Multi-Detector X-Ray Mapping and Generation of Correction Factor Images for Problem Solving

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Outline of Talk

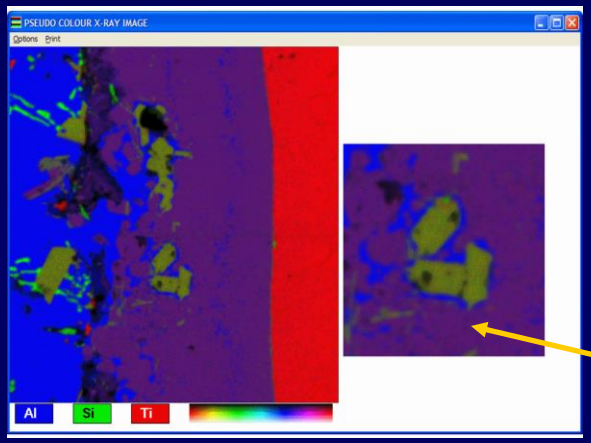
- Quantitative X-Ray Mapping (QXRM)
- Post processing of X-ray maps (Chemical Imaging)
- Quantitative Multi-Detector X-Ray Mapping
- Additional Information from Quantitative X-ray maps
- Correction Factor Images (CFI)
- Rough Samples

Introduction

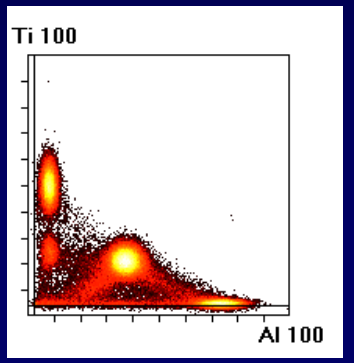
- X-ray mapping with Silicon Drift detectors (SDD's) and multi-EDS detector systems has become an invaluable analysis technique, because the time to perform an x-ray map is reduced considerably.
- Live x-ray imaging can now been performed with so much data collected in a matter of minutes.
- The use of multi-EDS detector systems has made this form of mapping even quicker and has also given users the ability to map minor and trace elements very accurately.
- How the data is collected and summed with multi-EDS detectors is very critical for accurate quantitative x-ray mapping (QXRM).

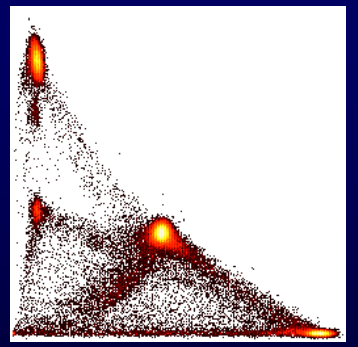
Introduction

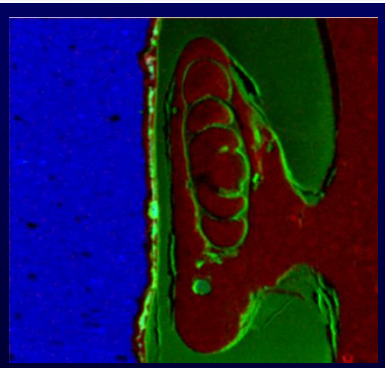
- There is so much information that can be obtained from x-ray maps. Some of which includes:



- elemental mapping
- scatter diagram creation
- rotational scatter diagrams
- pseudo colouring
- rotational colouring
- ratio mapping
- phase mapping and
- quantitative x-ray maps







Introduction

- In obtaining quantitative x-ray maps we are able to easily generate:

  - atomic number (Z)
  - absorption (A)
  - fluorescence (F)
  - theoretical back scatter coefficient (η) and a
  - quantitative total maps from each pixel in the image.

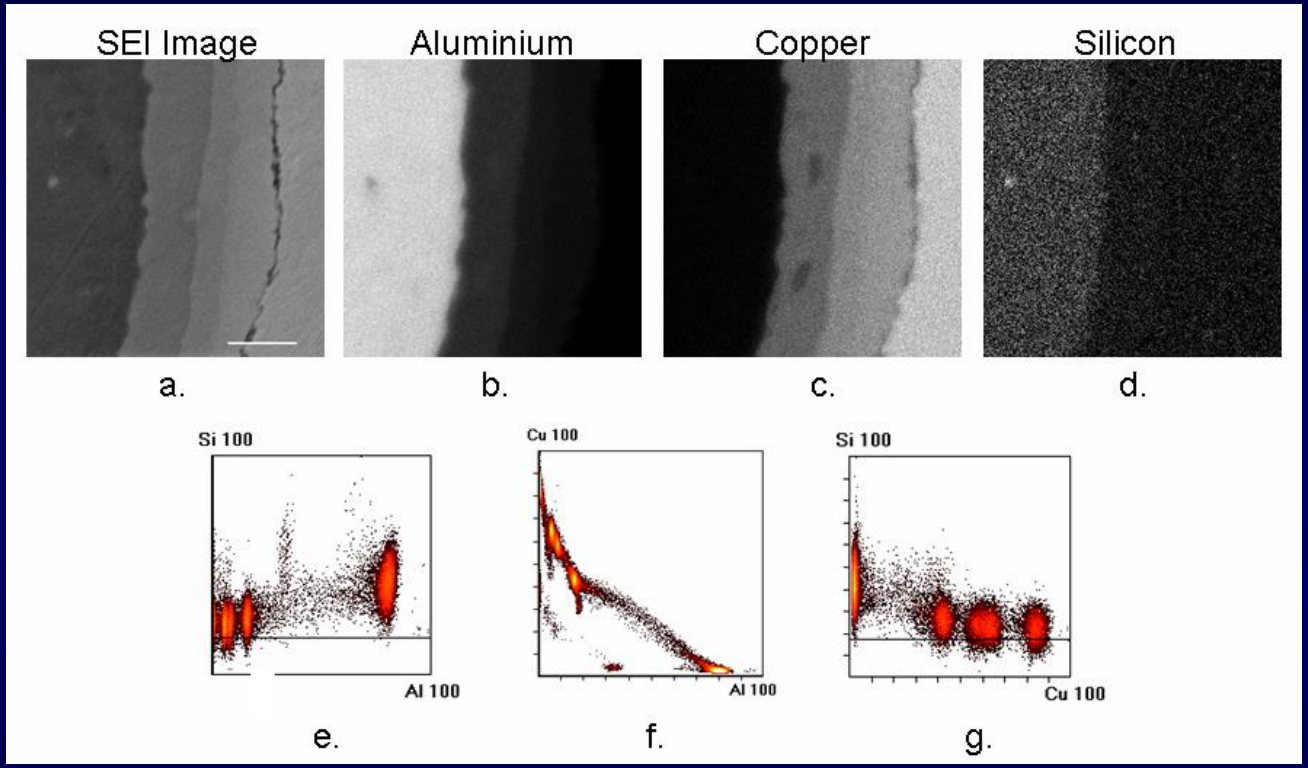
Introduction

- Quantitative total maps from each pixel in the image allows us to generate an image corresponding to each factor (for each element present).
- These images allow us to predict and verify where we are likely to have problems in our images, and are especially helpful to look at possible interface artifacts.
- For example, x-ray mapping at high magnification brings us into realm of secondary fluorescence, x-ray volume and electron volume artifacts and the user would be able to look at possible interface artifacts that exist.

Chemical Phase Mapping (CPM)

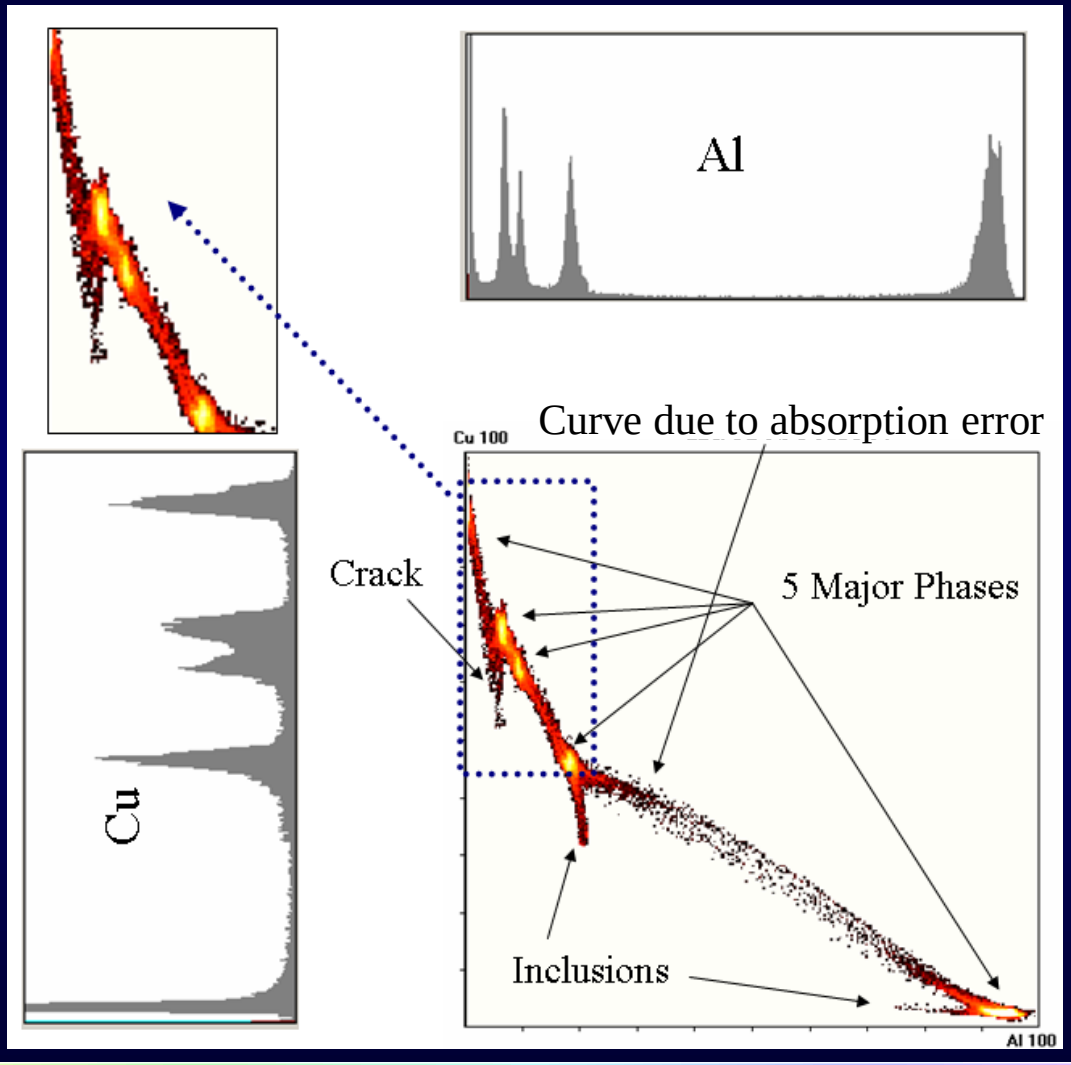
Copper-Aluminium Laminates

- Cu – Al roll bonded metal laminate after sintering at 430^{0}C for 1.5 hours.
- Maps collected at:
  - 20 keV
  - 256x256 pixel
  - 100 msec/pixel
  - 7 kcps



- High quality x-ray maps for Al, Cu and Si with their associated scatter diagrams. HWOF 45μm.
- Scatter diagrams are pixel frequency versus element concentration profiles plotted against each other in two dimensions for selected elements within the sample.

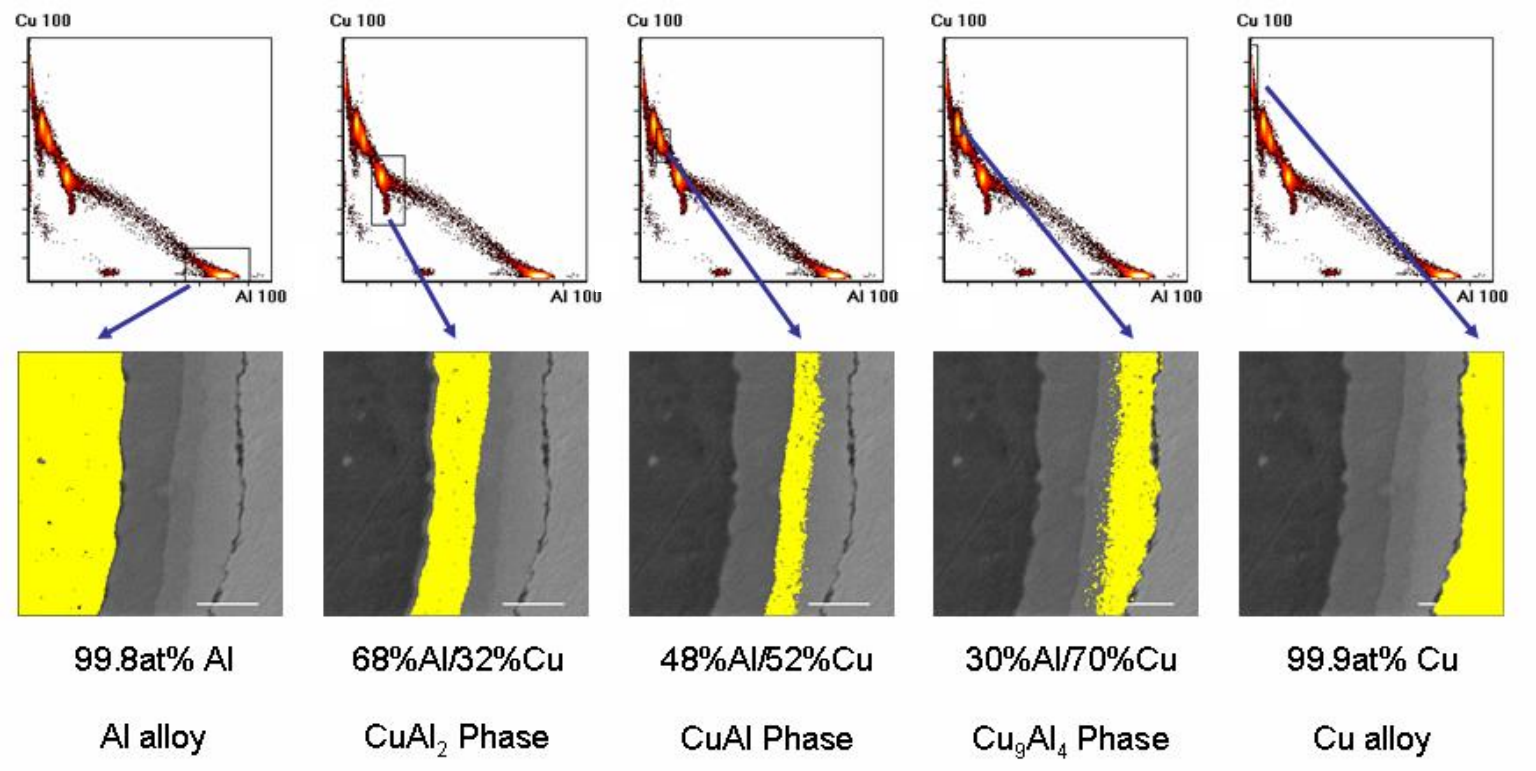
Scatter Diagram Production



Linear distribution of Aluminium

Linear distribution of Copper

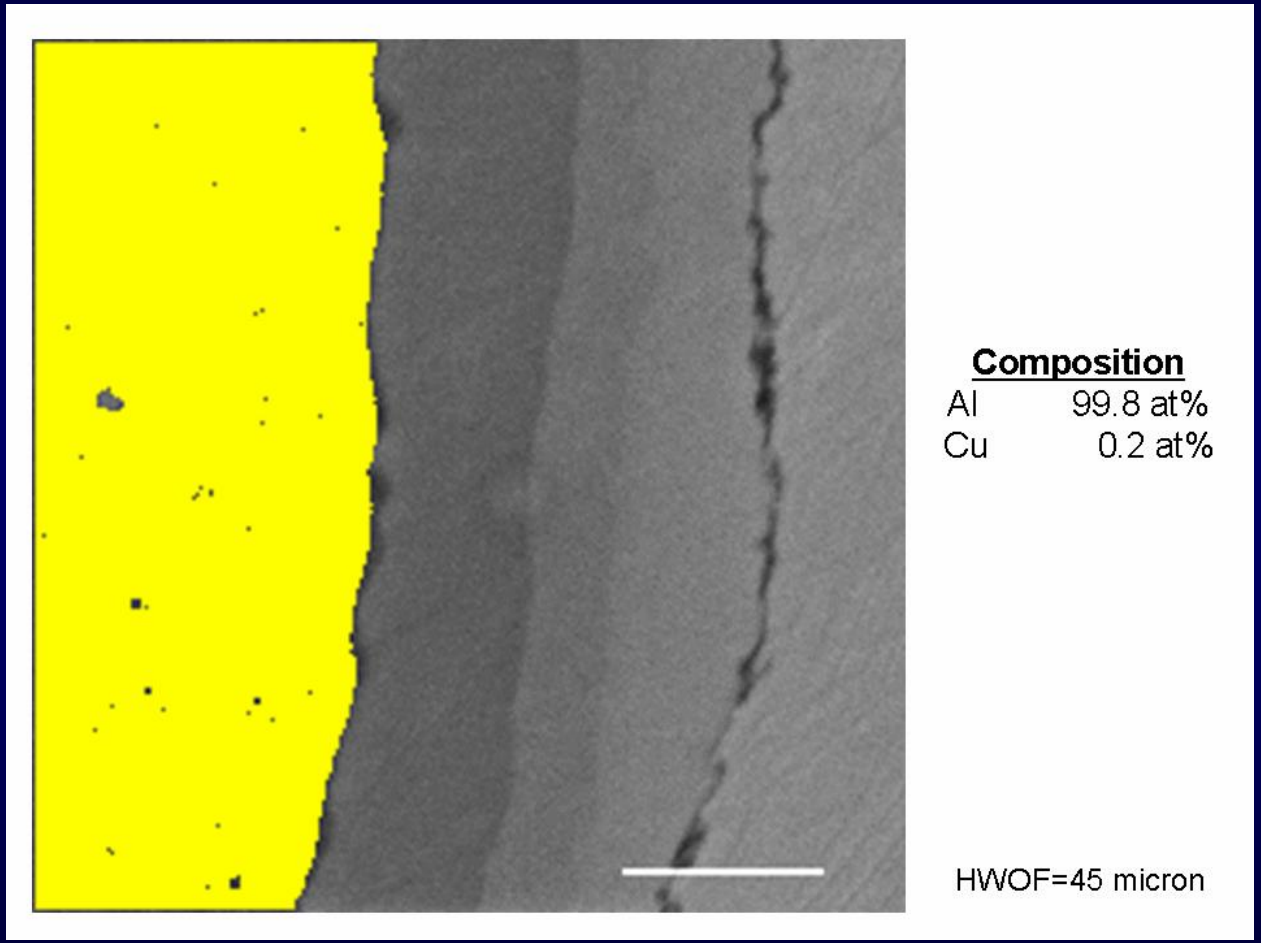
Chemical Phase Identification



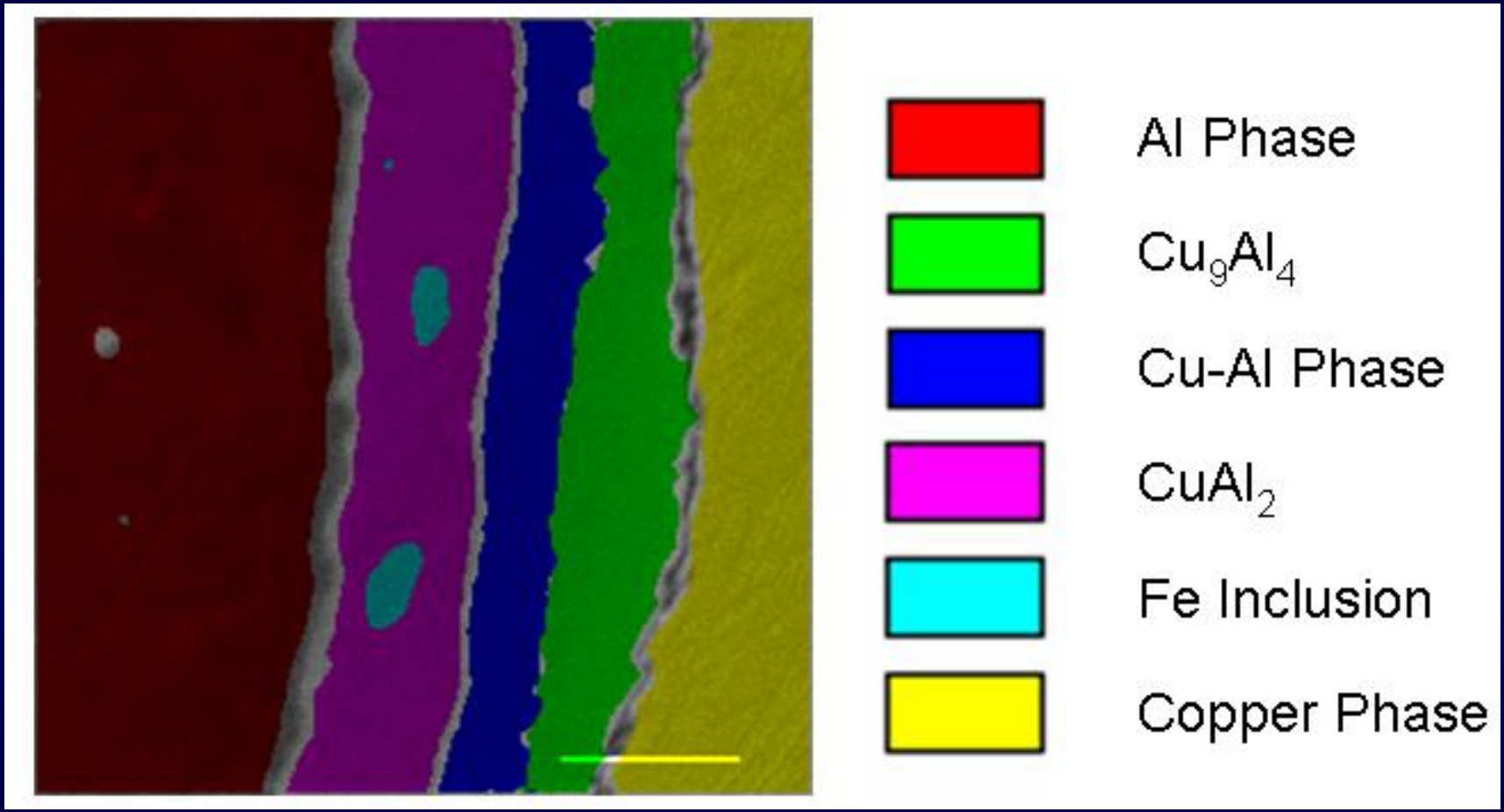
- The images below the scatter diagrams are secondary electron images with information from the different clusters of the scatter diagram superimposed over the image.

Copper-Aluminium Laminates

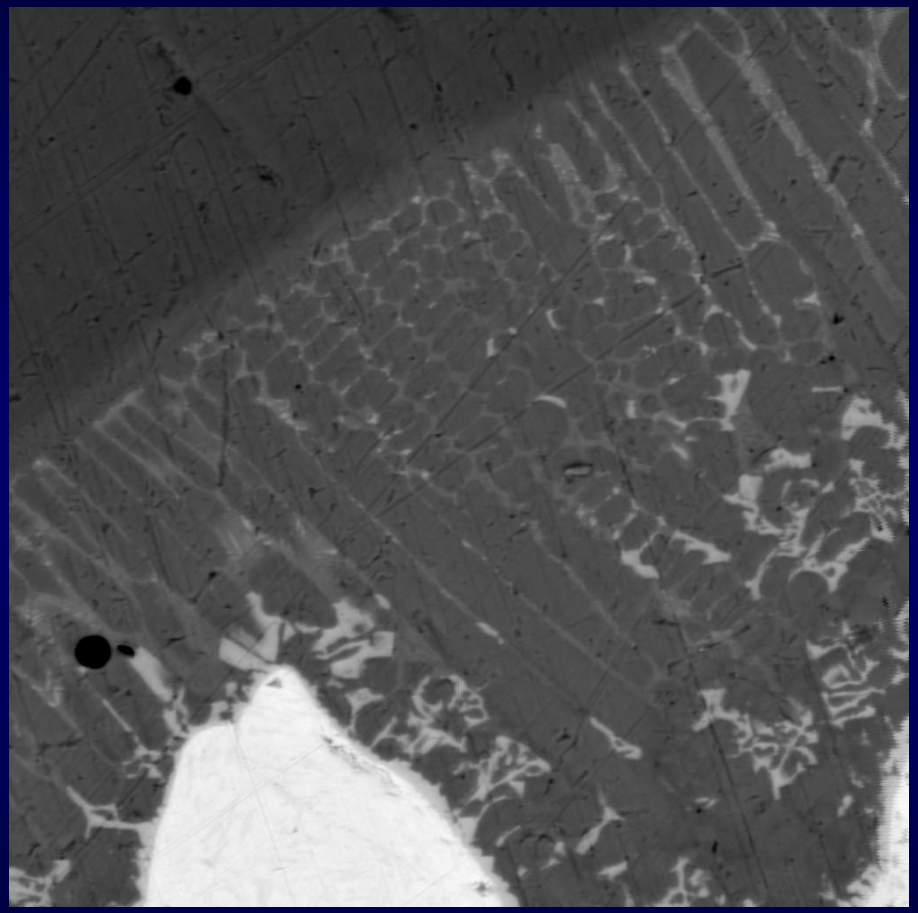
- From scatter diagrams, phases can be selected.
- The phases selected can be superimposed over image.
- After phase selection, data can be quantified.
- Composition of phases can be determined.



Chemical Phase Overlay



Quantitative XRM with Multi-EDS Detectors



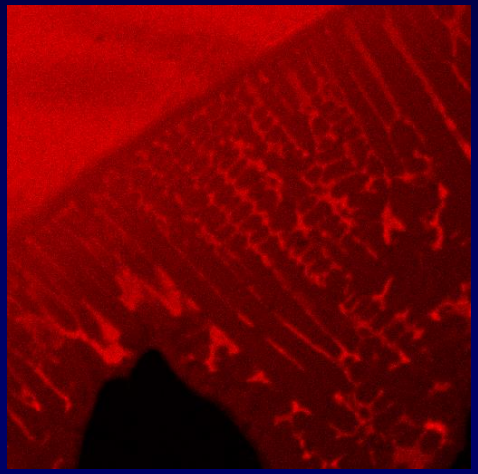
Tungsten Carbide Hard Facing Interface

- BSE image of a steel to nickel interface.
- Multi EDS map using 3 EDS detectors. Map duration 8 Hrs.
- 20keV
- 7.5kcps throughput (+15eV)
- 3nA (due to physical constraint of max ED count rate.)

BSE image - HWOF=85um 200msec/pixel, 512x512.

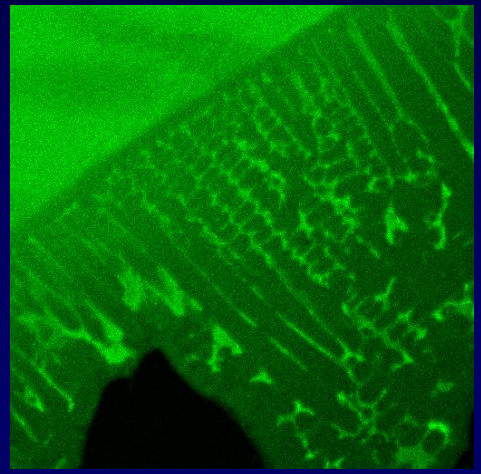
Colouring Verification Technique (CVT)

Fe



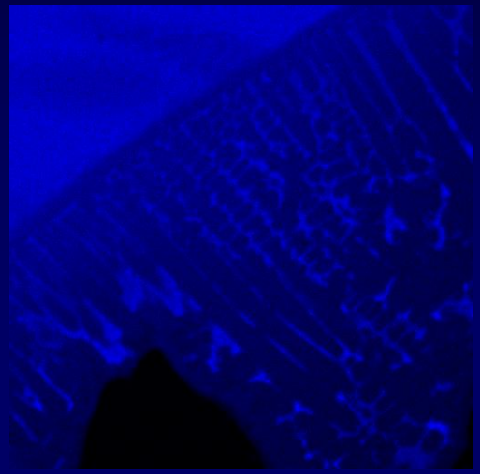
- **EDS1**

Fe



- **EDS2**

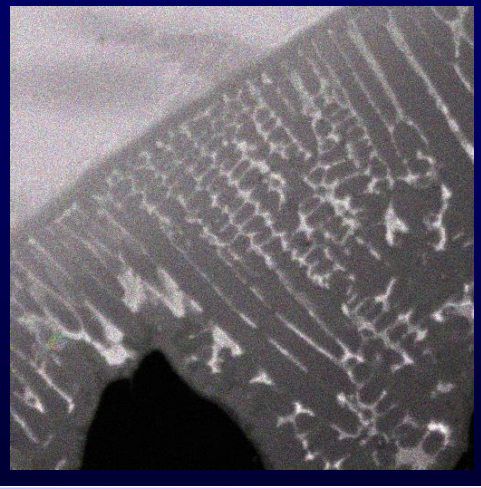
Fe



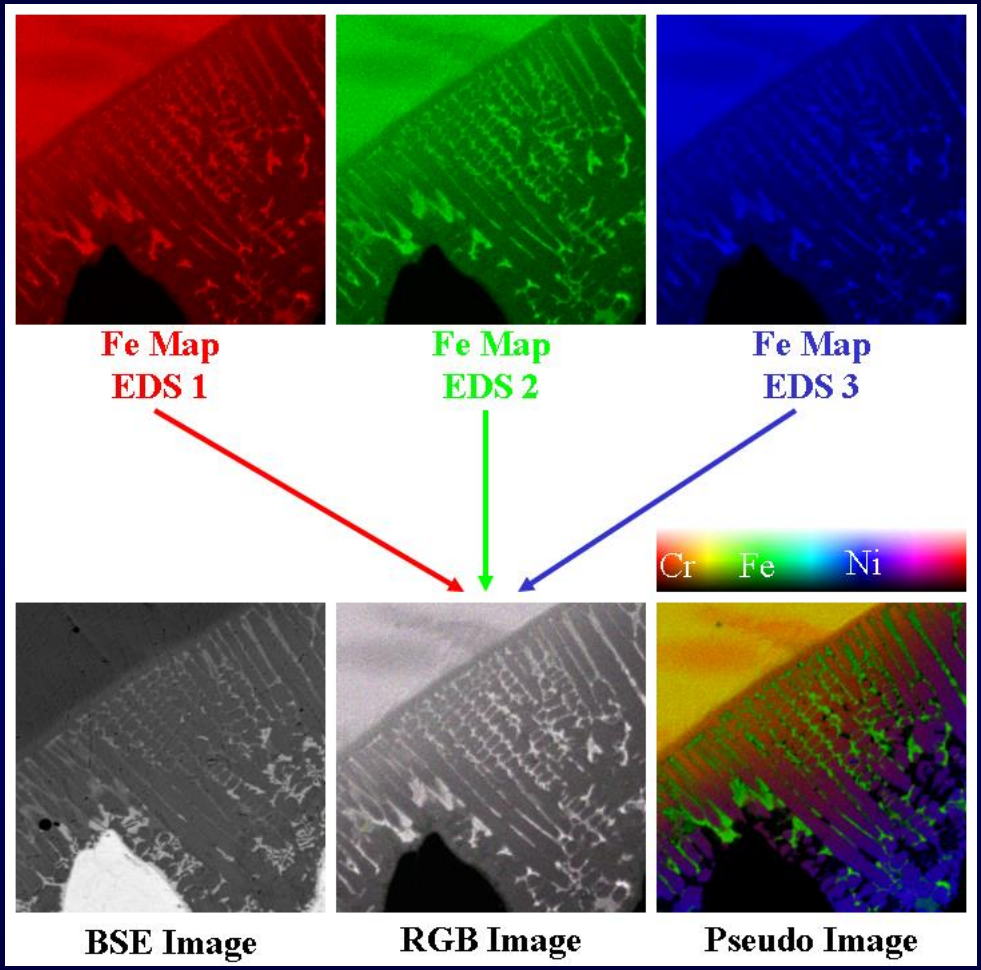
- **EDS3**

RGB

24 bit colour
Fe map



Colouring Verification Technique (CVT)

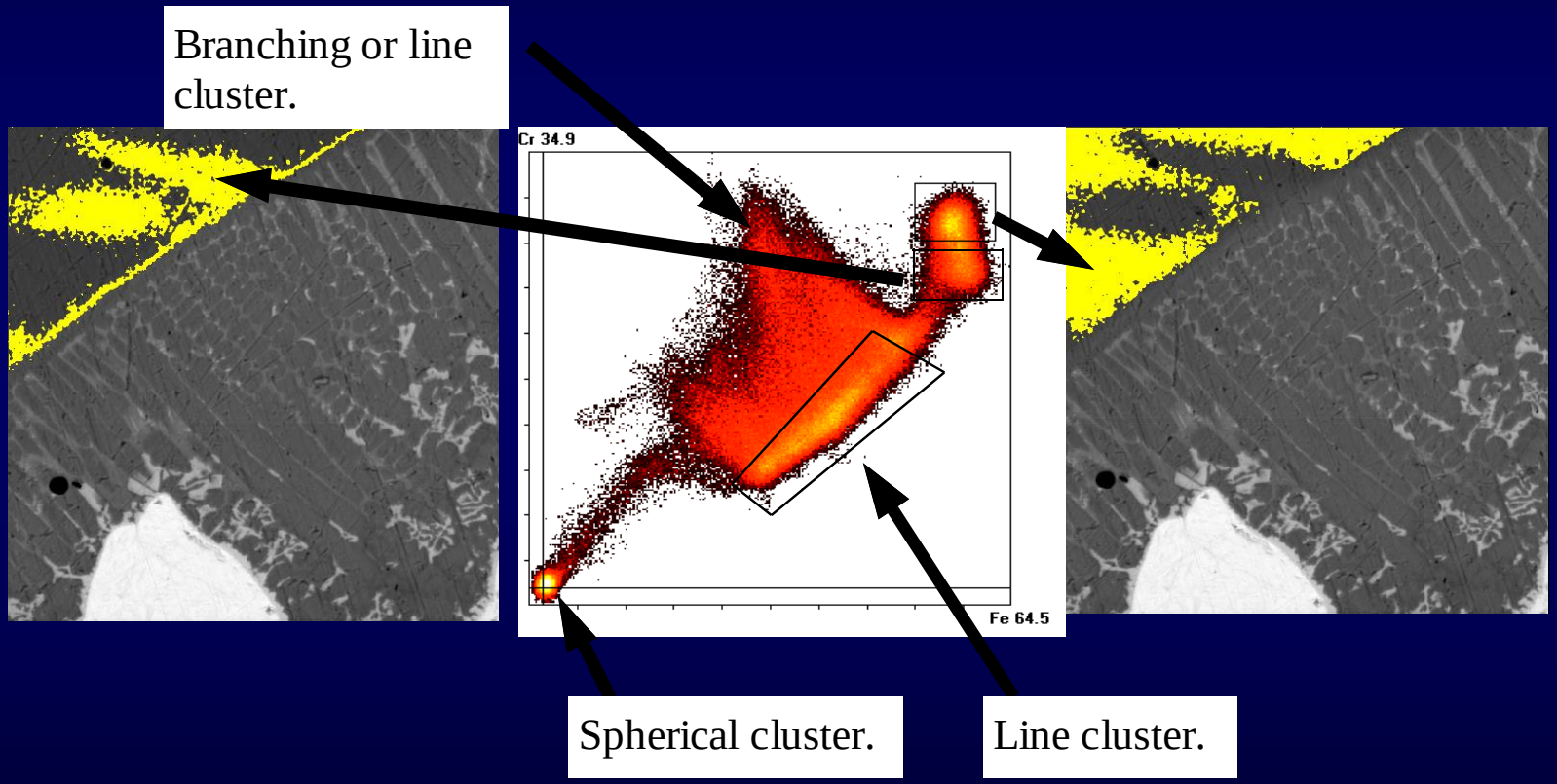


- A different RGB colour is assigned to each detector for the same element.
- The RGB image shows a grey scale map indicating total correlation between the three detectors at the most critical final stage of quantification.
- Also shown is the pseudo image for the three elements present (Cr, Fe, Ni).

BSE image - HWOF=85um 200msec/pixel, 512x512.

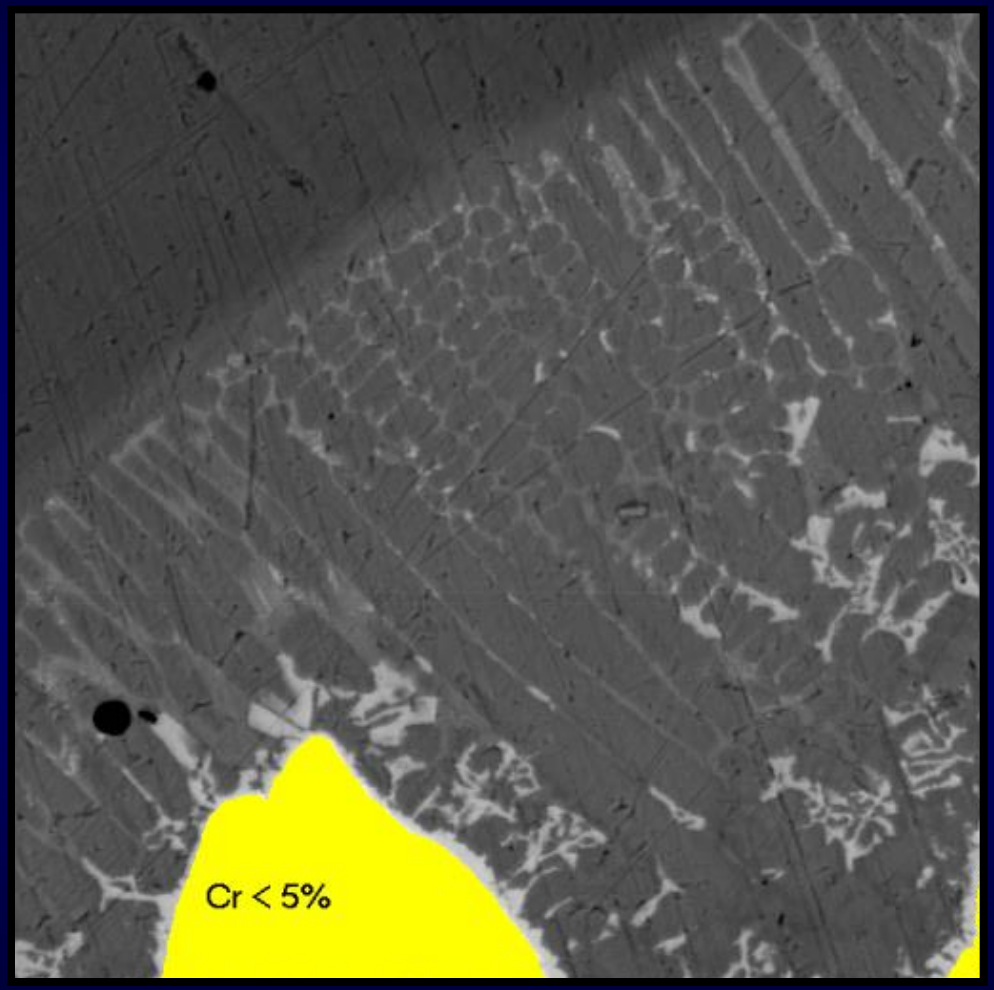
Selecting Phases from Scatter Diagrams

Tungsten carbide hard facing interface.
Superposition over BSE image.



BSE images - HWOF=85um 200msec/pixel, 512x512.

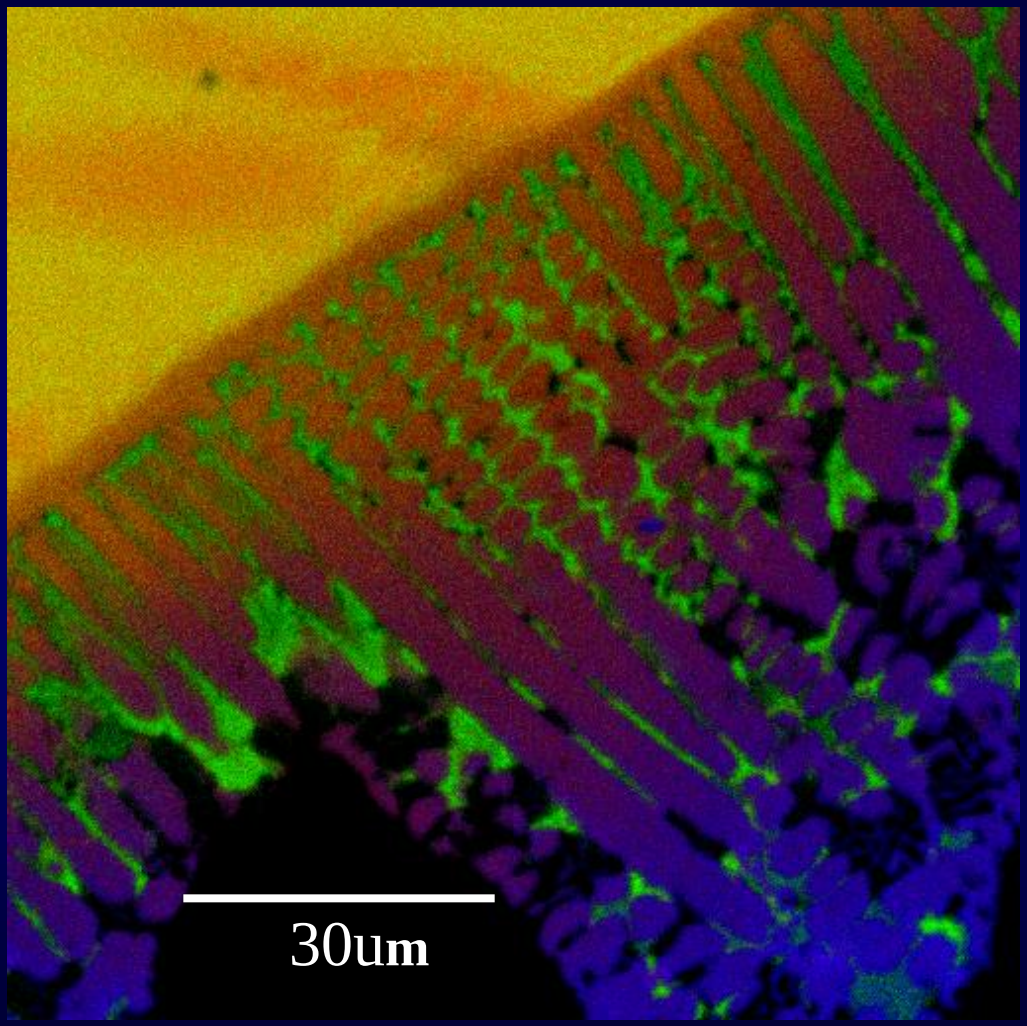
Distribution from min to max



Tungsten carbide hard facing interface.

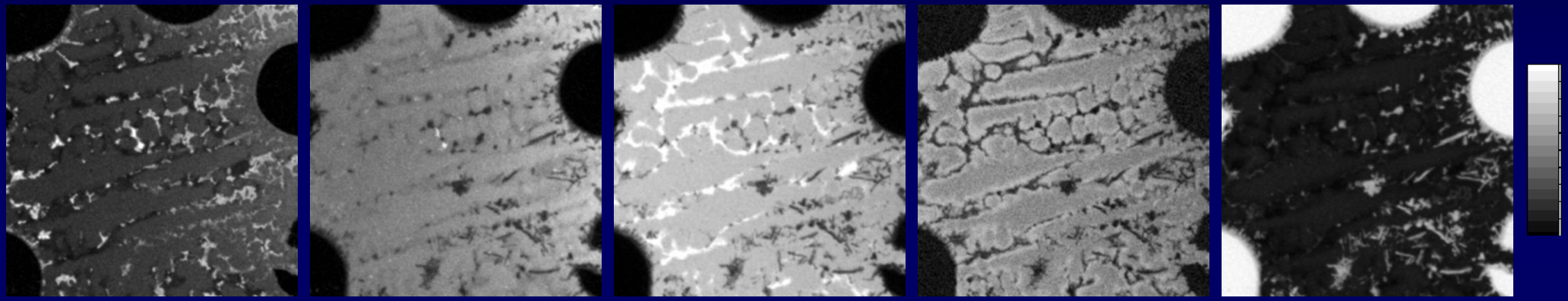
BSE image - HWOF=85um, 200msec/pixel, 512x512.

Coloured



Hard Facing Bonded to a Chrome Steel

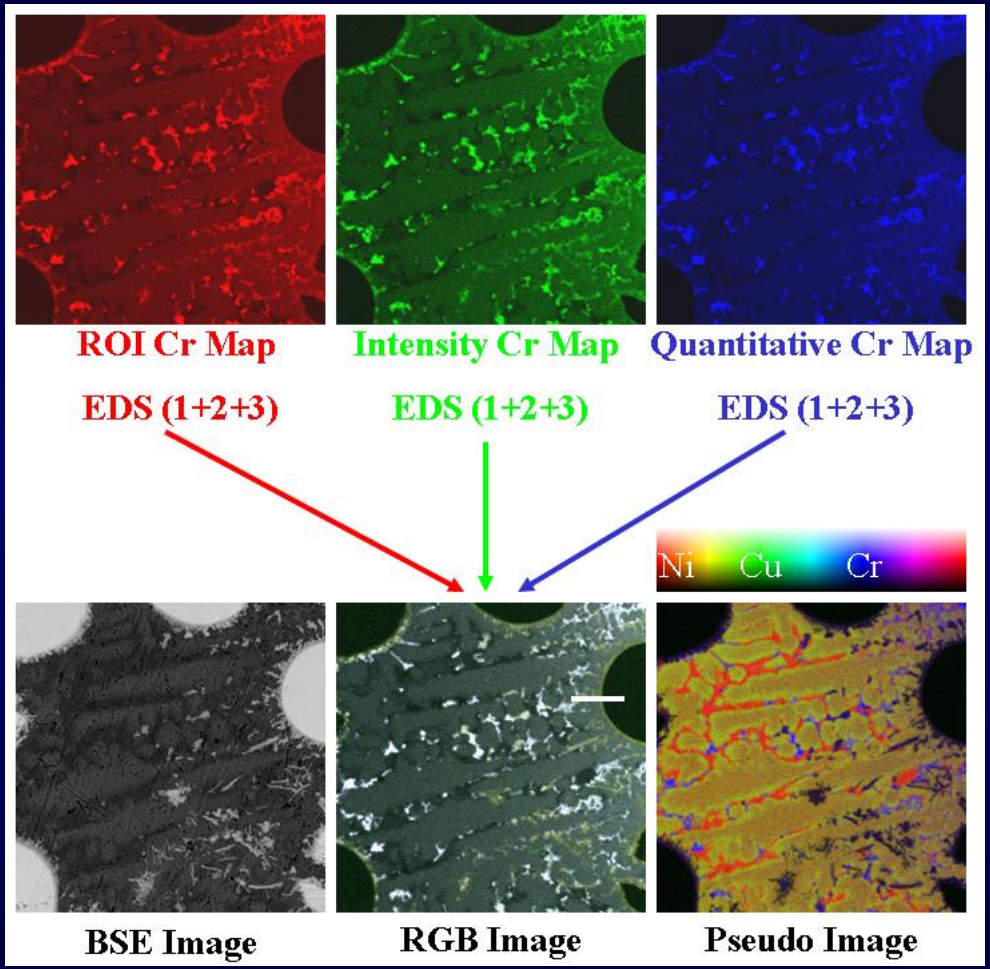
- Quantitative elemental x-ray maps produced from a hard facing bonded to a chrome steel.



Cr 0-20% Fe 0-30% Ni 0-70% Cu 0-30% WC 0-100%wt

- Maps collected at 25KV, 512x512 pixels and 12 hours (HWOF=110µm).
- The beam current for this image was 1nA with a combined input count rate of 20,000cps. The map was collected with three EDS detectors having a combined detector area of 70mm^2.

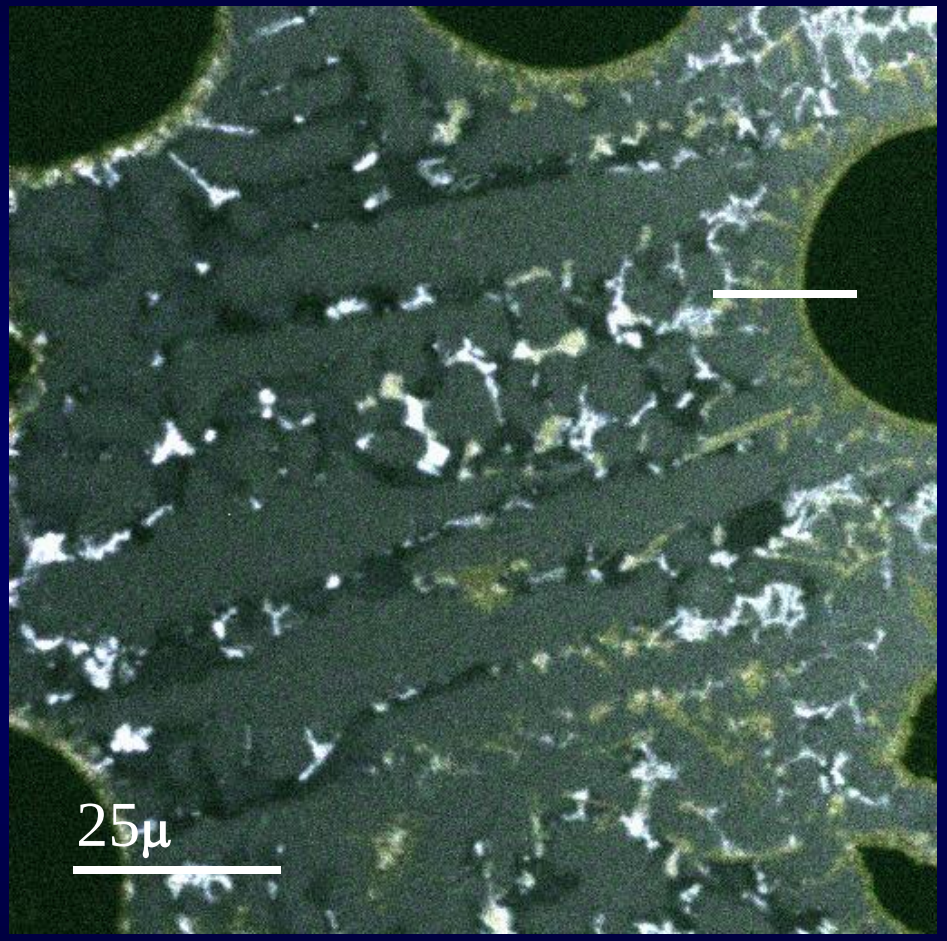
Quantification Performance Test (QPT)



- A different RGB colour is assigned to the combined sum of all detectors for an individual element.
- The information we are now looking for is the difference between the region of interest map, stripped intensity map and quantitative map for the same element.
- The RGB image shows a grey scale map indicating miss correlation between the three elements.
- Also shown is the pseudo image for the three elements present (Ni, Cu, Cr).

  HWOF=110µm.

Importance of Quantitation



Chromium image

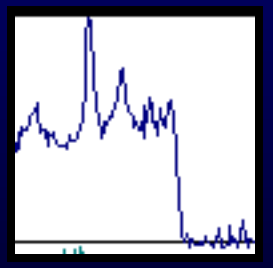
- Colour in this image is caused by a difference in one or more of the RGB layers.

  Layer 1 = ROI

  Layer 2 = Stripped Intensity

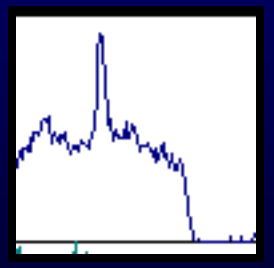
  Layer 3 = Quant

- This shows a 1% variation in composition from 5.5% to 6.5% Chromium.
- This affect is caused by no atomic correction (Z) done on the intensity profile



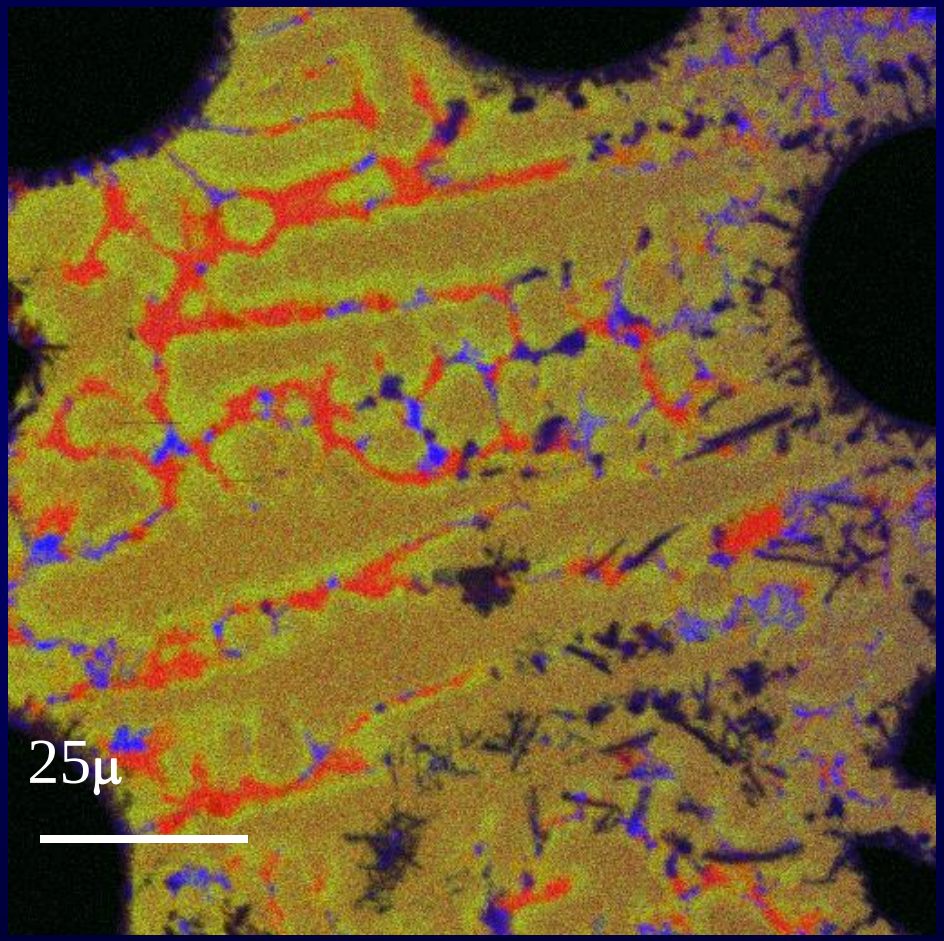
Intensity

Quant

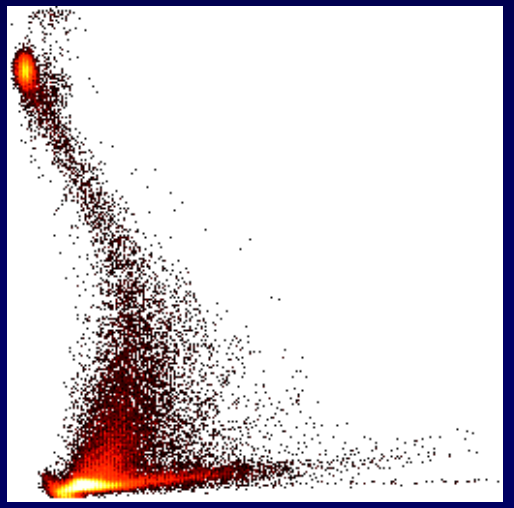


Chemical Phase Location

Ni Cu



Cr
W



Fe

Cr

Composite X-ray image

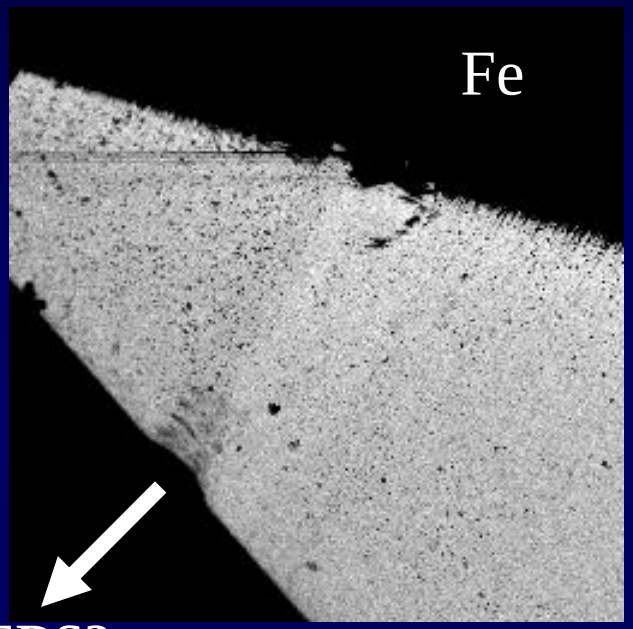
HWOF=110μ 100msec/pixel, 512x512

Cr and W forming distinct phases

(Blue swapping between W and Cr)

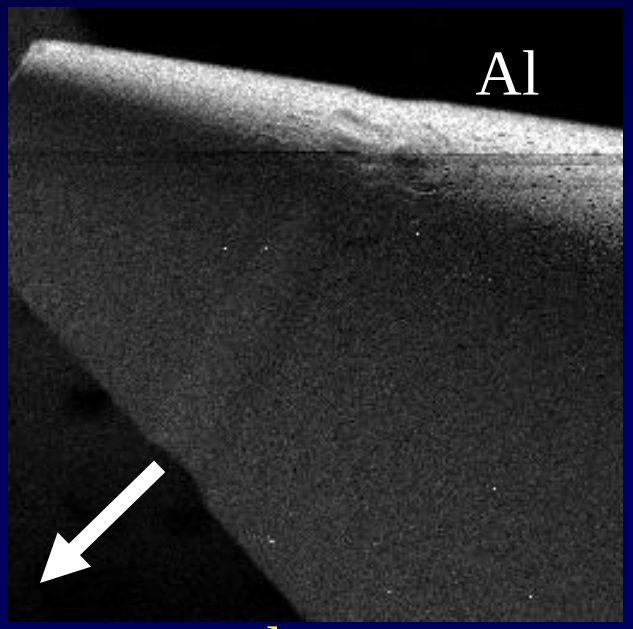
Geometry of Detectors (Needle Valve)

Si



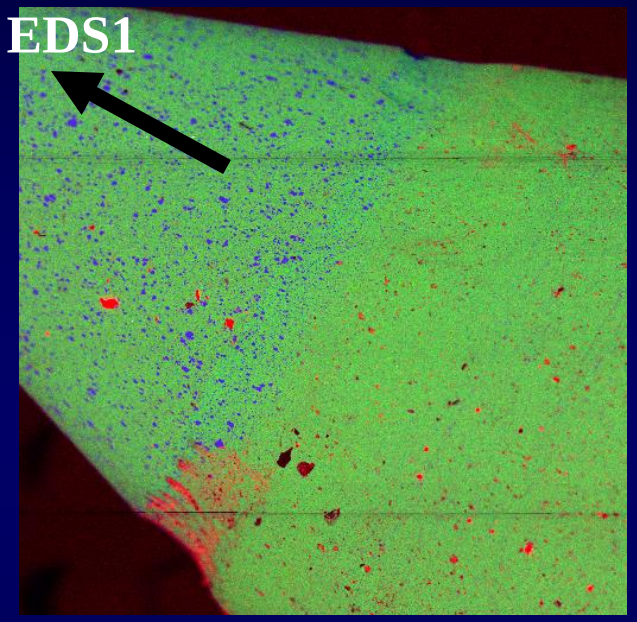
a

Fe



b

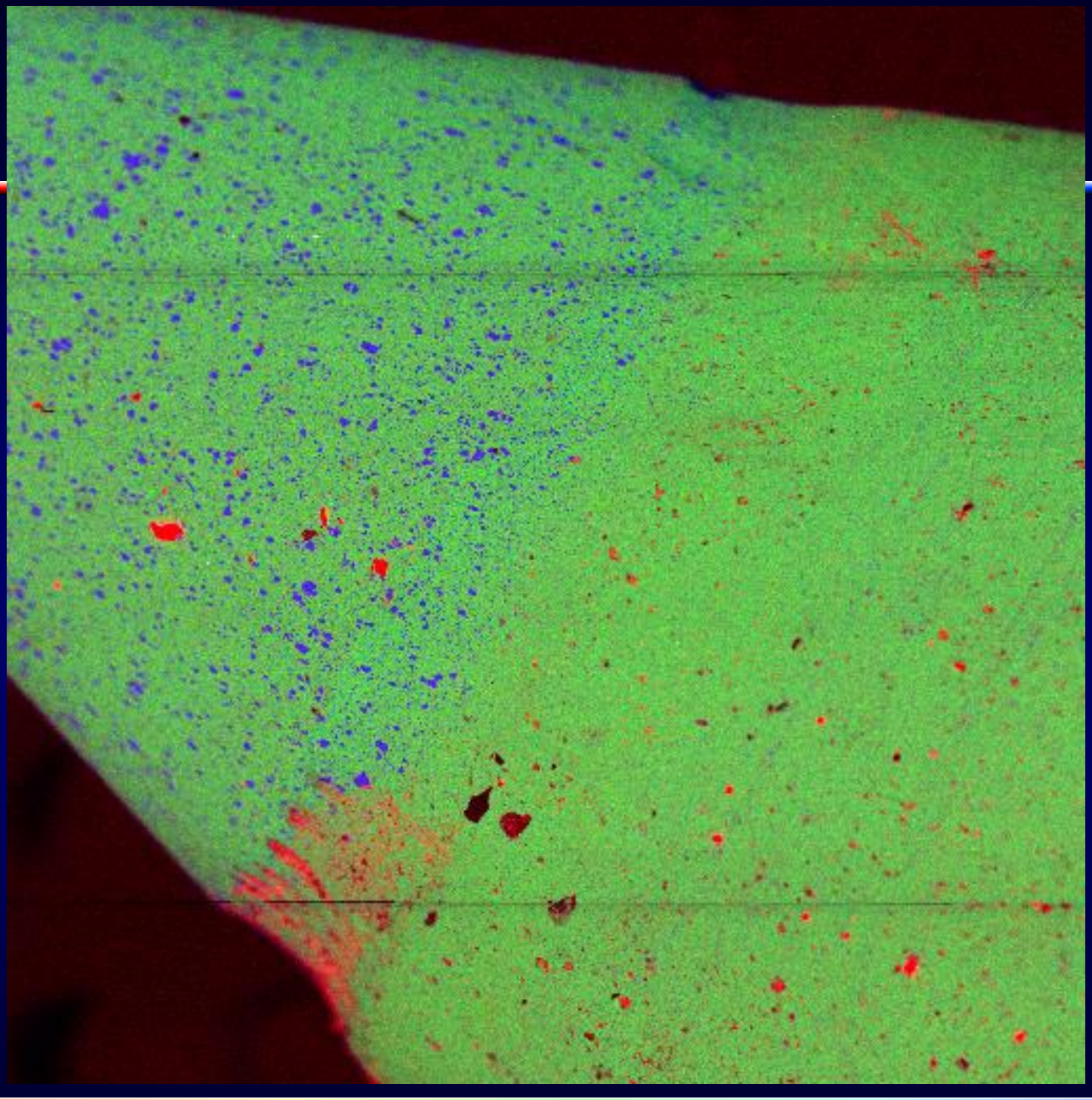
Cr



c

- Simultaneously mapped using 2 detectors. 512x512. Maps a and b are from detector 2, which cannot see the whole field of view. There is no Al in the sample. The amount of Al x-ray production is proportional to the BSE signal that reaches the specimen holder made from Al. Map c from detector EDS1 is more representative of the elemental distribution.

Needle Valve



Fe, Green

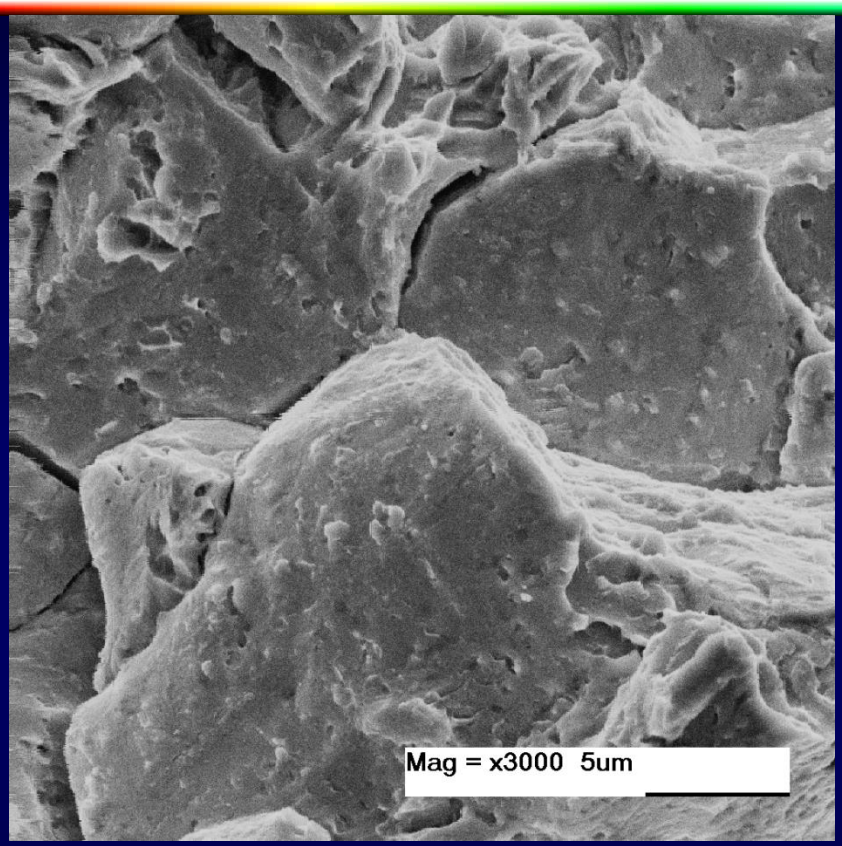
Cr, Blue

Si, Red

As seen from a different detector

Rough Samples
Inter Granular Fracture

EDS

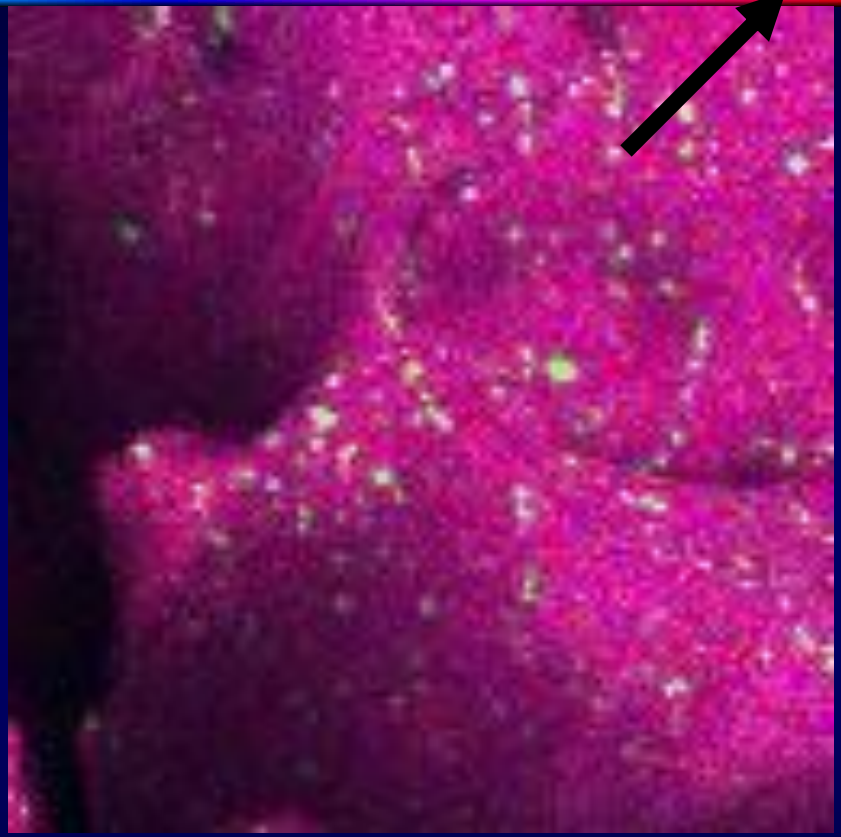


V

Cr

Cr-Fe Phase

Fe



- Sample is Vanadium Carbide precipitates in Fe-Cr Phase (Chromium Steel).
- Rough Surface x-ray map done at high magnification, which shows partial morphology for the steel but complete morphology for the carbides

Conclusion

- To completely characterise a sample, a number of post-processing methods should be employed. These include:
  - elemental mapping
  - pseudo colouring
  - ratio mapping
  - scatter diagram creation and rotational scatter diagrams
  - phase mapping
  - generation of theoretical BSE images
  - generation of correction images (Z, A, F)

- Through the use of x-ray mapping and post-processing techniques (chemical imaging), a better understanding of a materials chemical properties and chemical phase information can be determined.