

**A Miniature Electron Probe for In Situ Elemental Microanalysis.** L.F. Lim<sup>1</sup>, S.A. Getty<sup>1</sup>, C.A. Kotecki<sup>1</sup>, A. E. Southard<sup>1,2</sup>, J. Gaskin<sup>3</sup> <sup>1</sup>NASA/GSFC, Greenbelt, MD 20771 (lucy.f.lim@nasa.gov), <sup>2</sup>USRA, <sup>3</sup>NASA/MSFC

**Introduction:** The Mini-EPMA is an adaptation of the standard laboratory technique of electron probe microanalysis (EPMA) to a flight instrument suitable for a lander or rover mission. Mini-EPMA will combine an addressable-array carbon nanotube (“CNT”)–based electron source with a large-area silicon-drift X-ray detector (Fig. 1). The combination of efficient micro-scale compositional mapping with a new light-element (C/N/O) capability will significantly advance our capability for remote *in situ* determination of the elemental composition of planetary, asteroidal, and cometary material. Composition provides key evidence about the processes by which rocks, soils, and ices were formed and altered (for example, accretion, differentiation, and hydrothermal alteration) thus recording past stages in solar system evolution. The capability for rapid basic elemental analysis will also contribute to the location of resources to support exploration.

The high spatial resolution achievable with a focused electron beam will for the first time permit sub-millimeter scale compositional mapping in a flight instrument similar to that provided by a scanning electron beam in the laboratory. Preliminary modeling with SIMION [1] indicates that spot sizes under 100  $\mu\text{m}$  are achievable in a flight instrument with a miniaturized electrostatic lens stack. This represents an improve-

ment of a factor of over 100 in spatial resolution over the current generation of flight X-ray elemental analysis instruments, whose radiogenic sources limit their fields of view to several centimeters. The intrinsically micron-scale CNT emitters are well suited for production of such a high-resolution electron beam. Moreover, a single CNT cathode can be subdivided into an array of addressable emitters, which when activated sequentially will induce characteristic X-ray emission from a grid of 100- $\mu\text{m}$  spots on the planetary/asteroidal/cometary surface. The result will be similar to compositional maps routinely provided in laboratory EPMA by magnetically focused electron beams (*e.g.* Fig. 2) but with the simpler operational requirements, lower power consumption, and more robust hardware required for a flight instrument. This advance will thus provide a new capability to resolve key petrographic textures and fine-scale structures *in situ* and without consumables.

We expect the high S/N provided by the electron beam and large-area ( $\geq 100 \text{ mm}^2$ ) detector to enable integration times in the range of  $\approx 1$  minute per spectrum, or under two hours for a 10x10 element spectral map, with spectral sensitivity comparable to that of existing flight XRS instruments.

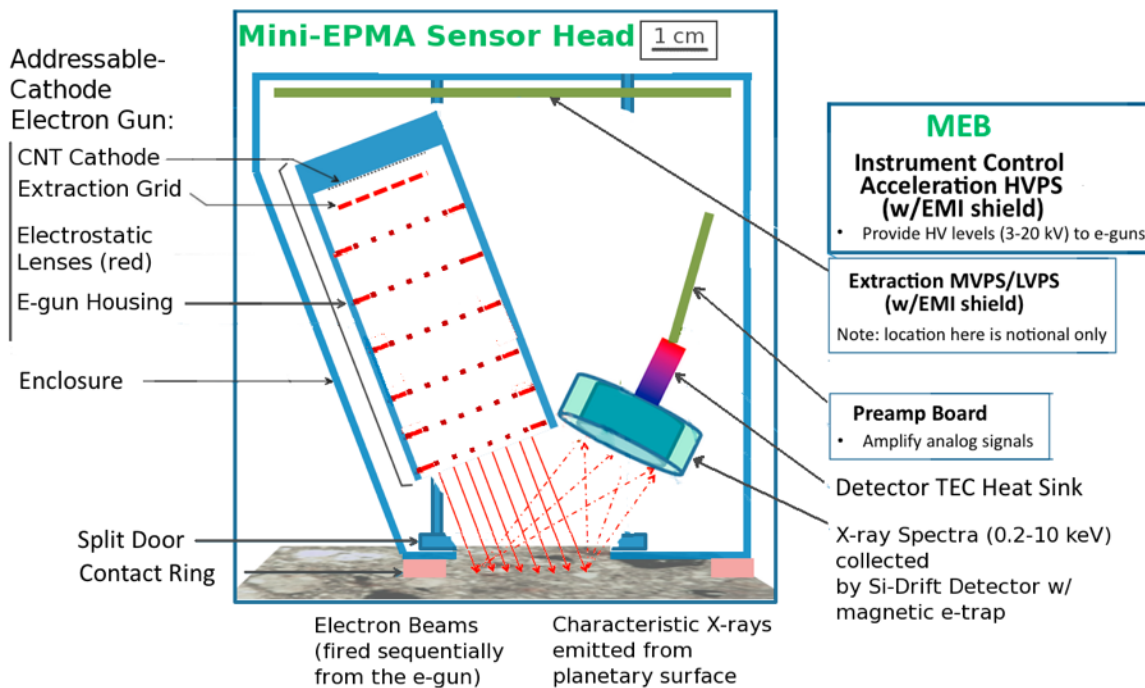


Fig. 1. The miniature electron gun will sequentially illuminate 100- $\mu\text{m}$  spots on the natural rock, soil, or ice surface, thus enabling *in situ* mapping of elemental composition on the scale of the beam diameter without moving parts.

**Mass, power, and spacecraft accommodation:** A systems-level treatment of the flight concept by a team of seasoned systems and discipline engineers in the areas of electronics, structures, mechanisms, thermal design, and contamination control (the GSFC Instrument Design Laboratory) resulted in a total flight instrument mass under 4 kg, peak power under 13 W, and average power under 6 W, including redundant (2x) electron guns and detectors for increased field-of-view and reliability. The device will thus be practical for use on any lander or rover missions to airless bodies, including those under the Discovery and New Frontiers programs.

The mini-EPMA system will be able to analyze unprepared planetary materials. The penetration depth of the electron beam is shallow, on the order of one micron (depending on the material and the accelerating voltage). This will permit analysis of surface coatings of dust or weathering rinds and enable (for instance) evaluation of the effects of space weathering in the upper few microns of an asteroid regolith.

For determining bulk rock compositions, however, this instrument would ideally be used in conjunction with a tool capable of exposing pristine rock surfaces, such as the MER Rock Abrasion Tool (RAT).

Performance of the active XRS system will be best when both source and detector can be positioned close to the surface to be analyzed, so a deployment arm or other means of positioning the mini-EPMA detector head will be desirable for an optimal configuration. We estimate that the ideal configuration will position the instrument approximately 2 cm from the sample to be analyzed.

**Science Operation:** In a minimal landed mission scenario, a single deployment of the mini-EPMA device onto the surface would return a single petrographic map of the surface material over a mission lifetime of a few hours. This would be an important advance over existing techniques as not only the bulk composition would be determined but also its heterogeneity on a submillimeter scale.

The electron beam is nondestructive, allowing other analytical techniques to be employed subsequently on the same sample. The elemental information provided by the proposed instrument would be well complemented by infrared spectroscopy, Mössbauer spectroscopy, or other mineralogical techniques.

**Light-element capability:** The electron beam will provide efficient excitation of characteristic  $K\alpha$  X-rays from elements lighter than Na (C/N/O/F). Together with the low-energy sensitivity of the polymer-window SDD detector system, this enables the measurement of X-ray emission lines from these very light elements, which are inaccessible to the current generation of flight XRS systems. Measurement of the 277 eV carbon  $K\alpha$  line in carbonates in this configuration has been demonstrated in laboratory settings. Carbon, apart from its astrobiological interest, is structurally important to a class of minerals (carbonates) key to understanding aqueous processing in solar system materials. Other forms of carbon (such as graphite, carbides, and organic carbon) are also important constituents of several classes of meteorites, notably ureilites and various carbonaceous chondrites.

Oxygen and the O/Si ratio in particular are also important for identifying carbonaceous chondritic sub-

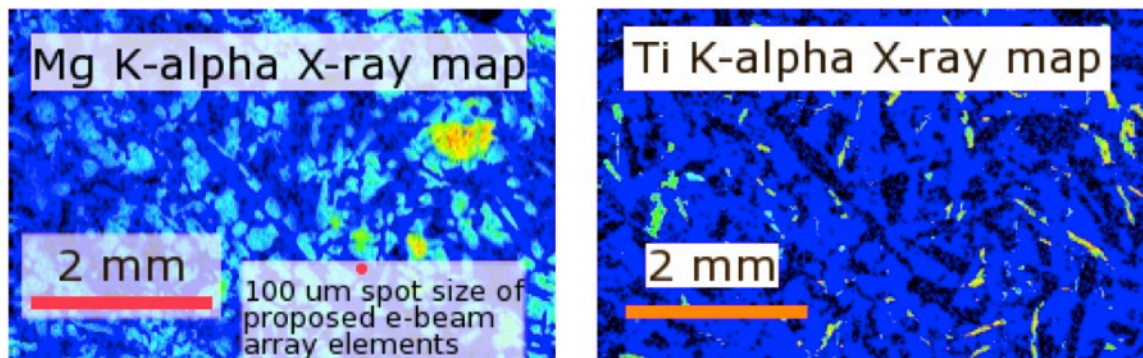


Fig. 2: The addressable array electron beam of the mini-EPMA will enable compositional maps such as these to be acquired *in situ*. X-ray compositional maps of unbrecciated lunar basalt LAP 02205 in Mg (left) and Ti; Fig. 1 from Righter *et al.* (2004). (The equipment used was a CAMECA SX100 electron microprobe at 15 kV accelerating voltage and 20–40 nA current.) The resolution of our addressable-grid electron source ( $\leq 100 \mu\text{m}$  spot size) will be sufficient to resolve both the olivine and pyroxene grains (yellow and cyan respectively in the left image) and the plagioclase and ilmenite laths (black and yellow respectively in the right (Ti) image). The compositions of individual mineral grains could thus be measured.

classes as relatively water-rich (CI and CM chondrites) or water-poor (e.g. CO, CV, CK), as O/Si in meteorites above the value found in olivine (O/Si = 2.3) is generally carried by water of hydration [3].

**Spatial resolution and sensitivity:** The addressable carbon nanotube cathode electron beam will permit compositional mapping with a spot size of 100  $\mu\text{m}$ . By activating the cathode elements in sequence, it will be possible to assemble concentration maps similar to those illustrated in Fig. 2 and to return compositional analyses of individual mineral grains and rock microstructure.

Laboratory electron microprobe analyses of mineral grains routinely achieve precisions of 1% or less for the weight percentages of major-element oxides as well as measurements of minor-element oxides <0.5% by weight using beam currents  $\approx 10\text{--}100$  nA, accelerating voltages 15–20 kV, cm-scale working distances, and exposure times  $\approx 20\text{--}30$  s. These levels of current and voltage will be readily achievable with the miniature electron gun albeit at a larger spot size ( $\approx 100$   $\mu\text{m}$  instead of <1  $\mu\text{m}$ ). We will also evaluate the benefit to the light-element analyses of collecting a second spectral map at a lower beam voltage (3–5 kV) where the C/N/O cross-sections are higher [4].

**Advantages of the CNT Electron Source:** The cathode of the miniature electron gun will consist of an array of CNT emitters that can be individually addressed. The primary advantage of this approach is that it requires no moving parts and is immune to nonlinearities that are inherent to scanning techniques. CNT cathode emission is especially well suited to this application owing to the demonstrated ability to pattern CNT films on a micron scale.

The CNT field emitter provides low operating power, scalability in size, addressability, current output, and long lifetime compared to other field emission materials [5, 6, 7]. In a prototype CNT e-gun developed at GSFC, we observed power consumption levels that were reduced by two orders of magnitude compared to a miniaturized thermionic e-gun competitor for similar operating conditions [8]. Furthermore, lifetesting investigations have shown that our CNT cathodes can produce a measurable beam current for over 450 hours in high vacuum ( $2\text{e-}7$  Torr). Moreover, due to the small size and mass of the e-gun (2.5 cm square by <10 cm long), longer life could be achieved by including redundant emitter arrays.

To achieve spatial resolution on the sub-mm scale, the extraction grid will be subdivided into addressable elements. The CNT emitter array will have 100 individual emission elements on a 10x10 array. Each ele-

ment can be engaged individually through a switching mechanism that requires a relatively small change in voltage.

**References:**

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