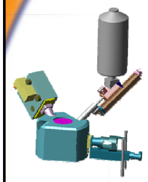
Integrated EBSD and EDS in the Scanning Electron Microscope

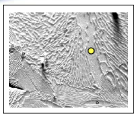


EDAX TSL
advanced microanalysis solutions
AMETEK

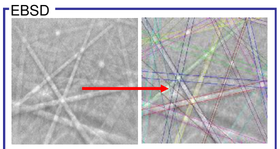
Outline

- **Integrated EBSD & EDS for Phase *Identification***
- **EBSD Based Phase Differentiation**
- **Integrated EBSD & EDS Phase *Differentiation***
- **Component Analysis**

Phase identification

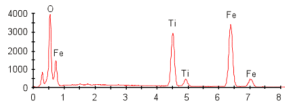


EBSD



Fe_2TiO_4

EDS Spectrum

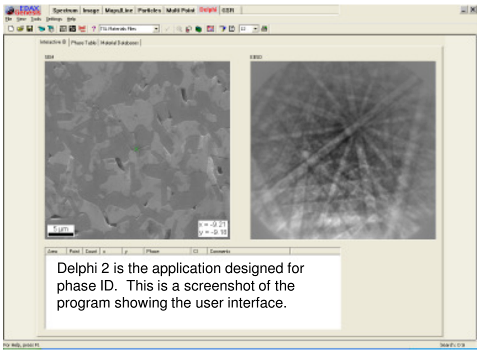


Database

Candidate Phases

Fe_2TiO_4	Cubic	a=11.297
Fe-Ti-O	Cubic	a=11.31
$Fe_4(TiO_4)_3$	Tetragonal	a=9.3, c=9.5
$FeTiO_3$	Trigonal	a=5.0884, c=14.093
Fe_2TiO_4	Cubic	a=8.5352
$Fe_3Ti_3O_{10}$	Orthorhombic	a=7.789, b=10.008, c=3.74162
$FeTiO_3$	Orthorhombic	a=5.026, b=5.174, c=7.245
$Fe_2Ti_3O_9$	Hexagonal	a=2.8667, b=4.5985

Delphi 2



Delphi 2 is the application designed for phase ID. This is a screenshot of the program showing the user interface.

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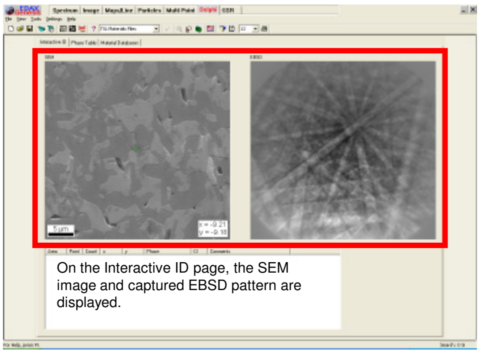
Delphi 2



On the Interactive ID page, the SEM image and captured EBSD pattern are displayed.

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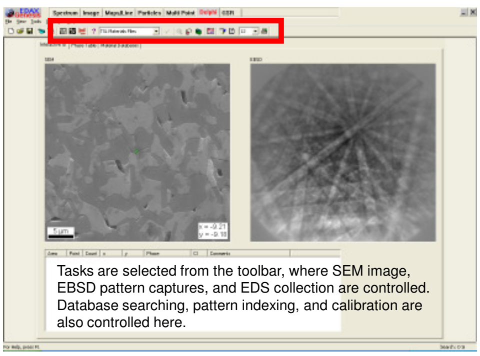
Delphi 2



Tasks are selected from the toolbar, where SEM image, EBSD pattern captures, and EDS collection are controlled. Database searching, pattern indexing, and calibration are also controlled here.

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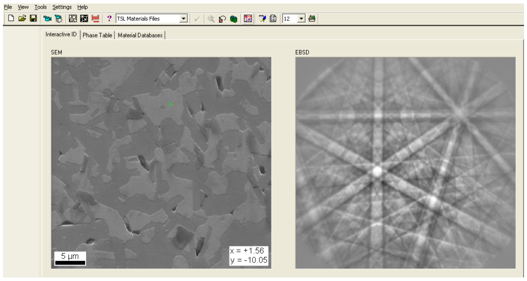
Delphi 2



An analysis location is selected in the SEM image, and a EBSD pattern collected from that position.

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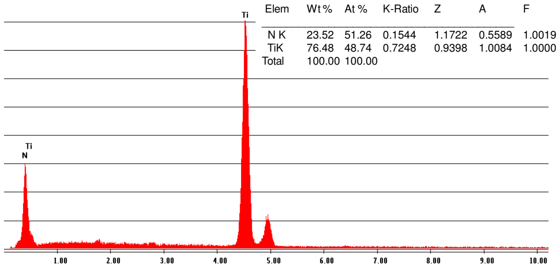
Example 1

EDAX PhiZAF Quantification (Standardless)
Element Normalized

Elem	Wt %	At %	K-Ratio	Z	A	F
N K	23.52	51.26	0.1544	1.1722	0.5589	1.0019
TiK	76.48	48.74	0.7248	0.9398	1.0084	1.0000
Total	100.00	100.00				



An EDS spectrum is also collected. Here Ti and N were identified as being present. Quant analysis indicates 51% N and 49% Ti

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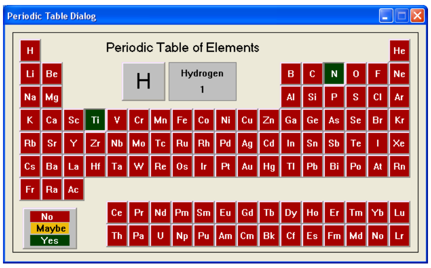
Example 1



The periodic table is used to specify which elements to search for in the different databases. A Yes/No/Maybe search for each element is possible. Here Ti and N are considered to be present.

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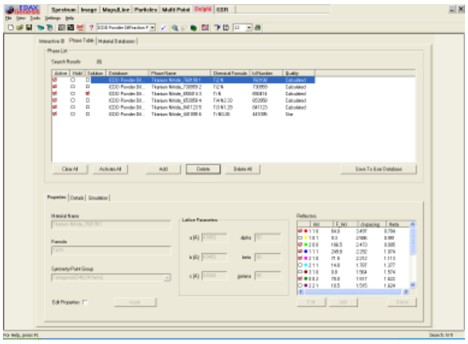
Example 1



After duplication filtering, 6 candidates with this elemental chemistry are found. The results are shown on the Phase Table page.

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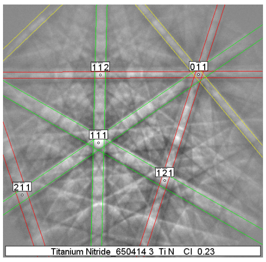
Example 1

The phase differentiation procedure is then used with these 6 candidate phases.

The TiN phase (PDF#6504143) initially has the highest Rank Factor, as well as the highest CI value. It also has the lowest fit value.

However, it has only the 2nd highest number of votes (20 vs. 30).

Visual inspection of the solution shows while the displayed bands match, only some of the observed bands are accounted for.



Phase	Votes	Fit [°]	CI	Rank Fact
☑ Titanium Nitride_650414 3	20	0.60	0.226	3.00
☐ Titanium Nitride_653958 4	5	1.17	0.012	1.07
☐ Titanium Nitride_730959 2	30	2.42	0.060	0.76
☐ Titanium Nitride_441095 6	4	2.65	0.000	0.45
☐ Titanium Nitride_760198 1	4	3.51	0.000	0.34
☐ Titanium Nitride_841123 5	4	5.56	0.000	0.21

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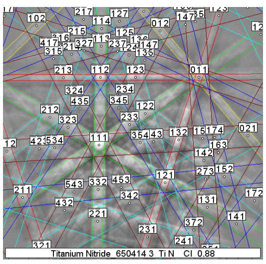
Example 1

Reflectors

	hkl	F_hkl	d-spacing	theta
☑	1 1 1	177.5	2.460	1.000
☑	2 0 0	333.0	2.130	1.155
☑	2 2 0	76.7	1.506	1.634
☐	3 1 1	14.8	1.284	1.916
☐	3 3 1	4.8	0.977	2.519
☐	4 2 0	9.9	0.953	2.584
☐	4 2 2	8.1	0.870	2.831
☐	5 1 1	4.4	0.820	3.003

Reflectors

	hkl	F_hkl	d-spacing	theta
☑	1 1 1	177.5	2.460	1.000
☑	2 0 0	333.0	2.130	1.155
☑	2 2 0	76.7	1.506	1.634
☑	3 1 1	14.8	1.284	1.916
☐	3 3 1	4.8	0.977	2.519
☑	4 2 0	9.9	0.953	2.584
☑	4 2 2	8.1	0.870	2.831
☐	5 1 1	4.4	0.820	3.003



Phase	Votes	Fit [°]	CI	Rank Fact
☑ Titanium Nitride_650414 3	82	0.59	0.881	3.00
☐ Titanium Nitride_653958 4	5	1.17	0.012	1.02
☐ Titanium Nitride_730959 2	30	2.42	0.060	0.56
☐ Titanium Nitride_441095 6	4	2.65	0.000	0.45
☐ Titanium Nitride_760198 1	4	3.51	0.000	0.34
☐ Titanium Nitride_841123 5	4	5.56	0.000	0.21

A better match is found by manually inspecting bands and activating reflectors initially deactivated due to intensity.

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Example 1

Reflectors

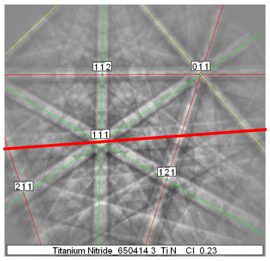
	hkl	F_hkl	d-spacing	theta
☑ ●	1 1 1	177.5	2.460	1.000
☑ ●	2 0 0	333.0	2.130	1.155
☑ ●	2 2 0	76.7	1.506	1.634
☐ ●	3 1 1	14.8	1.284	1.916
☐ ●	3 3 1	4.8	0.977	2.519
☐ ●	4 2 0	9.9	0.953	2.584
☐ ●	4 2 2	8.1	0.870	2.831
☐ ●	5 1 1	4.4	0.820	3.003

TSL Delphi

{hkl} = 4, 2, 2

Activate reflector?

Yes | No



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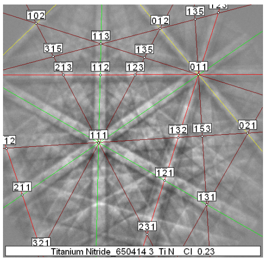
Example 1

When manually activating or adding reflectors, equivalent planes are also used. This can be used as a test to see if the correct structure is being used.



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EDAX TSL

Example 1

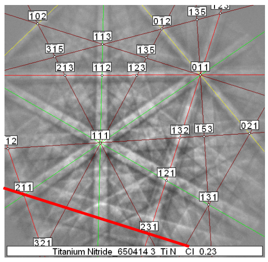
TSL Delphi

{hkl} = 4, 2, 0

Activate reflector?

Yes | No

	hkl	F_hkl	d-spacing	theta
☑ ●	1 1 1	177.5	2.460	1.000
☑ ●	2 0 0	333.0	2.130	1.155
☑ ●	2 2 0	76.7	1.506	1.634
☐ ●	3 1 1	14.8	1.284	1.916
☐ ●	3 3 1	4.8	0.977	2.519
☐ ●	4 2 0	9.9	0.953	2.584
☑ ●	4 2 2	8.1	0.870	2.831
☐ ●	5 1 1	4.4	0.820	3.003



Determines that this band is the {420} band.

Option to Activate this Band into the Indexing routine

Checks the database file to see if this {420} band is present.

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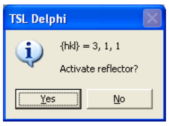
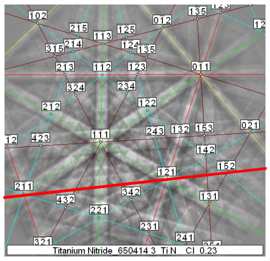
EDAX TSL

Example 1



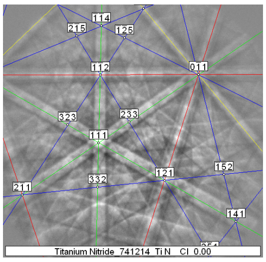
	hkl	F_hkl	d-spacing	theta
☑	1 1 1	177.5	2.460	1.000
☑	2 0 0	333.0	2.130	1.155
☑	2 2 0	76.7	1.506	1.634
☐	3 1 1	14.8	1.284	1.916
☐	3 3 1	4.8	0.977	2.519
☑	4 2 0	9.9	0.953	2.584
☑	4 2 2	8.1	0.870	2.831
☐	5 1 1	4.4	0.820	3.003



EDAX TSL

Example 1

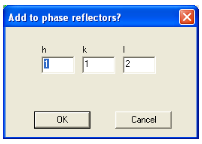
	hkl	F_hkl	d-spacing	theta
☑	1 1 1	174.5	2.540	0.969
☑	2 0 0	333.0	2.200	1.119
☑	2 2 0	77.3	1.556	1.582
☑	3 1 1	14.8	1.327	1.855

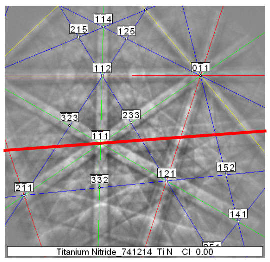


For some database files, the list of possible reflectors is incomplete.

EDAX TSL

Example 1





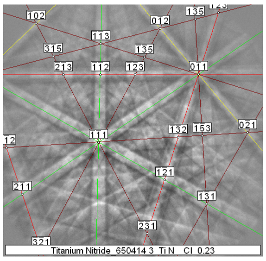
Instead of activating expected reflectors, manual inspection will add reflectors to the material file.

In this example, a low index (h+k+l=low number) plane is identified, which is what is expected for EBSD.

EDAX TSL

Example 1

Just as when activating reflector, when adding reflectors, if the symmetrically equivalent bands also match bands in the pattern then this is a good confirmation of the correct phase identification.



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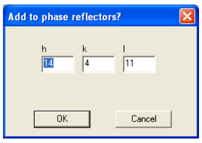
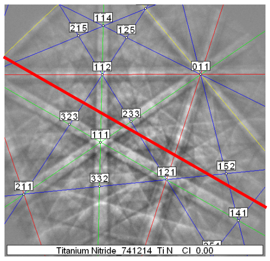
Example 1





For an arbitrary line however, a high index plane is identified.

Care and judgment must be used when adding a reflector.

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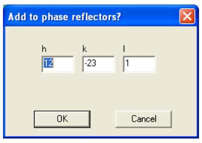
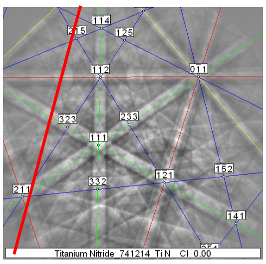
Example 1





Also watch for rounding errors when manually drawing lines. The plane should be a [1-20] plane.

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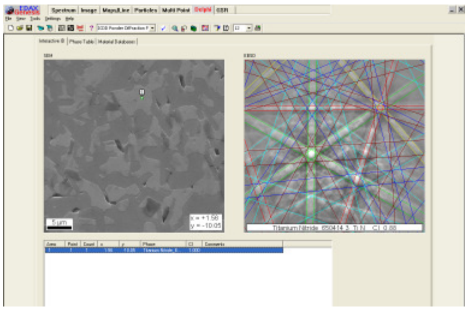
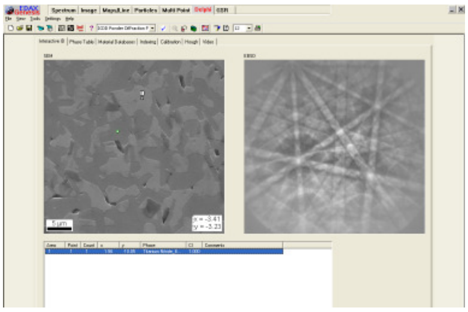
Example 1



The phase solution is then recorded for this analysis point. The phase, with reflector adjustment, can then be saved to a user database for OIM mapping and additional phase ID.

EDAX TSL

Example 1

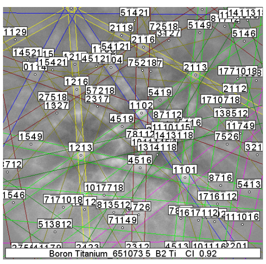


The darker "phase" is then selected in the SEM image, and a pattern obtained. EDS analysis indicated Ti and B are present.

EDAX TSL

Example 1

Phase	Votes	Fit [°]	CI	d-Space Fit	Rank Fac
☑ Boron Tita...	84	0.20	0.917	1.00	3.00
☐ Boron Tita...	35	1.72	0.298	1.00	0.56
☐ Titanium B...	4	1.98	0.024	1.00	0.23
☐ Boron Tita...	4	2.35	0.024	1.00	0.20
☐ Boron Tita...	2	2.38	0.012	1.00	0.18
☐ Boron Tita...	6	3.00	0.012	1.00	0.15
☐ Titanium B...	4	2.86	0.000	1.00	0.14
☐ Boron Tita...	3	3.19	0.000	1.00	0.13
☐ Boron Tita...	4	3.66	0.000	1.00	0.11
☐ Boron Tita...	1	3.76	0.000	1.00	0.11
☐ Titanium B...	1	5.83	0.000	1.00	0.07
☐ Boron Tita...	4	10.24	0.012	1.00	0.05



The second phase is identified as TiB_2 following the same procedure.

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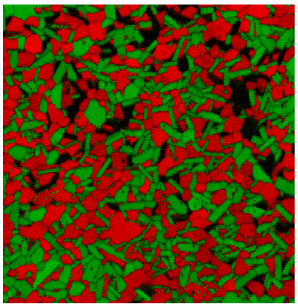
Example 1



Red: TiN
Green: TiB_2

With these material files, it is then possible to obtain a multi-phase OIM map.

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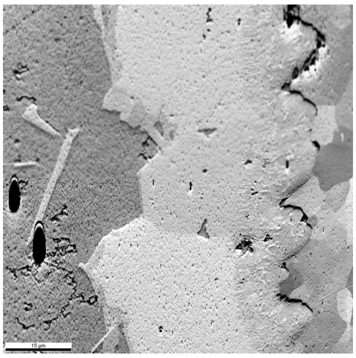
Example: phase identification



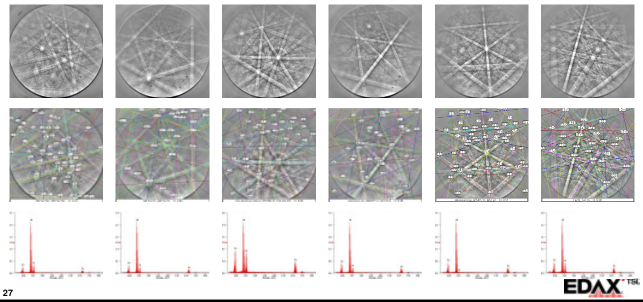
- FSD image of a Al – Fe weld interface.
- At the interface Si was added to facilitate the welding process.
- A large number of phases could be identified on the BSE and FSD (left) images.
- All the phases contain Al, Fe, and / or Si in different quantities and may be part of continuous solid-solution series.

EDAX TSL

Phase identification: Al-Fe (Si) welding interface

- The elements are identified for each phase
- This chemistry is used to search a database of candidate phases (e.g. ICDD database)
- The candidates are matched against the pattern using dedicated Phase ID software, Delphi.

$Al_9Fe_2Si_2$ – Monoclinic (C2h) [2/m]	Al_8Fe_2Si - Hexagonal (D6h) [6/mmm]	$Fe_3Al_2Si_3$ - Triclinic (S2, Ci) [-1]	$Al_{13}Fe_4$ - Orthorhombic (D2h) [mmm]	Al_5Fe_2 - Orthorhombic (D2h) [mmm]	Fe_2Si - Trigonal (D3d) [-3m]



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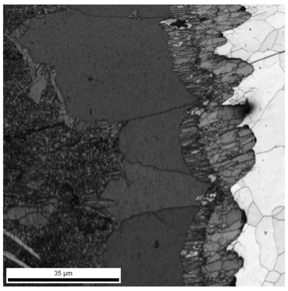
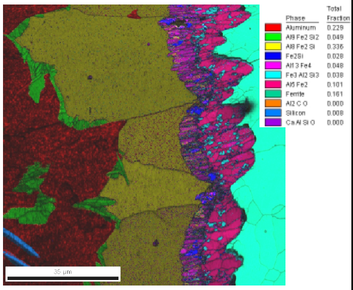
EBSD map results

After identification of all phases, an EBSD map was collected across the interface.





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Outline

- Integrated EBSD & EDS for Phase *Identification*
- EBSD Based Phase Differentiation
- Integrated EBSD & EDS Phase *Differentiation*
- Component Analysis

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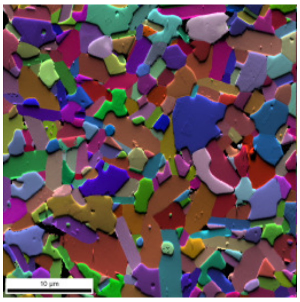
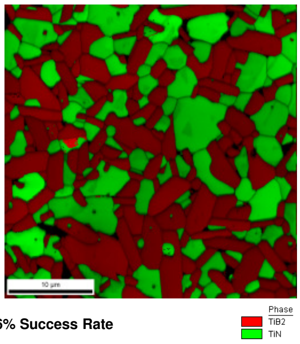
OIM – multiphase samples TiN - TiB_2 (cub – hex)





- 195 Indexed Points per Second with 96% Success Rate

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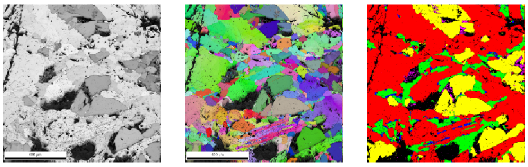
Common issues with EBSD on multiphase materials

- Large variation in EBSD pattern quality due to different polishing properties.



- Need to index each pattern with all possible phases and then select the best solution. This is slow and may produce errors.
- Phases may have the same crystal structure and cannot be differentiated from each other by EBSD (e.g. Ni vs. Cu).

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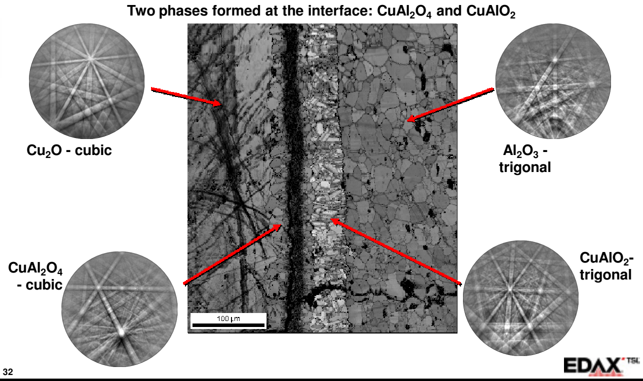
Multi-Symmetry Sample

Copper oxide – Aluminium oxide reaction couple

Two phases formed at the interface: $CuAl_2O_4$ and $CuAlO_2$



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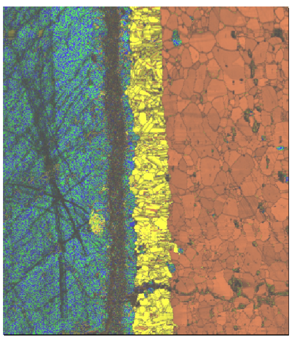
Conventional phase differentiation



Both trigonal phases, Al_2O_3 (orange) and $CuAlO_2$ (yellow) are sucessfully identified

The cubic phases Cu_2O (blue) and $CuAl_2O_4$ (green) could not be distinguished

Gray Scale Map Type: Image Quality
0...261.432 (0...261.432)

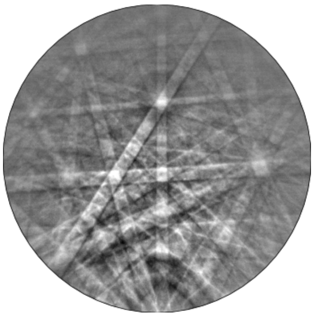
Color Coded Map Type: Phase

Phase	Total Fraction	Partition Fraction
Al2O3	0.417	0.422
CuAl2O4	0.140	0.142
CuAlO2	0.120	0.121
CuO	0.312	0.315

33 EDAX TSL

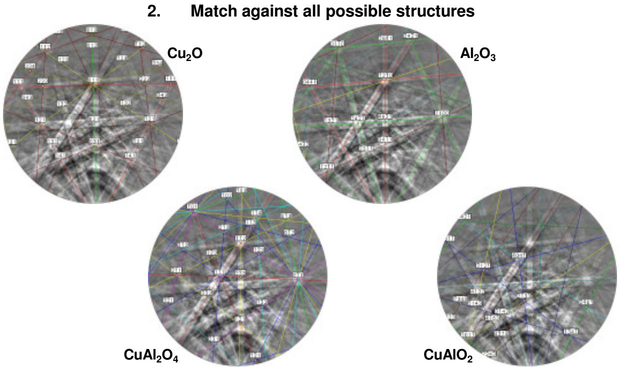
Scanning multiphase materials – phase 1

1. Obtain a pattern



Scanning multiphase materials – phase 1

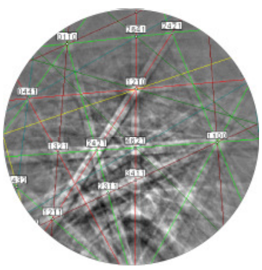
2. Match against all possible structures



Scanning multiphase materials – phase 1

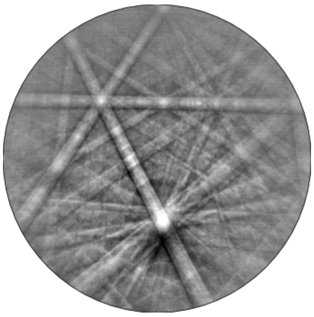
3. Select the best fit solution

phase	votes	fit (°)	confidence index	rank
CuO_2	6	2.10	0.000	0.48
Al_2O_3	80	0.86	0.500	3.00
$CuAlO_2$	12	2.10	0.017	0.59
$CuAl_2O_4$	8	2.11	0.008	0.52



Scanning multiphase materials – phase 2

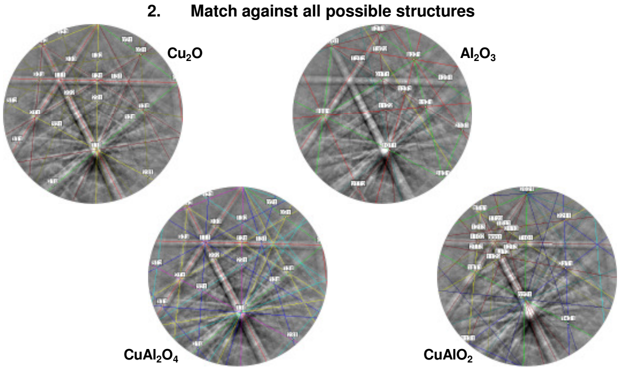
1. Obtain a pattern



37

Scanning multiphase materials – phase 2

2. Match against all possible structures



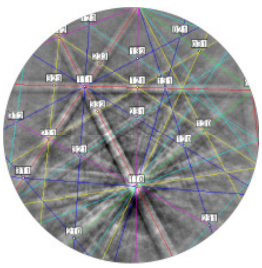
38

Scanning multiphase materials – phase 2

3. Select the best fit solution

Both phases are cubic

phase	votes	fit (°)	confidence index	rank
CuO_2	56	0.46	0.821	3.00
Al_2O_3	10	1.78	0.018	0.46
$CuAlO_2$	13	1.69	0.107	0.63
$CuAl_2O_4$	56	0.46	0.821	3.00



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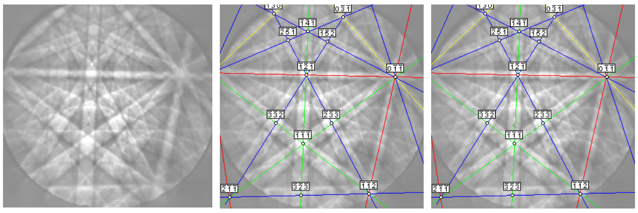
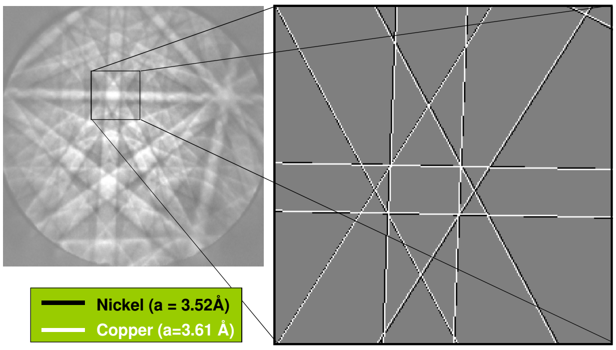
Phase differentiation

Nickel Pattern | Indexed as Nickel | Indexed as Copper



40

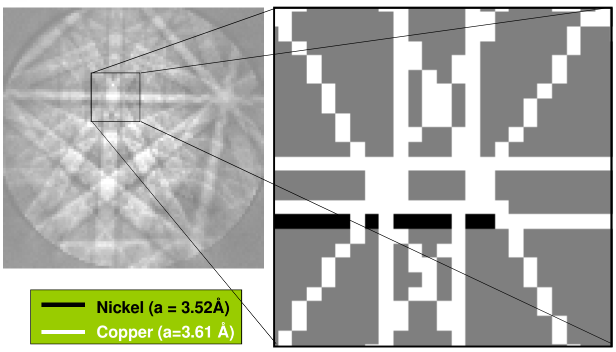
Phase differentiation – band widths



1024 x 1024 Image

41

Phase differentiation – band widths



96 x 96 Image

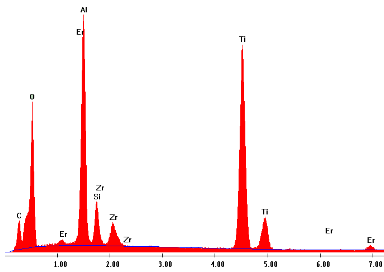
42

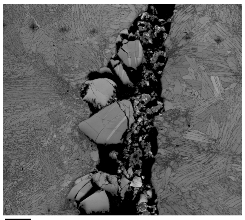
Sample

Titanium-aluminum sample with inclusions.

The EDS spectrum showed that titanium, aluminum, oxygen, zirconium and erbium were present in the material.

We knew a-priori that the two main phases were a hexagonal Ti phase and trigonal Alumina. We also identified an Erbium Oxide phase and two Zirconium Oxides.

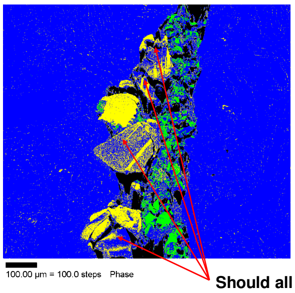


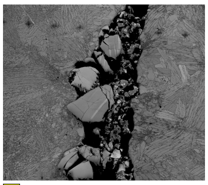


43

Conventional phase differentation

It was obvious from the phase map, that the software had a difficult time differentiating the Titanium and Alumina phases reliably. We tried modifying some of the indexing parameters but with about 870,000 data points these off-line iterations were fairly time consuming (about 3 hours each). Therefore, it was difficult to optimize the settings to improve the results.





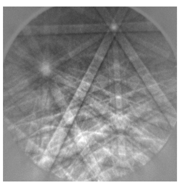
Alumina
Titanium
Erbium Oxide

Should all be yellow!

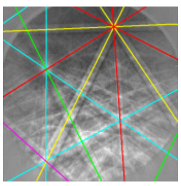
44

Conventional phase differentation

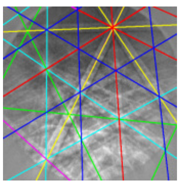
Pattern from Alumina



Indexed as Alumina

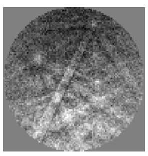


Indexed as Titanium



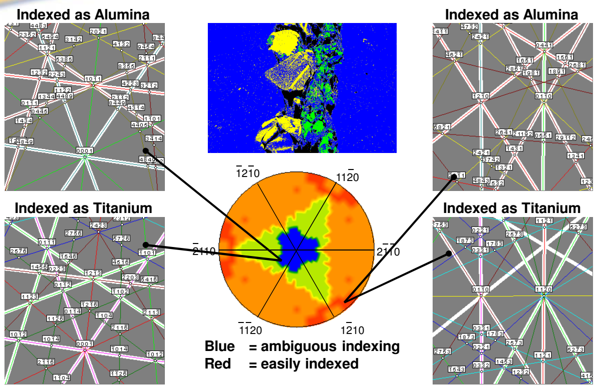
Real full speed scan patterns are much weaker than the beautiful high resolution pattern above.

8x8 binning & then compressed to 96x96



45

Orientation dependence



Blue = ambiguous indexing
Red = easily indexed

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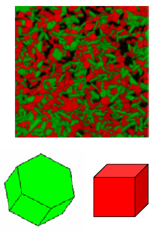
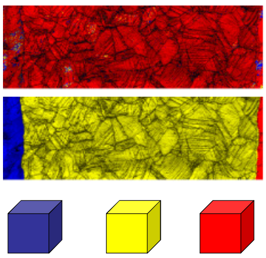
Summary of the Challenge?

EBSD works well when:
The crystallographic structures of the phases are very different.



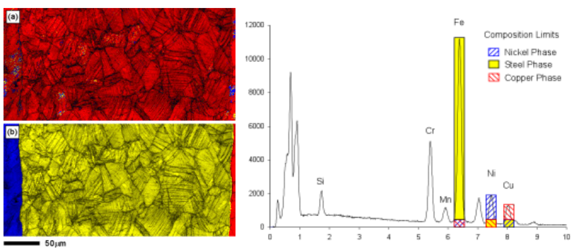
EBSD doesn't work well when:
The crystallographic structures are similar.

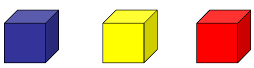


47

How do we overcome this challenge?

➔ **Use Chemistry!**





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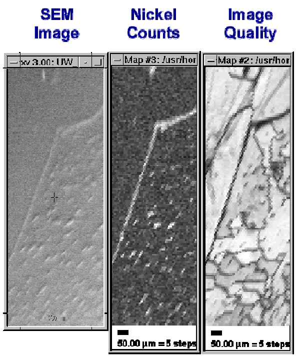
Outline

- Integrated EBSD & EDS for Phase *Identification*
- EBSD Based Phase Differentiation
- Integrated EBSD & EDS Phase *Differentiation*
- Component Analysis

49 EDAX TSL

Combined EBSD + EDS

- Simultaneous EDS and EBSD mapping have been available for 14 years.

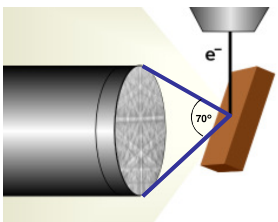


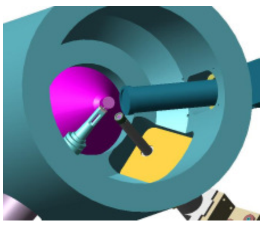
S. I. Wright (1997) Unpublished work at the University of Wisconsin-Madison. Presented at Microscopy and Microanalysis, 1997 Cleveland, Ohio.

50 EDAX TSL

Detector considerations

- For good simultaneous EDS - EBSD analysis, the sample surface has to be within the line of sight of both detectors.
- The EBSD detector must see a proper solid angle.
- There should be no detector shadowing.





51 EDAX TSL

Phase differentiation using simultaneous EDS and EBSD

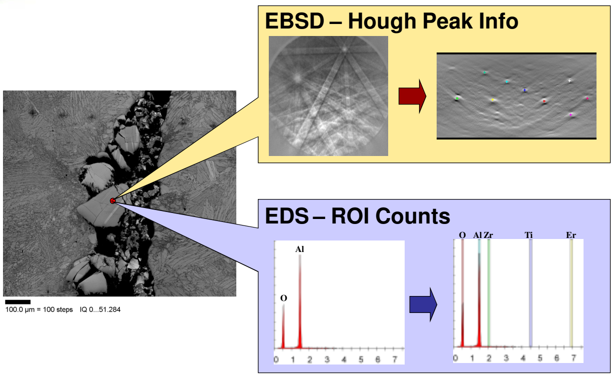
- **Analysis area is scanned and at each point the relevant pattern parameters are stored together with the EDS region-of-interest counts.**
- **Positions of phases are determined using X-ray maps.**
- **During off-line indexing, the recorded chemistry determines which phase / crystal structure file is used for indexing of each point**
- **Each pattern is indexed by only one phase**

ChI scan – Chemistry assisted Indexing

M. M. Nowell and S. I. Wright (2004). "Phase differentiation via combined EBSD and XEDS." *Journal of Microscopy* **213**: 296-305

EDAX TSL

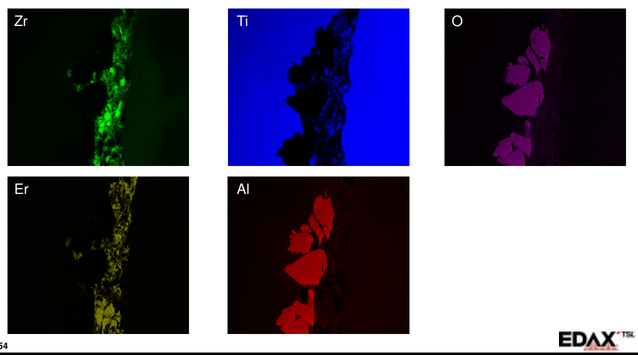
Data collected



EDAX TSL

Elemental maps

We were able to construct element maps from the EDS data collected simultaneously. This aided us in locating and identifying other phases. We were able to identify and index two more phases: a monoclinic ZrO_2 phase and a tetragonal ZrO_2 phase.

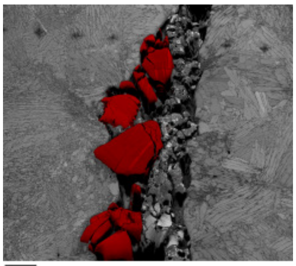


EDAX TSL

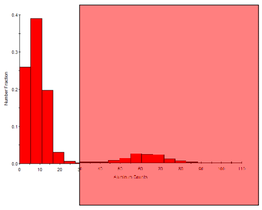
Chemical filtering (Al_2O_3)

All of the red grains will be indexed as Alumina

Al elemental map



Al > 30



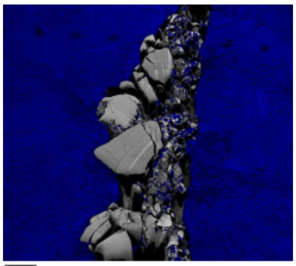
100.0 µm = 100 steps IQ 3.253...209.584, EDS [Aluminum] 30...110

55

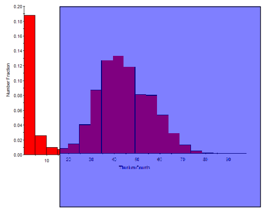
Chemical filtering (Ti)

Everything highlighted in blue will be indexed as titanium

Ti elemental map



Ti>15



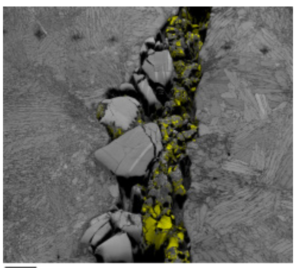
100.0 µm = 100 steps IQ 3.253...209.584, EDS [Titanium] 15...98

56

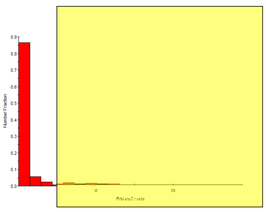
Chemical filtering (Er_2O_3)

Everything highlighted in yellow will be indexed as Erbium Oxide.

Er elemental map



Er>5



100.0 µm = 100 steps IQ 3.253...209.584, EDS [Erbium] 5...29

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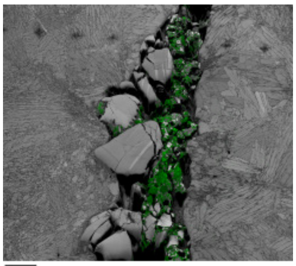
Chemical filtering (ZrO_2)

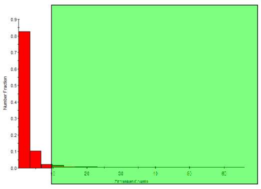
Everything in green will indexed as either tetragonal Zirconium Oxide or monoclinic Zirconium Oxide. EBSD will be used to differentiate between the two.

Zr elemental map



100.0 µm = 100 steps IQ 3.253...209.584, EDS [Zirconium] 10...66

Zr>10



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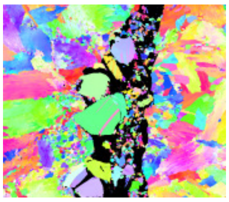
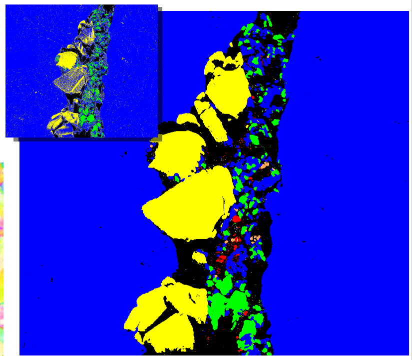
EDAX TSL

Chemical assisted indexing – ChI scan

By filtering the data based on chemistry as part of the phase differentiation process we get much better results as shown here. In addition it speeds up the indexing process quite a bit. It took 20 minutes to rescan this data set using all 5 phases with the chemical filter turned on and 3 hours without the filter turned on.





100.0 µm = 100 steps Phase

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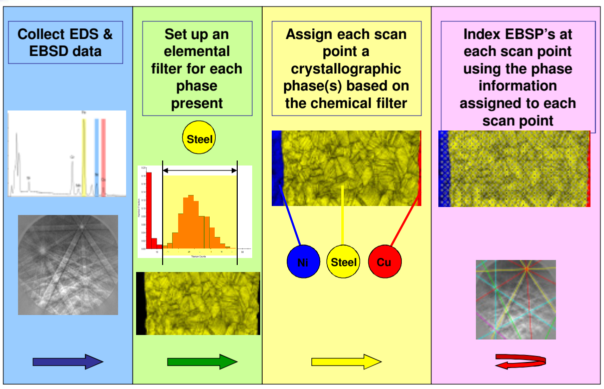
EDAX TSL

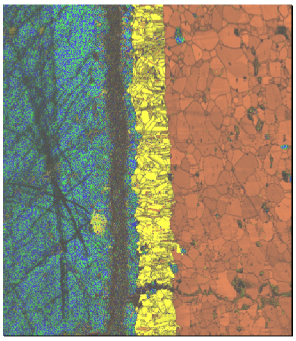
Flowchart



60

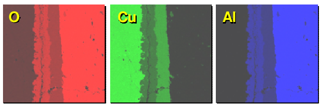
EDAX TSL

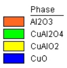
ChI scan phase differentiation – Example 2



Both trigonal phases, Al_2O_3 (orange) and $CuAlO_2$ (yellow) are sucessfully identified

The cubic phases Cu_2O (blue) and $CuAl_2O_4$ (green) could not be distinguished with EBSD alone, but are clear in the X-Ray maps (below)





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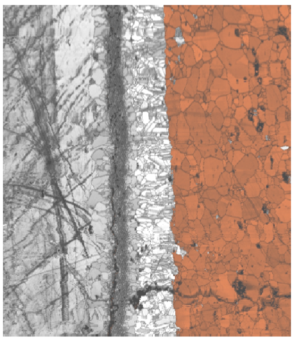
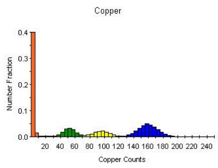
Chemical filtering





Cu_2O

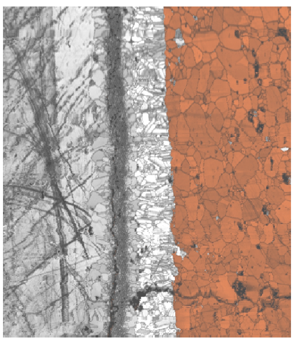
$CuAlO_2$

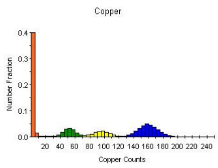
$CuAl_2O_4$

Al_2O_3

62

Chemical filtering





These phase definitions can now be used to select the proper crystal structure for each pixel in the scan, and distinguish crystallographically similar phases.

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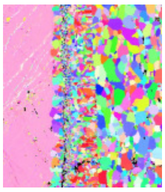
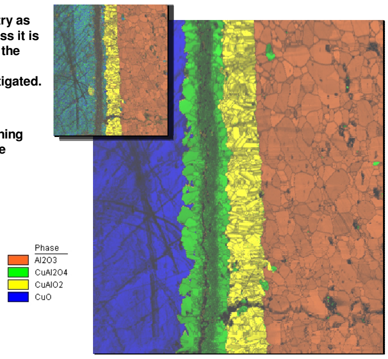
Chi-Scan results

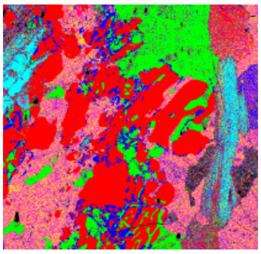
By filtering the data based on chemistry as part of the phase differentiation process it is now possible to differentiate between the cubic phases and the microstructural properties of all phases may be investigated.

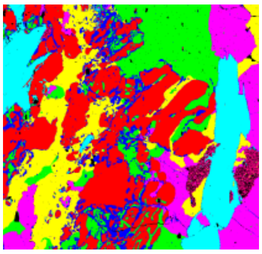
In addition, indexing speed is greatly improved from 1 hour (conventional scanning) to 10 minutes when rescanning this dataset using all 4 phases with the chemical filter turned on.





Example - mineral





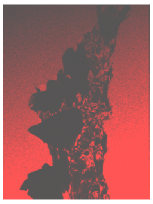
Phases	Chemical Limits (EDS Counts)
Nickeline	Ni>10, As>45, S<30
Pyrrhotite	Ni<10, S>20, As<10, Cu<10, Fe>40
Chalcopyrite	S>20, Ni<10, As<10, Cu>10, Fe>15
Cobalite	As>10, S>15
Pentlandite	S>20, Fe>15, Cu<10, Ni>10
Biotite	K>10
Carbon	C>20

Outline

- Integrated EBSD & EDS for Phase *Identification*
- EBSD Based Phase Differentiation
- Integrated EBSD & EDS Phase *Differentiation*
- **Component Analysis**

Automated phase recognition (PCA)

Issues with phase selection based on chemistry:



- Variation in EDS intensity over the scan area
  - -1- Because of the high-tilts required for EBSD, there is often a change in the EDS signal with WD
  - -2- Beam instabilites may cause variations in countrates during long scans
- Difference in spatial resolution of EDS and EBSD

  The spatial resolutions of the two techniques are approximately 50 nm and 1 micron for EBSD and EDS respectively.
  Thus, there will be some "smearing" at the boundaries where the EBSD must be used exclusively for the phase differentiation.

These issues can be minimised with Automated Phase Recognition

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Phase cluster analysis

A method of statistical analysis of chemistry data:

- Groups pixels based on similarity in the chemistry (EDS ROI counts).
- Allows the user to automatically find phases in the recorded data without prior knowledge.
- PCA ChI scan bridges the gap between the spatial resolutions of EBSD and EDS.
  - Grains down to 200 nm can now successfully be defined.

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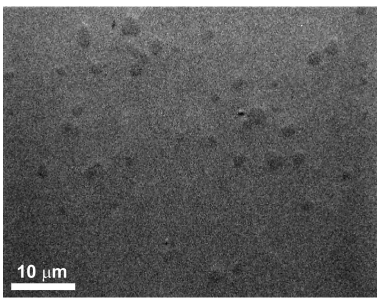
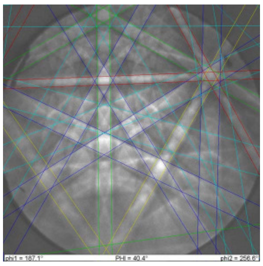
Silicon and Chromium Carbides in Cobalt Matrix



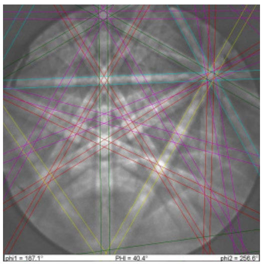
SE image

69

EBSD Patterns



Indexed as Co



Indexed as CrC

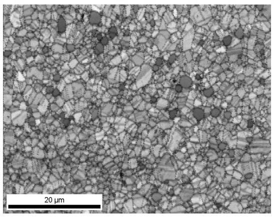
70

PCA example

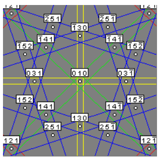
A three phase sample:

1) Cobalt
2) Chromium Carbide
3) Silicon Carbide

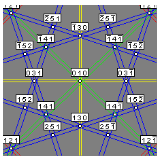
All three phases posses fcc cubic crystal symmetry making it very difficult to distinguish them from each other using EBSD alone.



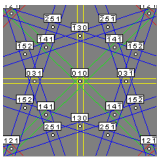
Cobalt



Chromium Carbide



Silicon Carbide



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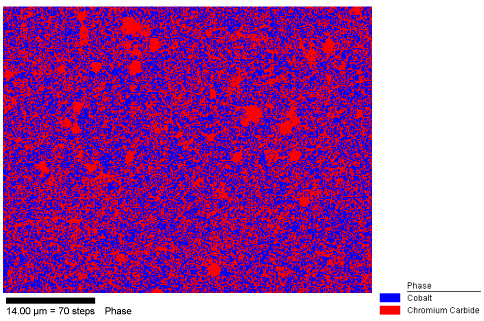
Indexing with crystallography only



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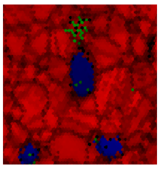
Results from the manually set limits

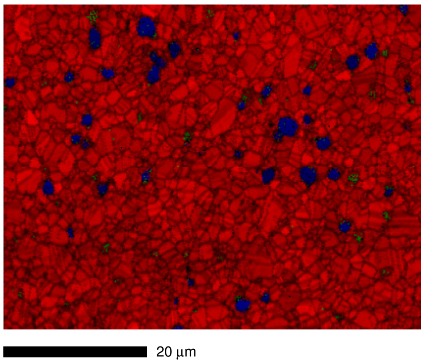
Cobalt

Chromium Carbide

Silicon Carbide

Difficult to find good sets of limits because of limited resolution.





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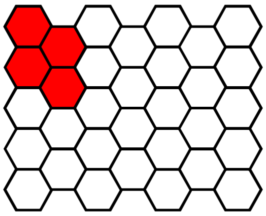
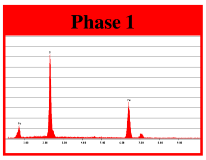
EDAX TSL

Phase cluster analysis



Step 1 - The spectra from the first 4 pixel block of measurement points are taken together and are assumed to be from a single phase.

The EDS signal from these pixels is defined as reference phase 1.



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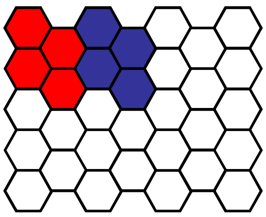
EDAX TSL

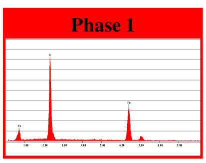
Phase cluster analysis

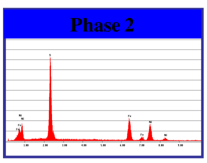


Step 2-The average spectrum from the second 4x4 block is compared to the first component. (Using a "contingency coefficient" based on the sum of squared differences calculated from normalized spectra)

$$\chi^2 = \sum_{i=1}^{N} \frac{(S_i - s_i)^2}{S_i} \qquad C = \frac{\chi^2}{\chi^2 + N}$$

The "spectra" are normalized before calculating the χ^2 values. N is the number of channels in the EDS spectra. S_i and s_i are the number of counts in the i[th] channel of spectra *S* and *s*. C is the contingency coefficient.





Reference: Numerical recipes in C: The art of scientific computing. E. H. Press, S. A. Teukolsky, W. T. Vetterling and B. P. Flannery, Cambridge University Press: Cambridge (1992).

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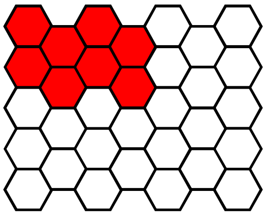
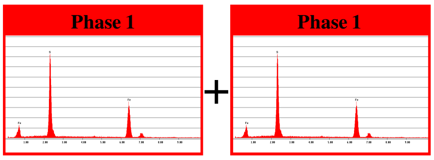
EDAX TSL

Phase cluster analysis



Step 3 - Step 3-If the second block matches the first component within a given tolerance of the contingency coefficient, this second spectrum is added to the first component.



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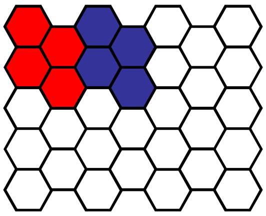
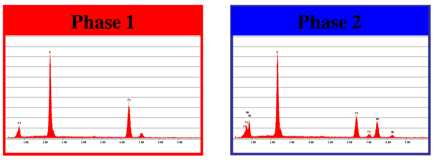
Phase cluster analysis



Step 4 - If the second block does not match the reference chemistry, it then defines a reference phase 2.



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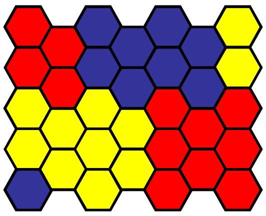
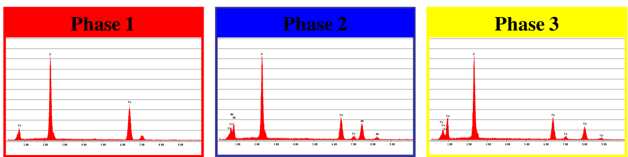
Phase cluster analysis



Step 5 - This comparative process is continued until each 4 pixel block has been matched to a phase.



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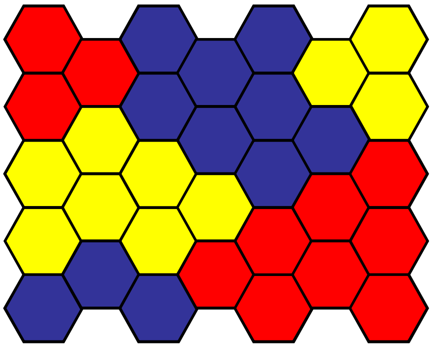
Phase cluster analysis



Step 6 –
When all reference phases are defined, the chemistry of each individual pixel is compared with and matched to one of the determined phases.

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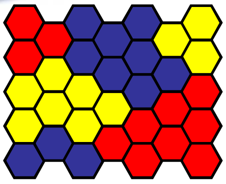
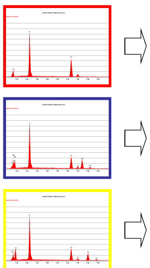
Phase cluster analysis



Step 7 - Candidate phases are assigned to each chemical component.



Phase A

Phase B
Phase C

Phase D

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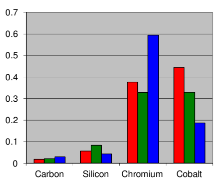
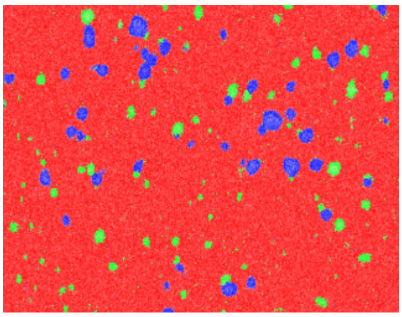
Component analysis

The component analysis found three components. With the following "spectra". The variation in the hue shows the difference of the individual pixel from the average for the component.





Component 1 → Cobalt
Component 2 → Chromium Carbide
Component 3 → Silicon Carbide

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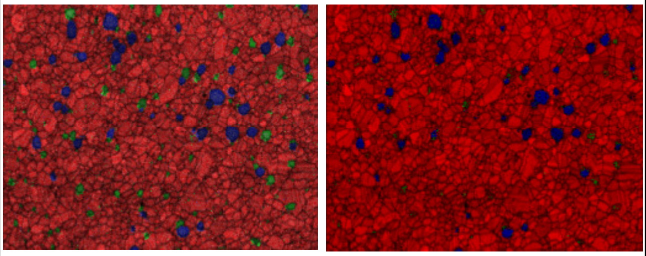
Component analysis - comparison

Component Analysis | Manual Limit Setting



Cobalt | Chromium Carbide | Silicon Carbide

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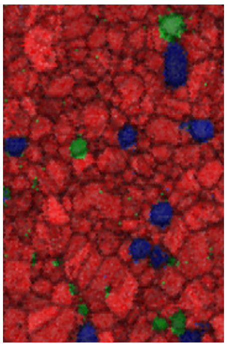
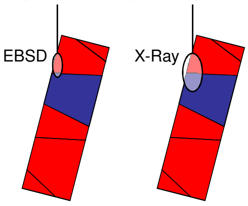
Comparison of PCA with Manual Limits



Note the “bleed” below some of the blue and green grains. This is a resolution difference effect. The bottom of the map corresponds to the top of sample.



Gray scale in the map is for EBSD, colors are for X-Ray

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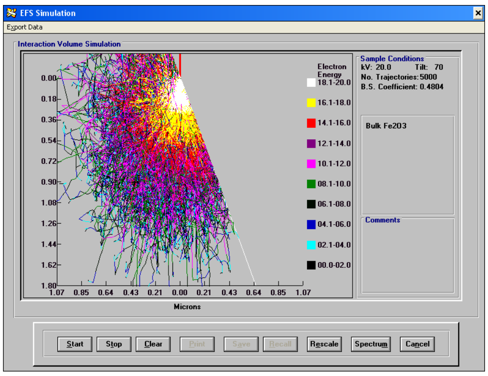
EDS interaction Volume



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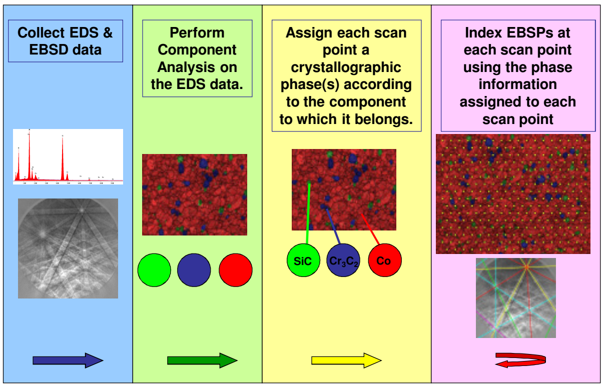
Flowchart



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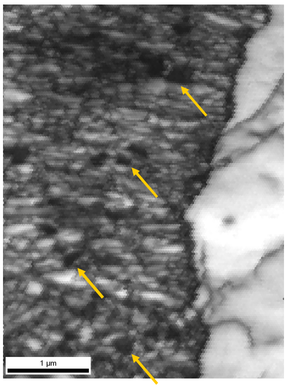
High resolution ChI-scan – submicron particles



- Ni film on iron substrate.
- In the film submicron oxide particles are present (arrowed).
- Differences in polishing properties caused poor patterns for the oxide phases.
- EBSD step size 30 nm.

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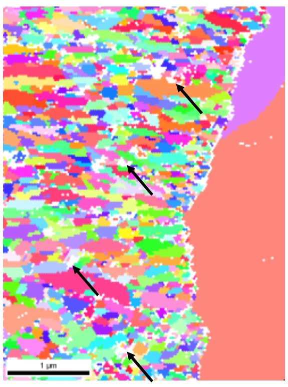
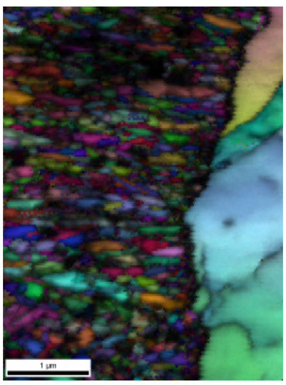
High resolution ChI-scan – submicron particles





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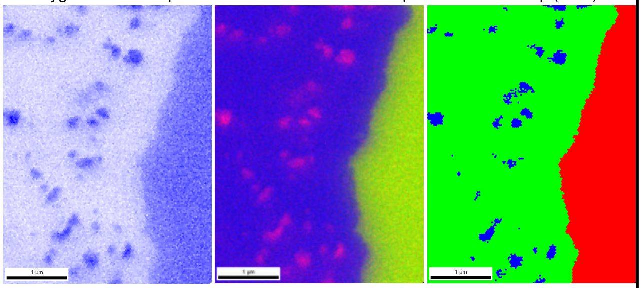
High resolution ChI-scan – submicron particles

Oxygen Counts Map | Element Color Blend Map | Phase Map (PCA)



Color Coded Map Type: EDS [Oxygen]EDS

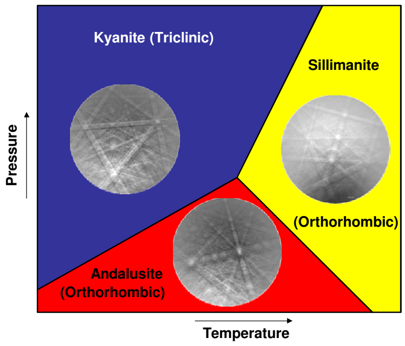
	Min	Max	Total Fraction	Partition Fraction
	14	156	1.000	1.000

Red Element: Oxygen
Green Element: Iron
Blue Element: Nickel

88 EDAX TSL

Example – polymorphs

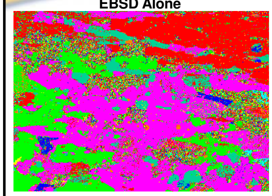
There are still many cases where chemistry does not provide good phase differentiation but EBSD can. Consider the Al_2SiO_5 polymorphs which have identical chemical composition but variable crystallographic structure



89 EDAX TSL

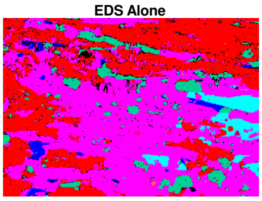
Example – polymorphs

EBSD Alone



- Kyanite
- Andalusite
- Sillimanite
- Muscovite
- Quartz
- Chlorite
- Rutile
- Hematite
- Zircon

EDS Alone



- Kyanite
- Andalusite
- Sillimanite
- Muscovite
- Quartz
- Chlorite
- Rutile
- Hematite
- Zircon

EBSD and EDS

- Kyanite
- Andalusite
- Sillimanite
- Muscovite
- Quartz
- Chlorite
- Rutile
- Hematite
- Zircon

90 EDAX TSL

Conclusions

Multi-phase indexing with (PCA) Chl-scan enables:

- Distinction of phases with similar crystal structure
- Distinction of phases with similar chemistry
- Greatly improved indexing accuracy
- Allows fast scanning of polyphase materials regardless of the number of phases present
- Minimises effects of different spatial resolutions of EDS and EBSD and variation in EDS intensities
- Typical Chl-scan collection speeds are between 10 and 100 points per second.
  (100 to 10 msec EDS dwell time)

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EDAX TSL