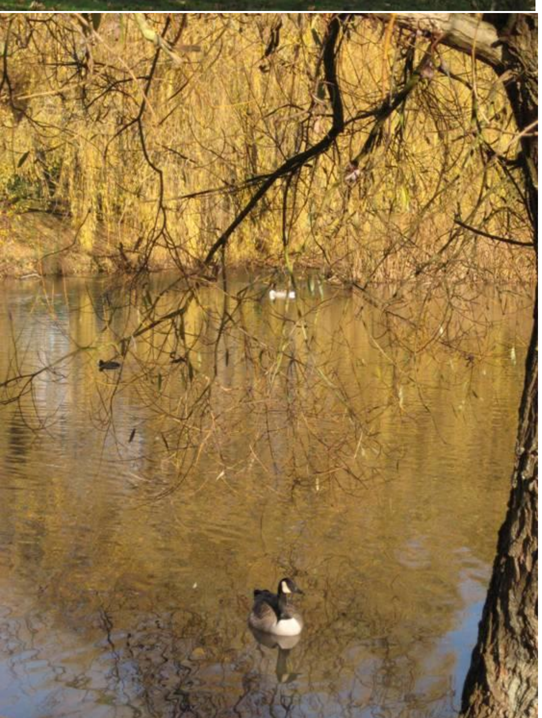

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Lost image recovery for stained glass panels from the Rosslyn Chapel

IoP meeting: Surface Modification and Analysis
Coventry, 15th October 2014
Wednesday 15th October 2014, 4.30-5.00 pm
5th Vacuum Symposium (part of Vacuum-EXPO, 15-16 October 2014), Ricoh Arena, Coventry

C. Jeynes, A. Hamilton*, G.W. Grime, V.V. Palitsin, J.L.Colaux, L. Antwis, R.P. Webb, M. Bambrough**

University of Surrey Ion Beam Centre, Guildford, England
** Dept. Civil and Environmental Engineering, University of Strathclyde*
*** Scottish Glass Studios, Glasgow*

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  - ***Glass deterioration***
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  - ***Imaging by Ion Beam Analysis (IBA)***
  - ***Why XRF doesn't work***
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- Discussion: materials analysis
  - ***Total-IBA***
  - ***Accuracy***

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Acknowledgements

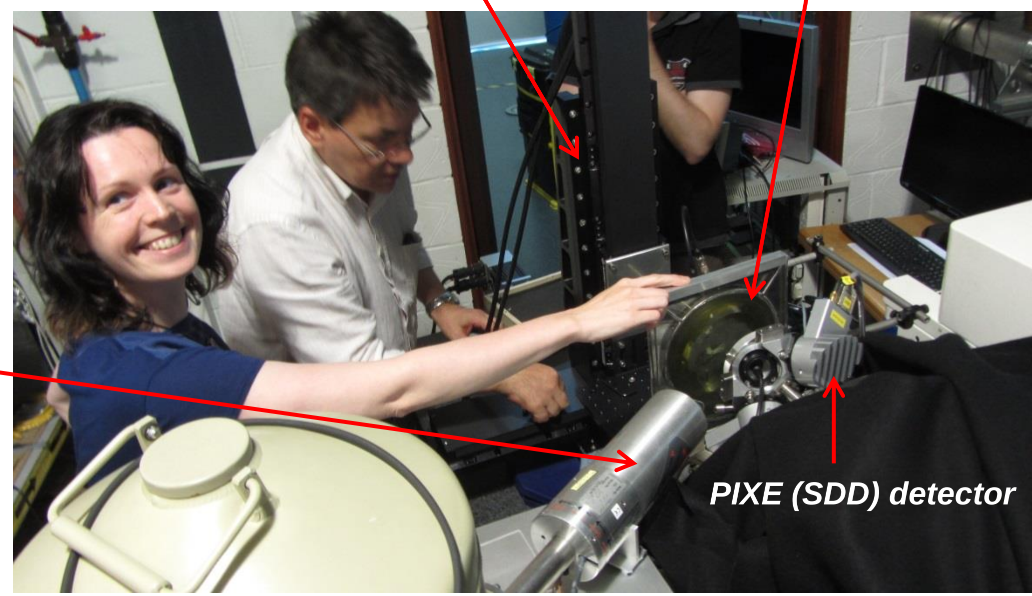
This project is partially funded by The Rosslyn Chapel Trust (Registered Charity number SC024324, Lord and Lady Rosslyn, Owners and Trustees), and WREN Heritage Fund (www.wren.org.uk)

Mark Bambrough
(still from the video on the Rosslyn Chapel website about the glass restoration project).

Motorised XYZ stage

Rosslyn glass window sample

Gamma-ray detector (HPGe)



Andrea Hamilton and Vladimir Palitsin
setting up the external beam.

Rosslyn Chapel

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- 7 miles from Edinburgh
- Category A listed building
- Scheduled Ancient Monument

- Built 1446-1484
- St.Clair family trace their line back to Rognvald, Jarl of the Orkneys and Norwegian Earl, born 835 AD: his son fought in France and became 1st Duke of Normandy 912 AD: his son William was related to William the Conqueror and fought at Hastings in 1066. St.Clairs served the Scottish kings prominently until the Union.
- 1441: ***Grand Master Mason*** title granted by James II
- 1571: Rosslyn Chapel ***endowments*** seized in the Reformation
- 1592: Altars destroyed, Chapel described as "***an house and monument of idolatrie***"
- 1736: windows ***glazed*** for the first time
- 1862: church services restart
- Visited by Robert Burns, Walter Scott, Dorothy Wordsworth, Queen Victoria, Dan Brown

Rosslyn Chapel: Lost Image Research Project

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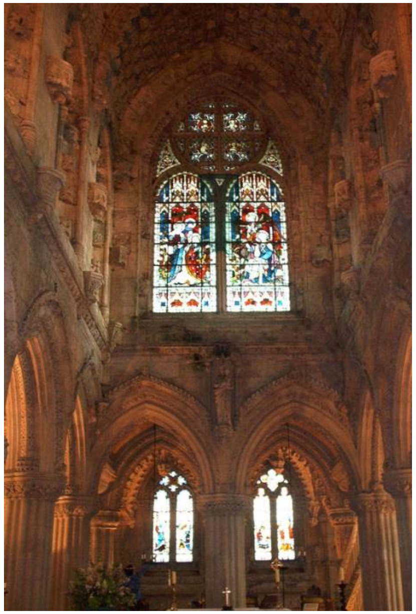
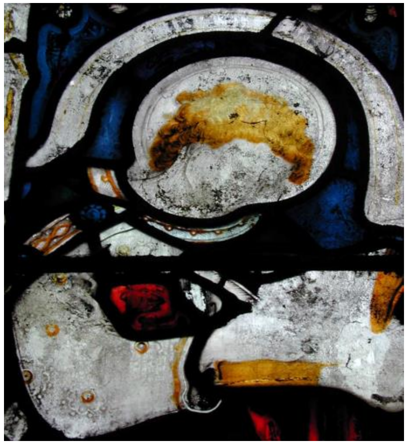


1867-1887: Windows manufactured by Clayton & Bell of London, with the "grisaille" method.

Stained glass is an essential part of the visual language of mediaeval religious architecture intended to generate a sense of the "*beauty of holiness*".

"The stained glass windows that are in churches and through which… the clarity of the sun is transmitted signify the Holy Scriptures, which *banishes evil from us and enlightens our being*" (Pierre de Roissy, Chartres Cathedral, *circa* 1200).

East window: "*Three Marys*" (post-restoration)



Grisaille: glass paint fired into window

Severe environmental degradation has resulted in loss of grisaille: the liturgical context makes *image legibility* crucial.

Research Problem: *can the lost images be recovered?*

Window nII: "*John the Baptist, with lamb*"

Glass Deterioration

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Ion Beam Centre

- Conditions
  - *Rosslyn Chapel very exposed*
  - *Building not waterproof for >50 years*
- Deterioration
  - *Internal surfaces had poor conditions (high humidity, cold etc)*
  - *External surfaces suffer severe weather conditions*
  - *Different interior/exterior mechanisms*
  - *Poor kiln firing conditions*
- Previous work on glass deterioration
  - *Used only spot analyses*
  - *Little depth information*
  - *Glass science still sketchy*

Lost Image Recovery ?

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The Resurrection Panel

Analytical Methods to see the Lost Image

- XRF: poor lateral resolution
- NIR reflectography: not sensitive enough
- X-ray radiography: not sensitive enough
- PIXE/EBS/NRA: sensitive, good resolution

Ion Beam Analysis

- External beam! (PIXE = XRF)
- Ion beam can be focussed to <10 microns
- PIXE gives elemental specificity
- Backscattering gives depth sensitivity
- He-PIXE gives near-surface Na
- PIGE gives bulk Na

Lost Image Recovery: Imaging by IBA

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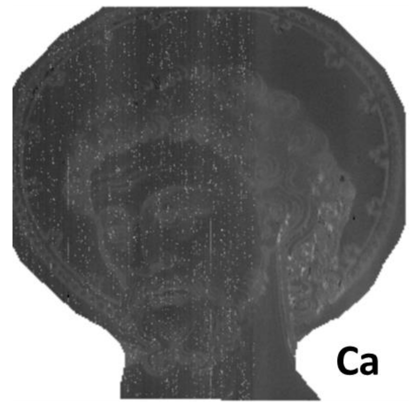
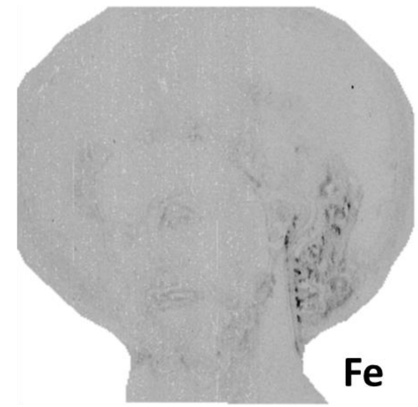
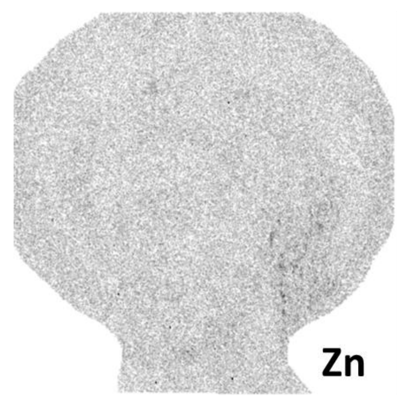
Ion Beam Centre

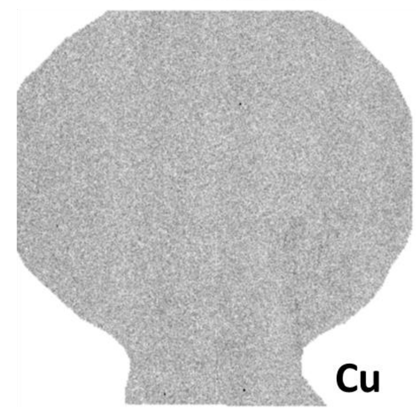
- Long run (overnight) with mechanical scanning
- 2.5 MeV protons with PIXE (particle-induced X-ray emission)
- Particle backscattering and gamma rays also collected

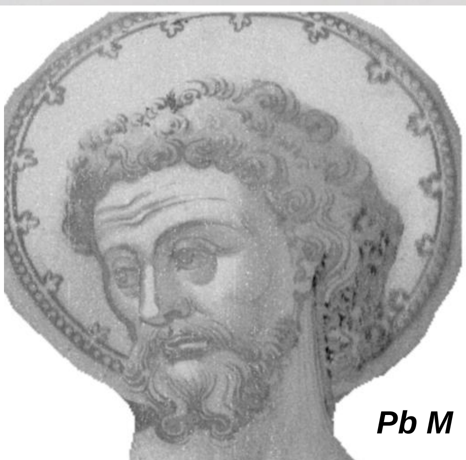
- These data are “low” resolution
- Plan to run He^+ too
- New spectrometry
- New software
- New hardware
- New beamline











Deus ex Machina !

Ion Beam Analysis Methods

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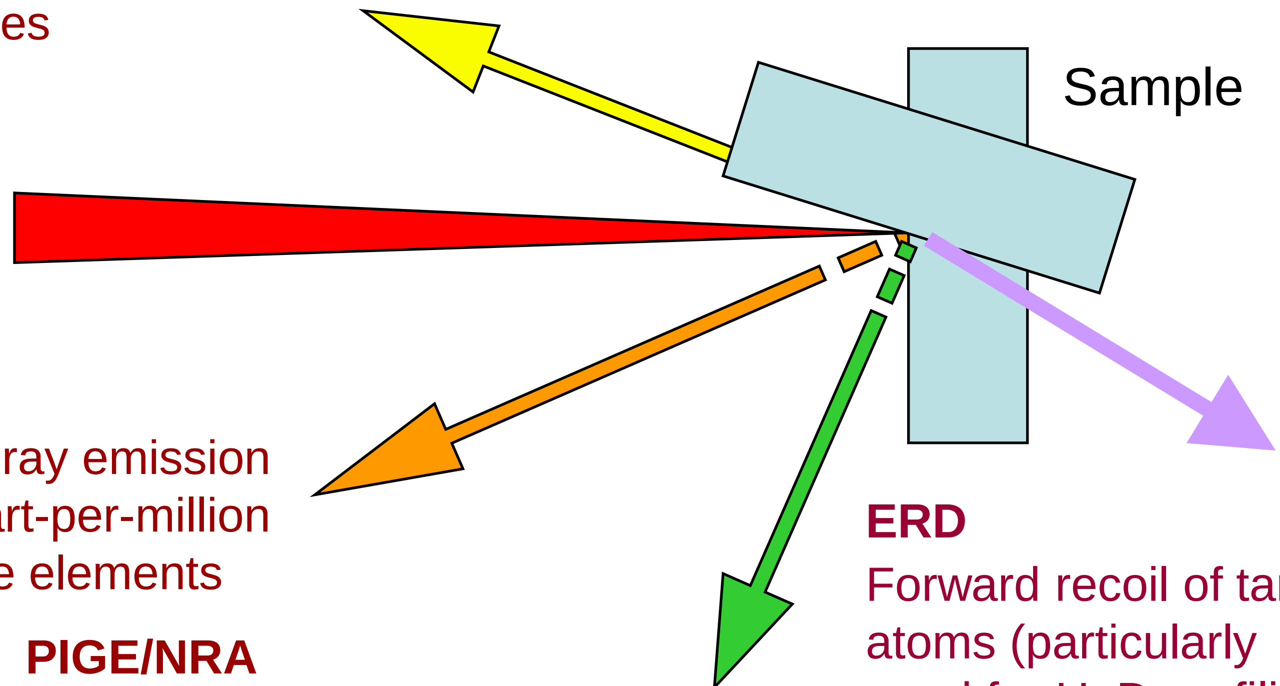
IBIL:	ion beam induced luminescence
IBIC:	ion beam induced current
STIM:	scanning transmission ion microscopy
MeV-SIMS:	secondary ion mass spectrometry

EBS/RBS
Energy of elastically scattered particles gives light element composition and elemental depth profiles

3 MeV protons



PIXE
Characteristic X-ray emission
Simultaneous part-per-million detection of trace elements from Na to U

PIGE/NRA
Nuclear reactions give characteristic gamma rays and/or particles from light nuclei (e.g. Li, B, F)

ERD
Forward recoil of target atoms (particularly good for H, D profiling)

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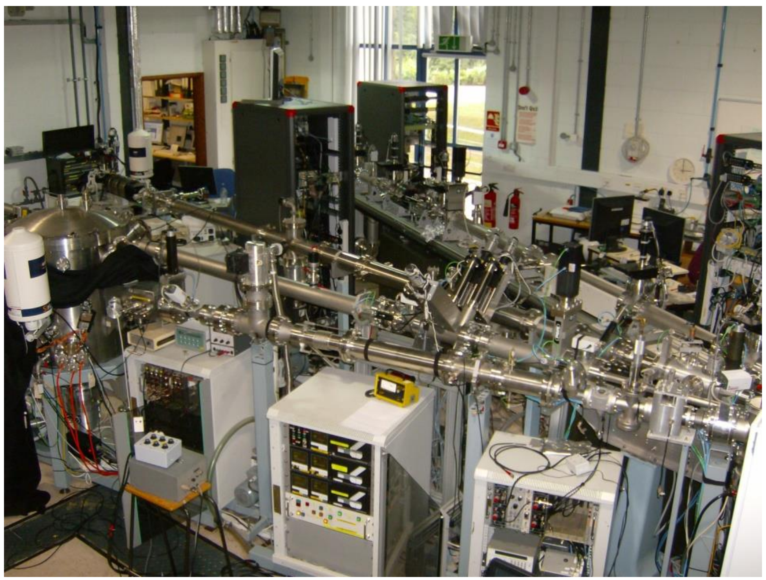
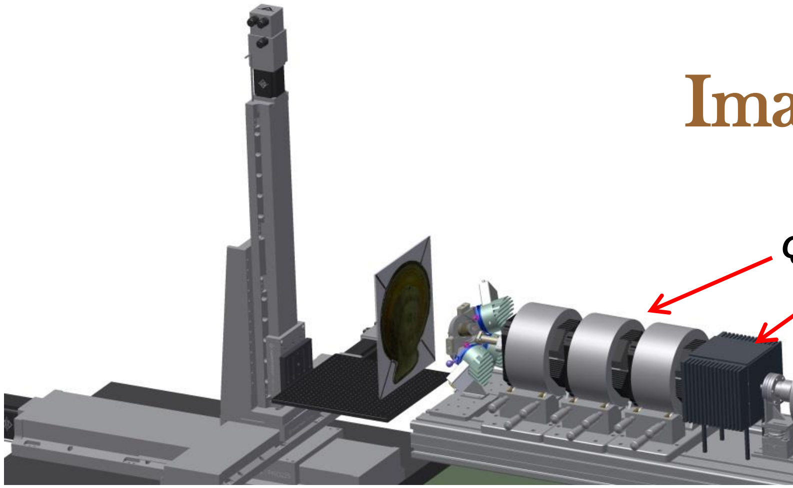
Imaging by IBA

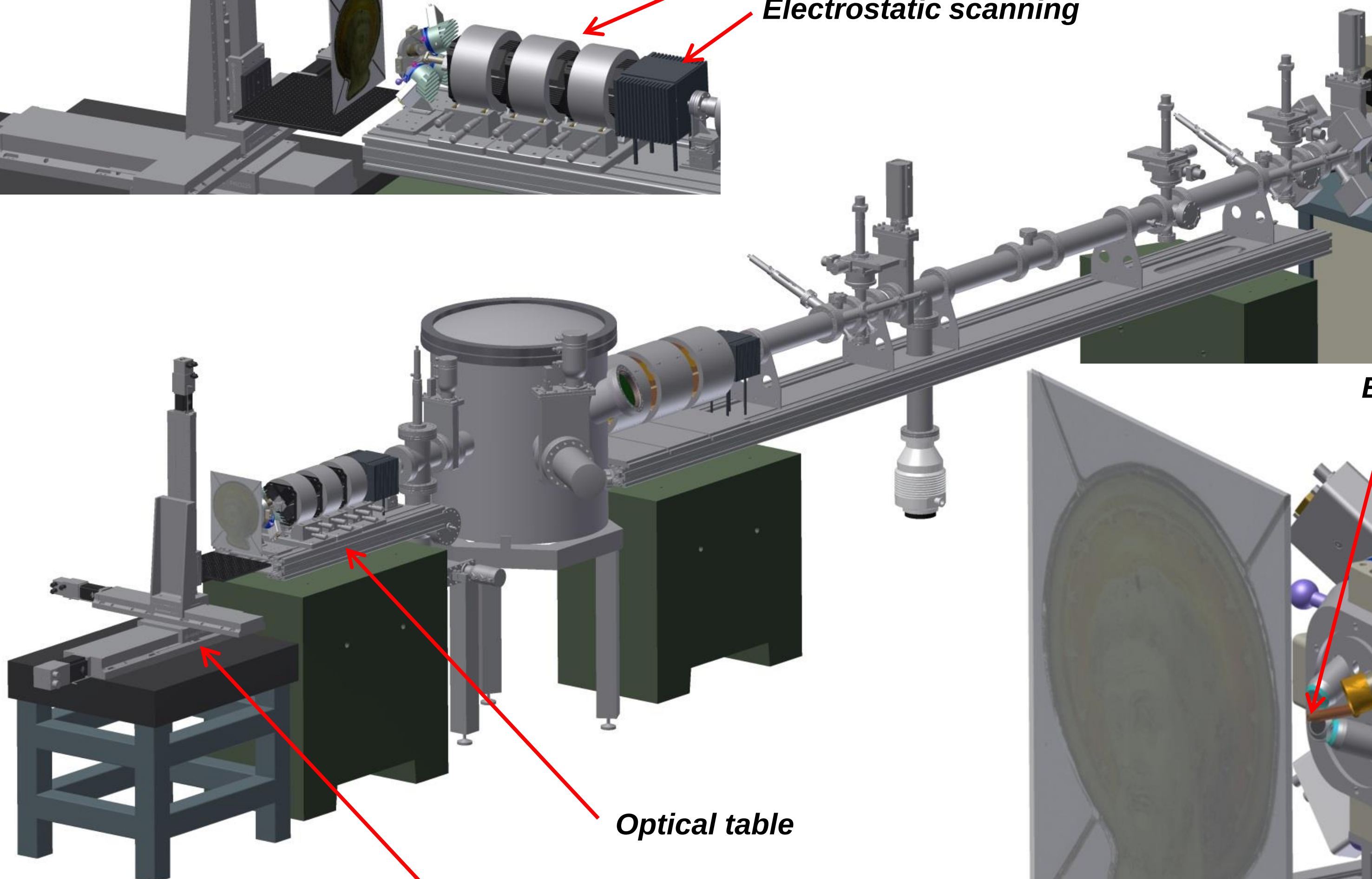


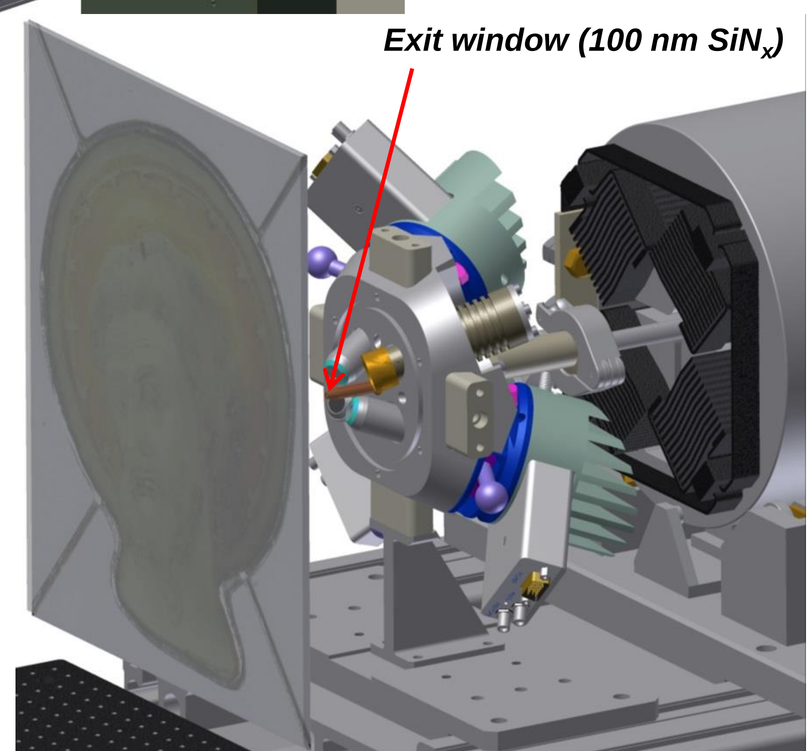
Beamlines for 2 MV Tandem

2 MV Tandem, installed at Surrey 2002

Imaging by IBA







IBA of large, valuable, or wet samples

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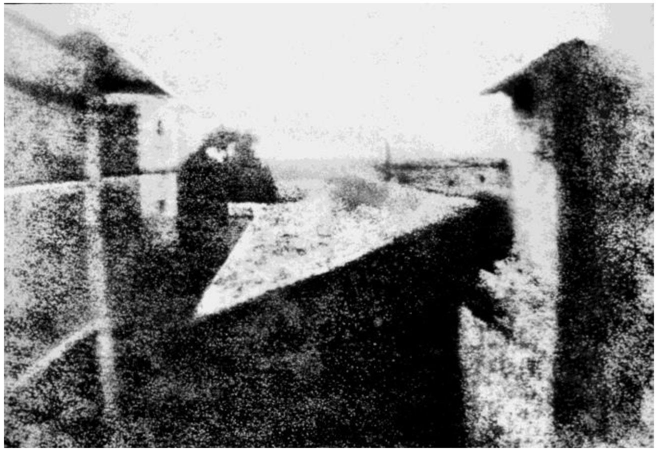
- External beam (atmospheric)
- Old glass cannot be put into a vacuum (internal bubbles)
- Large samples may be too big for vacuum chamber
- Wet samples cannot go into vacuum
- Precious samples are chained to curator

L. Beck (Louvre), C. Jeynes (Surrey), N.P.Barradas (Lisbon)
Nucl. Instrum. Methods B, 266 (2008) 1871-1874

"La Bohémienne", Frans Hals 1630

AGLAE : Accélérateur Grand Louvre d'Analyse Élémentaire

C.Pascual-Izarra (Madrid), N.P.Barradas, M.A.Reis (Lisbon), C. Jeynes (Surrey), M.Menu, B.Lavdrine, J.J.Ezrati, S.Röhrs (Louvre)
Nucl. Instrum. Methods B, 261 (2007) 426-429



Niepce's first Heliography: Paysage à Saint-Loup de Varennes (1827)

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Complementary or Competing?

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- XRF vs IBA footprint!
- XRF push-button, IBA complex

(Florence, LABEC)

Why XRF doesn't work

- X-ray fluorescence is like PIXE (particle-induced X-ray emission) except that ionisation is by photons instead of charged particles
- Main background is from secondary electrons for both XRF and PIXE
- Information depth is ~100 μm for XRF, ~20 μm for PIXE
- Microfocussed XRF needs capillary lenses (and high intensity X-ray source). Usually a synchrotron technique. Desktop XRF is mostly low resolution.
- Depth information hard to extract from XRF, also very low depth sensitivity.
- Depth information directly available in IBA from particle backscattering (energy loss).
- Electrostatically scanned ion beam gives image very rapidly. This is very useful for verifying the imaged area.
- For XRF (and IBA), mechanical scanning is slow.
- XRF is blind to light elements (low ionisation and small fluorescence yield).
- Backscattering and NRA are sensitive to light elements, and hydrogen content can be obtained with elastic recoil (or NRA) methods.
- IBA has reliable quantitation and mass closure. Desktop XRF quantification depends on uniformity in depth, and even so is always troublesome because the tube spectrum is hard to characterise.

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Glass science issues: test sample

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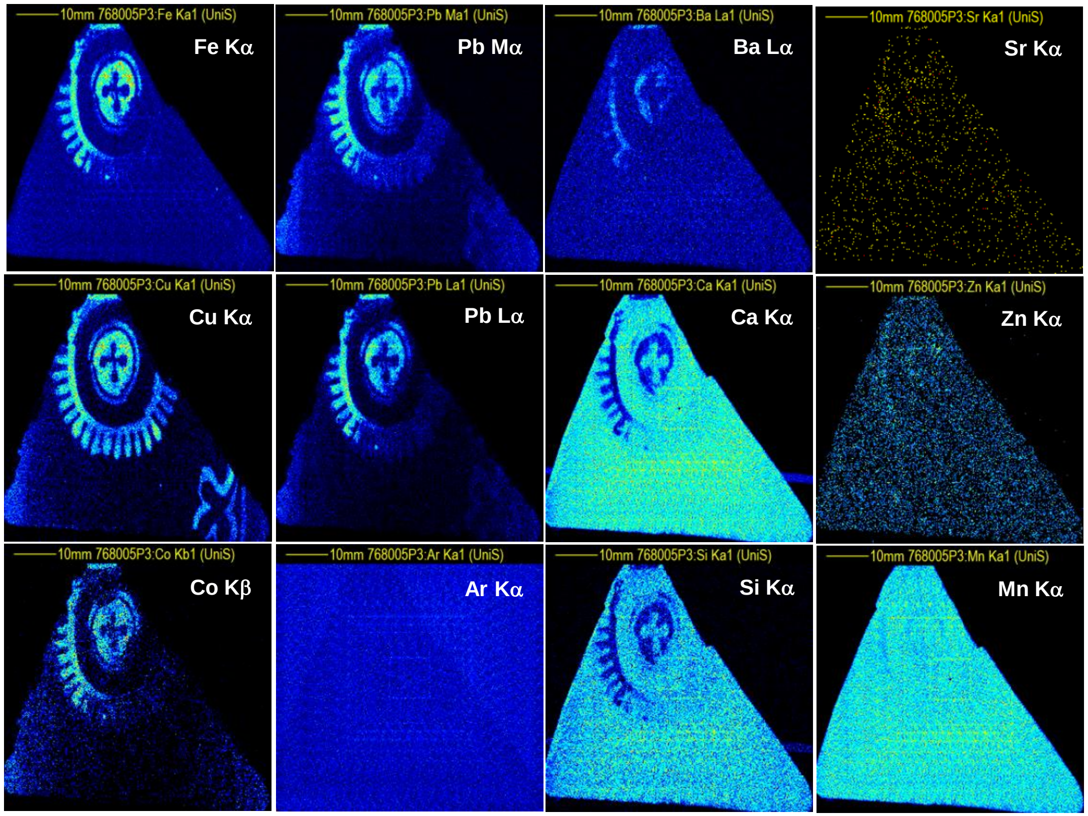
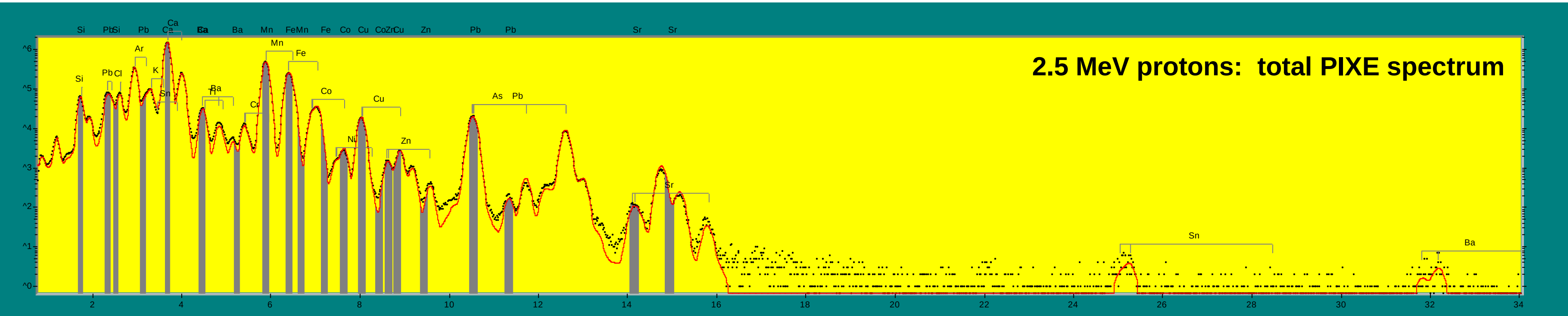


- Ar is in atmosphere
- Mn is in glass bulk
- Low energy Ca & Si signals absorbed by surface layers
- Pb L/M ratio >> process info
- Ba & Ca ***not*** correlated
- Transition metals in grisaille
- Mapping quantitation problems remain: see artefacts in Ar map
- PIGE not collected
- EBS faulty (collection unsuccessful)
- 42287 secs collection time (~12 hr)
- 3 * 1.7 kHz (SDDs can run 100 kHz)
- 1024×1024 pixels (200 mm sample)



Rosslyn Glass: test sample (1024×1024 pixels)

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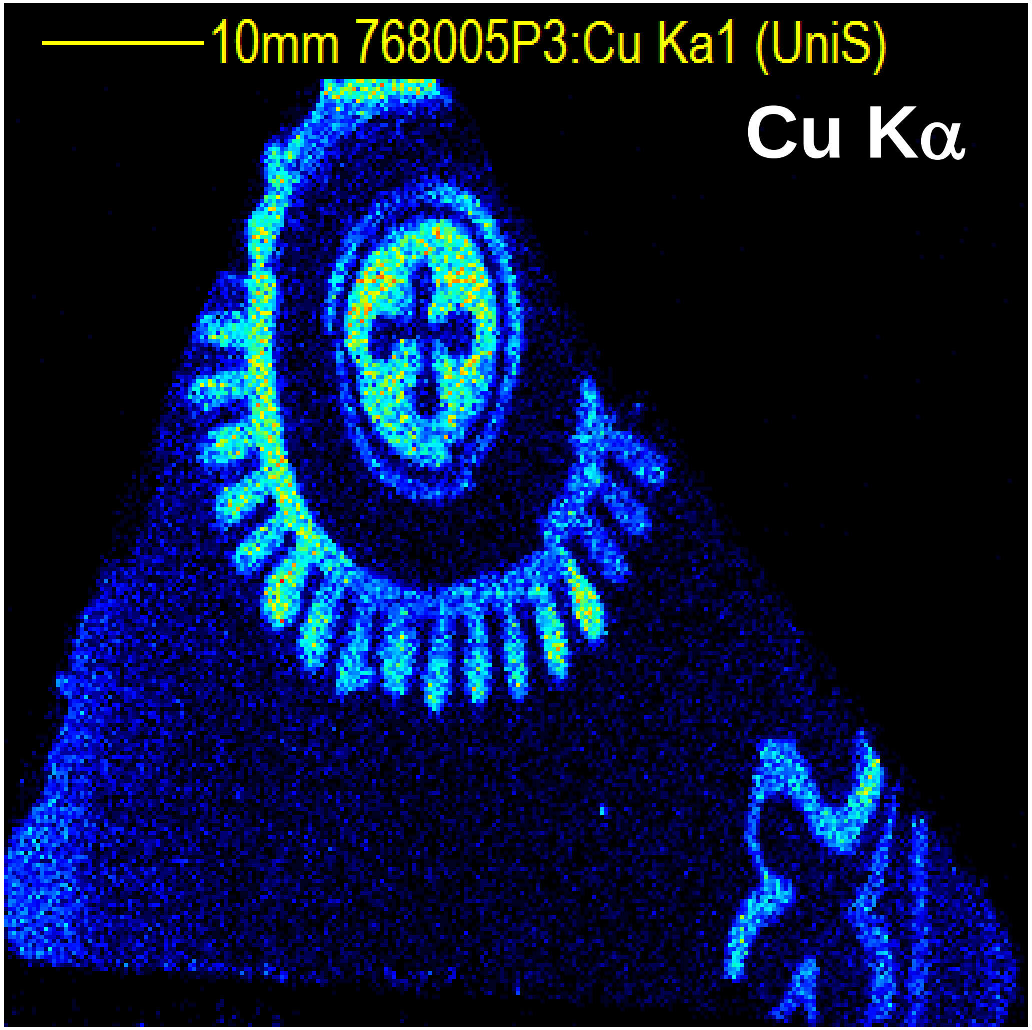

External Scanning Microbeam: Rosslyn Chapel glass

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52 mm

R1

39 mm

R2

60 mm

R3

50 mm

R4

50 mm

R5

82 mm R6

- Preliminary analysis from 2009
- No digital spectrometry
- Slow Si(Li) detectors, not fast SDD detectors
- No motorised XYZ stage

External Scanning Microbeam: Rosslyn Chapel glass

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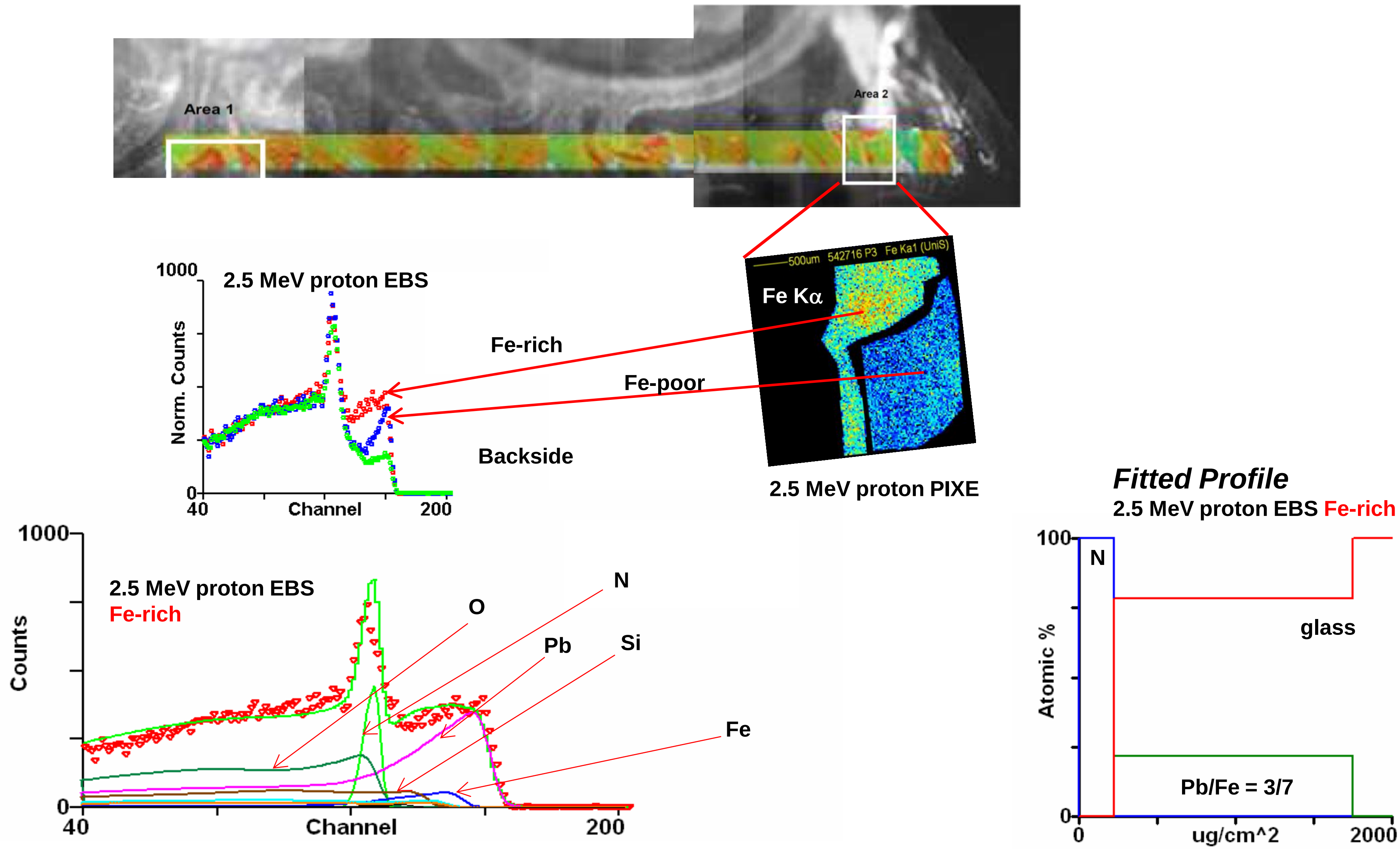
- Comparing 3 different Rosslyn samples with literature (“GOY7”)
- R#2 is a similar glass to GOY7
- R#1 and R#6 are different glasses from R#2
- The “inside” and “outside” surfaces are different

Glass Substrate composition (at%)

		Rosslyn #2			Rosslyn #1				Rosslyn #6			
	GOY7	back	Fe-poor	Fe-rich	back, off SS	back, on SS	Fe-poor	Fe-rich	back, off SS	back, on SS	Fe-poor	Fe-rich
Si	22.70	22.39	21.85	22.52	18.34	18.82	10.11	8.16	26.40	16.34	26.64	26.67
Na	7.25	7.10	6.93	7.14	18.57	17.54	20.64	24.51	8.53	13.87	8.61	8.62
Ca	5.83	5.72	5.58	5.76	4.15	4.68	9.35	8.02	5.40	9.87	5.45	5.46
K	5.17	4.88	5.70	4.30	0.30	0.30	0.80	0.46	0.70	0.83	0.54	0.40
Pb	0.91	0.87	0.84	0.96	0.0082	0.0084	0.0220	0.0960	0.0007	0.0227	0.0001	0.0005
Fe	0.48	0.49	0.48	0.49	0.20	0.20	0.53	0.25	0.47	0.55	0.26	0.41
Mn	0.12	0.11	0.16	0.35	0.02	0.02	0.05	0.08	0.07	0.05	0.09	0.05
O	59.49	58.25	58.23	58.20	58.26	58.26	58.15	58.26	58.26	58.23	58.28	58.30
Al	0.0921	0.0920	0.1032	0.1247								
Ti	0.0715	0.0713	0.0799	0.0965	0.0178	0.0191	0.0393	0.0182	0.0180	0.0236	0.0138	0.0097
Ni	0.0119	0.0120	0.0179	0.0087								
Cu	0.0106	0.0107	0.0144	0.0215	0.0011	0.0011	0.0024	0.0011	0.0033	0.0014	0.0037	0.0026
Zn	0.0040	0.0040	0.0047	0.0185	0.0018	0.0019	0.0039	0.0018	0.0054	0.0023	0.0062	0.0044
Sr					0.0082	0.0088	0.0181	0.0084	0.0083	0.0109	0.0063	0.0044
Ba					0.0858	0.0917	0.1893	0.0877	0.0868	0.1137	0.0665	0.0465
As					0.0460	0.0491	0.1014	0.0470	0.0465	0.0609	0.0356	0.0249

External Scanning Microbeam: Rosslyn Chapel glass (R#2)

University of Surrey Ion Beam Centre



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Accurate measurement of implanted fluence

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SPIRIT

Colaux & Jeynes, *Analytical Methods*, 6 (2014) 120-129

"… few techniques capable of non-destructive and model-free measurement at 1% absolute accuracy without the use of sample-matched standards"

Analytical Methods

ROYAL SOCIETY OF CHEMISTRY

PAPER

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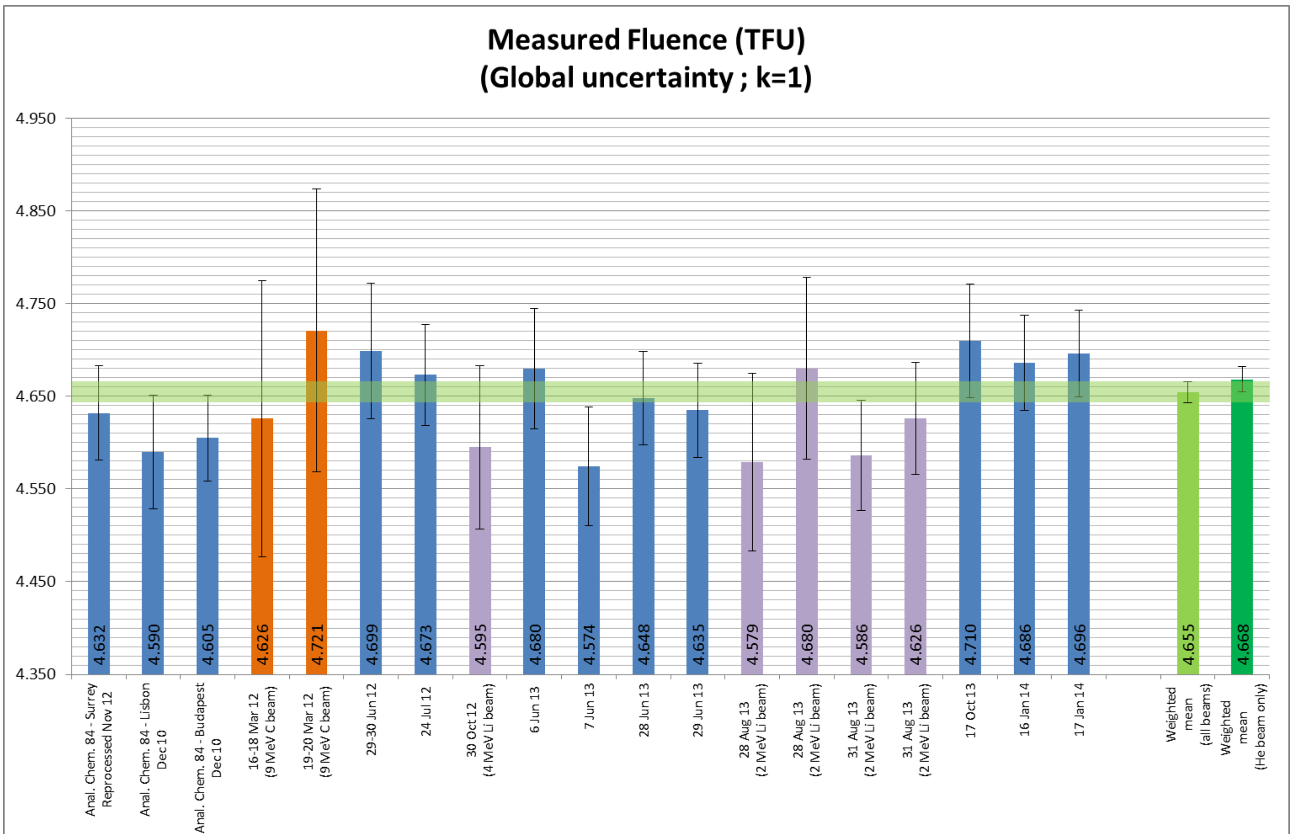
Cite this: *Anal. Methods*, 2014, **6**, 120

High accuracy traceable Rutherford backscattering spectrometry of ion implanted samples

J. L. Colaux* and C. Jeynes

There are few techniques capable of the non-destructive and model-free measurement at 1% absolute accuracy of quantity of material in thin films without the use of sample-matched standards. We

Repeated RBS over 2 years
"SPIRIT21" sample (150 keV ~5.10^{15}As/cm^2)



Absolute (traceable) 1% accuracy confirmed:

- ***SE of mean is 0.3%***
- ***Shows error well estimated***
- ***DF line 1 accurate at 1%***

- ***2 detectors essential***

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Analytical Methods

PAPER

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High accuracy traceable Rutherford backscattering spectrometry of ion implanted samples

Cite this: *Anal. Methods*, 2014, 6, 120

J. L. Colaux* and C. Jeynes

RBS Uncertainty Budget

Colaux & Jeynes, *Analytical Methods*, 6 (2014) 120-129

Table 1 Uncertainty budget obtained for the fluence determination of the As-implant sample using a $^4He^+$ 1.5 MeV ion beam (Q = 40 μC)[a]

<table>
<tr><th></th><th>Type</th><th>Det A</th><th>Det B</th></tr>
<tr><td>Pileup correction (As signal), %</td><td></td><td>0.3</td><td>0.8</td></tr>
<tr><td>Pileup uncertainty (10% of total correction), %</td><td>B_1</td><td>0.03</td><td>0.08</td></tr>
<tr><td>Counting statistics, implant signal, %</td><td>A_1</td><td>1.60</td><td>0.85</td></tr>
<tr><td>Counting statistics, a-Si signal, %</td><td>A_2</td><td>0.25</td><td>0.13</td></tr>
<tr><td>Scattering angle $\sim 1/\sin^4(\theta/2)$ and $1/\cos(\theta)$, %</td><td>B_2</td><td>0.08</td><td>0.41</td></tr>
<tr><td>Electronics calibration uncertainty, %</td><td>A_3</td><td>0.26</td><td>0.14</td></tr>
<tr><td>Relative uncertainty (dataset), %</td><td>U_1</td><td>1.64</td><td>0.97</td></tr>
<tr><td>Relative uncertainty of average of 2 detectors (dataset), %</td><td>U_2</td><td colspan="2">0.83</td></tr>
<tr><td>Beam energy, %</td><td>B_3</td><td colspan="2">0.20</td></tr>
<tr><td>Disagreement between both detectors, %</td><td>A_4</td><td colspan="2">0.00</td></tr>
<tr><td>Pileup uncertainty (from model), %</td><td>B_4</td><td colspan="2">0.20</td></tr>
<tr><td>Code uncertainty, %</td><td>B_5</td><td colspan="2">0.20</td></tr>
<tr><td>Rutherford cross-section (screening uncertainty), %</td><td>B_6</td><td colspan="2">0.16</td></tr>
<tr><td>Si stopping power (uncertainty on the a-Si yield), %</td><td>A_5</td><td colspan="2">0.80</td></tr>
<tr><td>Si stopping power (uncertainty on the implant counts), %</td><td>A_6</td><td colspan="2">0.03</td></tr>
<tr><td>Combined extra systematic uncertainty, %</td><td>U_3</td><td colspan="2">0.89</td></tr>
<tr><td>Total combined standard uncertainty (accuracy), %</td><td>U_4</td><td colspan="2">1.22</td></tr>
<tr><td>Reduced uncertainty</td><td>U_5</td><td colspan="2">0.83</td></tr>
</table>

[a] $U_1^2 = \sum_{i=1}^{3} A_i^2 + \sum_{i=1}^{2} B_i^2$; $U_2^{-2} = U_{1A}^{-2} + U_{1B}^{-2}$; $U_3^2 = \sum_{i=4}^{6} A_i^2 + \sum_{i=3}^{6} B_i^2$; $U_4^2 = U_2^2 + U_3^2$. Measurement uncertainties of type A (statistical estimate

Complementary PIXE/backscattering

In layered structures, need depth profile to correctly calculate PIXE absorption

PIXE strength

- High sensitivity
- Excellent specificity

BS strength

- Traceable accuracy
- Excellent depth resolution

BS weakness

- Low sensitivity
- Poor mass resolution

PIXE weakness

- Poor traceability
- Poor depth resolution

NB: PIXE information depth is significantly larger than for BS

Characterization of paint layers by simultaneous self-consistent fitting of RBS/PIXE spectra using simulated annealing

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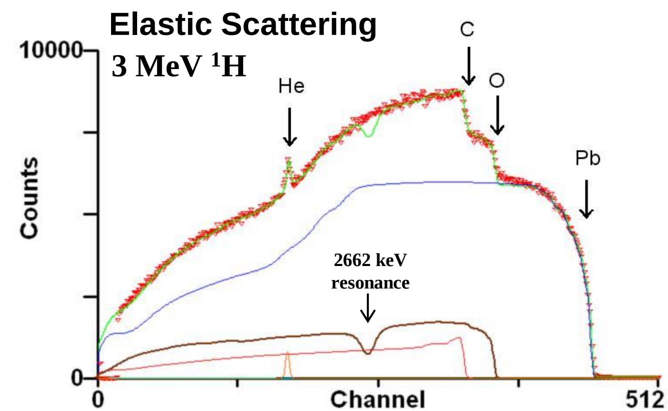
Ion Beam Centre

L. Beck (Louvre), C. Jeynes (Surrey), N.P.Barradas (Lisbon)

***Nucl. Instrum. Methods B*, 266, 1871-1874 (2008)**

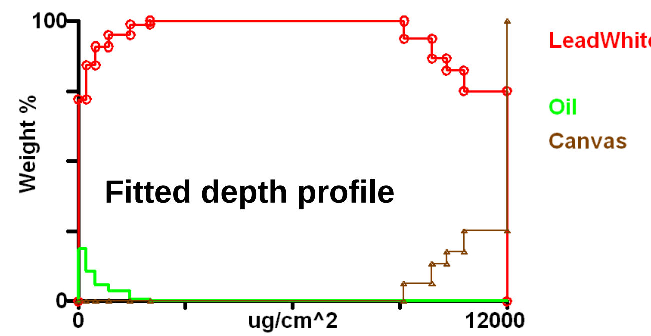
"La Bohémienne", Frans Hals 1630

AGLAE : Accélérateur Grand Louvre d'Analyse Élémentaire



Ochre (haematite) pigment located and quantified by PIXE/RBS/EBS

(PIXE not shown)



See also:

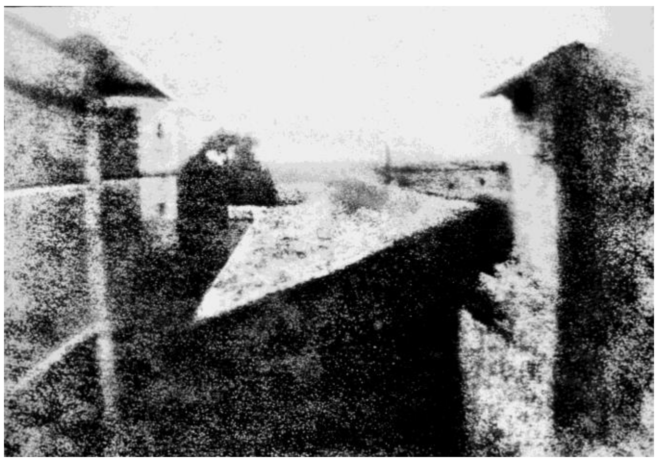
L.de Viguerie, L.Beck, J.Salomon, L.Pichon, Ph.Walter, Composition of Renaissance paint layers: simultaneous PIXE and BS, *Analytical Chemistry*, 81 (2009) 7960-7966

Towards truly simultaneous PIXE and RBS analysis of layered objects in cultural heritage

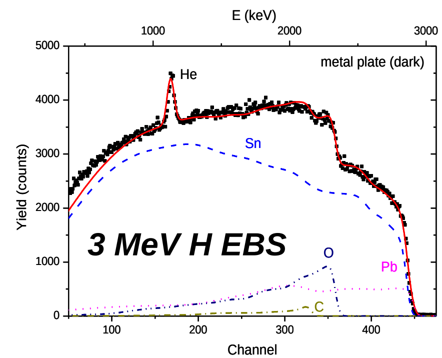
University of Surrey Ion Beam Centre

C. Pascual-Izarra (Madrid), N. P. Barradas, M. A. Reis (Lisbon),
C. Jeynes (Surrey), M. Menu, B. Lavdrine, J. J. Ezrati, S. Röhrs (Louvre)

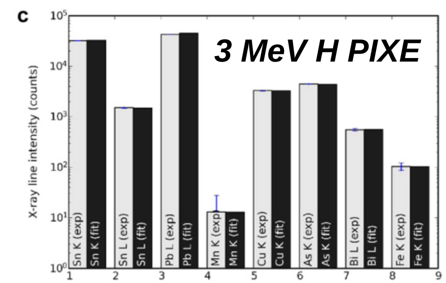
Nucl. Instrum. Methods B, 261 (2007) 426-429

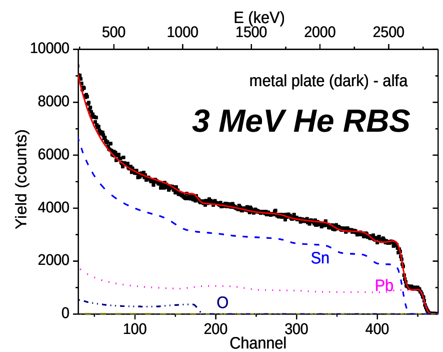


Niepce's first Heliography:
Paysage à Saint-Loup de Varennes (1827)

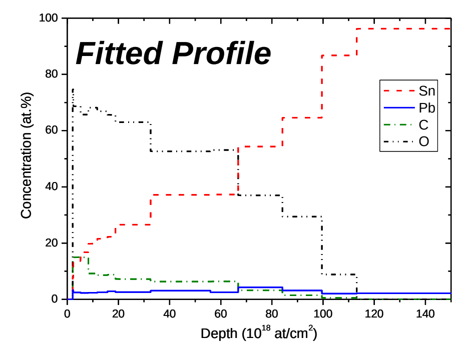


Fitted RBS spectrum for 3 MeV H⁺ beam on the dark spot (corroded area). Calculated partial spectra for each element are also shown





Fitted RBS spectrum for 3 MeV ⁴He⁺ beam on the dark spot (corroded area). Calculated partial spectra for each element are also shown



Concentration profiles for the dark spot (corroded area), as obtained from a simultaneous fit to 3 MeV proton PIXE and RBS, and 3 MeV alpha RBS.

Corrosion products demonstrated by PIXE/RBS/EBS to be tin oxide in a tin/lead matrix

PIXE/EBS of Darwin glass

Bailey, Howard, Jeynes+, *Nucl. Instrum. Methods B*, 267 (2009) 2219-2224

Howard, Bailey, Jeynes, Stolojan++, *Nature Geoscience* 6 (2013) 1018–1022

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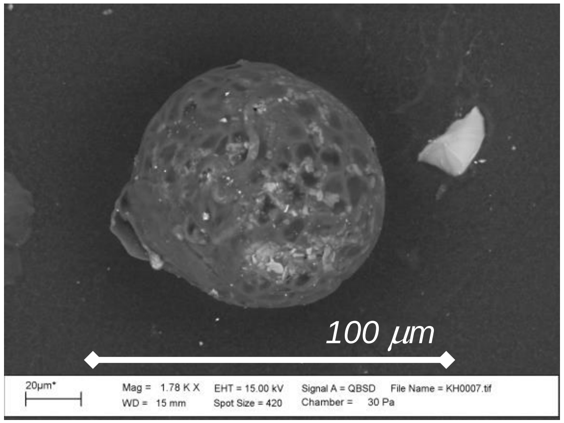
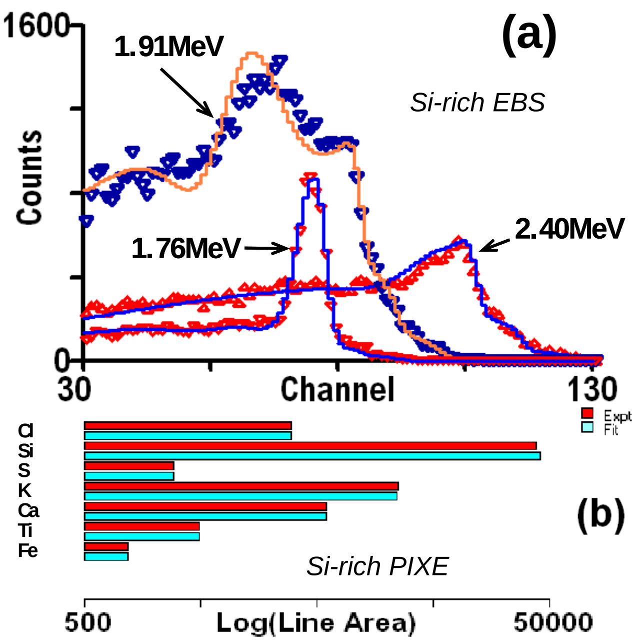
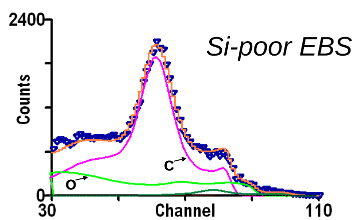


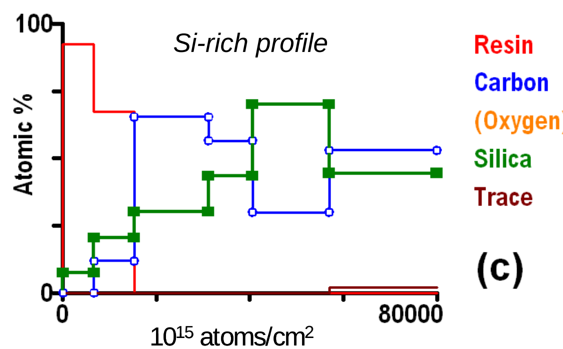
Impact glass from 800,000 year old meteor strike crater at Mt.Darwin, Tasmania

Inclusions are Carbon and Silicon (silica) rich: confirmed by PIXE/EBS

Inclusions highly heterogeneous: silica observed by EBS/PIXE







Silica (quartz by XRD) not observable without PIXE.

Unequivocal profiling down to ~15µm with EBS

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RBS/EBS/PIXE measurement of single-walled carbon nanotube modification by nitric acid purification treatment

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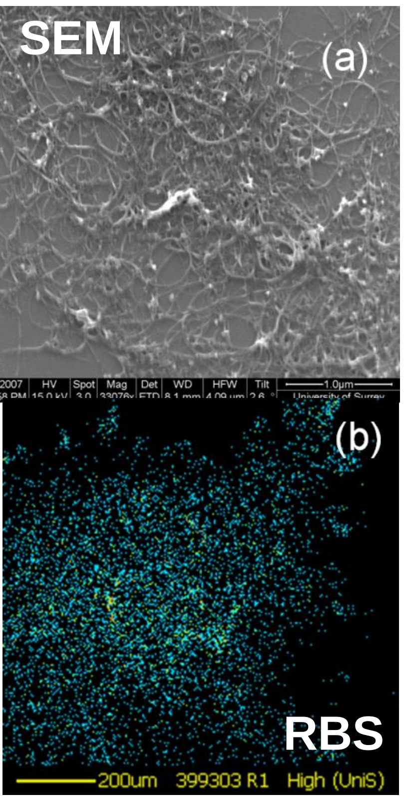
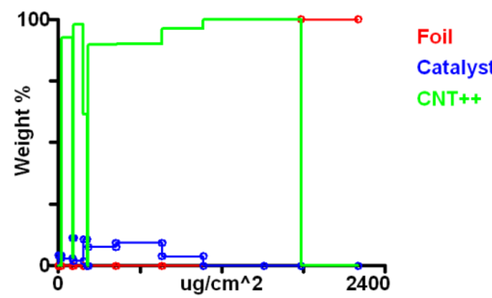
J.C.G. Jeynes, C. Jeynes, K.J. Kirkby, M. Rümmeli, S.R.P. Silva
Nucl. Instrum. Methods B, 266 (2008) 1569-1573



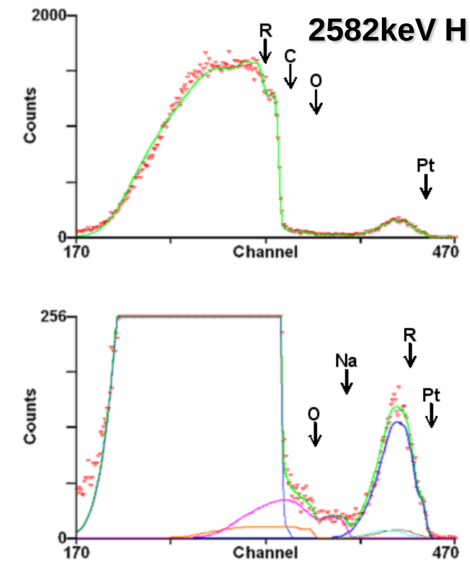
SWCNT on mylar

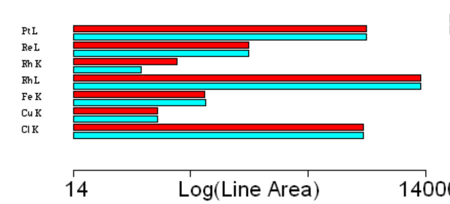
O quantified by PIXE/RBS/EBS in the presence of mixed heavy metal catalyst content of CNT with heavy roughness

Catalyst contains Pt, Rh, Re, Fe, Cu, Cl, Na. Roughness effects marked "R"



Fitted depth profile





RBS/EBS & PIXE data & fits

Measuring and modelling cell-to-cell variation in uptake of gold nanoparticles

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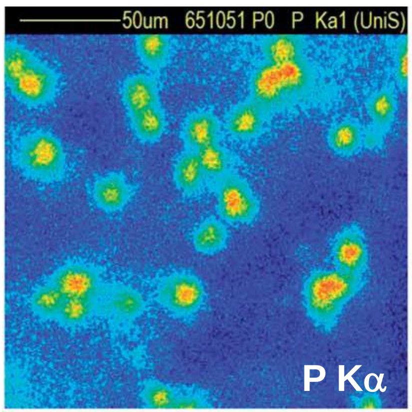
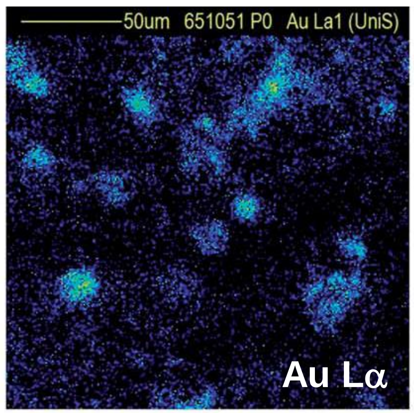
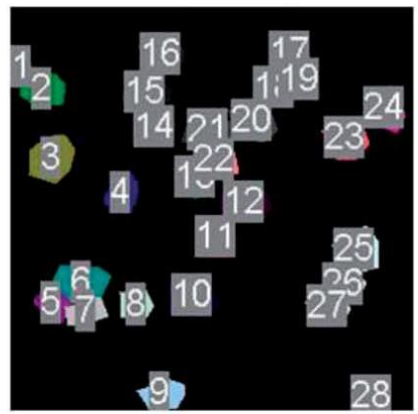
J.C.G. Jeynes, C. Jeynes, M.J.Merchant, K.J. Kirkby

***Analyst*, 138 (2013) 7070-7074**

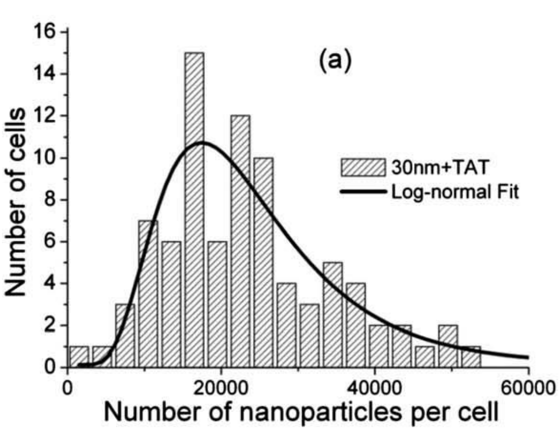
GNPs are used to carry drugs into tumour cells in cancer treatment: preferential takeup is due to the rapid metabolism in these cells

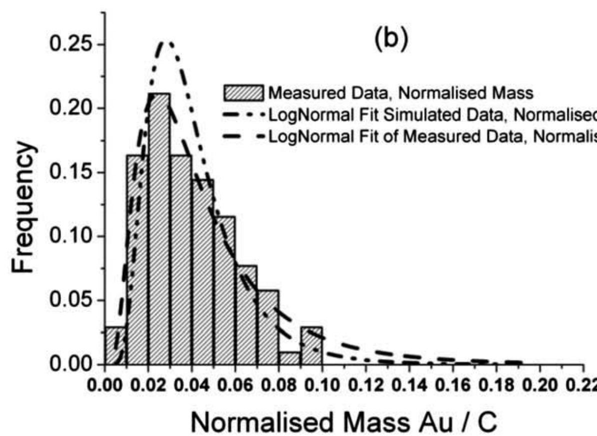






- We have counted GNPs per cell, using PIXE to image and RBS for quantitation
- Absolute measurements unprecedented
- Log-normal distribution both of numbers/cell and of cell uptake rates





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Sample H1 : CIGS/Mo/Cr/SS

(CuInGaSe chalcogenide semiconductor for PV: photovoltaics)

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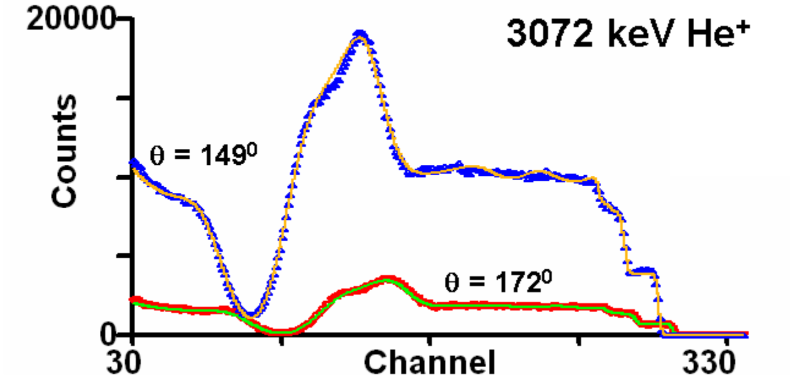

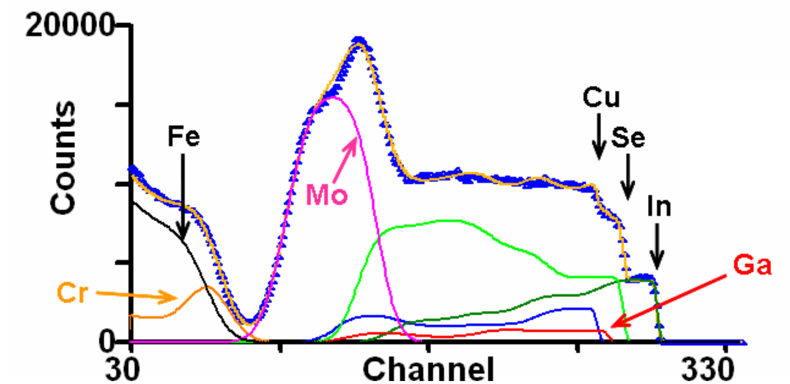

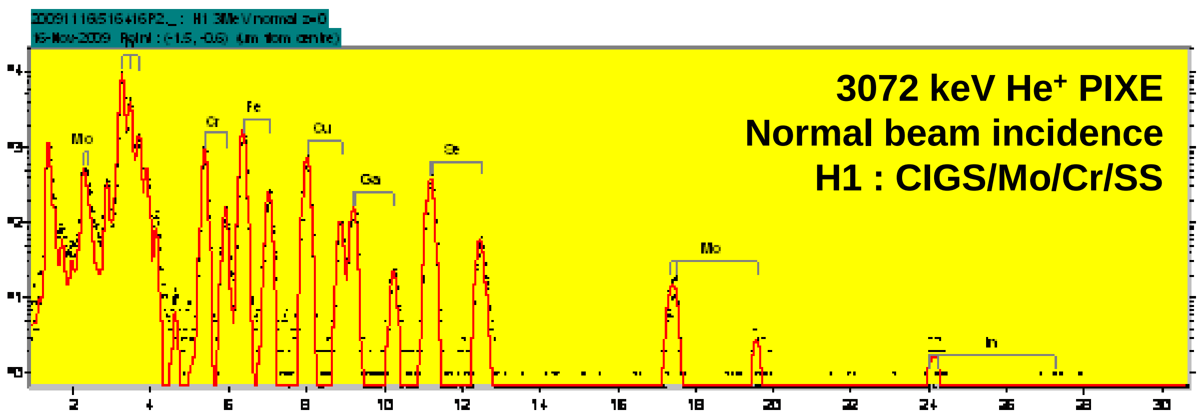

RBS spectra of CIGS/stainless steel

- All Rutherford
- RBS of CIGS multiply ambiguous
- Unambiguous at surface
- Mo energy loss 93% of database
- Layer thickness roughness required
- Thicknesses well determined

Excellently fitted!

Sample H1 : CIGS/Mo/Cr/SS

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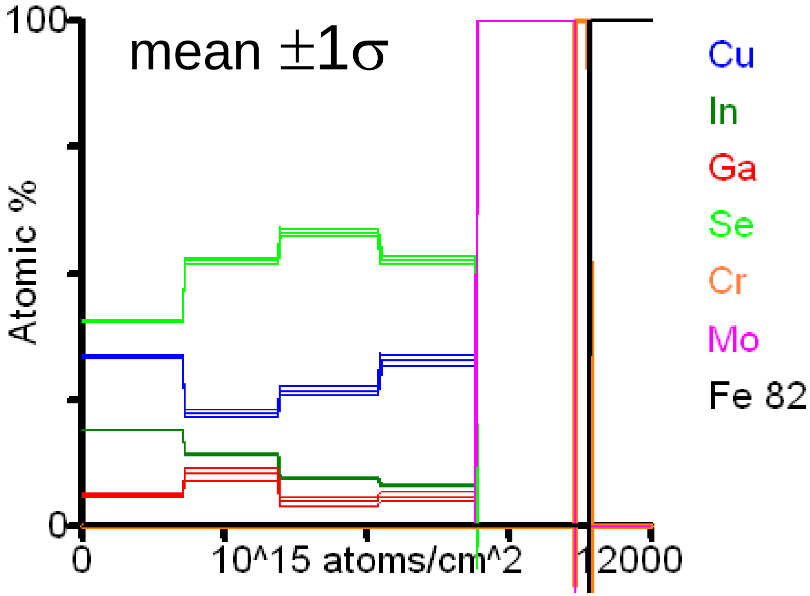

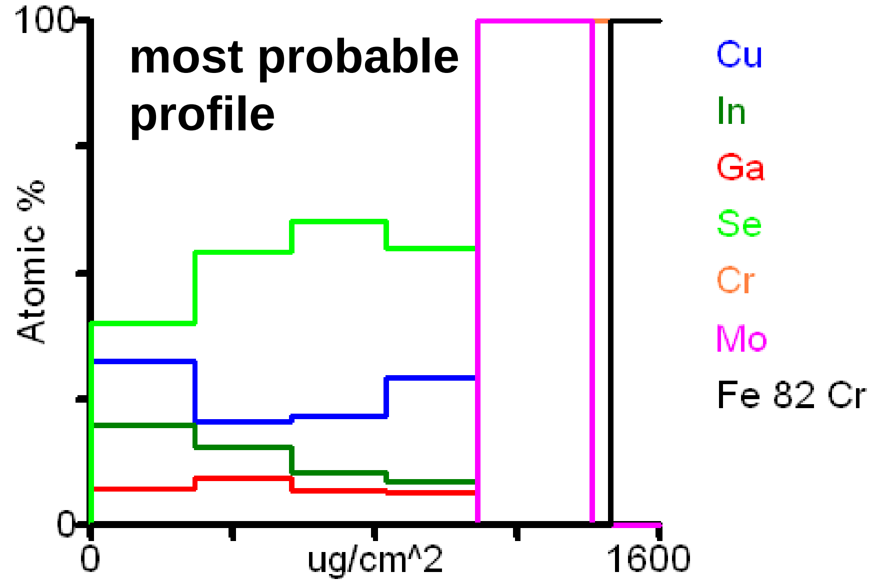

- CIGS = ~1860 nm (5.8 g /cc), 5% roughness
  XRF = 1900 nm
- Mo = 322 nm (15% roughness), Cr = 70 nm
- Self-consistent fit of ALL <u>photon</u> and <u>proton</u> data (15 spectra!)
- Bayesian estimate of uncertainty (<u>Markov chain</u> mathematics)
- CIGS = (27, 18, 8, 47) (top half); (26, 10, 7, 58) (bottom half); (262,138,77,522) (av)
  XRF = (219,133,90,558)

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Characterisation of gunshot residue particles using self-consistent IBA (2570 keV $^{1}H^{+}$)

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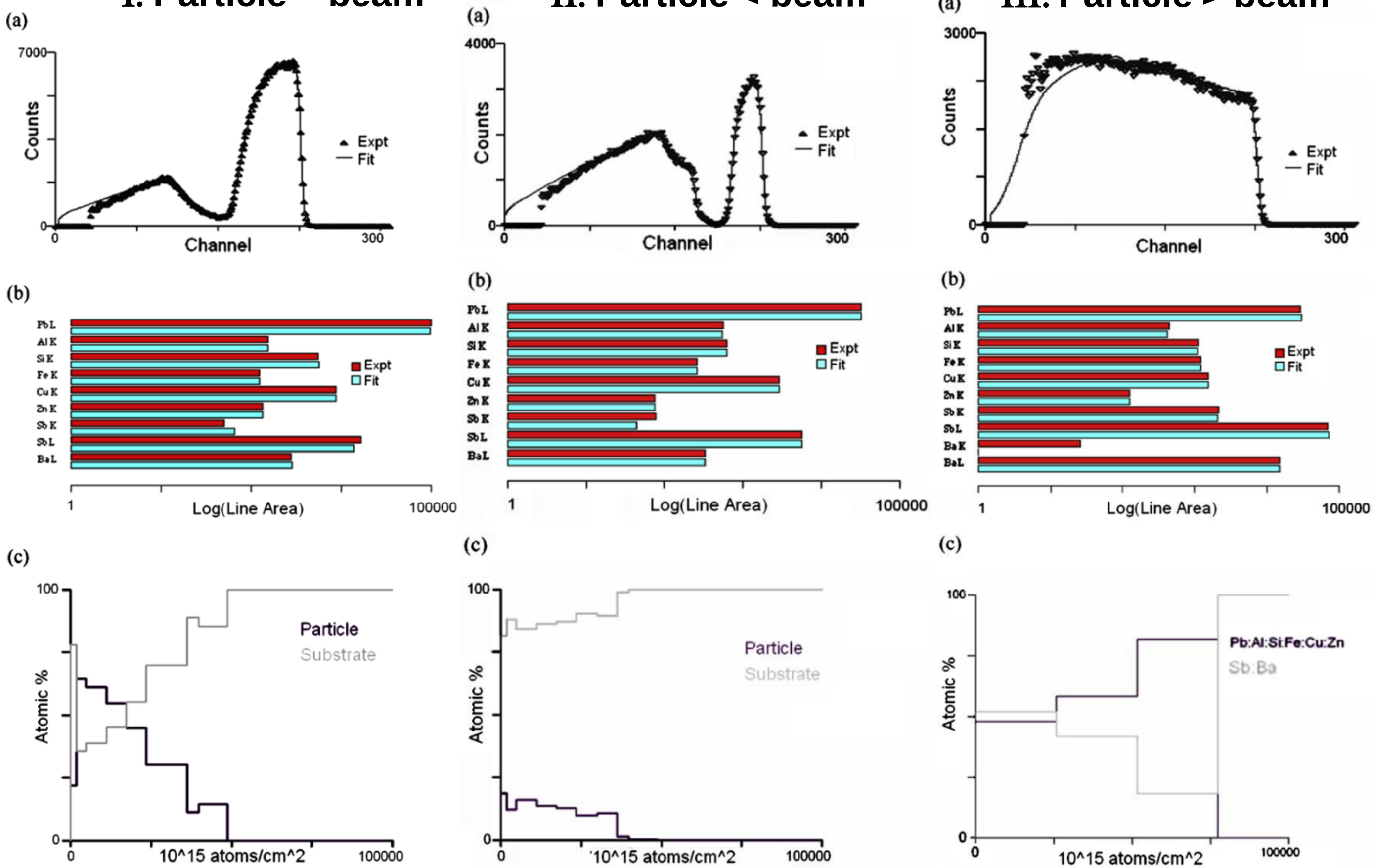
I: Particle ~ beam | **II: Particle < beam** | **III: Particle > beam**



- 4 μm spot
- Case III: good fit but bad model !
- Cases I & II: good fit but (different) bad model !
- Accuracy elusive but qualitative results appropriate
- SEM-EDS used routinely, but unable to cope with new lead-free materials

Bailey & Jeynes, *Nucl. Instrum. Methods B*, 266 (2008) 1569-1573

See also: Romolo, Bailey … Jeynes *et al*, *Forensic Science International* 231 (2013) 219–228

and: Christopher, Bailey … Jeynes *et al*, *Analyst*, 138 (2013) 4649-4655

Summary

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- "Total-IBA" :
  - *full use of all analytical signals using MeV ion probe*
  - *self-consistent handling of all signals*
  - *PIXE + RBS + EBS + ERD + NRA*
  - *1% accuracy for RBS: implant fluence QA*
    - **Total-IBA inherits accuracy of best signal**
- Glass analysis (external beam):
  - *Mapping large (precious) samples on new system*
    - **Fast (100 kHz) digital acquisition (3×SDD PIXE detectors + BS & PIGE detectors)**
    - **Motorised XYZ sample handling**
    - **Controlled atmosphere (in principle)**
  - *Quantitation in layered samples*
    - **Better than XRF: scan speed, quantitation, depth sensitivity**
    - **Differential PIXE (use of proton & alpha beams)**
  - *Glass science*
    - **Different (internal/external) weathering mechanisms**
    - **Evaluate technology (firing errors, paint composition etc)**

Summary (continued)

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- "Total-IBA"
  - **1% accuracy for RBS: implant fluence QA: Total-IBA inherits accuracy of best signal**
- Glass analysis (external beam)
  - **Mapping large (precious) samples on new system**
  - **Quantitation in layered samples**
  - **Glass science**
- Other Total-IBA applications (only a mere selection):
  - **Cultural Heritage (external beam: Louvre, Florence etc)**
  - **Darwin glass (geoscience)**
  - **Carbon nanotube surface treatment (nanotechnology)**
  - **Gold nanoparticles in cell biology**
  - **CuInGaSe (CIGS): materials for photovoltaics**
  - **Gunshot residue (forensics)**

Thanks for listening!

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