

EPMA meets XPMA: Testing a Microfocus XRF Source in an Electron Probe Microanalyzer

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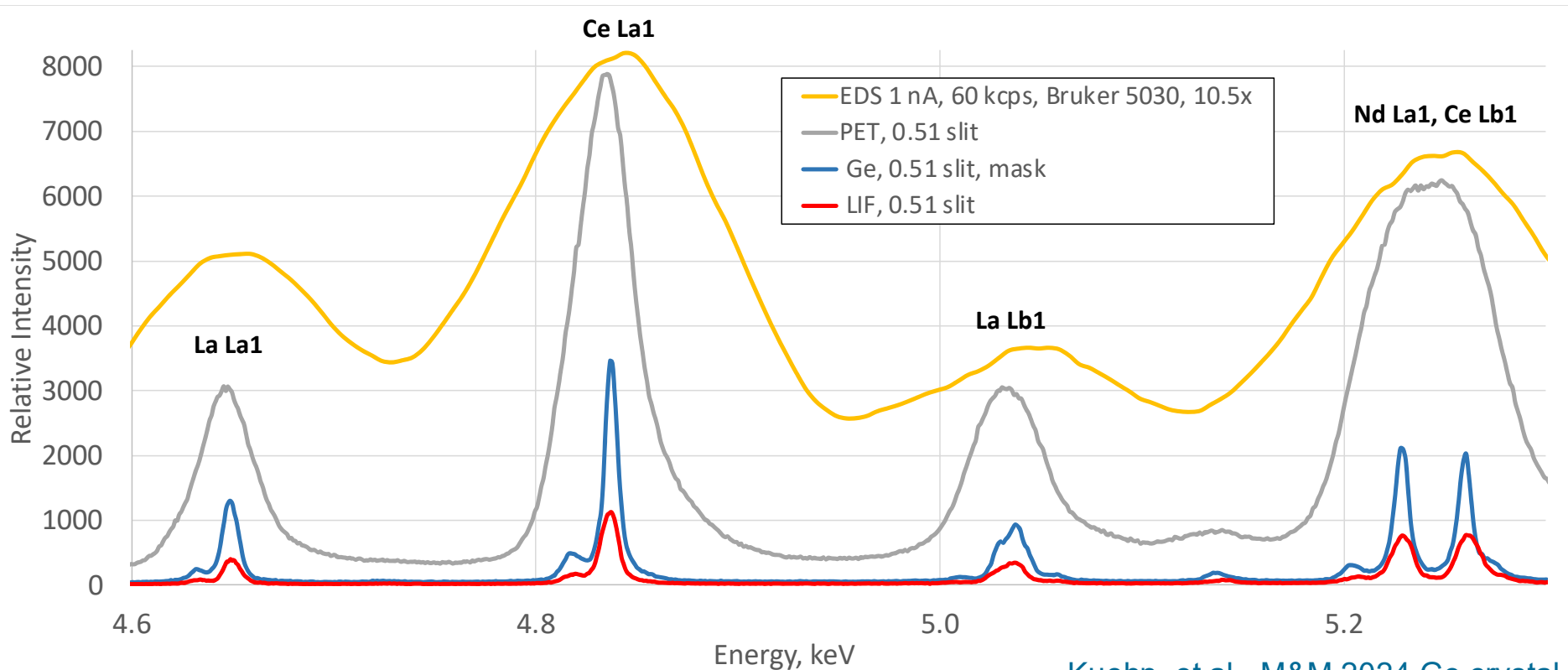
Concept and benefits

- **Change excitation:** E-beam to X-ray beam in SEM
 - Much lower backgrounds due to lack of Bremsstrahlung
 - Detection limits in the 10s of ppm even with EDS (up to 2 orders of magnitude lower)
 - More efficient excitation of higher energy X-ray lines which have fewer spectral interferences/overlaps (up to 50 kV X-ray tube)
 - Powder samples possible
 - Non-conductive samples possible without addition of a conductive coating
 - Extended thickness range possible for multilayer thin film samples
 - Less sensitive to quality of surface polish (with EDS detection)
 - Correlative analysis when combined with e-beam
 - Larger spot size
 - No e-beam damage
- **Change detection:** EDS to WDS
 - Much greater spectral resolution
 - Reduced spectral interferences
 - Greater peak to background ratios
 - Detection limits in the 10s of ppm with e-beam readily achievable, even single digit ppm with specially optimized methods
- **What if we put an XRF source on an EPMA and do both?**

Spectral resolution comparison

EDS vs WDS with E-beam:

REE in Monazite (Ce,LREE,Th,U,Ca)PO₄



Kuehn, et al., M&M 2024 Ge crystal

Techniques with potential overlap in capabilities with XRF in EPMA (XPMA)

Could XPMA take on some work currently done with other techniques?

Considerations:

- major element selection
- trace element sensitivity
 - sample size/type
 - powders, films
- destructive vs non-destructive
 - micro vs macro vs bulk
- conductive vs nonconductive
 - mapping
- cost, availability, accessibility

- Benchtop micro-XRF with EDS
- Benchtop XRF with EDS
- Floor model WD-XRF
- SEM with EDS
- SEM micro-XRF with EDS
- EPMA WDS+EDS
- LA-ICP-MS
- ICP-OES
- LIBS
- SIMS
- Etc.

Applications



- All the applications of SEM-EDS and EPMA
- Overlap with benchtop micro-XRF
- Overlap possible with LA-ICP-MS, SIMS, and others
- Geology and astro-materials
- Cultural heritage
- Materials Science
- Manufactured devices
- Forensics
- Metallurgy
- Biology
- Etc.

A few relative strengths and weaknesses (1)

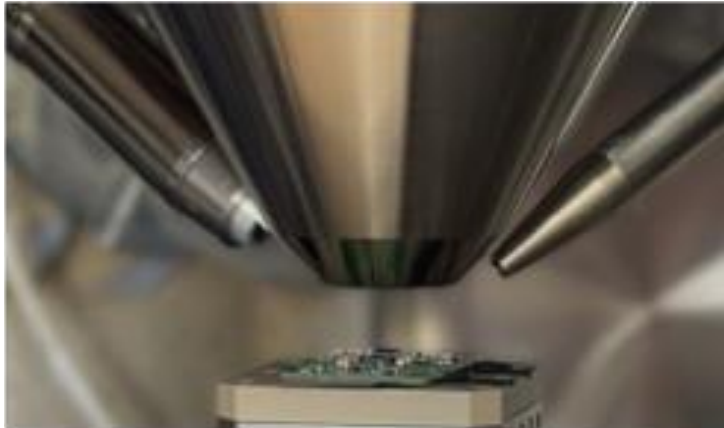
- SEM with EDS
 - Well-established, widely-used platform and technique with robust hardware and software ecosystem developed over decades
 - Combines routine e-beam imaging with (semi)quantitative compositional analysis of major-minor elements
 - Can host variety of add-on capabilities
- EPMA
 - Well-established technique with robust hardware and software also developed over decades
 - Specialized type SEM instrument hosting up to 6 WDS* and commonly 1 EDS
 - Also combines routine imaging with compositional analysis of major-minor and trace elements
 - All WDS quantitative analysis is fully standards-based

*12 fixed-element WDS + 1 EDS possible with existing hardware, but no implementations currently known to exist

A few relative strengths and weaknesses (2)

- Micro-XRF on SEM with EDS

- Relatively recent add-on to SEMs offering new modes of data acquisition and more sensitive trace-element analysis
- Micro-XRF optic enters below pole piece, approaching sample from an oblique angle thus resulting in oval spot.
- On SEM, multiple and large EDS can be possible, very close proximity to sample possible (high solid angle)
- Accurate Z positioning in SEM can be challenging - Often estimated from (an ideally well-calibrated) combination of kV, beam current, and final lens drive current – If Z too is high or too low, e-beam and XRF spots lose co-alignment



A few relative strengths and weaknesses (3)

- Micro-XRF on EPMA with simultaneous WDS and EDS
 - NEW and emerging combination
 - Potential to mount the micro-XRF source at a higher angle for a more circular spot – May be above, below, or through the pole piece depending on EPMA instrument design
 - Up to 7 simultaneous channels of X-ray signal collection combining WDS and EDS
 - EPMA is typically limited to 1 EDS detector, and the EDS may be farther from the sample than in SEM
 - Accurate Z-positioning of sample surface is routine using an integrated optical microscope - Optimal analytical geometry and beam co-alignment is easier and more reproducible.
 - Depending on the micro-XRF optic and details of the instrument mounting, the optic may be further from sample
 - Greater sample distance results in a larger XRF spot which could be broad enough to produce some defocusing of the WDS spectrometers

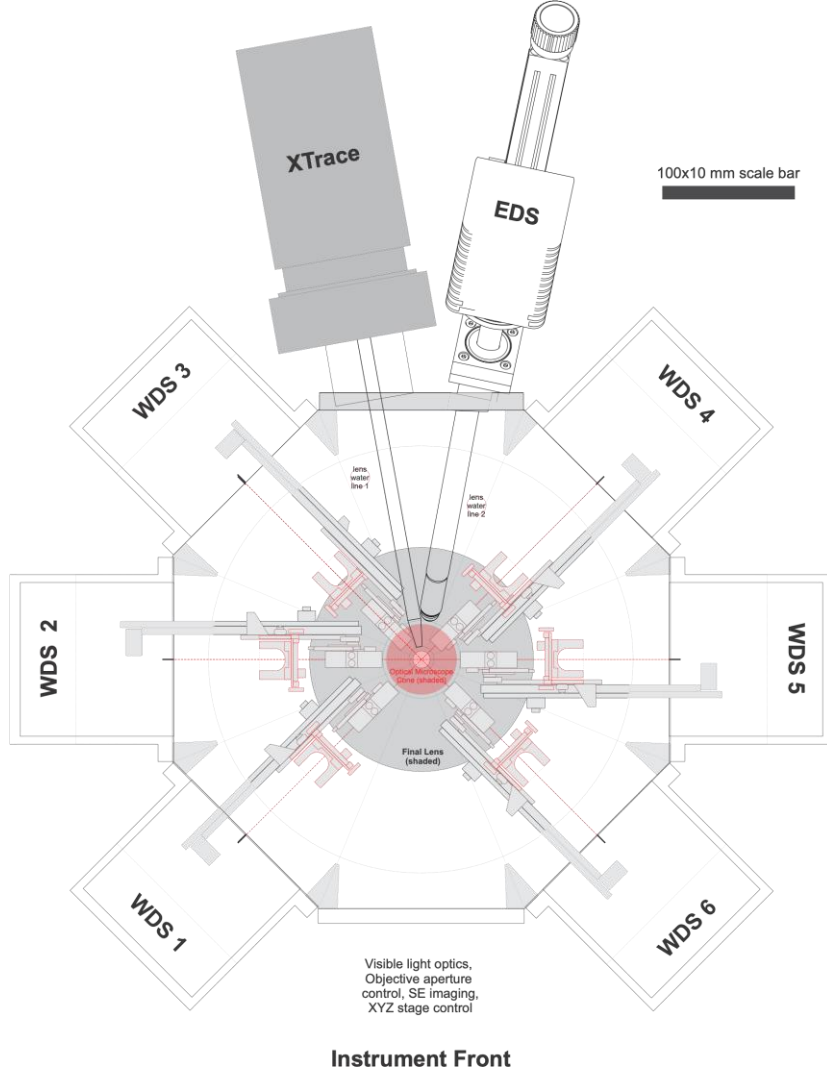
Field test instrument configuration:



- ARL SEMQ electron probe microanalyzer
- 6 WDS spectrometers - 4 with sealed xenon detectors (LIF, PET, Ge) and 2 with thin-window P-10 flow detectors (PET, RAP, OV60)
- Bruker 730M EDS
- Bruker XTrace 1 – on rear port next to EDS
- 52.5 degree take-off for WDS, above the final lens or pole piece
- Center of X-ray path between final lens and light microscope is slightly higher at 53.5 degrees (opening spans 47 to 60 degrees) – Thus the optimal XRF installation is at 53.5 degrees

Field test instrument configuration:

- Octagonal column frame
- Optical microscope (over final lens) and SEI detector (below lens) on separate front ports
- 6 WDS ports, 3 on each side – WDS views sample over lens, but the same ports could support below lens devices
- One rear port (over lens), used for XTrace and EDS *
- With custom mounting plates, can put what you want where you want, above or below the final lens

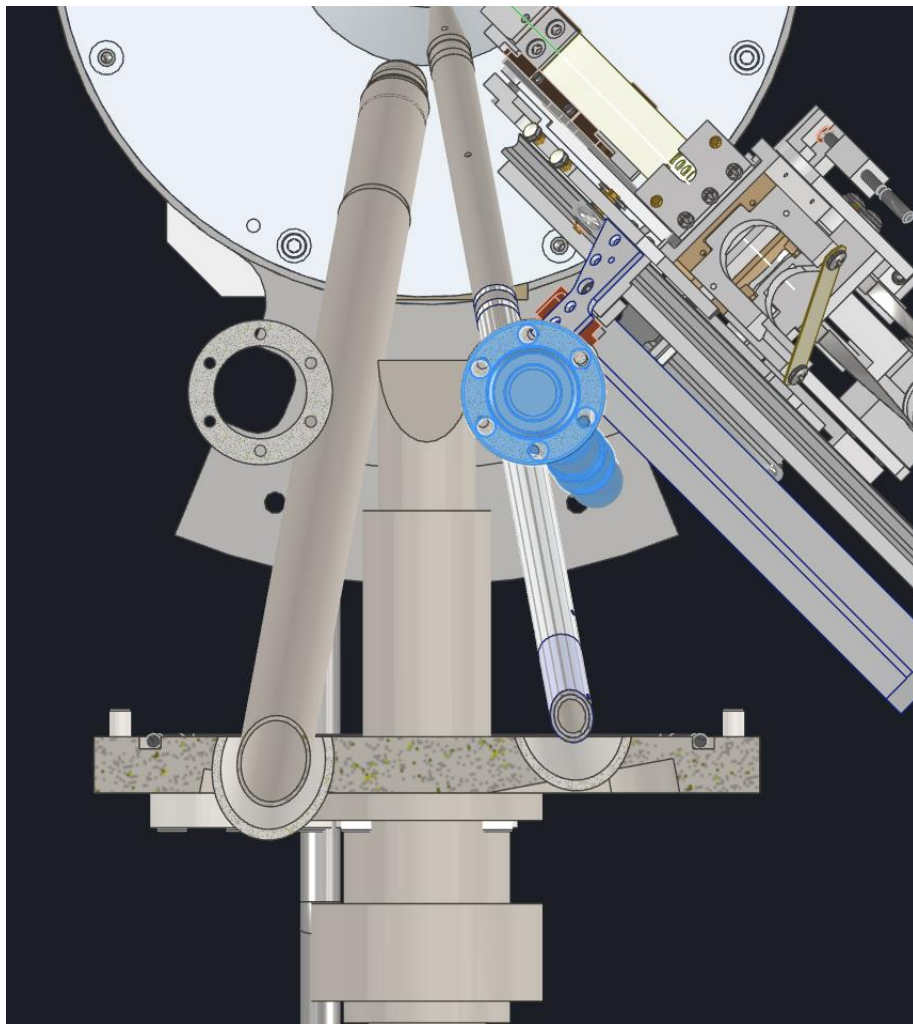


* Long-reach rear under lens port possible through diffusion pump mounting chamber

Field test instrument configuration:

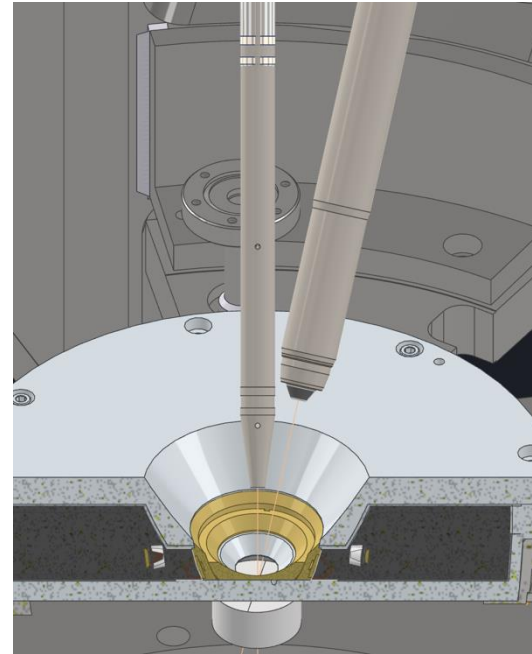


- Modern SEM electronics and software – SEM View 8000 system
 - “SEMQ 8000” configuration
- Modern EDS software – Bruker Esprit
 - EDS, XRF, Imaging/Mapping, Thin films, etc.
 - E-beam, XRF, and combined quantification
- Modern EPMA analytical software – Probe for EPMA 14.x
 - WDS, EDS, WDS+EDS
 - Advanced background, time-varying intensity, and spectral interference capabilities



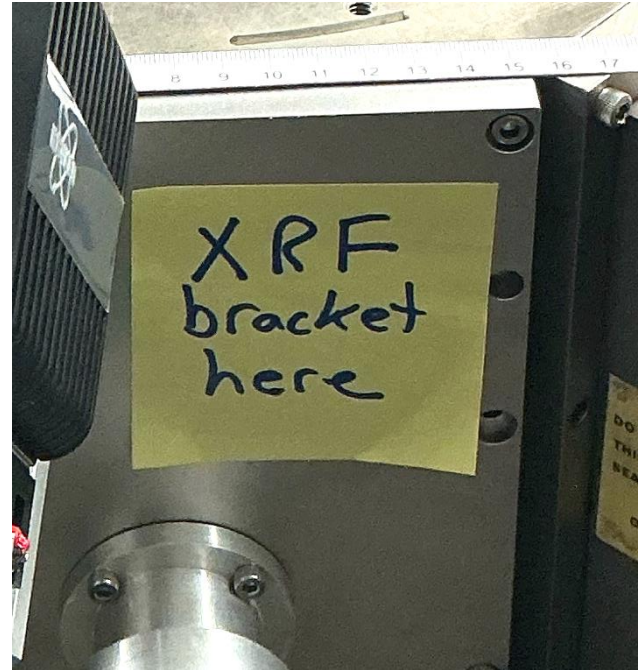
Preparation:

- Planning started in 2024
- Measurements and engineering diagrams
- Condenser lens cooling water line modification



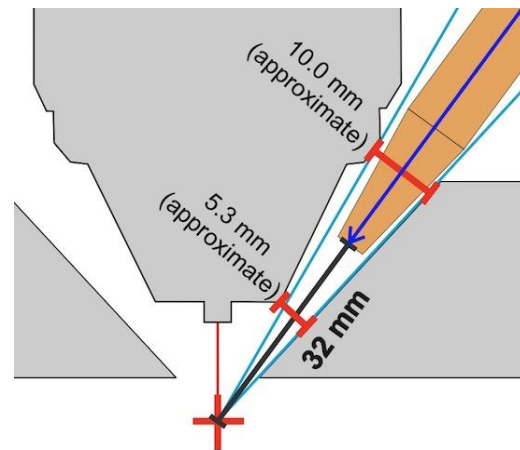
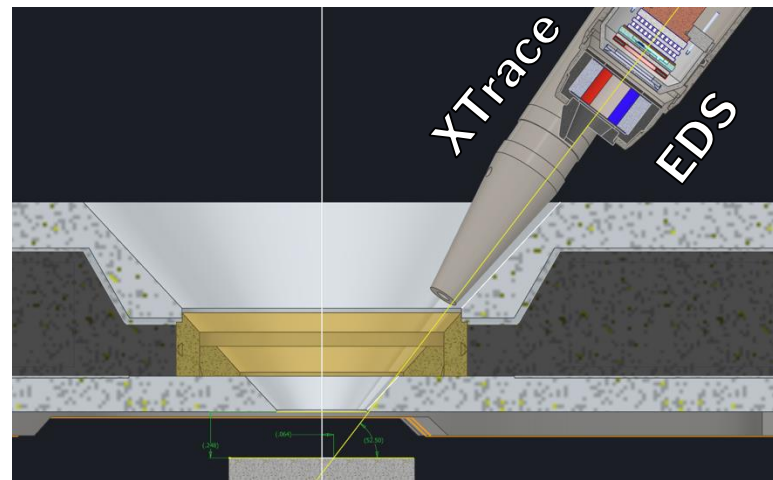
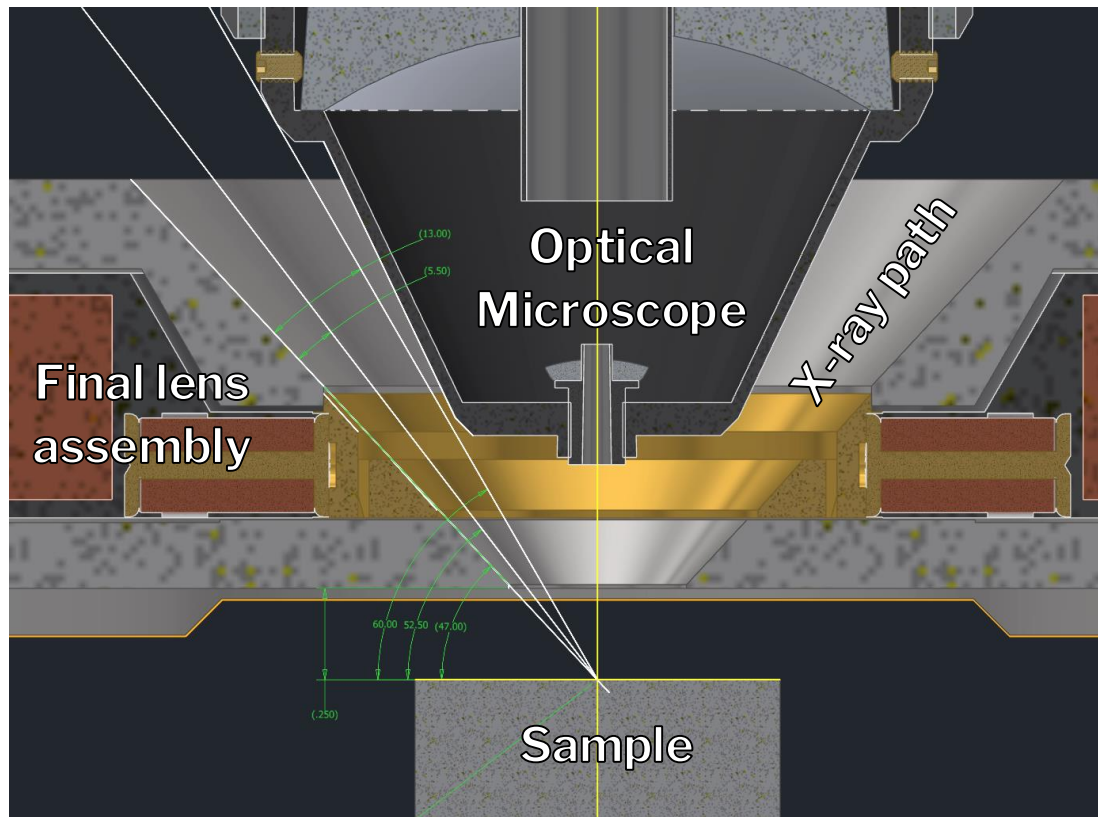
Preparation:

- Rear port installation via custom plate



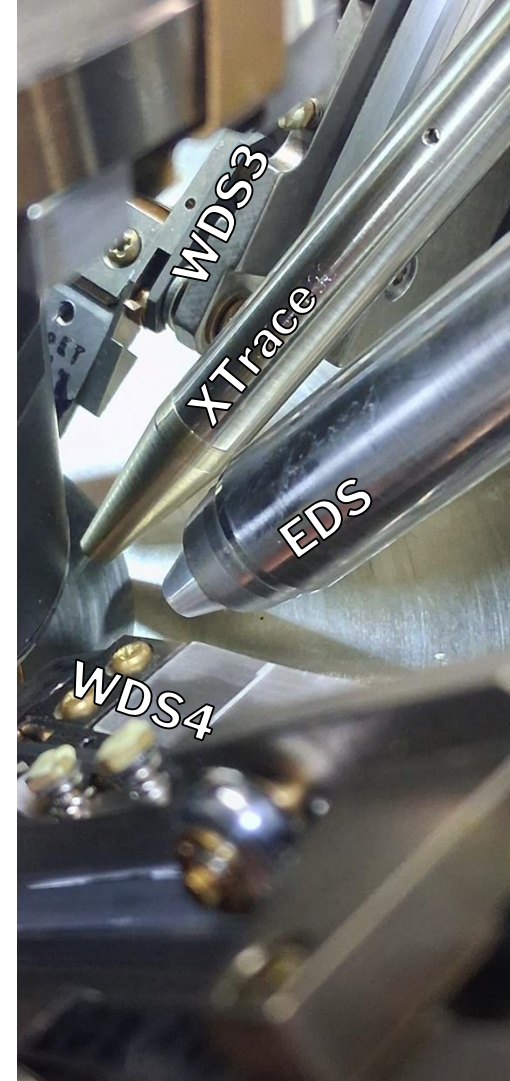
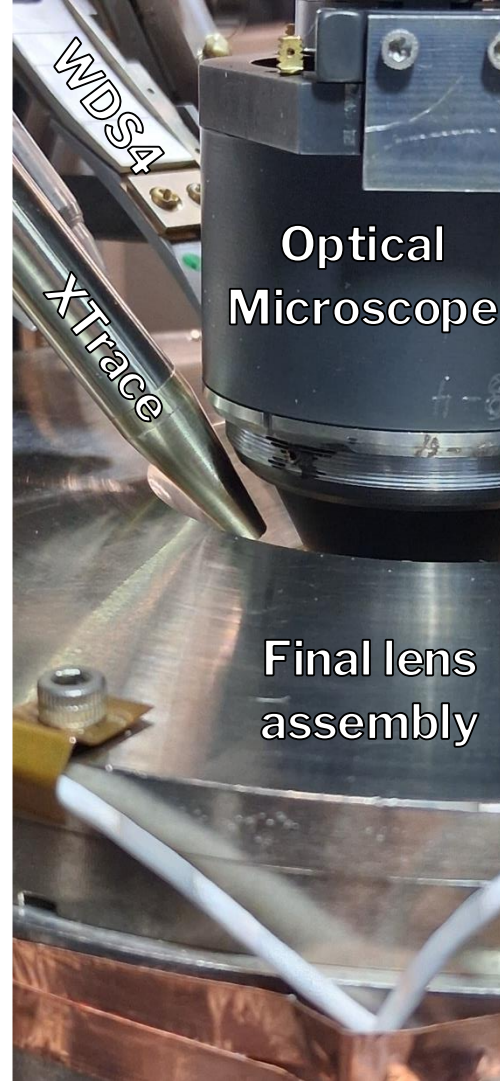
Preparation

— engineering diagrams



Installation in
late July
2026

just days
before M&M



To Do List

- Hardware
 - Optimize and finalize XTrace and EDS mounting and alignment
 - Move XTrace and EDS apart so don't collide – change azimuth
 - Better align with center of X-ray path at 53.5 degrees
 - Closer approach to sample, yielding smaller spot (probably a few hundred microns)
 - With final alignment, baseline calibration for quantification can be done
 - XTrace 2 would be smaller and higher power than XTrace 1
- Reference material targets
 - For larger spots and deeper X-ray penetration versus e-beam
 - Ten large fragment standard mounts prepared – just glasses to start
 - Need to prepare minerals, synthetic compounds, metals, REE, etc.

To Do List

- Software:
 - Need one software which can do XRF while using all of the WDS and EDS spectrometers of an EPMA, ideally supporting some form of combined e-beam and XRF quantification.
 - Add full WDS spectrometer control to Esprit?
 - Stage motor control is already implemented
 - Spectrometer motors use the same controller and command set
 - Add XRF quantification to Probe for EPMA?
 - Already has full WDS control and can request EDS spectra via API
 - Or some third option?
- Method development and verification