluation of sigma phase in high alloyed steels, inclusion rating in steels, evaluation of retained ferrite and austenite measurement in steels.

Whatever the purpose of an EBSD analysis, it requires a specific, careful preparation of specimens and specific solutions to ease handling of specimens by mounting or using some clamping devices, as the specimen's size is limited by the capacity of the SEM chamber.

For a successful mechanical polishing, it is suggested that:
- coarse abrasives for plane grinding are avoided.
- fine grinding and polishing, with diamond abrasive, should be thorough in order to remove all deformation from plane grinding.
- a final oxide polish with colloidal silica is carried out to provide a nearly deformation free surface.

Electrolytic polishing, which gives a deformation free surface, is a fast and good preparation technique for homogeneous ferrous metals.

Suitable mechanical and electrolytical metallographic preparation processes of ferrous metals for Electron Backscatter Diffraction (EBSD) analysis are described. For optimal results, a combination of both preparation techniques, combined with good laboratory practices, a thorough cleaning and proper protection of prepared surfaces are recommended.

Glossary

[1] **EDS:** Energy dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. As a type of spectroscopy, it relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing x-rays emitted by the matter in response to being hit with charged particles. Its characterization capabilities are in large part due to the fundamental principle that each element has a unique atomic structure allowing x-rays that are characteristic of an element's atomic structure to be identified uniquely from each other.

[2] **Bragg:** Bragg condition: The Bragg condition defines the conditions for diffraction to occur. This can be expressed as a formula:

$$n\lambda = 2d_{hkl} \sin\Theta_B$$

- Θ_B is the Bragg angle
- n is an integer and defines the diffraction order, e.g. first.
- d_{hkl} is the interplanar spacing for the (hkl) plane
- λ is the wavelength of the incident radiation

For SEM electrons, the wavelength (Å) is

$$\lambda = \frac{0.387}{\sqrt{kV}}$$

where kV is the acceleration voltage (kV).

[3] **Kikuchi bands:** Kikuchi bands are linear features that appear in an EBSP (Kikuchi pattern). They correspond to a difference in electron intensity from the background level. The width of a Kikuchi band is twice the Bragg angle for the relevant plane.

[4] **Hough transform:** The automated detection of Kikuchi bands in the CHANNEL acquisition software is based on a Hough transformation. The Hough transform maps the EBSP image (X,Y) into Hough space ("theta", "distance") by calculating the average intensity along lines inclined at an angle "theta" and displaced from the centre of the image by "distance". A point in the EBSP transforms into a sinusoid in Hough space, a thin line transforms into a point. A Kikuchi band of width d transforms into a pair of local maxima (or minima) that are separated by a distance d. A butterfly filter is used to identify these maxima.

[5] **IPF:** The inverse pole figure map component allows the crystallographic orientations to be quickly interpreted in terms of sample coordinate system (e.g. rolling direction).

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Application Note

Preparation of ferrous metals for Electron Backscatter Diffraction (EBSD) analysis

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Structure 38 Preparation for Electron Backscatter Diffraction with LaboPol-4/Labo-Force-1, Danka Katrakova, Morten J. Damgaard and Frank Mücklich

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Journal of Microscopy, 213, 3 (March 2004) 13 papers on the EBSD technique

Quantitative metallography by electron backscattered diffraction, F.J. Humphreys, Journal of Microscopy, V195, Pt 3 Sept 1999, P170-185.

Reference to equipment used

EBSD: NORDLYS S detector and the CHANNEL5 software

SEM: Zeiss LEO Supra55 VP - field emission type

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