New and future analytical capabilities for lunar return samples - a case for SIMS

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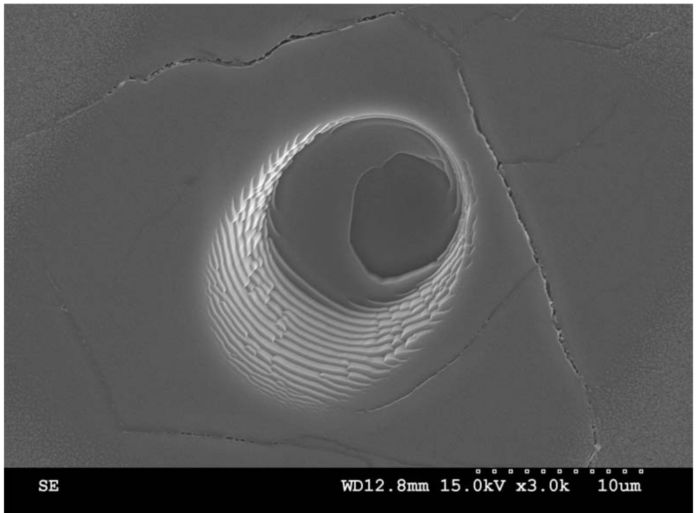
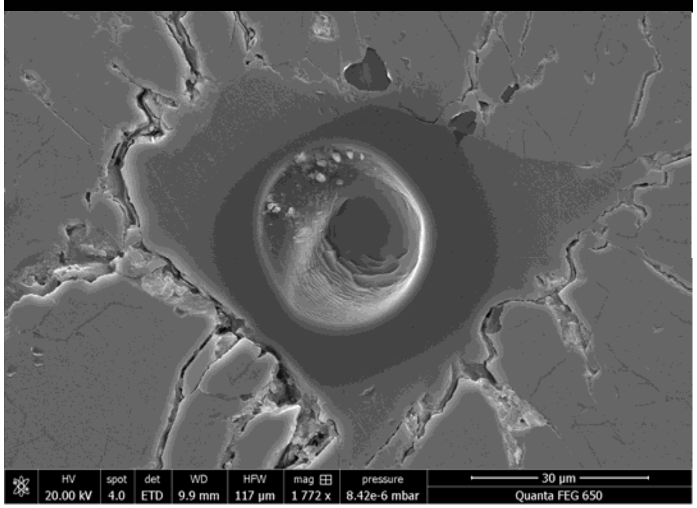
Analytical considerations for lunar samples:





- sample volume will always be severly limited cf. terrestrial geological samples
- minimise sample preparation by *in situ* work on thin section/polished rock chips
- minimise terrestrial contamination by avoiding analysis of grain boundaries/cracks (especially critical for e.g. Pb isotopes)
- versatile sampling probe to provide both light element (e.g. volatives) and trace metal analysis
- small sampled volume to permit multiple analysis of small and/or trace minerals

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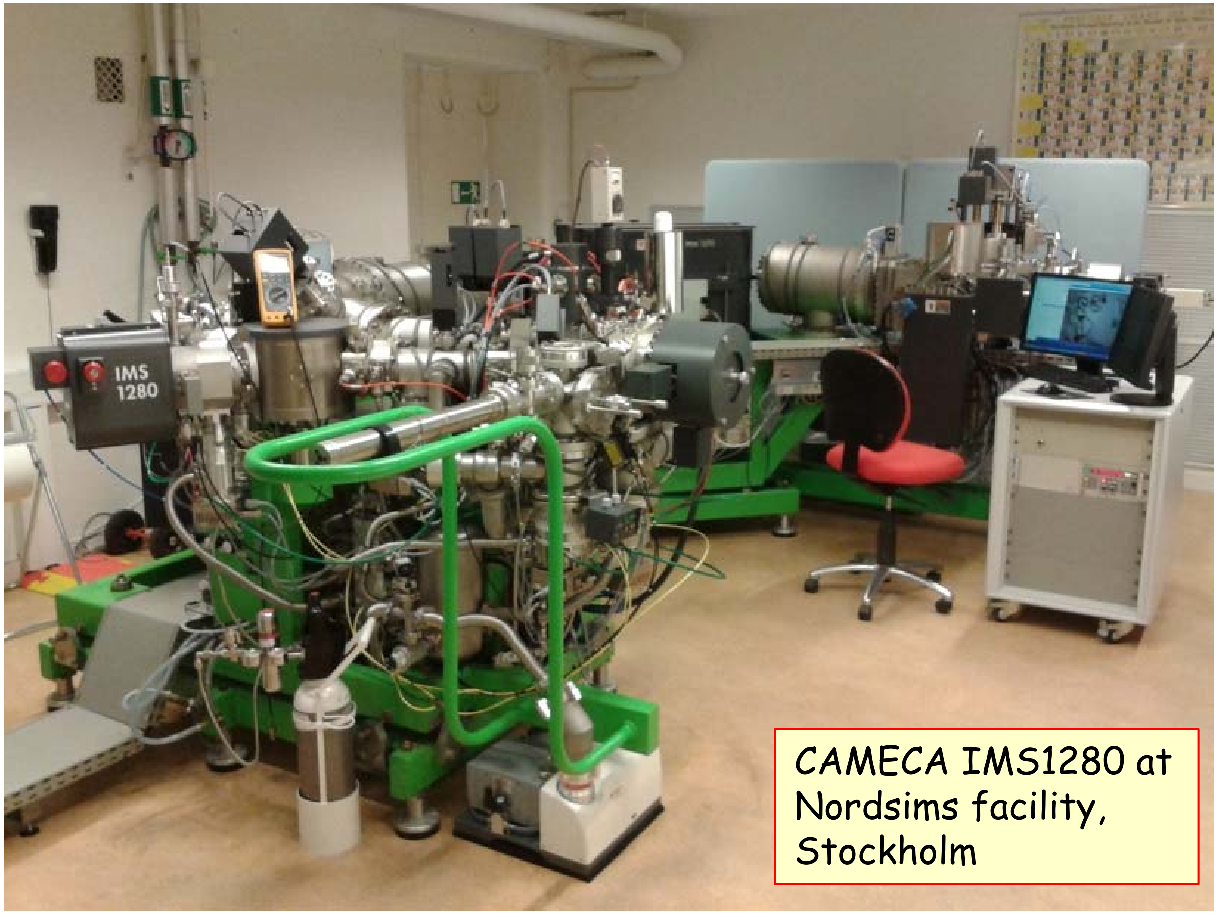
Large-geometry Secondary Ion Mass Spectrometer (SIMS) (a.k.a. ion microprobe) effectively fulfils all these requirements



CAMECA IMS1280 at Nordsims facility, Stockholm

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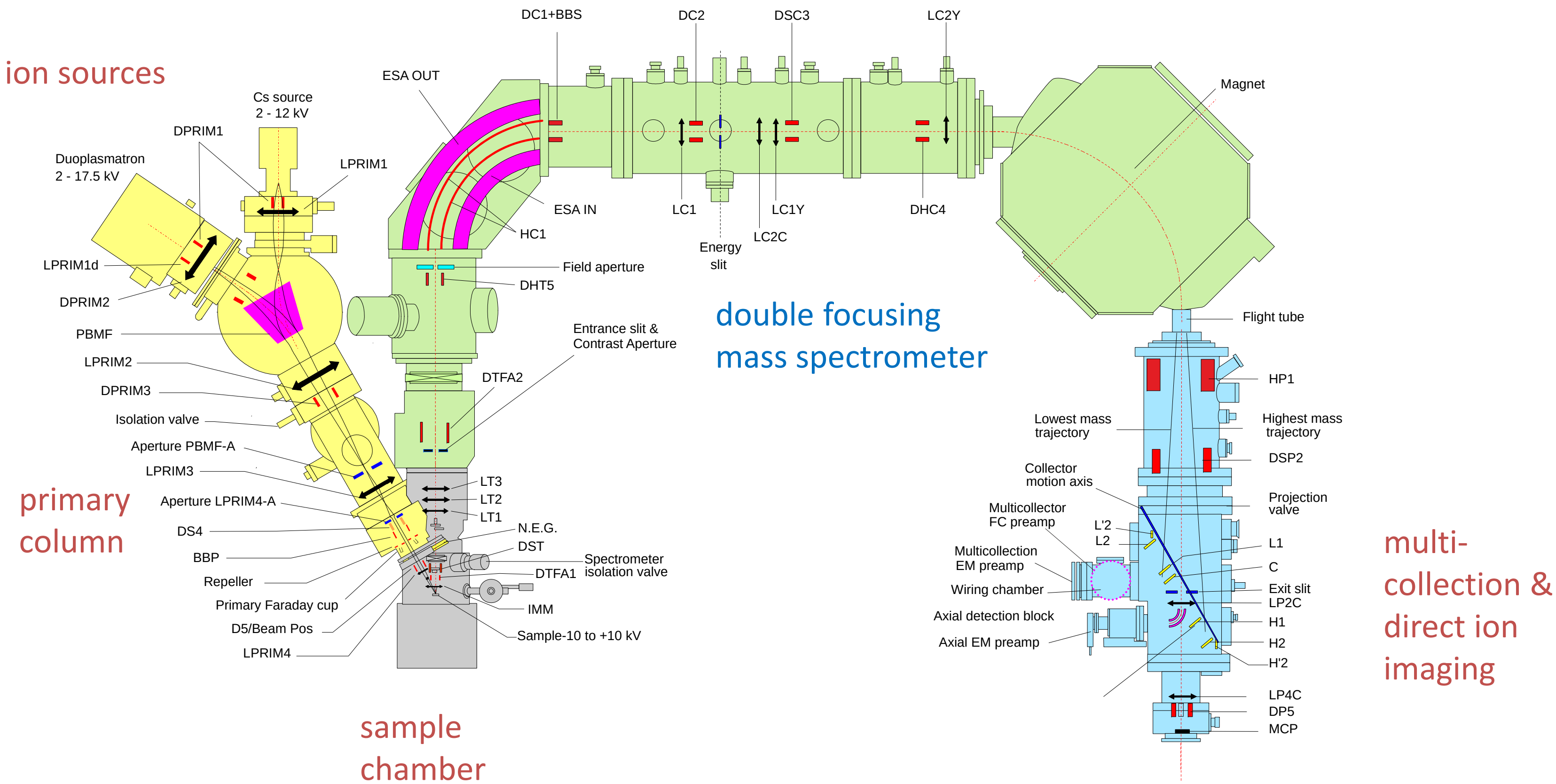
Key elements of a large geometry SIMS instrument



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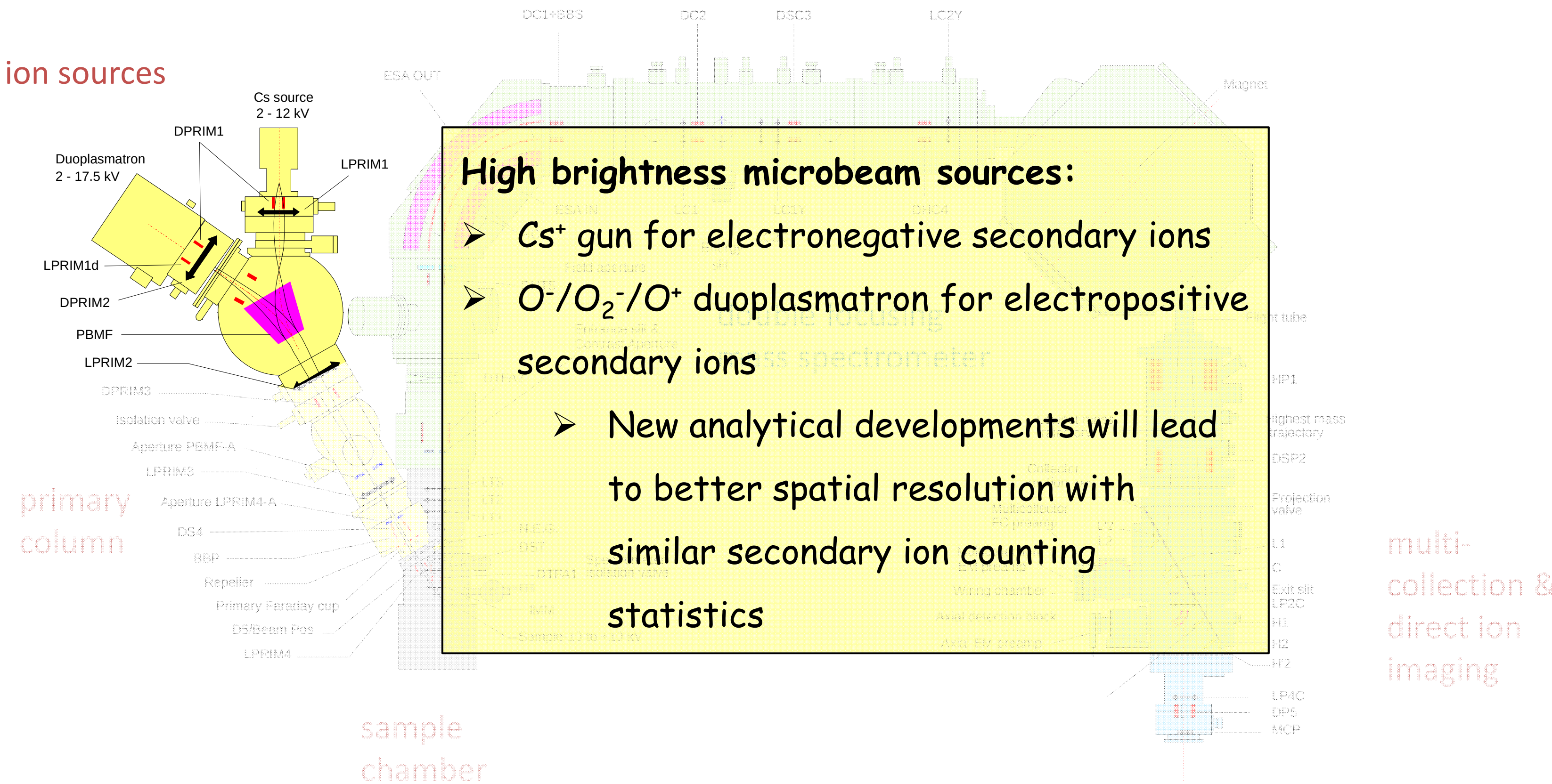

High brightness microbeam sources:

- Cs^+ gun for electronegative secondary ions
- $O^-/O_2^-/O^+$ duoplasmatron for electropositive secondary ions
    - New analytical developments will lead to better spatial resolution with similar secondary ion counting statistics

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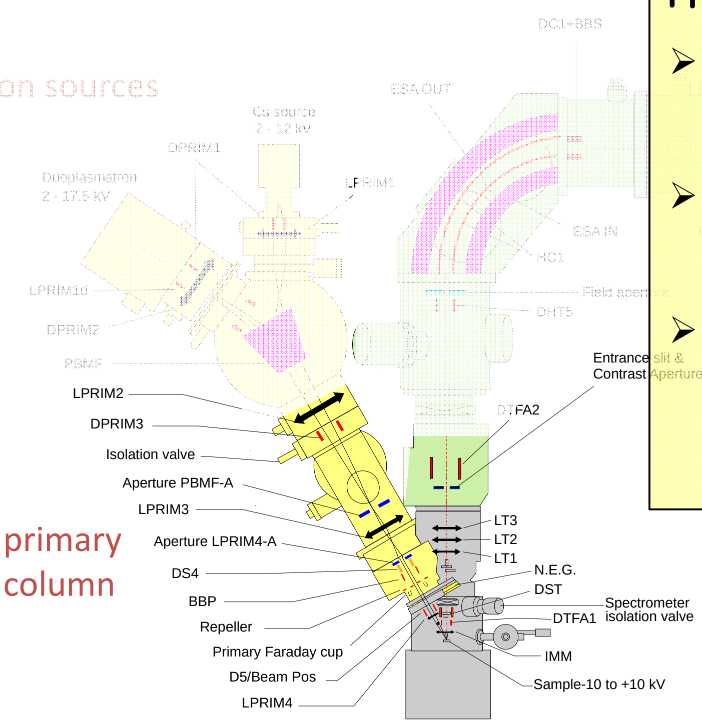
Key elements of a large geometry SIMS instrument

ion sources



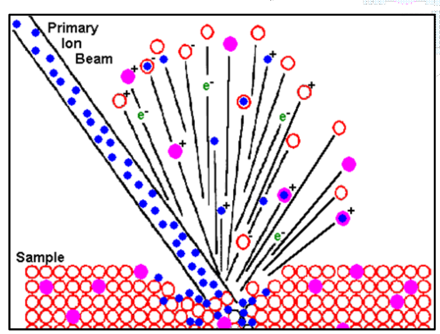
primary column

sample chamber

multi-collection & direct ion imaging

Primary column, sample & transfer:

- aperture illumination mode for flat-bottomed probes, Ø ~ 2 to 30 µm
- critical focussing for smallest probes (Ø ~ 0.5 µm Cs, 1 µm O-).
- up to 500 µm raster for ion mapping, synchronised with transfer raster to preserve high transmission mode.



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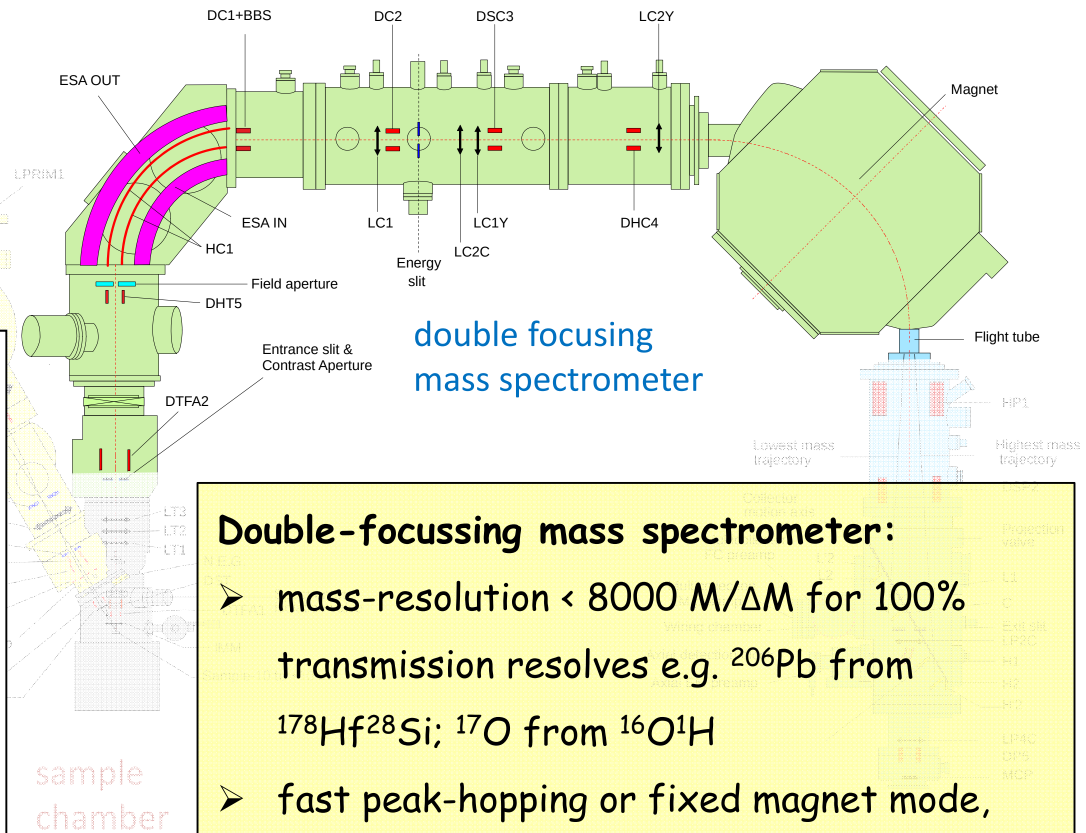

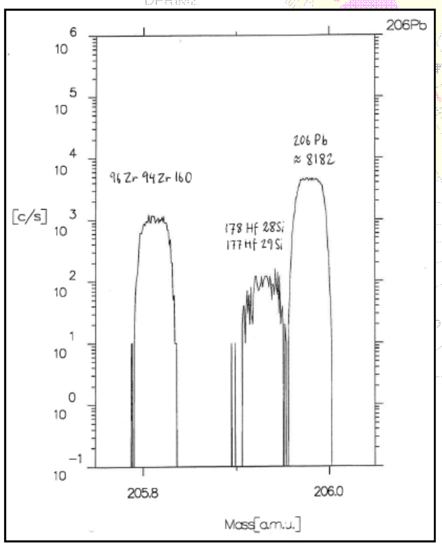

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Double-focussing mass spectrometer:

- mass-resolution < 8000 M/ΔM for 100% transmission resolves e.g. ^{206}Pb from $^{178}Hf^{28}Si$; ^{17}O from $^{16}O^{1}H$
- fast peak-hopping or fixed magnet mode, with NMR regulation to <0.2 ppm/24hours
- stigmatic optics for direct ion imaging

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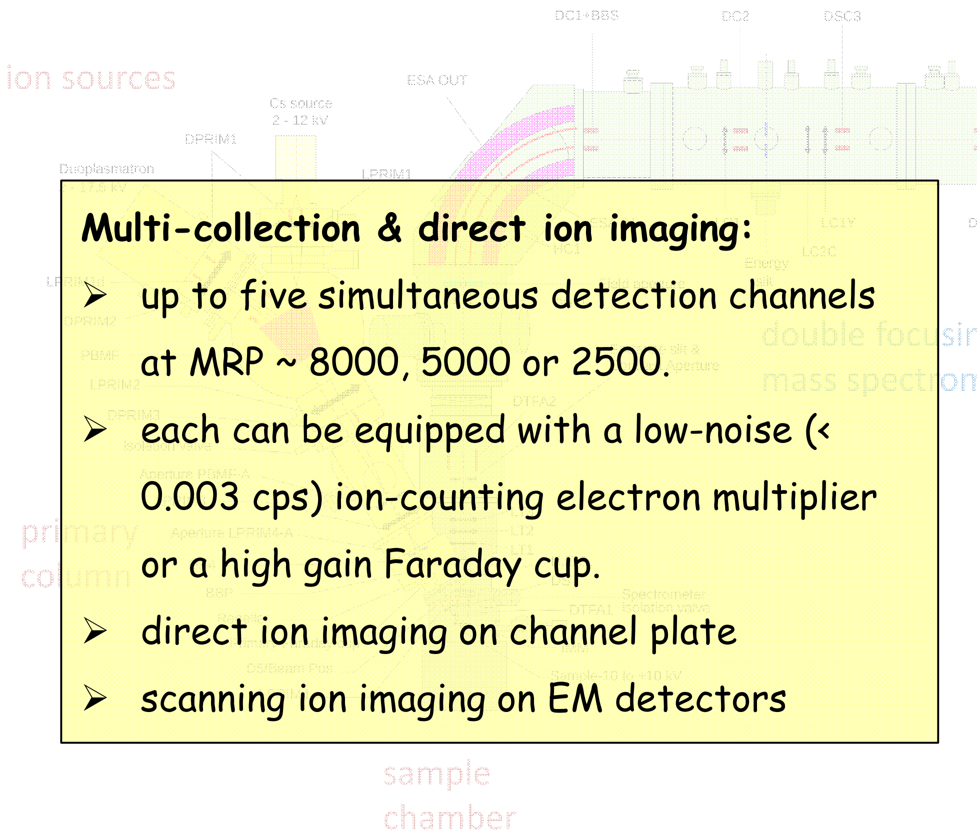
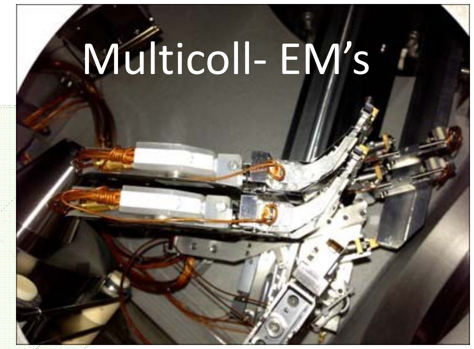
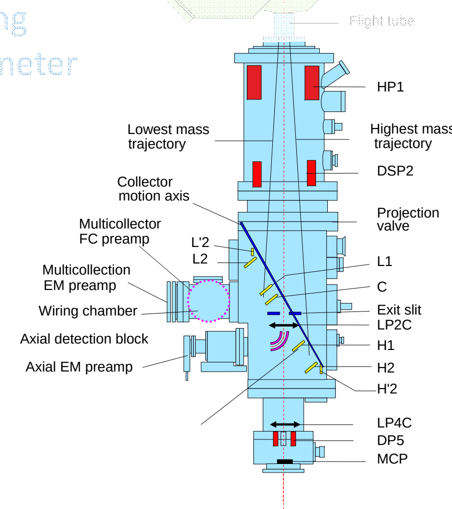


Multi-collection & direct ion imaging:

- up to five simultaneous detection channels at MRP ~ 8000, 5000 or 2500.
- each can be equipped with a low-noise (< 0.003 cps) ion-counting electron multiplier or a high gain Faraday cup.
- direct ion imaging on channel plate
- scanning ion imaging on EM detectors





multi-collection & direct ion imaging

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Recent applications in lunar science (list far from exhaustive!):

- Geochronology of U-bearing phases (e.g. zircon, apatite)
    - Magmatic and impact histories
- Pb-isotope dating of lunar basalts
- Pb-isotope systematics of major lunar silicate reservoirs
- Scanning ion imaging geochronology
- Volatile concentrations and light stable isotopes

- See Joshua Snape's poster tonight for more in-depth scientific discussions

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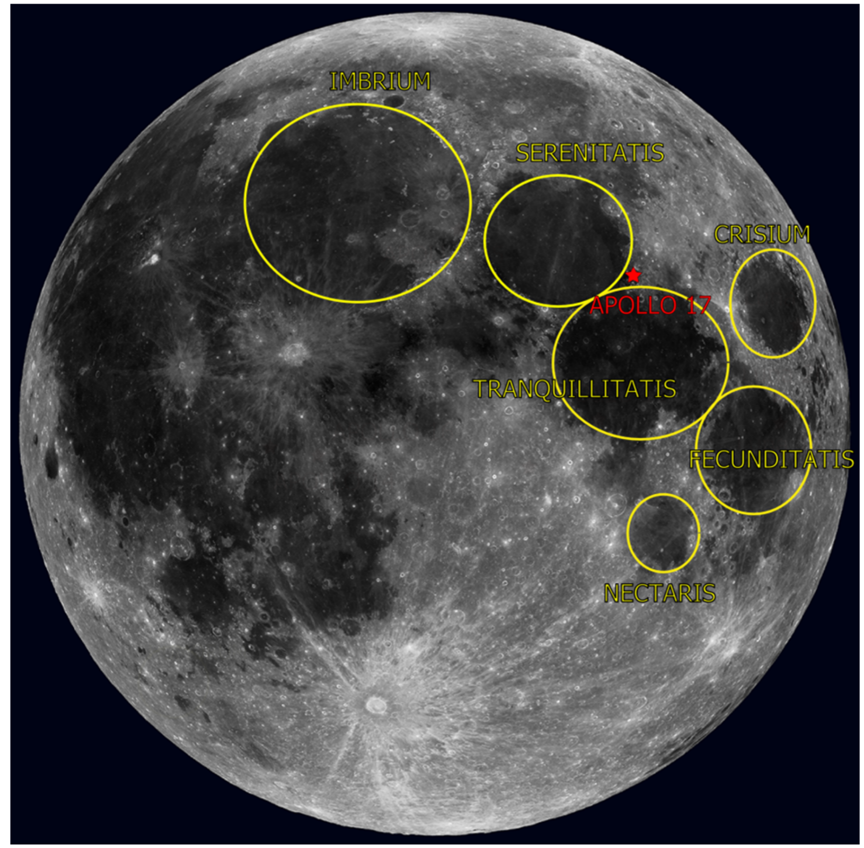
Resolving conflicting basin chronology by dating of lunar breccias

Stöffler et al. (2006)		Fassett et al. (2012)	
Imbrian	Orientale	Imbrian	Orientale
	Schrödinger		Schrödinger
	Imbrium		Imbrium
Nectarian	Bailly	Nectarian	Humboldtianum
	Sikorsky-Rittenhouse		Humorum
	Hertzsprung		Crisium
	Serenitatis		Moscoviense
	Crisium		[Planck]
	Humorum		Mendel-Rydberg
	Humboldtianum		[Grimaldi]
	Mendeleev		Hertzsprung
	Korolev		Mendeleev
	Moscoviense		Korolev
	Mendel-Rydberg		Nectaris
	Nectaris		
Pre-Nectarian	Grimaldi	Pre-Nectarian	Freundlich-Sharonov
	Apollo		Apollo
	Freundlich-Sharonov		[Serenitatis]
	Birkhoff		[Ingenii]
	Planck		[Birkhoff]



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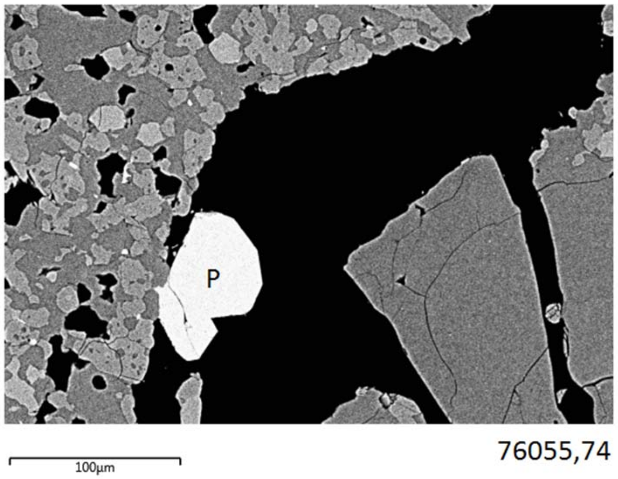
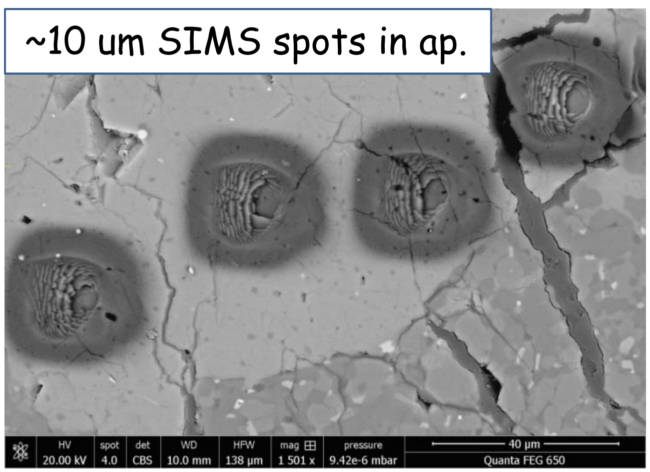
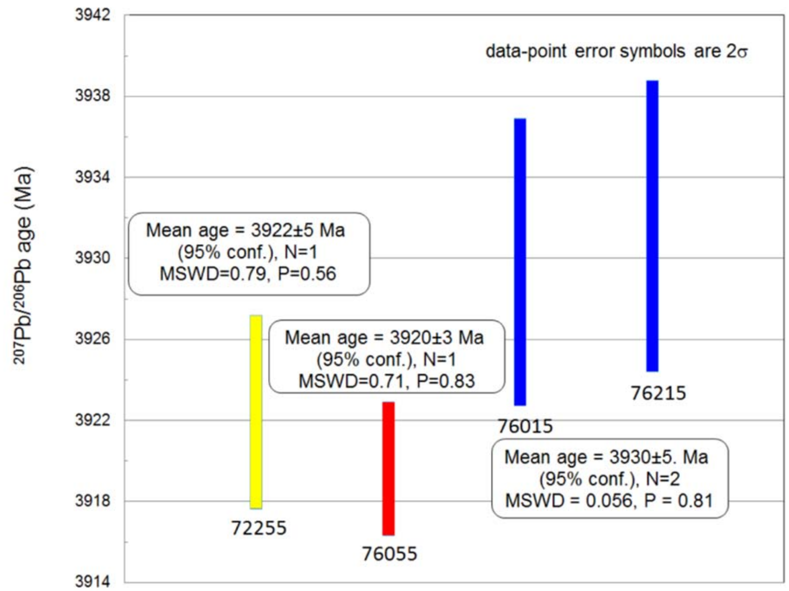




Apollo 17 data

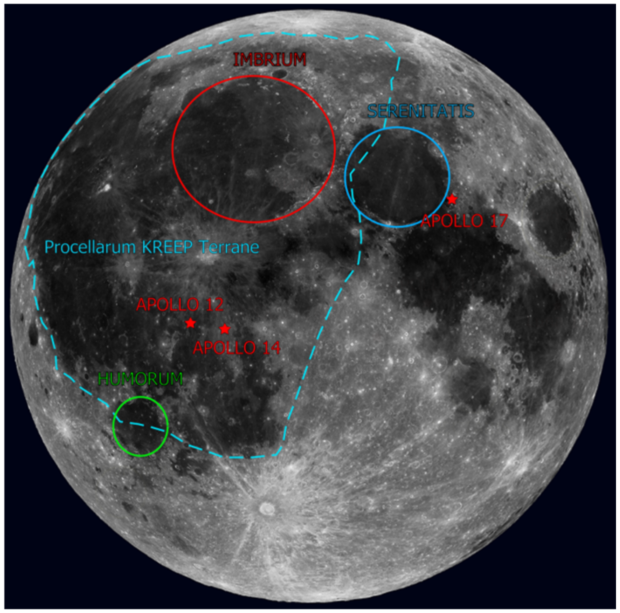


Precise U-Pb SIMS ages combined with textural information suggest:

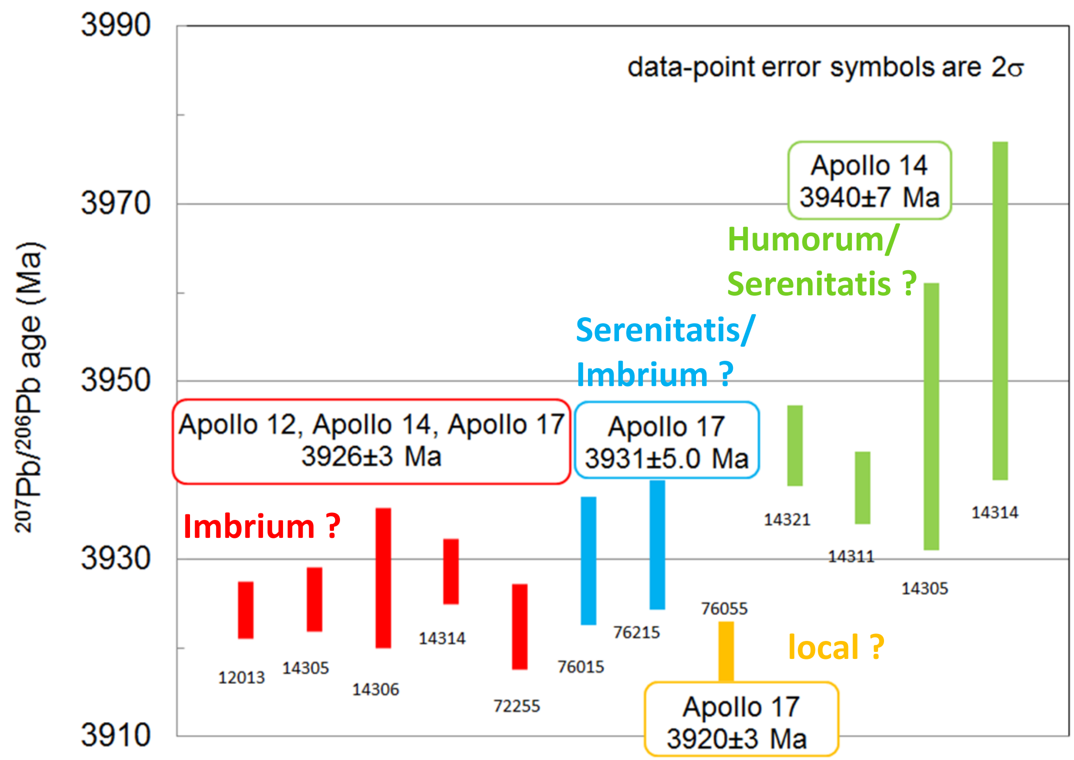
- Three different impact events
- Two basin forming events within ~8Ma

Possible origins: Nectaris, Crisium, Serenitatis, Imbrium

Resolving conflicting basin chronology by dating of lunar breccias



Apollo 12, 14 and 17 data



- U-Pb SIMS dating gives more precise ages but the link between breccias and impact basins remains uncertain.
- Combined with textural and chemical information, there are at least three distinct impact events between 3920 and 3940 Ma.
- LHB model may or may not be supported by these data – work in progress

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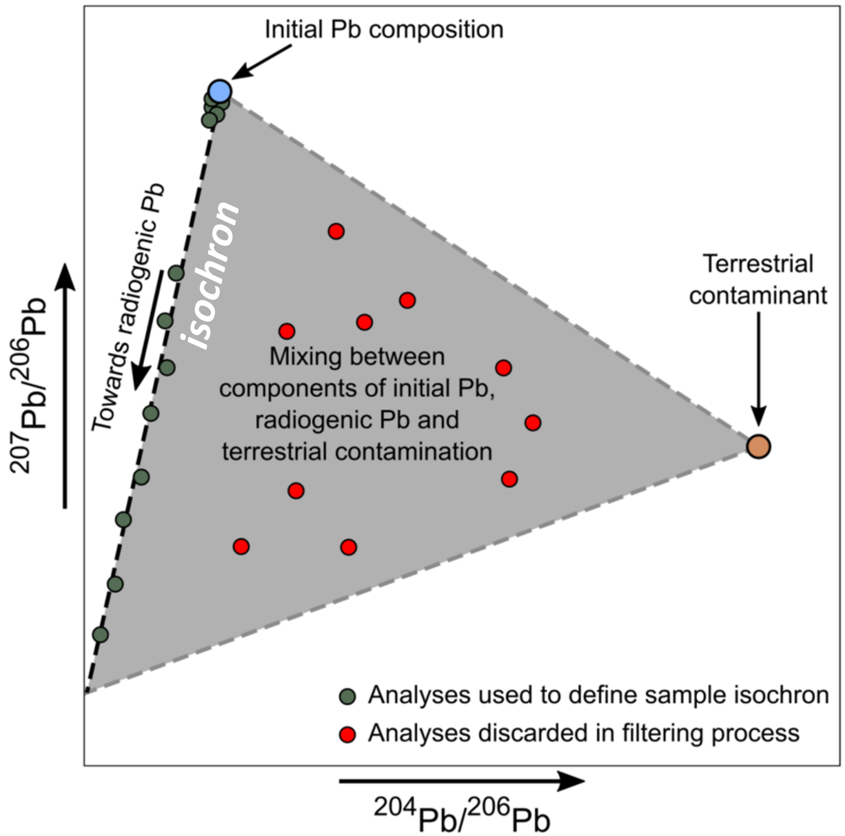
Basalt dating and lunar differentiation from Pb isotopes



Pb isotopes in lunar rocks are highly susceptible to contamination during sample preparation due to their low Pb contents and evolution in a very high μ ($^{238}U/^{204}Pb$) reservoir.

- **Multicollector ion-counting SIMS** permits **rapid *in situ*** targeting of diverse mineral phases and mesostasis in lunar basalts, while avoiding obvious cracks and/or grain boundaries where contamination lurks.

- Comprehensive data sets allow clear delineation of analyses that define an **isochron age** from those that are likely terrestrial Pb contaminated. Additionally, the **initial Pb composition** of the isochron is commonly determined and can be used in reservoir modelling.

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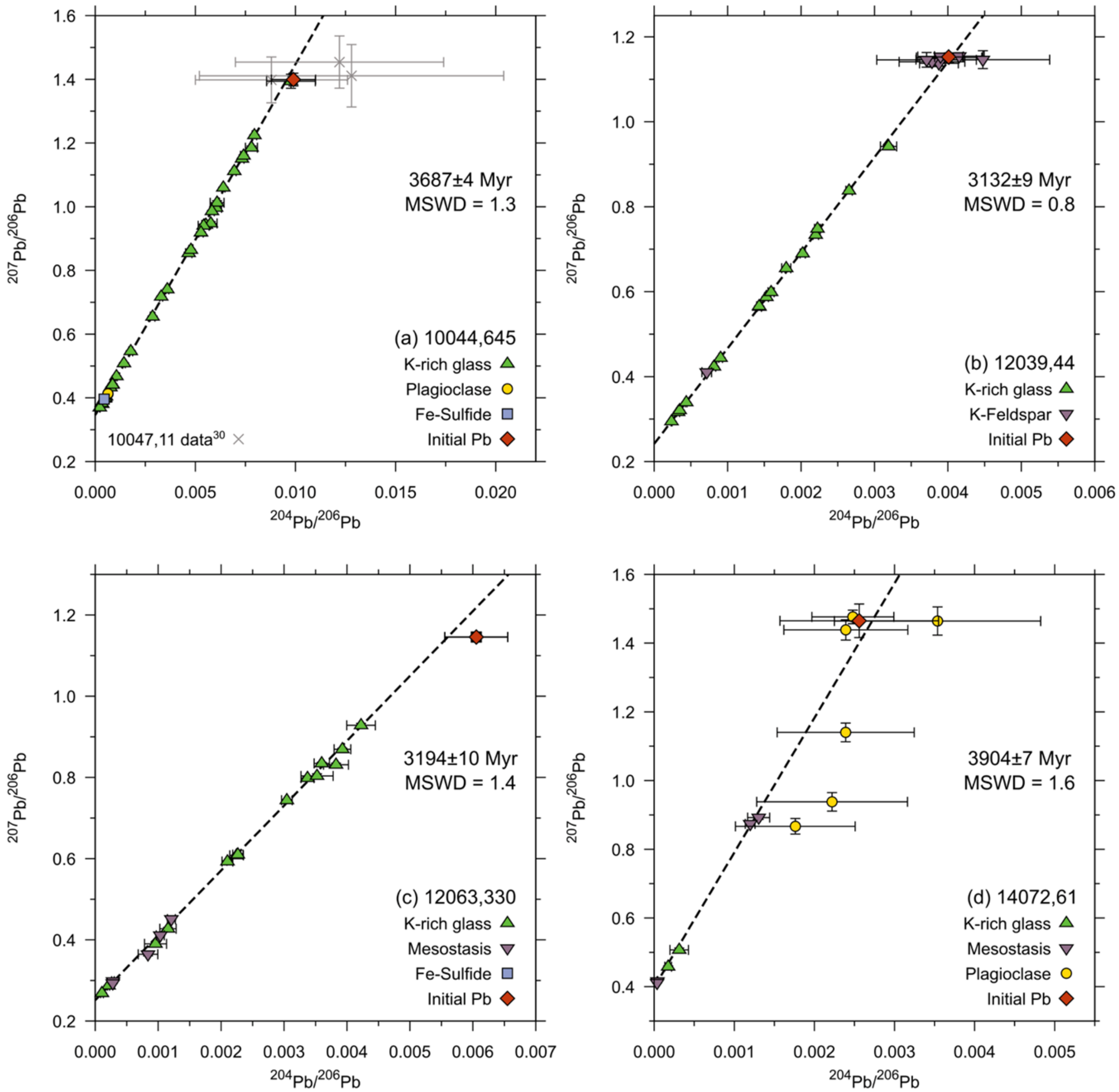
Basalt dating and lunar differentiation from Pb isotopes



- **Pb isochron ages** for basalts broadly agree with previous studies (mostly Ar-Ar), but are almost an **order of magnitude more precise**, demonstrating the potential of SIMS applied to lunar geochronology.
- In most cases, **initial Pb** is also well-defined by unradiogenic analyses on the isochron and permit multi-stage Pb-isotopic modelling.

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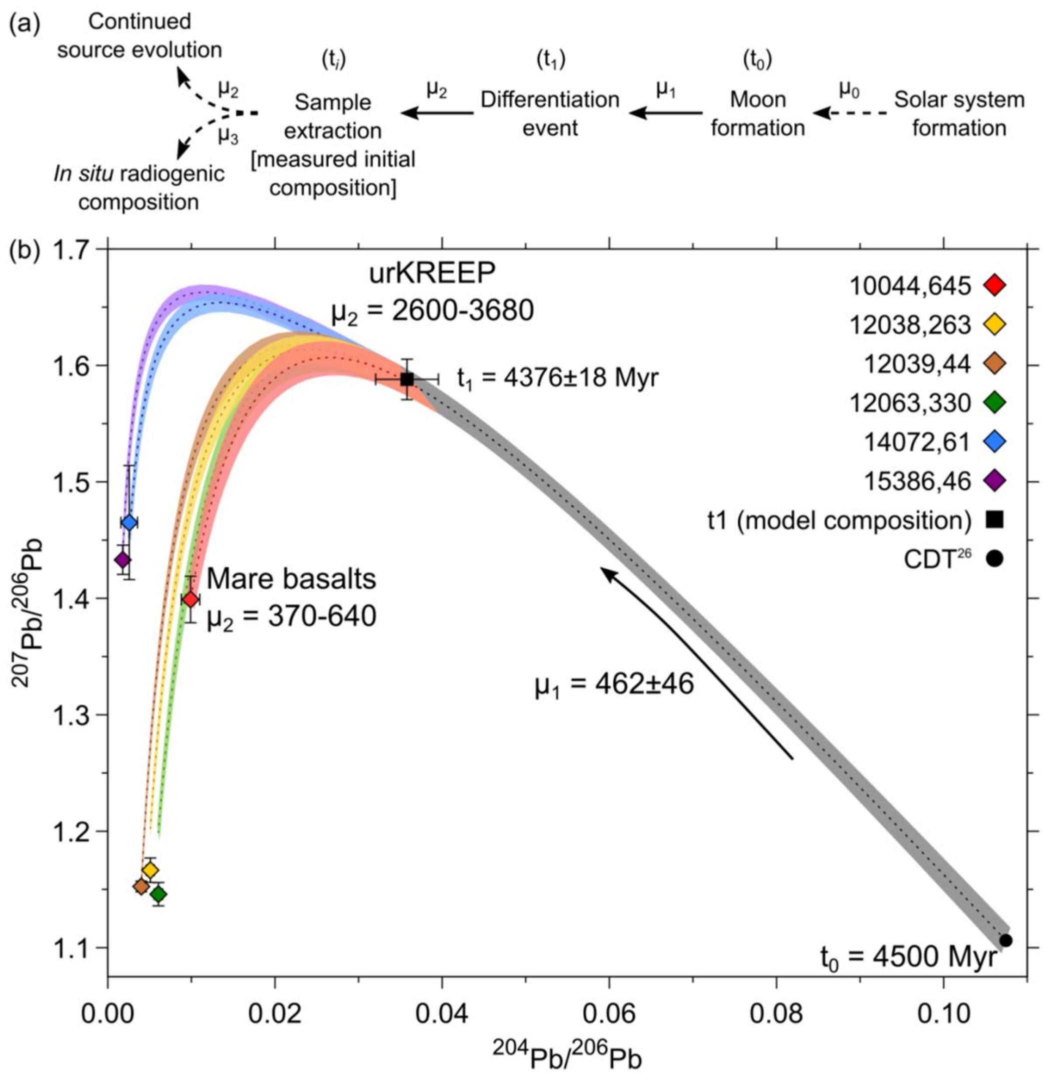
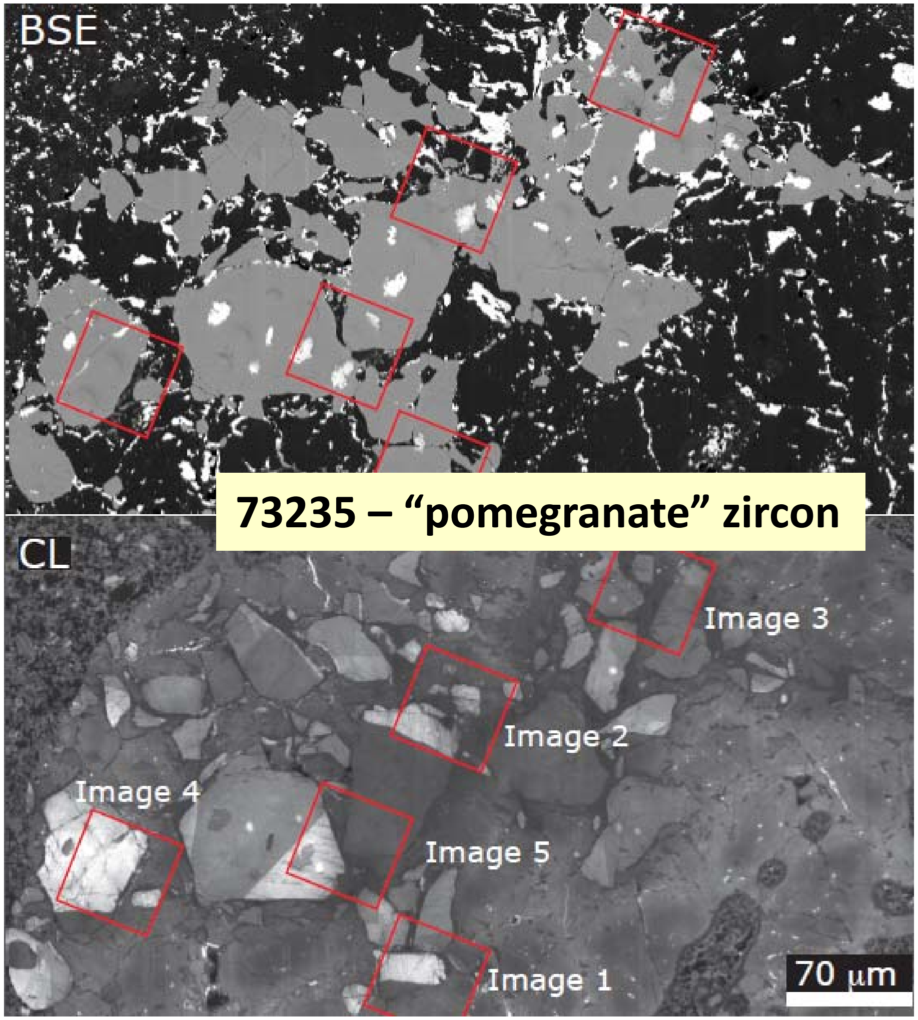
Basalt dating and lunar differentiation from Pb isotopes



- Initial Pb of basalts can be modelled back to a **common diffentiation event** in their source, dated at **4376 ± 18 Ma.**
- Similarity to Nd and Hf model ages suggest this could be **formation of urKREEP** in the final LMO crystallisation stages.
- Alternatively, a **post-LMO major impact** on the lunar nearside may be recorded.
- High-μ first stage also contrains the **minimum age** of the Moon to ~4500 Ma.

Scanning ion imaging geochronology of lunar zircon



"Pomegranate" zircon spot analyses yield ages of 4.315 ± 0.015 Ga for primary zoned igneous zircon and 4.187 ± 0.011 Ga for secondary CL-dark amorphous zircon (Pidgeon et al, 2007).

- Investigating U-Pb systematics of secondary zircon is complicated by small size relative to typical spot analysis.
- Ion image acquisition of full U-Pb data over 70 x 70 mm regions.
- Spatial resolution determined by primary beam, typically 5 mm.
- Complex areas can be treated as a single analysis.

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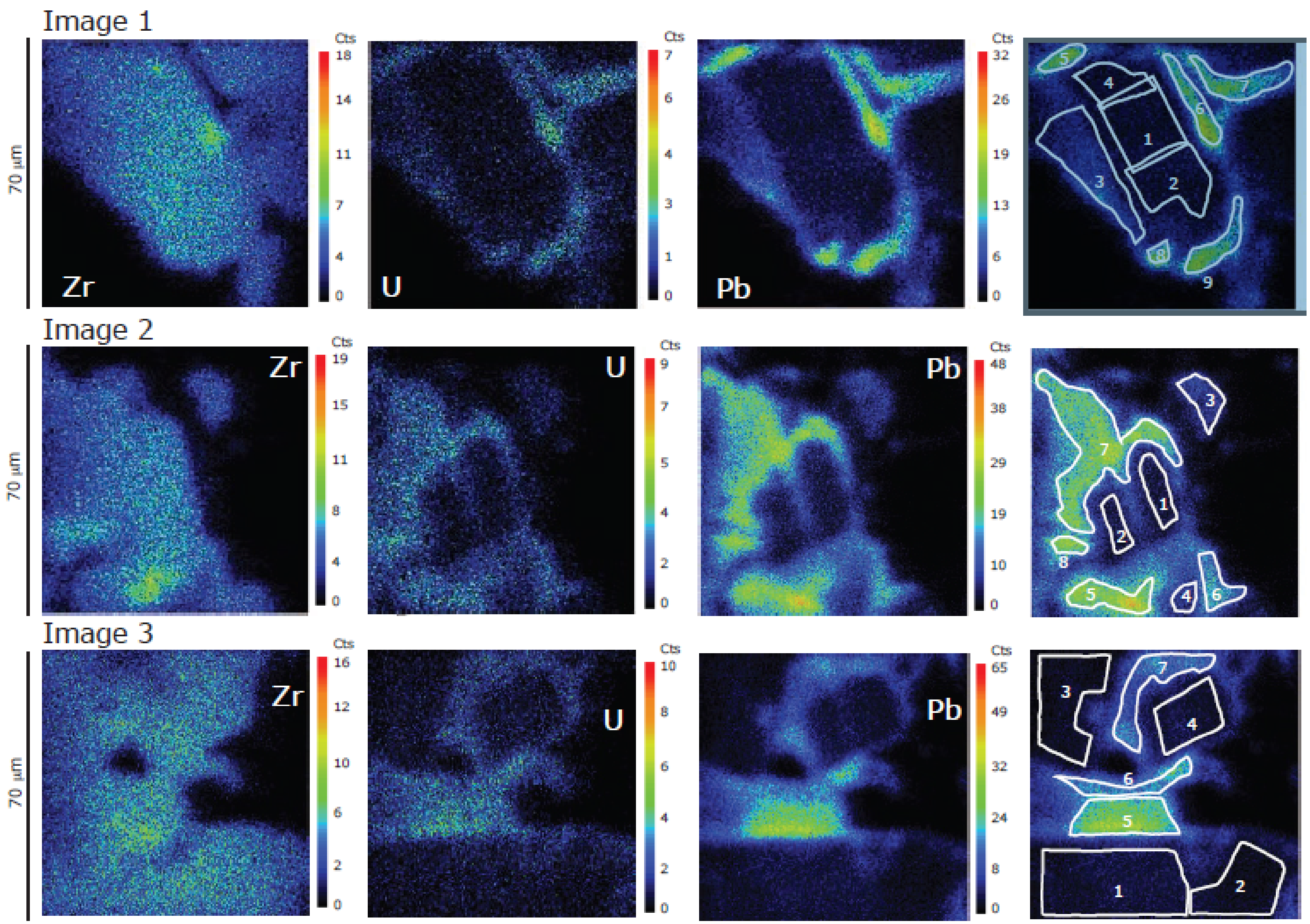
Ion imaging geochronology of lunar zircon



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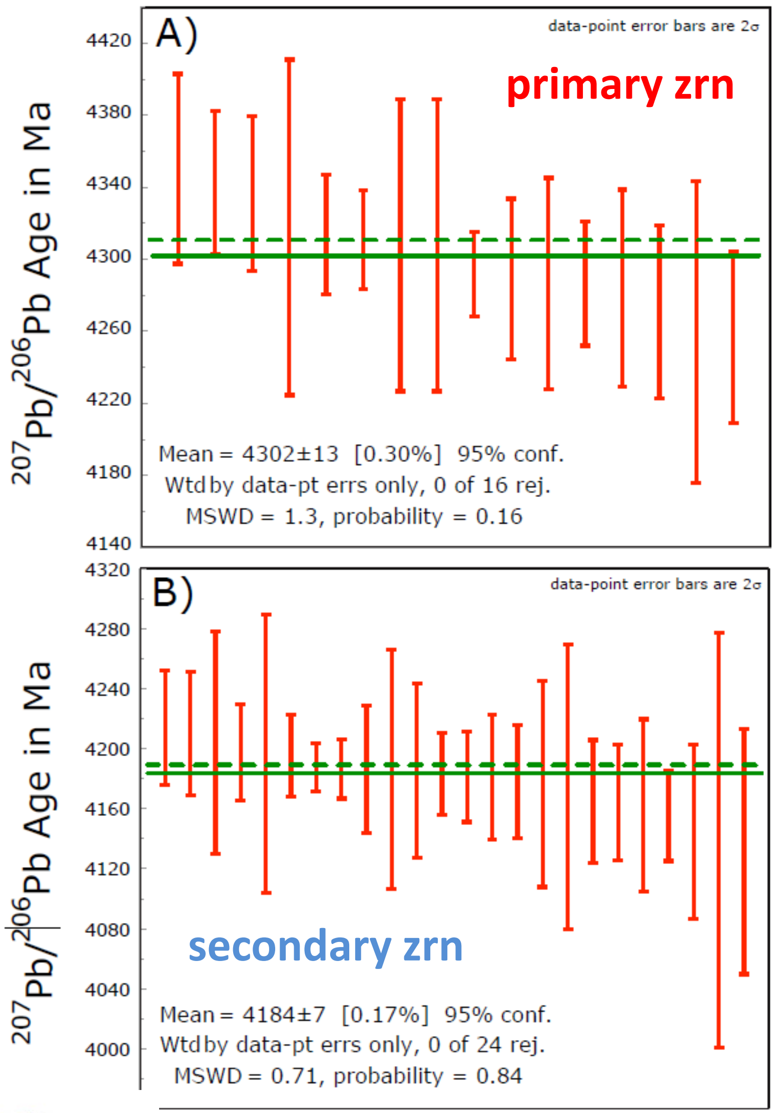

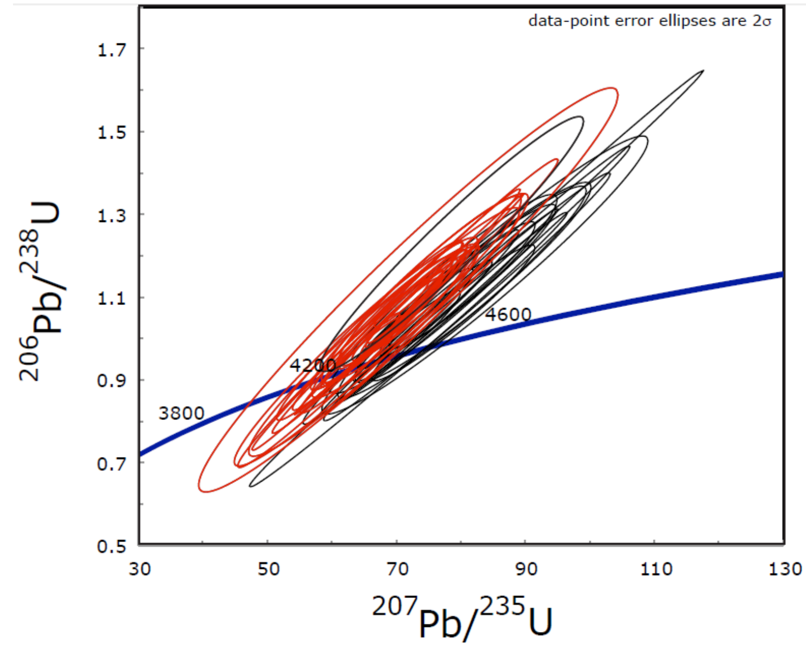

SII presents a new approach to high spatial resolution geochronology in complex lunar zircon

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Summary

- Modern large-geometry SIMS instruments provide an unmatched analytical platform for analysis of lunar samples.
    - New, critical data and techniques being collected/developed despite the last lunar sample returned in 1972
- Minimal sampling depth preserves valuable material for subsequent analysis of different elements/isotopes.
- High-sensitivity coupled with extremely low-noise signal detection is critical to high-accuracy, high precision analysis of e.g. Pb-isotope systematics.
    - Continued developments in source technology will only push the boundaries of spatial resolution
- Spatial resolution enhanced by ion imaging and selection of small/complex areas.

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