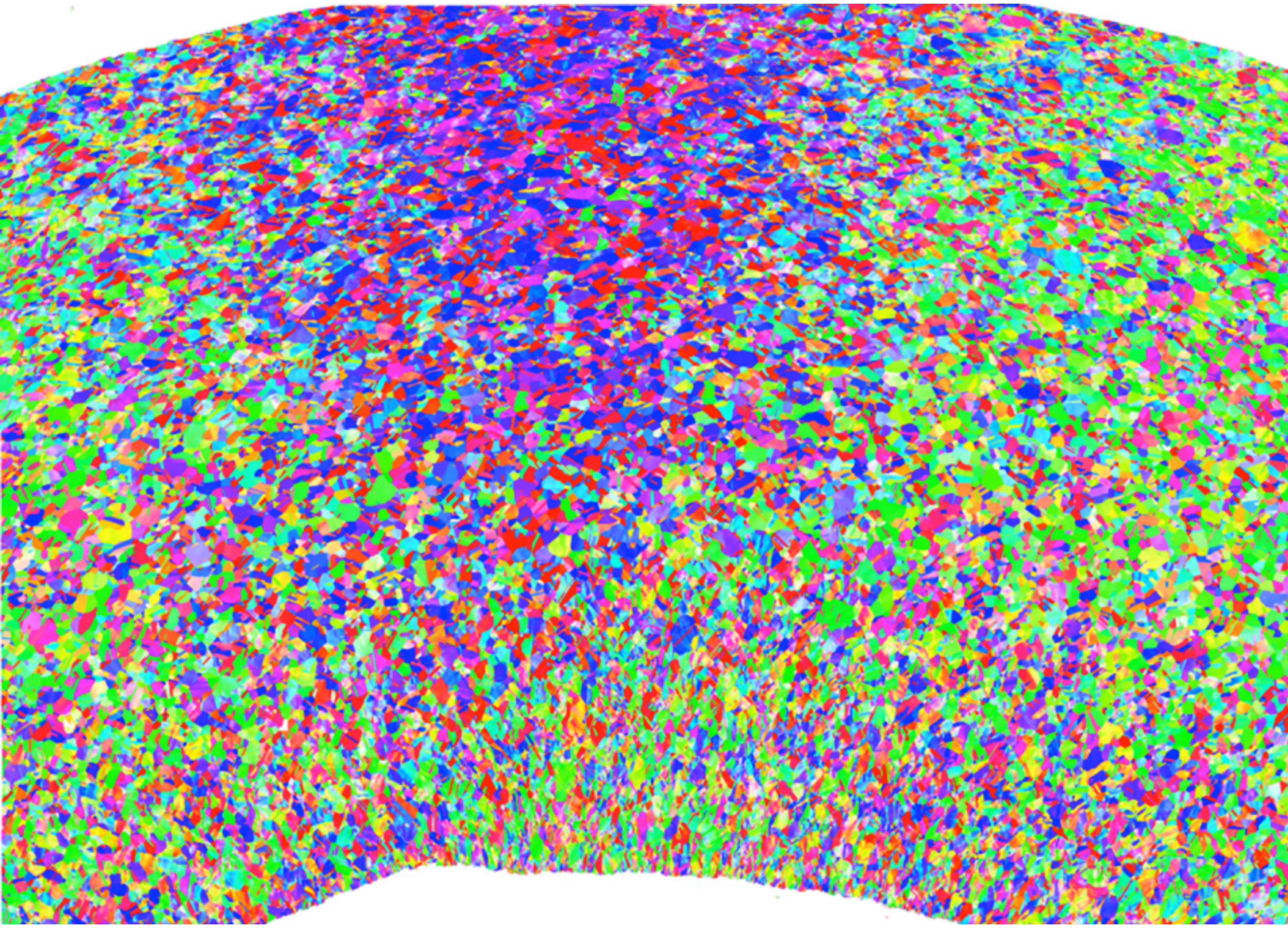

Electron Backscattered Diffraction (EBSD) for assessment of metals and alloys

Solutions from ZEISS for Advanced Analytical Microscopy

Electron Backscattered Diffraction (EBSD) for assessment of metals and alloys

Solutions from ZEISS for Advanced Analytical Microscopy

Author: Dr. Roger Barnett
Carl Zeiss Microscopy, Cambridge UK

Date: January 2018

Electron backscatter diffraction (EBSD) is a powerful characterization method for the microstructural study of materials using a ZEISS scanning electron microscope (SEM). An electron beam interacts with a polycrystalline/crystalline sample, giving rise to backscattered electrons. The generated diffraction patterns contain pairs of dark and bright lines, named Kikuchi bands (Figure 1). Effectively, these dark and bright lines are projections of the lattice plane geometry of the grain targeted by the electron beam. Combining indexed Kikuchi bands with crystallographic structural information on the relevant materials yields information on the crystallographic orientation, as well as identification of individual phases.

Introduction

As diffracted electrons arise from the first few nanometers of the crystal lattice, sample preparation is critical. Ideally, there should be minimal sample deformation, no mechanical damage, and low lattice distortion within the first 15 nm. Removing any significant damage (e.g., large scratches) first, then applying standard metallographic mounting and grinding/polishing methods (to a 0.25 µm finish) are sufficient for initial preparation. A subsequent finer preparation stage is needed to achieve a suitable finish, with a contamination-free surface. Vibratory polishing with colloidal silica, electro-polishing, or broad argon ion beam milling are methods that yield a surface with minimal relief and etching.

Scanning the electron beam over a defined region, when combined with crystal structure information, generates a map of grain orientation. These maps contain information on grain size, grain boundaries, phase identification, phase distribution, and orientation relationships between phases.

Grain orientation maps can have an extremely high lateral resolution (in 2D), higher even than energy-dispersive X-ray spectroscopy (EDS). This capability is particularly useful for definitive identification of the phase of very small grains (<0.2 µm). 2D lateral resolutions may be extended to 3D by employing a focused ion beam (FIB) in combination with serial sectioning of the EBSD maps generated for each section. Using an *in situ* testing stage allows assessment of microstructure at various points during heating, cooling, or the application of mechanical stress.

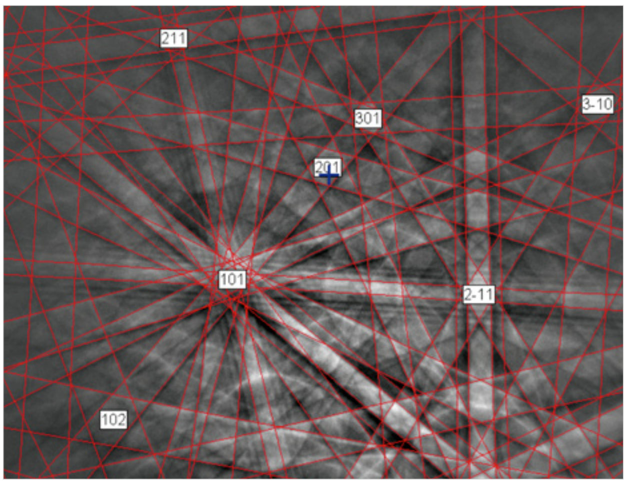

Figure 1 *Example electron backscattered diffraction (EBSD) pattern, showing indexed Kikuchi bands.*

General Microstructural Characterization

The microstructure of a metal or alloy, whether based on iron, nickel, aluminum, copper, or any other element, strongly influences the bulk properties. These properties include strength, hardness, ductility, formability, toughness, and corrosion behavior, among others. As examples: more martensite in steel microstructures leads to increased hardness; smaller grain size leads to increased yield strength due to grain boundaries impeding dislocation movement; carbide precipitations along grain boundaries may lead to higher susceptibility to corrosion in stainless steels.

Light microscopy of etched structures gives a broad range of information – including grains, precipitate/inclusions, sensitivity to corrosion, phase distribution, and mechanical damage. Secondary electron imaging offers high-resolution information on surface topography. Backscattered electron imaging will give contrast based on atomic number. Quantitative elemental analysis is available using energy dispersive X-ray spectroscopy (EDS) for most elements. Correlating quantitative and qualitative information from all of these techniques significantly aids understanding of bulk properties and performance.

However, none of the above techniques gives information on grain orientation, localized stresses/strains, nor quantitative identification of grains too small for EDS. Figure 2 displays the same sample assessed using both light microscopy and EBSD. Light microscopy shows stronger etching along the grain boundaries, indicative of a number of possible effects including sensitization, local compositional variation at the boundaries, or residual stress or strain in the central region, with the grain boundaries more strongly affected by etching. However, the ~125 µm-thick central band of the material shows a clear pattern of crystallographic orientation by EBSD, likely due to some combination of thermal history, rolling, or mechanical processing/deformation.

Electron Diffraction

Optical Polarized

Optical Etched

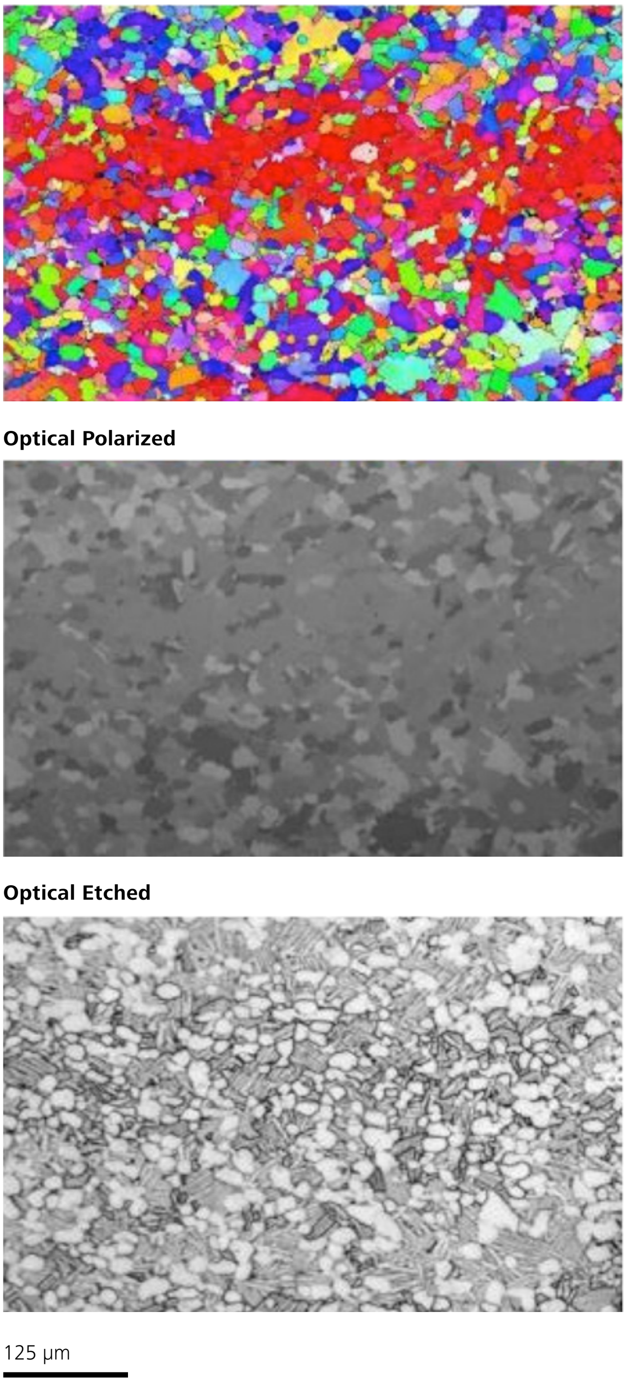

Figure 2 *Proprietary aircraft alloy imaged using three different microscopic techniques. Courtesy: Swansea University, U.K.*

In situ EBSD of microstructural transformations

Austenite is a face-centered cubic (fcc) form of iron/steel, stable at higher temperatures with a high solubility for carbon. Ferrite is a body-centered cubic (bcc) form of iron, which has lower carbon solubility, stable at lower temperatures. Typically, ferrite occurs in conjunction with pearlite – a two-phase lamellar structure of alternating ferrite and cementite (Fe_3C) layers. Upon heating past the lower critical point, ferrite and pearlite transform to austenite, with the carbon starting to dissolve into the austenite solid solution. Heating past the higher critical temperature is needed to ensure complete transformation. Upon cooling, the fully austenitized material may transform into various iron-carbon microstructures – ferrite, pearlite, martensite, bainite or a mixture of the above, dependent on steel composition and the cooling rate. Controlling these phase transformations is key to managing microstructural properties and bulk properties.

EBSD can visualize these microstructural transformations in steel *in situ*, by use of a temperature-controlled stage. Placing a prepared steel sample upon a proprietary heated stage EBSD patterns were taken during heating up to 945°C (to fully austenitize the material) then subsequently cooled below the critical temperature, Figure 3. Grain recrystallization upon cooling is visible, with a coarser ferrite structure forming. Understanding these transformations leads to control of steel grain size, identification of ideal quench points, and assessment of the impact of different heat treatments on a particular steel – annealing, tempering, normalization, or quenching.

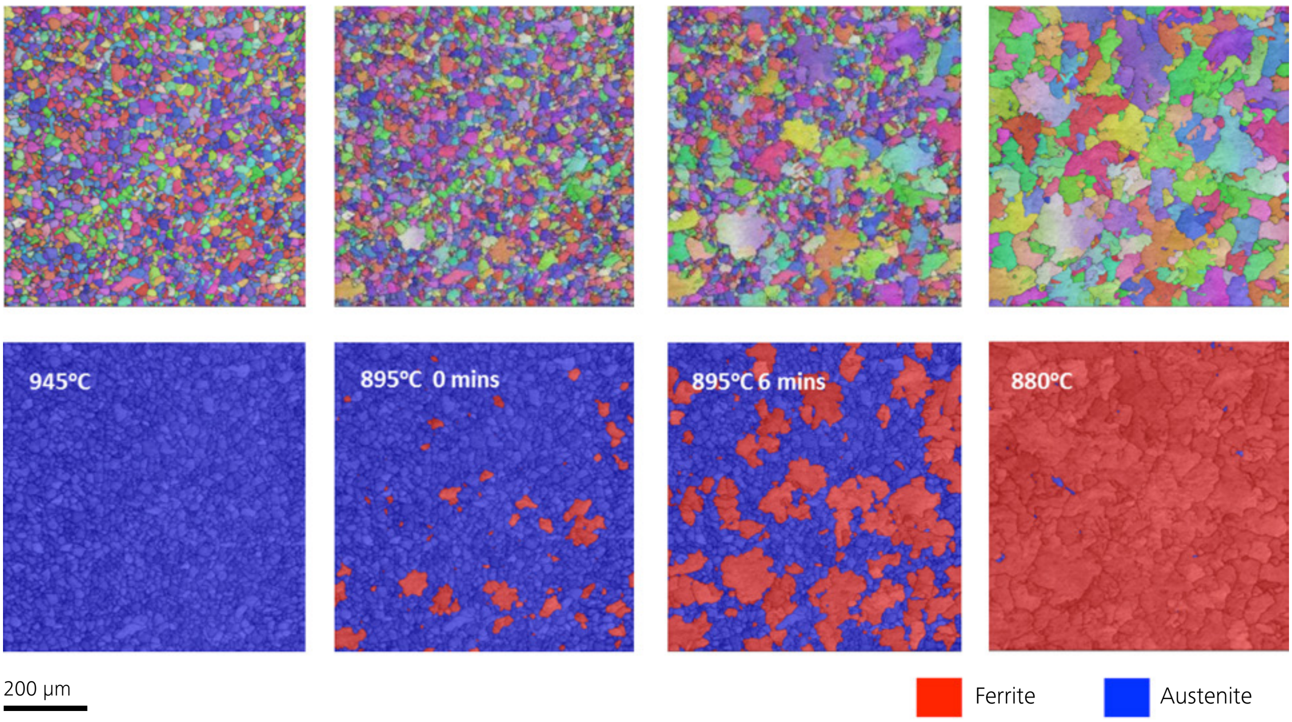

Figure 3 In situ *EBSD of steel (during heating to 945°C and subsequent cooling) showing ferrite recrystallization. EBSD data provided by Oxford Instruments Nanoanalysis.*

EBSD for analysis of texture and strain during bending/forming and heat treatment

Determination of plastic strains and misorientations in metals is another use for EBSD. Reference data is generated either by computer simulation of EBSD patterns or by measurement of a material/region known to be free of residual stress or strain. Comparing diffraction patterns from strained material with the reference allows for identification of equivalent plastic strains. Distinguishing between recrystallized and deformed grains, or identifying misorientation between grains caused by this plastic straining are examples of what this method offers.

Identification and location of residual strains in components provide a greater understanding of failure mechanisms and corrosion performance in service. This information can be used for detailed modeling of components and improved component design. Indirect observation of strain is even possible by use of the ZEISS AsB detector, which can separate backscattered electrons by both Z contrast and angle contrast, showing evidence of dislocations.

The majority of metallic materials are polycrystalline. Typically, the orientation of grains is not random. Clustering close to particular orientations is present – referred to as texture. Texture is a function of the material and its history, including solidification, heat treatment, and mechanical deformation. In particular, the formability and corrosion resistance of sheet materials are often dependent on the texture. Differences in grain size, as well as local stress/strain, can lead to preferential pitting corrosion or even stress corrosion cracking in service. EBSD is, therefore, a vital tool for visualizing texture and monitoring how different processing steps affect it. Example EBSD maps of folded 2 mm thick Ni200 (>99% pure nickel, fcc), and mild steel (bcc) sheets indicated misorientations of up to 3° in regions under strain. There was no significant difference between regions under compression or tension. The central un-strained band had much lower levels of misorientation (<1°), and this band was wider in steel than nickel, Figure 4.

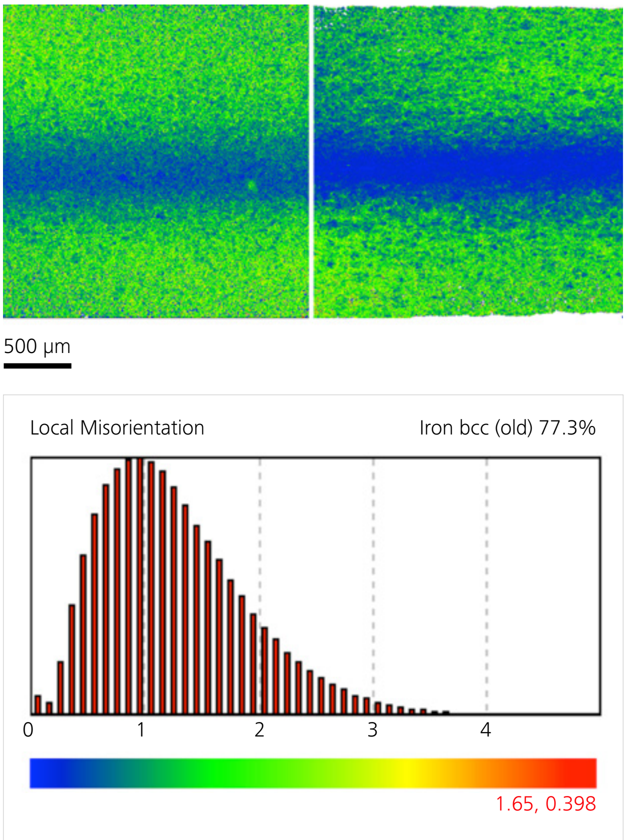

Figure 4 *EBSD map of cross-sections through Ni200 (left) and mild steel (right) sheet bending, showing grain misorientations in strained regions. A color scale of misorientation is provided. EBSD maps and data provided by Oxford Instruments Nanoanalysis.*

Applying a heat treatment to these folded sheets leads to grain growth by recovery/recrystallization in both materials. The nickel grains grew from 20 µm to ~40 µm in the strained regions and up to 100 µm in the center. For steel, the pattern reversed; grains grew from 15 µm to 23-30 µm in the strained regions but did not grow significantly in the center of the fold, leaving a band of relatively unchanged microstructure, Figure 5.

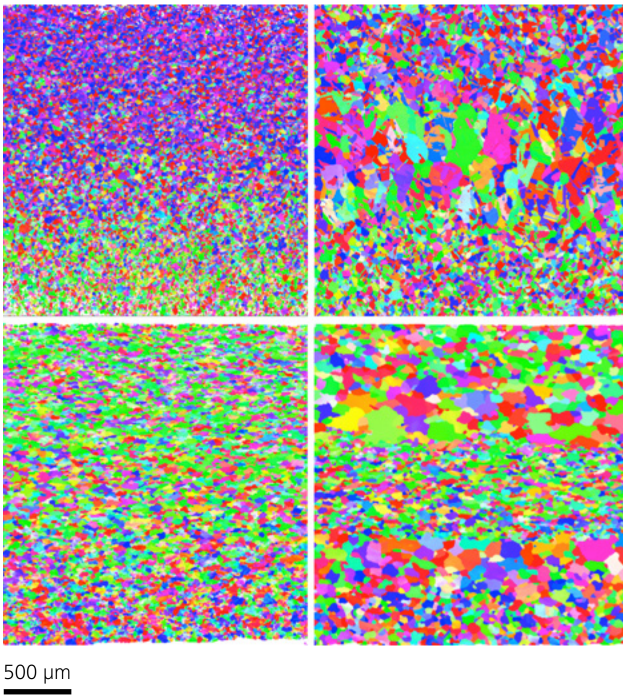

Figure 5 *EBSD maps of cross-sections of through-folded Ni200 and mild steel sheet. As-folded sheet (left), same sheet after heat treatment (right), at 600°C for 40 minutes. EBSD maps and data provided by Oxford Instruments Nanoanalysis*

Quality control of metal-metal joints

The ability to make strong connections between metallic materials is key to modern industry, from the very large (vehicles, buildings, pipelines) to the very small (electronics). In most cases, the weakest points of any structure are the joints. Joining behavior and joint properties must be thoroughly understood, and strict quality control observed to prevent catastrophic failure. Figure 6 shows an example of EBSD mapping to understand texture and solidification in a metal-metal weld.

As an additional example, duplex and superduplex stainless steels are specialized steel grades for high-performance applications. The amounts of alloying elements (e.g., Cr, Mn, Ni, Mo) lead to a mixed microstructure of austenite and ferrite. Compared to austenitic stainless steels, they have higher strength and improved resistance to localized attacks such as stress corrosion cracking or pitting. The austenite-ferrite ratio is typically 50:50 though in some alloys, the ratio may be closer to 40:60. In weld metal this ratio can further vary, affecting properties and corrosion resistance. Both destructive and non-destructive methods are available for characterization of joints in duplex stainless steel structures. Only EBSD gives information on the crystallographic orientation of grains at the same time as the ferrite-austenite distribution.

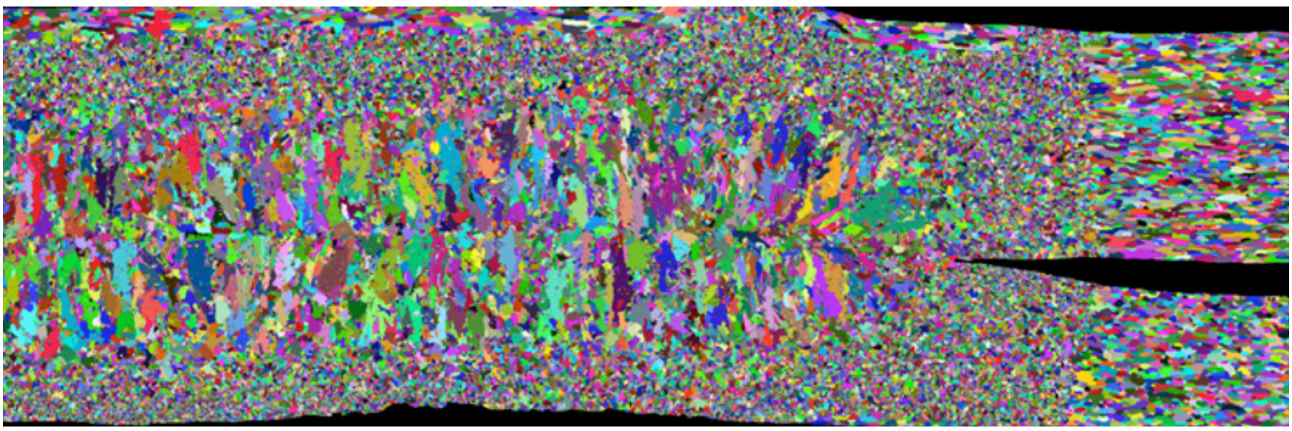

Figure 6 *EBSD map of a weld between two metallic sheets, provided by Oxford Instruments Nanoanalysis*

A large area of welded duplex stainless steel is mapped in Figure 7, showing the distribution of austenite and ferrite in the weld, heat-affected zone, and parent material. Too little ferrite leads to lower resistance to stress corrosion cracking, too much may lead to a loss of ductility and toughness (depending on application/grade) and thus control of this ratio is key. Figure 8 shows matching orientations between individual austenite grains arranged in clusters in a subset specified in Figure 7.

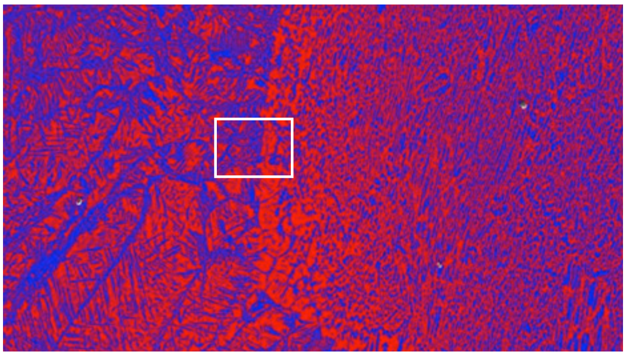

Figure 7 *Composite EBSD map of duplex stainless steel weld, formed from 96 individual maps with 50 million data points. Austenite (fcc) is in blue, ferrite (bcc) in red. Image provided by Oxford Instruments Nanoanalysis*

Recent Developments in EBSD

Of recent developments in EBSD, the primary one is the increased speed and resolution of commercially available EBSD detectors. As an example, Oxford Instruments' Symmetry EBSD Detector is capable of detection speeds of over 3,000 patterns per second, capturing 30 times as many data points as a conventionally sensitive CCD EBSD detector.

Acquiring a crystallographic map of a 300x300 μm area (1.4 million points, 0.25 μm step) of a deformed nickel superalloy can now be achieved in under 8 minutes with 99.4% correct indexing. Earlier generations of detectors could take several hours to generate a map of the same area at much lower resolution. Corresponding increases in computer processor power and data analysis have added to this improvement, bringing EBSD closer to near real-time.

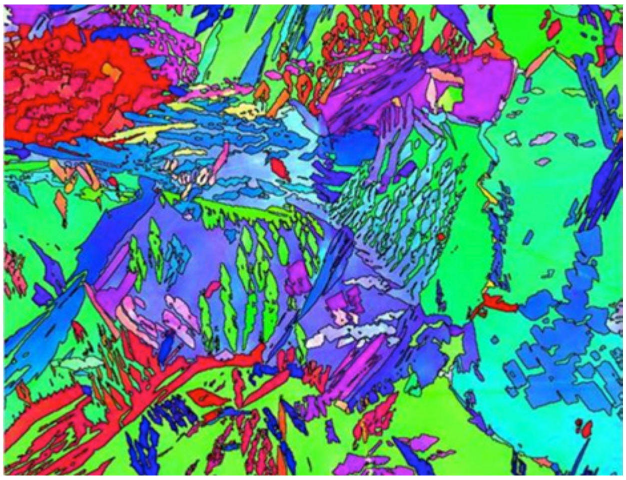

Figure 8 *Higher resolution EBSD map of the region marked in Figure 7. Crystallographic orientation is indicated by color. Image supplied by Oxford Instruments Nanoanalysis*

Ion-beam milling (separately, or *in situ*, via FIB-SEM) is becoming more widespread and practical, allowing reliable preparation of small areas for EBSD. Analysis of multiple sequential slices enables full 3D EBSD mapping. Advanced electron microscope stages permit a wide variety of *in situ* EBSD under conditions of extreme temperature and different mechanical loadings. This is ideal for alloy development, high temperature performance testing and many other applications.

Transmission Kikuchi Diffraction (Figure 9) is another relatively recent development related to EBSD. The electron beam passes through thin foil samples rather than reflecting from bulk materials. Existing EBSD detectors capture transmitted electron diffraction patterns, using tilts of 0-20°. Spatial resolutions of ~2-5 nm are viable, comparable to nano-diffraction in a TEM, with the capture of indexable patterns from particles <10 nm. This result is an order of magnitude greater than EBSD.

Correlative microscopy has also grown in importance. Features visible using different imaging techniques and at different length scales must be correlated so the data can be used most effectively. ZEISS Shuttle & Find software allows the location of features by standard or confocal light microscopy, then rapid re-location of these same regions in the SEM. ZEISS Atlas 5 software offers the import and alignment of data from disparate sources – light microscopy, X-ray microscopy, electron microscopy – into a single 3D data set. The user can build a single consistent picture of the sample with data at multiple length scales, ideal for research, quality assurance or even visualization of complex components.

Column development is another significant step forward in EBSD. The Gemini optics found in the columns of all FE-SIMs and FIB-SEMs in the Sigma, Gemini, and MERLIN series of electron microscopes from ZEISS offer superior low-distortion EBSD patterns. Even at low voltages, it is possible to obtain extremely high-resolution images and diffraction patterns using the novel Nano-twin lens of the GeminiSEM 500 (which significantly reduces chromatic and spherical aberration) and applying a deceleration voltage on the sample. Imaging resolutions of ~0.8 nm are possible at 1 kV. There is no magnetic field below the lens in the Gemini column. This ZEISS design feature enables distortion-free EDS and EBSD analysis for large fields of view, as shown in Figure 10.

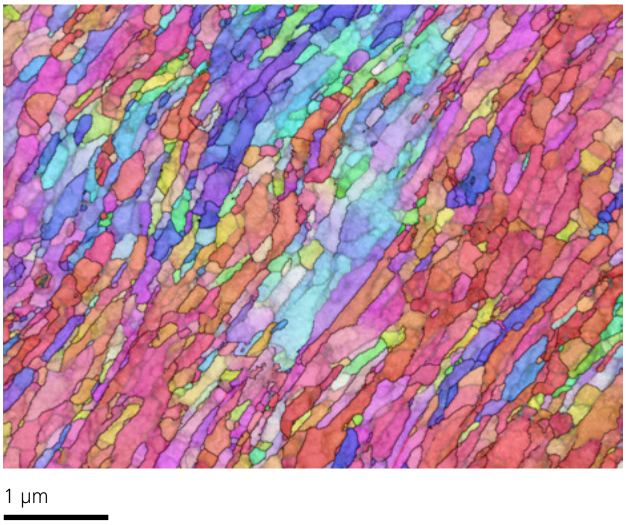

Figure 9 *Transmission Kikuchi Diffraction scan of heavily deformed Al-Mg-Cu alloy. Image provided by Oxford Instruments Nanoanalysis*

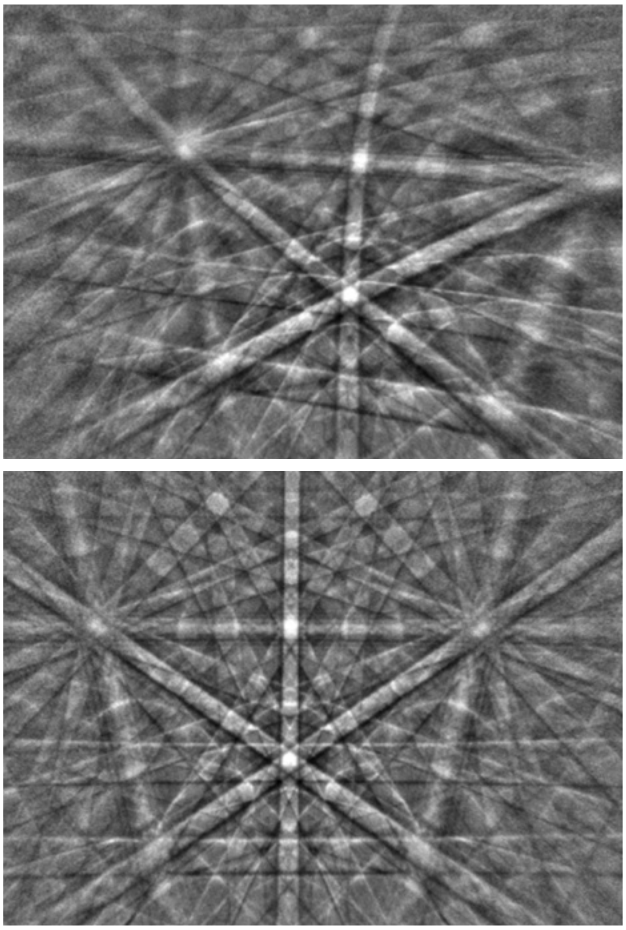

Figure 10 *EBSD pattern obtained under conventional high field lens (top) and under the Gemini lens designed by ZEISS (bottom).*
EBSD patterns provided by Oxford Instruments Nanoanalysis

Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/microscopy