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# **Recent & Historical Geochronology Instrument Developments @ JPL**

**Paula Grunthaner**

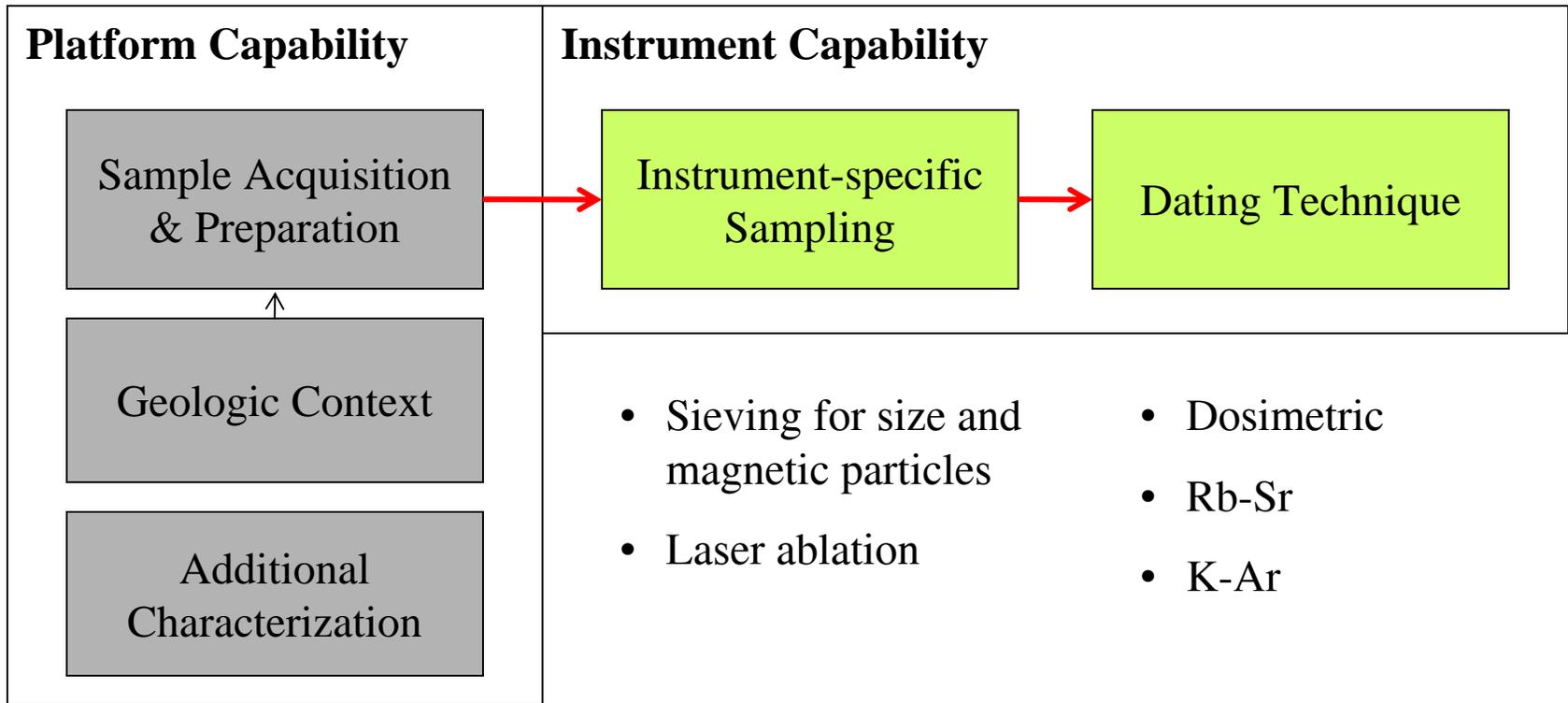
**Special thanks to J. P. Kirby**

**Jet Propulsion Laboratory, California Institute of Technology**

# Topics

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- What are the challenges for an *in situ* system?
- What approaches have been (or are being) tried?
  - Intent is to provide a sense of the development status of geochronology instrumentation
- What challenges do these developments face going forward to flight?



- Chronological techniques not applicable to all rock types
- Sample must be free of post-crystallization disturbances
- Dust contamination
- As a minimum, expose fresh surface
- Desirable, pre-selection of grains

# In Situ Geochronometer Developments

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- **Luminescence/ESR technique**
  - JPL, Ulmer Systems, Oklahoma State U, McMaster U, LPI
- **$^{87}\text{Rb}$  –  $^{87}\text{Sr}$  decay**
  - JPL, U Pittsburgh → Scott Anderson's talk
  - JPL, U. Wisconsin, Tangent Technologies, OI Analytical
- **$^{40}\text{K}$  –  $^{40}\text{Ar}$  decay & cosmogenic nucleotide buildup ( $^3\text{He}$ ,  $^{20,21,22}\text{Ne}$ ,  $^{36,38}\text{Ar}$ )**
  - UA, JPL, LANL → Tim Swindle's talk

## Luminescence and Electron-spin Resonance Dating

S. Kim (JPL), S. McKeever (Oklahoma State U), W. Rink (McMaster U),  
S. Clifford, LPI, A. Yen (JPL)

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- Dosimetric approach that integrates TL, OSL, and ESR
- All 3 used for dating time since some dynamic geological resetting event
  - Time since last exposure to sunlight (solar reset) and thus burial ages of soils in sediments
  - Time since last thermal event such as volcanic heating, mechanical stresses
  - Measures the accumulation of absorbed radiation dose within mineral grains as  $f(\text{time})$ 
    - Ionizing radiation accumulates trapped charge in the mineral, which is depleted when exposed to solar radiation (resetting the clock)
    - Luminescence intensity  $\propto$  dose since last exposure to sunlight  $\propto$  depositional age
- Utility: dating eolian, fluvial, periglacial, impact, volcanic processes spanning the past 100 ka – 1 Ma)

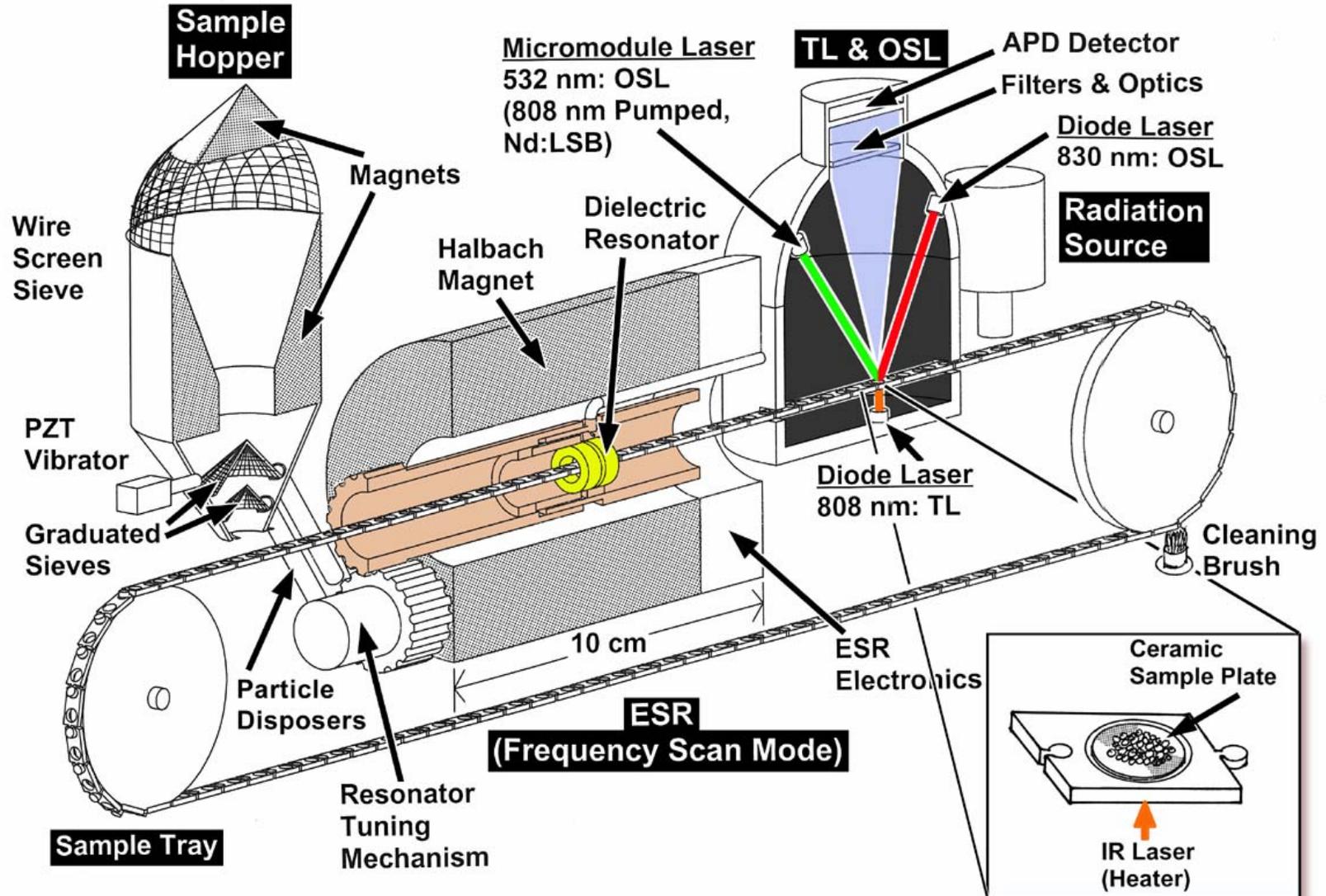
# Applicability of techniques, as practiced on Earth

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	TL	OSL	EPR
Eolian/Fluvial deposits (age of burial, solar reset)	M	H	L
Volcanic Rocks (crystallization age)	H	H	L
Sedimentary Volatile Deposits (age of crystallization of carbonates, sulfates)	L	L	H
Dust Particles in Polar Ice Caps (age of incorporation into ice)	L	H	L
Material characterization	L	L	H

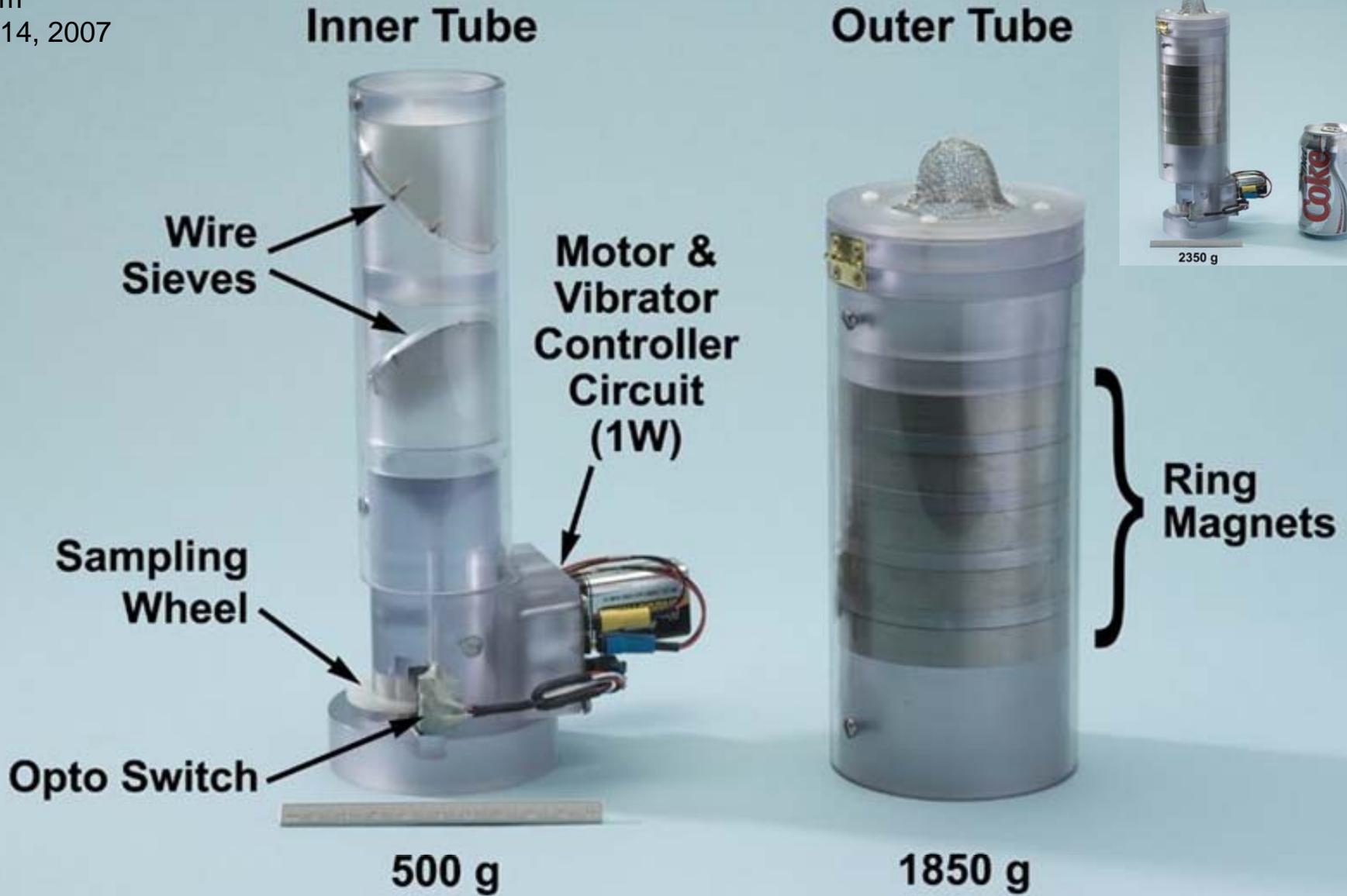
Applicability H: High M: Medium L: low

# Luminescence and Electron-spin Resonance Dating Breadboard Concept



# Luminescence and Electron-spin Resonance Dating Development Status—Sample Preparation Unit

S. Kim  
May 14, 2007



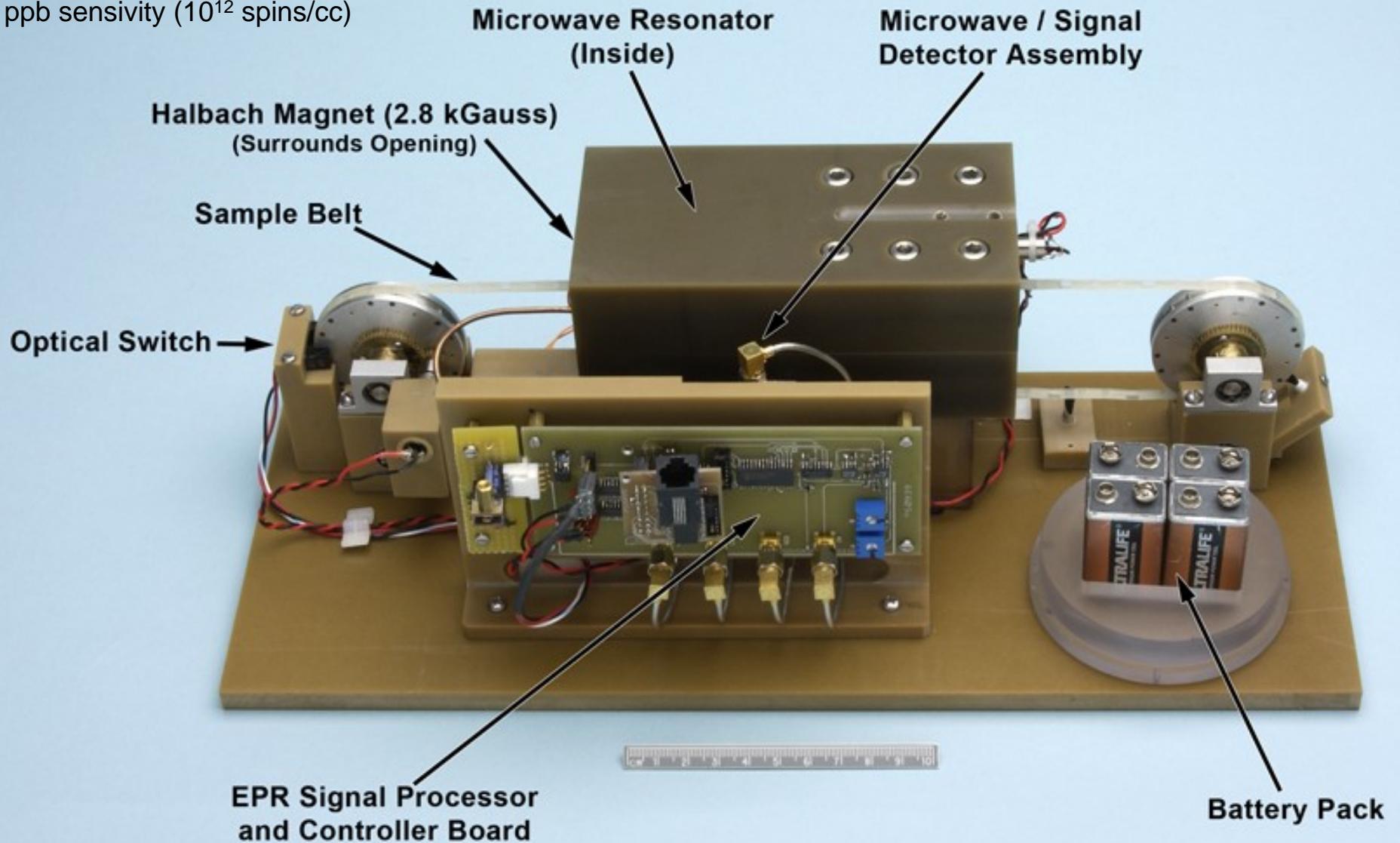
# Luminescence and Electron-spin Resonance Dating Development Status—ESR Spectrometer subsystem

S. Kim

May 14, 2007

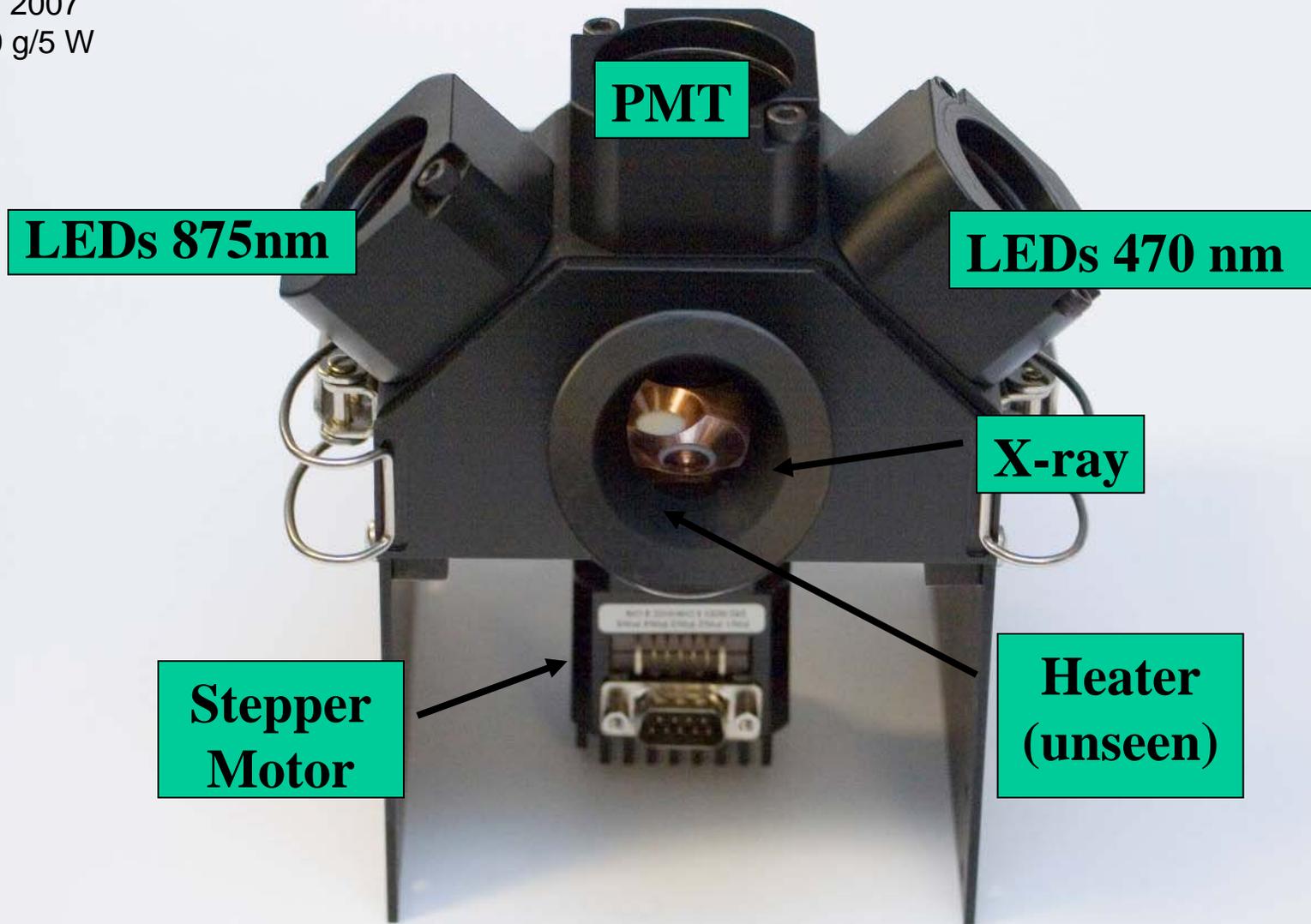
ppb sensitivity ( $10^{12}$  spins/cc)

(3W, 3.8 kg)



# Luminescence and Electron-spin Resonance Dating Development Status— OSL and TL subsystem

S. Kim / Oklahoma State  
May 14, 2007  
ppb/400 g/5 W



# Luminescence and Electron-spin Resonance Dating Challenges Going Forward

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- No currently funded task
  - JPL PI submitted PIDDP on a different topic because geochronology not a priority in the NRA
- Science— Do we understand dosimetry on Mars well enough?
  - On Earth, use mineral separates; on Mars likely polymineralic
  - On Mars, lower natural dose rate but 1000x higher cosmic ray flux
  - Necessitates need to know burial rate
  - Gradual accumulation over time => variable dose rate
  - Efficiency of solar bleaching in low T of Mars

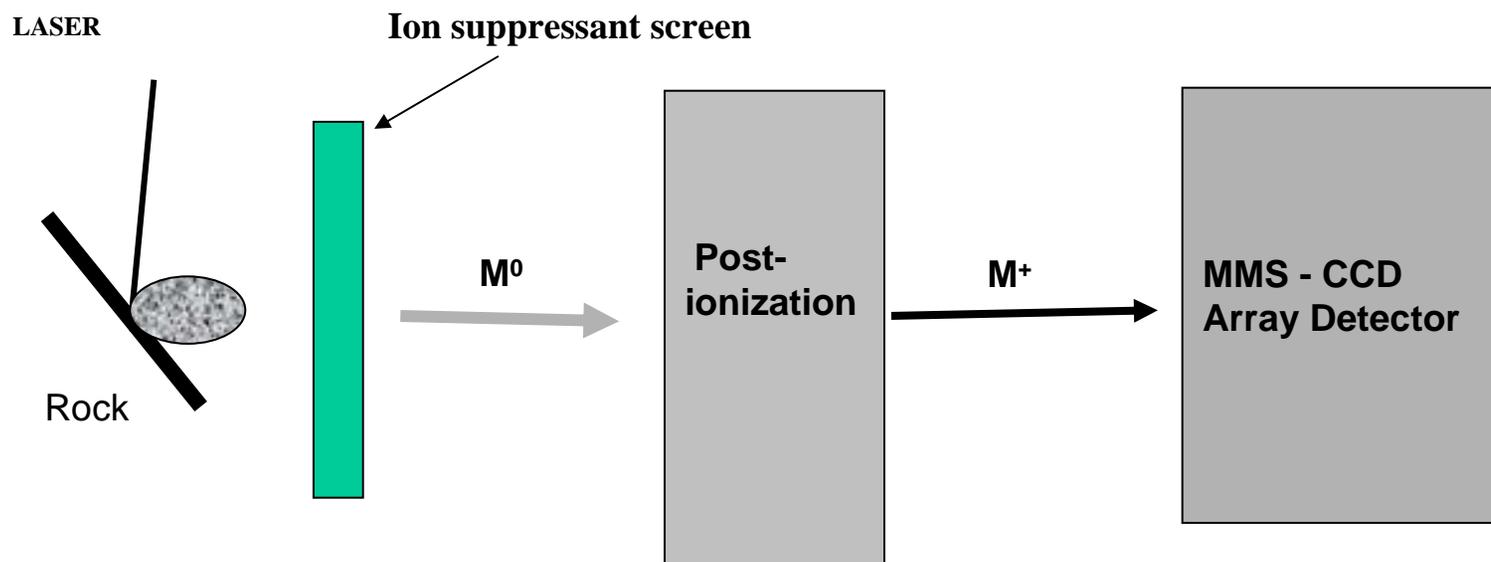
# Rb-Sr Geochronology

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- A few reminders about the technique
  - $^{87}\text{Rb}$  to  $^{87}\text{Sr}$  by  $\beta^-$  decay
  - $(^{87}\text{Sr}/^{86}\text{Sr})_i = 0$  not valid nor is it possible to estimate. So ratio is calculated by measuring 2 or more minerals in same rock with spread in Rb/Sr ratios
  - Measurement masses are 85, 86, 87. Use fact that  $^{87}\text{Rb}/^{85}\text{Rb}$  is constant in all SS materials to derive desired  $D_r/D_s$  and  $P/D_s$  for isochrons

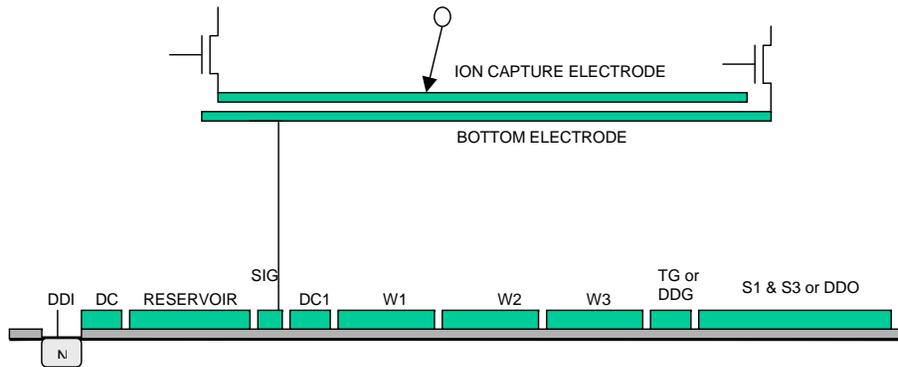
# Laser Ablated – Electron Impact Ionization – MS for Geochronology (Rb-Sr)

M. Sinha (JPL), B. Beard (U WI), M. Wadsworth (Tangent), OI Analytica

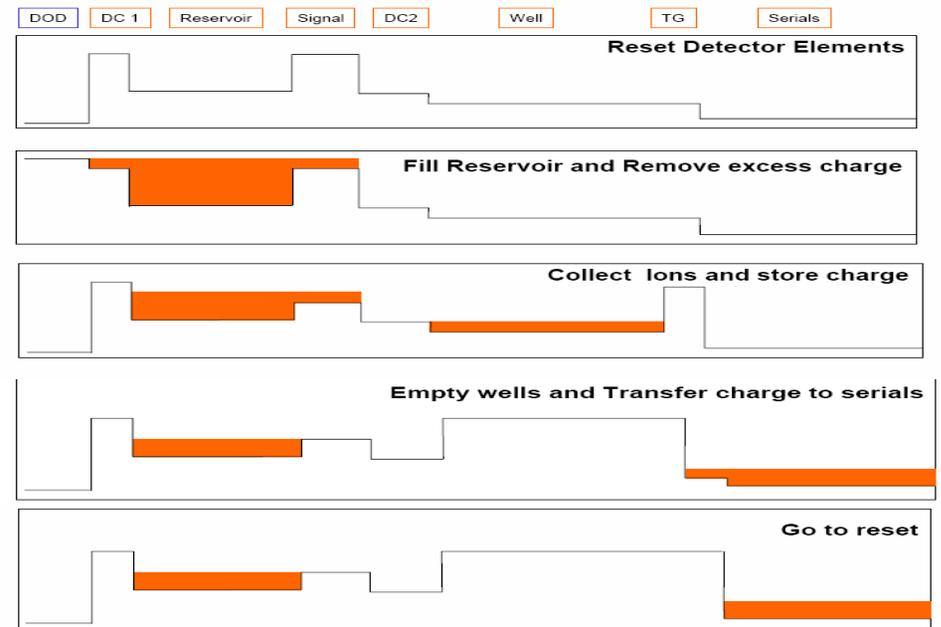


- Laser-ablation and sampling of neutrals only
  - Potential for cleaner mineral separates & better understanding of internal isochrons
  - $10^2$ – $10^6$  more neutrals than ions; may better represent elemental abundance
- Electron impact ionization of neutrals creates 100–1000  $\mu$ s ion pulse
- Non-scanning magnetic sector MS with custom focal plane array detector integrates entire ion pulse simultaneously
- Estimated precision of age dating: 95% confidence of  $\pm 150$  Ma for a 500 Ma rock

# Direct Ion Detection with a Modified CCD Focal Plane Array



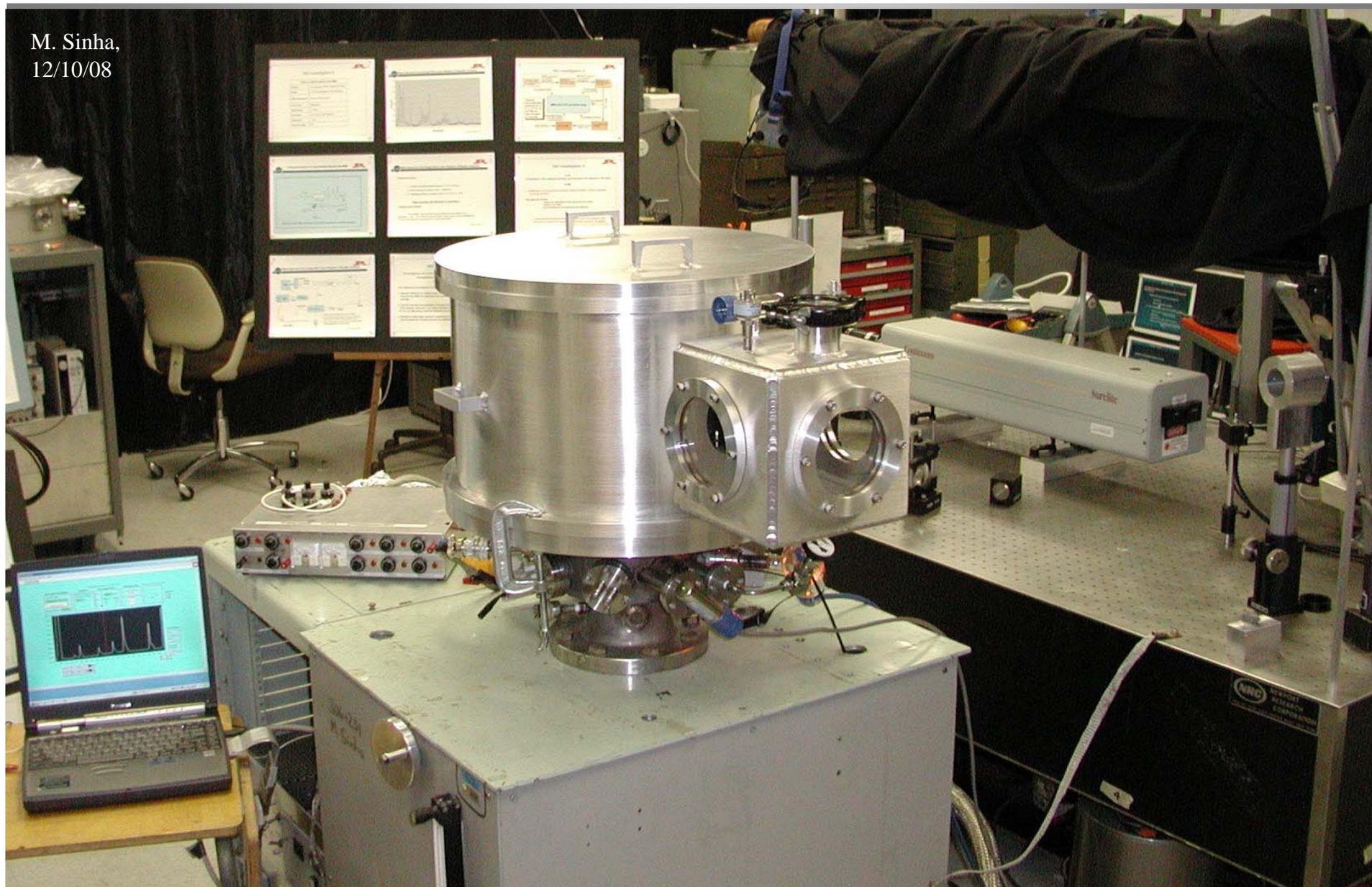
- Linear array of CCD pixels
- Photodiode replaced by a capacitive sensing element that serves as ion detector
- Capacitive element is coupled to CCD shift register and creates a packet of signal charge proportional to the charge on the capacitor
- 2<sup>nd</sup> generation CCD improves sensitivity, reduces noise, increases dynamic range and resolution



# Laser Ablated – Electron Impact Ionization – MS for Geochronology

Sinha, et al

M. Sinha,  
12/10/08

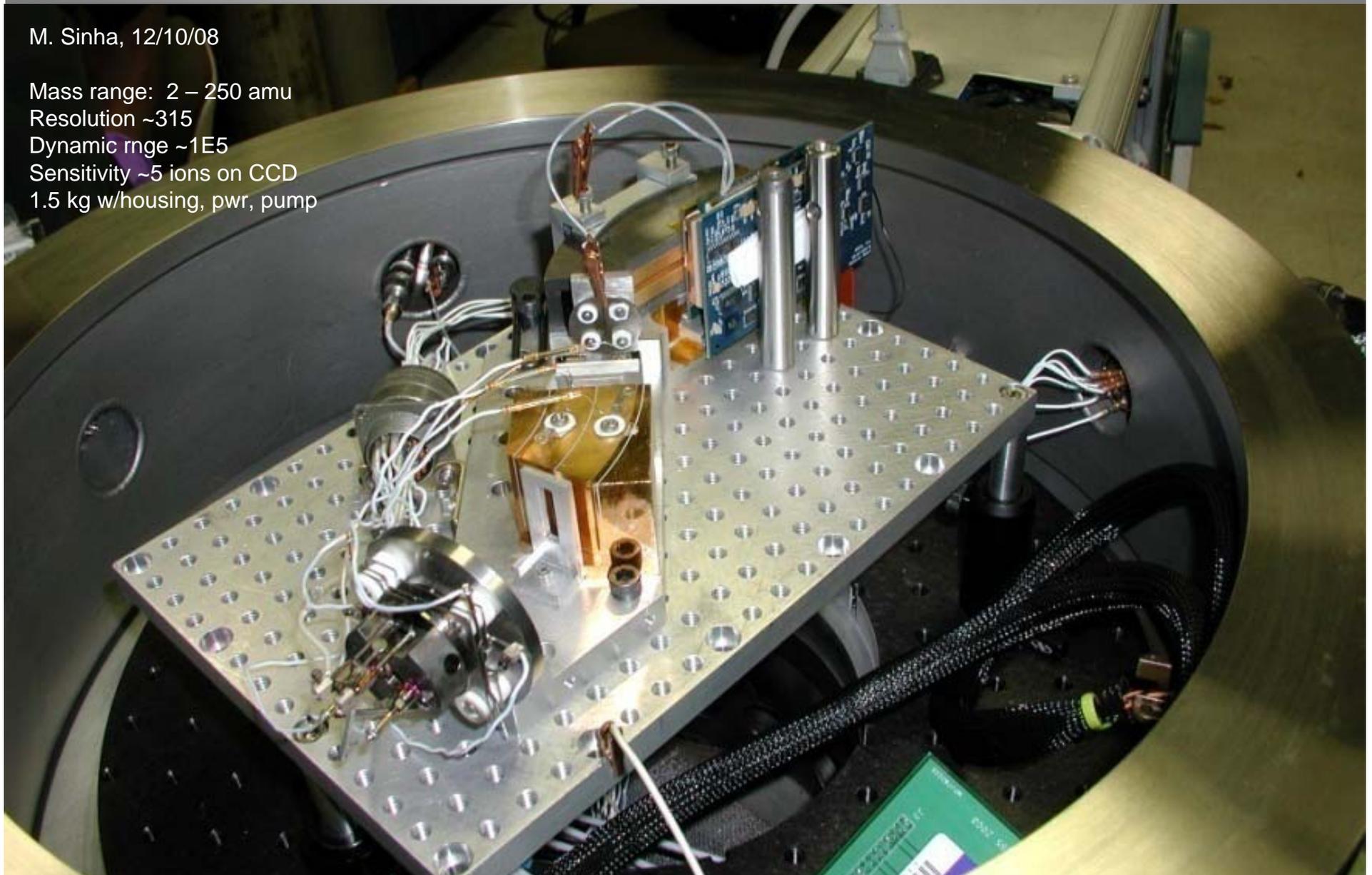


# Laser Ablated – Electron Impact Ionization – MS for Geochronology

## Sinha, et al

M. Sinha, 12/10/08

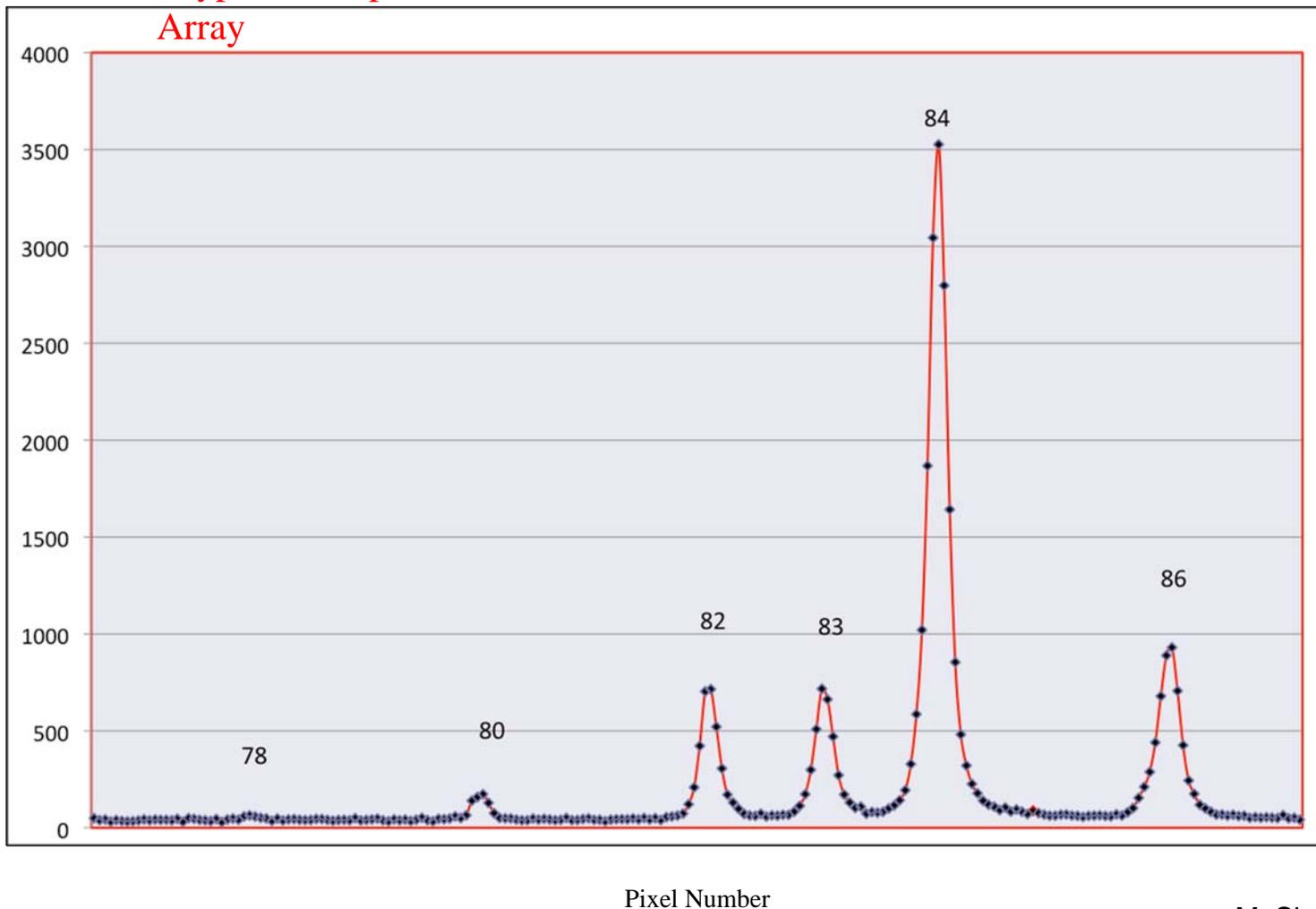
Mass range: 2 – 250 amu  
Resolution ~315  
Dynamic range ~1E5  
Sensitivity ~5 ions on CCD  
1.5 kg w/housing, pwr, pump



# Laser Ablated – Electron Impact Ionization – MS for Geochronology

## Sinha, et al

Krypton Isotope Measurement with CCD Direct-Ion-Detector Array



## Error Estimates for Rb-Sr ratios

	Plagioclase	Clinopyroxene	Orthopyroxene	Olivine	
Sr ppm	100	10	3	1.5	
$^{87}\text{Sr}/^{86}\text{Sr}$ and % error	$0.70537 \pm 0.0793 \%$	$0.70990 \pm 0.1770 \%$	$0.71134 \pm 0.5499 \%$	$0.70836 \pm 0.5869 \%$	yesterday $\pm 1\%$
$^{87}\text{Rb}/^{86}\text{Sr}$ and % error	$0.0513 \pm 1.277 \%$	$0.687 \pm 0.349 \%$	$0.8901 \pm 0.5869 \%$	$0.4717 \pm 1.203 \%$	$\pm 10\%$

- Assumes  $1 \times 10^6$  atoms/laser shot, 10% of atoms delivered to ion source, 1 in 1000 atoms ionized, 20% of ions arrive at detector, ions accumulated for 100 shots, all minerals had  $(^{87}\text{Sr}/^{86}\text{Sr})^i$  of 0.705 and are 500 Ma old
- For a 4-mineral isochron using the above estimates, 95% confidence of age error  $\pm 140$  Ma for 500 Ma rock

# Challenges Going Forward for Both Rb-Sr Systems

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- Rb and Sr must be ionized in direct proportion to their abundance in the sample
- Laser ablation for sampling
- Potential for cleaner mineral separates & better understanding of internal isochrons
- Both techniques sample neutrals, not ions;  $10^2$ – $10^6$  more neutrals than ions; may better represent elemental abundance
- But, laser ablation (w/ICPMS) has shown significant elemental fractionation for elements of different volatility. Neutral generation is thermal.
  - Rb (688 °C) and Sr (1382 °C) have very different melting points
  - The extent of elemental fractionation must be characterized for different minerals
- Ionization efficiencies must be understood. In this respect, electron impact is more commonly used and may be better understood than resonant ionization (but, heck, I'm not sure)
- Resonant ionization is highly sensitive for ionization and highly selective. Electron impact is not selective and can, therefore, be used to characterize other neutral species.
- Both approaches must develop differentially pumped systems to keep sample outside (or bring the sample inside and pump down each time)

# Summary

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- There are several age dating systems operating at TRL 4 with potential to meet performance requirements
- None of these systems have yet demonstrated they can deliver the performance demanded
  - Elemental sampling and/or ionization bias effects need to be examined
  - H/W needs miniaturization & subsystems
- KISS should identify the tall tent poles that may not be funded under existing PIDDP/MIDP/etc funds
- To paraphrase Gregg, development needs to proceed *tout de suite*