Specifications for Modified Secondary Ion Mass Spectrometer (SIMS)

General Description

The Naval Research Laboratory (NRL) has a requirement for a modified commercial Secondary Ion Mass Spectrometer (SIMS) to be used as an ion source for NRL's existing Trace Element Accelerator Mass Spectrometer (TEAMS) system. The TEAMS system is built around a 3 MV National Electrostatics Corporation (NEC) Pelletron tandem accelerator. It currently includes a NEC 40-cathode Model MC-SNICS negative ion source that injects ions into a NEC switching electrostatic analyzer (ESA), followed by a unique Danfysik-built "Pretzel" magnet used as a notch mass filter. The negative ions are then accelerated to the positive terminal of the tandem accelerator where they transit through an Ar-gas stripper cell and are converted to positive atomic ions. These positive ions are further accelerated back to ground potential, deflected electrostatically by 3° to select one charge state, energy analyzed by a Danfysik ESA, and finally mass analyzed by an Enge split pole magnetic spectrograph. All focusing elements in the system are electrostatic so that parallel mass transport can be provided. The only components that disperse masses are the Pretzel magnet, which then recombines only the masses chosen anywhere in the range between H and U, and finally the spectrograph, where mass analysis is performed.

The desired modified SIMS will be installed at the second input to the low-energy NEC switching ESA, with all subsequent components of the TEAMS system shared between the two different ion sources. Since the modified SIMS will generate much lower energy ions than currently available from the MC-SNICS ion source, some modifications to the NRL TEAMS ion optics will be required. When these modifications are completed, NRL expects that the NRL TEAMS system will have an ion optical acceptance of 1-3 π mm mrad $MeV^{1/2}$. The physical layout of the area in the vicinity of the low energy switching ESA is shown in Figure 1. Quantitative details of the ESA are listed in Table I. The modified SIMS instrument and associated coupling components will need to mate with this existing equipment, both physically and with matching ion optics. NRL will provide a gate valve, bellows, and coupling chamber between the modified SIMS and the NRL low energy switching ESA. The offeror must allow space for these components to be inserted between the ESA entrance flange and the modified SIMS exit flange.

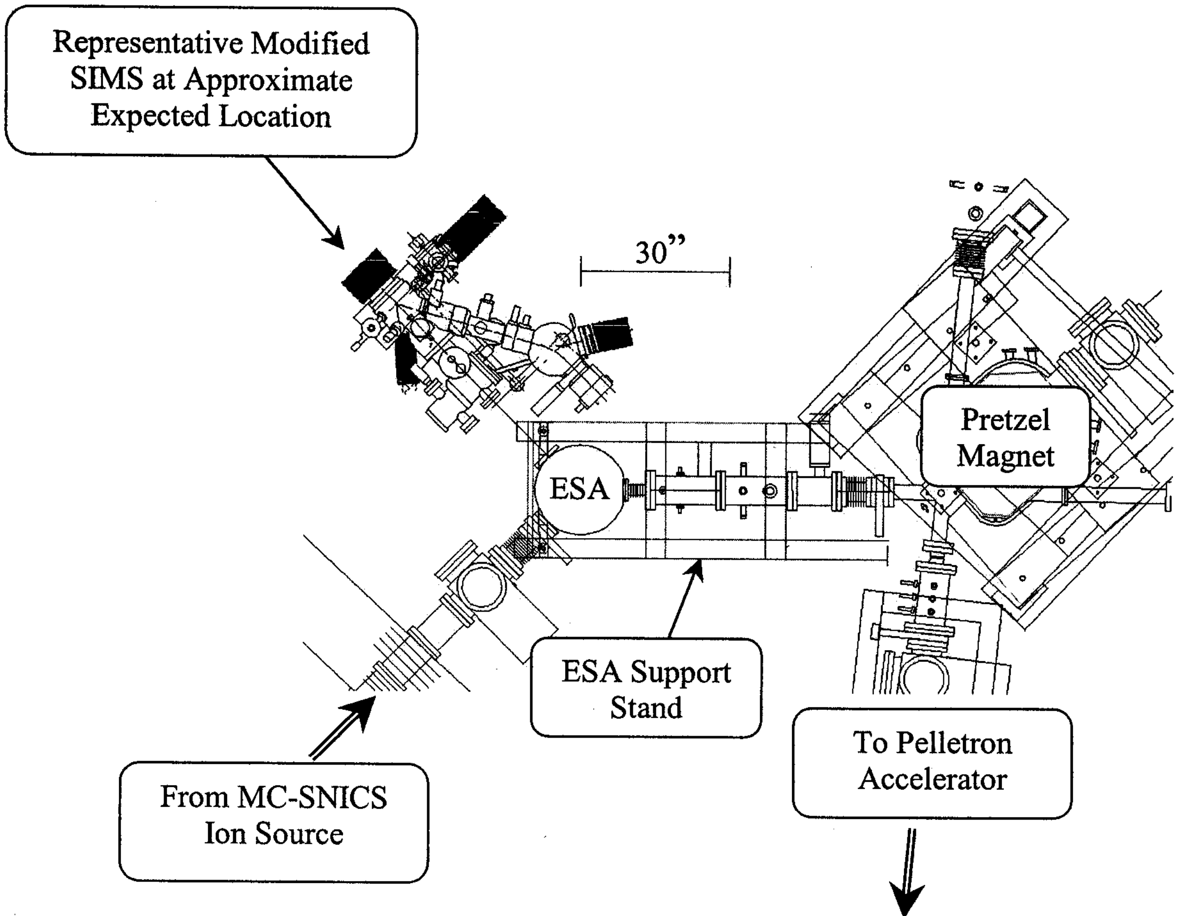

Figure 1. Layout of low-energy components of NRL TEAMS system. Intended location of modified SIMS is shown in diagram.

Table I. Specifications for switching ESA to be connected to modified SIMS

Component	Characteristic	Specification
Spherical Electrodes	Deflection	± 45°
	Radius (Central Beam Axis)	300 mm
	Plate separation	50 mm
Entrance and Exit Apertures	Size	1" diameter
High Voltage Power Supplies	Positive Supply	+15 kV, 1 mA
	Negative Supply	-15 kV, 1 mA
Vacuum Chamber Housing	Ion beam entrance port	6" OD ConFlat flange

The desired modified SIMS system is similar to a commercially available magnetic sector based SIMS, without the final electrostatic and magnetic analysis components, projector lens, or standard secondary ion detectors. The function of these components will be provided instead by the existing NRL TEAMS system. The instrument offered must not introduce mass dispersion of the secondary ions to be provided for injection into the NRL TEAMS system, since the TEAMS system is designed to provide parallel mass analysis. This instrument shall include a high brightness cesium microbeam source, a primary beam mass filter, a charge autocompensation electron gun, software support, and preliminary acceptance testing. Option items include a high resolution charge coupled device (CCD), Z-axis manual sample-position movement, an oxygen flooding attachment, a closed loop water cooled chiller, and a 3 phase line conditioner and step up transformer.

The modified SIMS must meet or exceed the minimum specifications described below:

General Specifications of Modified SIMS

The modified SIMS must:

1) Provide a mass-filtered high-brightness Cesium (Cs) primary ion beam, able to sputter a sample in a raster pattern to produce a flat-bottom sputter crater.

2) Enable routine operation of the modified SIMS so only atoms and molecules sputtered from the flat bottom of the crater can be measured by the existing NRL TEAMS system.

3) Provide high spatial resolution, both in depth and laterally. Specifically, it must be able to provide 50 nm full-width-at-half-maximum depth resolution using 10-keV secondary ion energy, and 0.3 μm lateral resolution, consistent with generation of 0.3-μm-diameter primary ion beam probe size.

4) Provide high efficiency extraction of secondary ions as described in section d below, and generate up to 10-keV negative secondary ions while maintaining the modified SIMS vacuum chamber at ground potential.

5) Provide a horizontal non-mass-dispersed secondary ion beam, and be emittance matched not to exceed the ion optical acceptance of the NRL TEAMS system, physically matched to existing hardware of the NRL TEAMS system, and use a dynamic transfer lens to maximize transmission of secondary ions. Details are provided in Fig. 1 and sections e through g below.

6) Provide a bakeable ultra high vacuum environment with a rapid sample load lock system, sample motion for analysis of various regions of a sample, and a magnified sample viewing capability. For details, see section b below.

7) Provide a means to automatically compensate for charge buildup on an insulating sample using an electron source,

8) Provide capability to field install a second mass-filtered high-brightness primary ion source of O, N, or Ar ions that is commercially available.

9) Provide an automation system, including hardware and software with a complete command set to operate the modified SIMS instrument and which permits external LabVIEW software to be co-hosted on the instrument's automation system.

Detailed Specifications of Modified SIMS

a) Primary Ion Column

Ion-optical column consists of lenses, stigmator, double deflector for primary beam rastering and Faraday cup for primary ion current measurement. Faraday cup must be capable of measuring current of smallest diameter primary ion beam offered.

Ion source:
Surface ionization, cesium microbeam source, with Einzel lens system.

Accelerating voltage:
Cs^+: 2 to 13 kV, continuously adjustable for any ion source

Beam diameter on sample, operator controlled within range of:
Cs^+: ≤0.30 µm to 200 µm (source HV = 10 kV, sample HV = - 10 kV)

Beam current and density, at sample position, operator controlled from zero up to maximum:
Cs^+ (source HV = 10 kV): maximum primary beam intensity > 0.6 µA
For a beam of 20 µm diameter the beam density is:

Primary ions	Beam density
Cs^+ (source HV = 10 kV, sample HV = - 10 kV)	> 50 mA/cm^2

Beam current stability:
For 10 kV Cs^+: $\delta I/I < 1$ % over 10 min

Lateral beam drift:
< 100 nm/10 min for Cs^+

b) UHV Specimen Chamber Including

Fast entry load lock

Fast pumping of the load lock with turbomolecular pump, so that empty load lock chamber can be pumped to 5 x 10^{-9} Torr within 10 hours.

Allows storage of two sample holders for changing holders without an additional pump down

Heating facility for sample degassing.

Sample stage

Accepts samples up to maximum size:

1 inch in diameter

1/2 inch in thickness

X, Y movements

By stepping motors controlled by vendor supplied automation system

Resolution of 1 µm, reproducibility ± 5 µm.

Movement: 20 mm in X and Y directions.

Optical microscope

Magnification: 70 X

Resolution: 3 µm

Field of view: 2 mm

UHV baking capability

c) Secondary Ion Optical System

Immersion and transfer optical system operates in both ion microscope and microprobe modes:

In the microscope mode

Field of view continuously adjustable from 35 µm to 250 µm.

Image magnification: on the 26 mm image detector, the sample area must be adjustable from 250 to 35 µm via the combination of the transfer lenses and the projection lenses.

In the microprobe mode

Maximum field of view: 400 µm x 400 µm

Includes a manual slit with continuous width adjustment of 0–400 μm

Includes a four position opening aperture holder with manual X and Y centering capability

d) Secondary extraction

The system shall be delivered with a secondary extraction system ensuring collection efficiency better than 70% while working at an extraction potential of 10 kV (positive or negative). The extraction voltage shall be continuously adjustable over a range of +/-10 kV. Low extraction voltages will be accessed when low primary impact energies are required.

e) Dynamic transfer optics

For optimized sensitivity, the secondary beam emittance must be matched to not exceed the acceptance of the mass analyzer. The existing NRL TEAMS system has an acceptance between 1 and 3 π mm mrad $MeV^{1/2}$, depending on mode of operation. The modified SIMS instrument shall be equipped with a set of transfer lenses for shaping the secondary beam in both the entrance slit and the opening aperture. This shaping is required to optimize the overall transmission according to the primary beam size. The dynamic transfer system must allow operation at constant transmission while rastering a given primary beam up to 500 x 500 μm.

f) Secondary beam transport

The modified SIMS instrument must be mounted on a support frame and oriented to produce a horizontal secondary ion beam. It must not produce mass dispersion of the secondary ion beam, must provide a beam emittance matched not to exceed the ion optical acceptance of the NRL TEAMS system, and generate an image of the sample at an appropriate location for injection into the TEAMS system. The modified SIMS instrument and associated support frame must not interfere with the existing NRL TEAMS system, including any existing support structures shown in Fig. 1, and must enable secondary ion injection at the beam axis height of the NRL TEAMS system. This means the secondary beam must be provided at a beam height $\leq$1470 mm above the floor. A gating signal must be provided to enable selective measurement of secondary ions only from the flat bottom of the sputter crater.

g) NRL Electrostatic Analyzer (ESA) compatibility

The modified SIMS instrument must be optically and mechanically compatible with the NRL electrostatic analyzer (ESA).

Optically, the modified SIMS must produce an image of the sample at the object position required for the NRL ESA, located 74-84 cm from the NRL ESA flange. NRL will supply the coupling chamber, which will have an enlarged field or opening aperture. The instrument provided must contain all other needed beam apertures and slits.

Mechanically, the modified SIMS must allow space between the end of the modified SIMS and the entrance to the NRL ESA for NRL to install the bellows, gate valve, and coupling chamber that NRL will provide. These components will need at least 360 mm of space.

h) Micro channel plate (MCP) / fluorescent screen assembly

The offeror must provide a micro channel plate (MCP) / fluorescent screen assembly. This assembly is to be located after an opening aperture at the exit of the modified SIMS. This assembly will be used for viewing the stigmatic total ion image of the sputtered area during the final stage of acceptance testing. This assembly must be removed after instrument acceptance testing to join the instrument to the NRL TEAMS facility, but it will remain the property of NRL. This assembly must provide lateral resolution better than 10 µm. The offeror must include the cost of this assembly in the cost of the modified SIMS instrument.

i) UHV Vacuum System

In the sample chamber with the primary ion source off $\leq 5\times10^{-10}$ Torr

In the sample chamber with the cesium source operating $\leq 7\times10^{-10}$ Torr

Vacuum conditions in remainder of system must be sufficient to meet stated performance specifications. Vacuum valves and gauges required to operate modified SIMS must be supplied with the instrument.

System must include:

Turbomolecular pump for primary column.

Turbomolecular pump with titanium sublimator for specimen chamber.

Anticontamination cold plate surrounding the specimen.

Fail safe fully interlocked microprocessor vacuum control and display.

UHV baking system with temperature sensors and timer.

Oil-free mechanical pump for backing the turbo pumps

j) Electronics

Electronic supplies and control chassis will be provided for the following units:

Cesium microbeam source

Accelerating voltage 1 – 12 kV

Primary beam mass filter

All lenses and deflectors of the primary and secondary ion columns.

Digital primary beam rastering system.

Includes beam blanking.

Dynamical transfer optical system.

Charge compensation electron gun with perpendicular incidence on sample.

k) Automation system

An automation system with computer controlled electronics and a data system will be provided. Instrument set-up parameters are to be stored and recalled by the computer. Hardware shall include:

Microcontroller units dedicated to digital control of the instrument settings.

Microprocessor for control of the vacuum system.

Workstation with at least the following minimum capabilities:

500 MHz processor clock rate

512 KB cache

512 MB RAM memory

40 GB hard disk drive

DVD ROM drive

SmartCard driver

ZIP drive 750 MB

Video board

Serial/parallel multiplexer

18" high resolution (1280 x 1024) LCD color monitor

Inkjet Color Printer

Ethernet interface

PCI interface

Dedicated color 18" high resolution (1280 x 1024) LCD monitor and keyboard for instrument control

Supplied hardware and software must be capable of operating the modified SIMS instrument including at a minimum, the ability to perform the following functions:

Save and restore the instrument set up files containing the setting of the parameters controlled by an instrument keyboard

Tuning for adjusting the primary ion current reading

Start & Stop the Cs source

Vacuum for displaying and controlling all vacuum and baking systems

Holder for controlling the sample stage and displaying the sample holders

Specifications for Advanced High-Brightness Micro-Beam Cesium Source

The modified SIMS instrument shall have a high brightness cesium source, providing a Cs^+ beam of more than 50 mA/cm^2 density, with a beam spot size over the range: 10–50 µm diameter, and a maximum primary ion beam current of at least 600 nA. It must be mounted on a 3-stage primary column and be possible to form a Cs probe as small as 0.3 µm at the sample surface. The Cs ion source shall be charged with a solid, safe-to-handle Cs compound, such as Cs-carbonate.

Specifications for Primary Beam Mass Filter (PBMF)

The modified SIMS instrument shall have high performance mass filtering of the primary ion beam. The mass resolution $M/\Delta M$ shall be adjustable up to 60. This means that the full width at half maximum (in mass) of a single mass peak must be less than $1/60^{th}$ of the mass of the peak itself. During operation, the primary beam mass filter must provide mass separation of alkalis such as Na, K, and Rb from Cs, and remove any neutral beam component, thus producing a clean Cs beam at the sample surface. The PBMF shall be designed to accommodate a commercially available ion source, such as a Duoplasmatron, that can be installed in the field. The PBMF must provide high performance mass filtering on that source as well.

Specifications for Charge Compensation Device Electron Gun

The modified SIMS instrument shall be equipped with an electron gun capable of directing electrons to have perpendicular incidence on the sample of interest. The design shall focus the electron beam at the sample surface through the same lens used to collect the secondary ions. This design must enable charge compensation with very low energy electrons (<10 eV) and must not cause damage in the sample. For ease of operation, there must be a self-adjustment mode that automatically controls the charge compensation to maintain a constant surface potential on the sample, without regard for primary beam intensity and the nature of the sample. Detailed requirements are listed below.

Electron beam intensity

- Greater than or equal to 80 µA

Electron beam size

- Up to 250 µm for positive secondary ions
- Up to 100 µm for negative secondary ions

Secondary ion intensity stability with charge neutralization

- $\delta I/I \leq 10$ % for negative secondary polarities (sample HV = -10kV). This will be demonstrated before splitting the instrument into two parts.
- Measured with a 1 µm layer of SiO_2 on Silicon

Documentation, Software Support, and Warranty

A) A full set of all written documentation customarily provided to the public with a commercial item shall be provided. This shall include users manual(s) or equivalent as well as copies of any software, and any manuals for the software included with the system, if customarily provided. This documentation must be received at NRL with the system hardware, unless specifically stated otherwise below, or unless the authorized Government representative agrees to other arrangements. In addition to the above documentation which is customarily provided, the following specific documentation is required, and shall be included in the price of the Modified SIMS instrument:

1) Documentation of interface commands (including vacuum securities) and control voltages for all power supplies
2) Diagram relating electronics to the ion optics, showing interconnections and functional relationships
3) Interconnecting diagrams of assemblies and sub-assemblies (inter- and intra-chassis)
4) Programmable array logic (PAL) equations
5) Information on the PAL development system, sufficient to allow NRL to alter the operation of modified SIMS
6) Mechanical drawings for delivered instrument, including component parts
7) List of Al-seals and other replacement parts (ceramics, etc.), with diagrams or explanatory text relating each part to its physical location in the instrument
8) Information needed to prepare for installation and testing of delivered instrument. This includes instrument and support frame drawings with dimensions, and requirements for utility services, utility connections, space, and environmental controls. NRL also needs sufficient information to construct a coupling vacuum chamber to house an enlarged field aperture and an MCP imaging device. Critical dimensions and requirements for the vacuum chamber, its flanges and feedthroughs, the field aperture, and an MCP device must be provided. All this installation and testing information is to be provided to NRL within 60 days of contract award. NRL will notify the successful offeror of its ability to meet the specifications required for installation within 90 days of receipt of the information required by this paragraph.

Within these specified documents, the successful offeror shall provide sufficient information to permit external LabVIEW software to be co-hosted on vendor's automation system and to issue required commands to operate the modified SIMS instrument. Specifically, the vendor must provide the information necessary for NRL to initialize, tune, control, monitor, shutdown, secure, and service the partial SIMS instrument's primary and secondary columns, and associated vacuum system, electronic, and power components. The provided information must enable normal operational capabilities such as switching between and using different modes of operation, for example, dynamic transfer mode, raster mode, and imaging mode. Other examples include the use of charge compensation for insulating samples, motion of the sample to different viewing areas, gating the secondary ion signal to identify when it originated from the central region of the sputter crater, and a means to monitor pressure and valve conditions for the vacuum system.

As a guide to the some of the information that will be needed, if a control keyboard using serial I/O controls instrument functions using commands, then for each of these commands NRL needs addresses for all components, needs to understand the packing structure and format for the commands, and needs to understand details of any acknowledge responses. For commands operating through a server, NRL needs to know the server protocol. For server commands, NRL needs to know how to establish client and server communications and how to share information between them, including obtaining access to any computer source code required to communicate with the server. NRL also requires detailed knowledge of the command and acknowledge string structure, including syntax and content. This includes guidance to their generation and a map of locations.

B) The successful offeror shall provide at least 24 hours of software support to assist in implementation of NRL-supplied LabVIEW software to control the modified SIMS instrument at no additional cost. The support includes telephone, email, and fax communications, and associated off-line research and related activities by the offeror. The support must be available over a period of 15 months from instrument acceptance, up to a maximum of 8 hours in a given week, and during business hours of the offeror. Additional support beyond the 24 hours shall be offered at hourly and daily rates, over the same 15-month period and under the same weekly time limit and business hours limit.

C) The contractor shall offer the Government at least the same warranty terms, including offers of extended warranties, offered to the general public in customary commercial practice. These warranty terms must be included in the price of the instrument. The period of the warranty shall begin upon acceptance.

Specifications for Installation, Acceptance Testing and Training

NRL requires preliminary acceptance testing at the factory with a complete SIMS instrument using the standard protocols of the manufacturer. This testing (Part A) includes use of electrostatic analyzer (ESA), mass spectrometer, and secondary ion detectors normally employed with a complete SIMS. All specifications stipulated above must be demonstrated to the satisfaction of a representative from NRL, either within a period of one week during a site visit to the factory, or subsequently by other means agreeable to NRL.

Subsequently, the front-end portion that is to be delivered to NRL (the modified SIMS) is to be separated from the complete SIMS instrument and subjected to additional acceptance tests (Part B) at the factory to confirm acceptable operation after separation. These tests are performed without the ESA, mass spectrometer, and normal secondary ion detectors used with a complete SIMS. Secondary ions are detected instead by an MCP / fluorescent screen assembly mounted at the location of their exit from the modified SIMS. The image detector allows the formation of a total ion image (non-mass filtered) with a lateral resolution better than 10 µm. This detector will be used to evaluate the beam density of the Cs beam for a medium primary beam size (25µm) and the electron beam shape and centering.

During Part B, for a Cs^+ beam of 25-µm-diameter, the beam intensity must be at least 200 nA. It corresponds to a beam density of 40 mA/cm^2 (instead of 50 mA/cm^2 as stipulated for a complete SIMS) to take into account the approximate method used to evaluate the beam diameter. The beam diameter is evaluated from the total direct image (using a contrast aperture of 50µm) based on the contrast given by the mesh (25µm) of a Cu grid deposited on an Al test sample. Testing must demonstrate the instrument can meet the listed specifications for beam current stability for a 10 kV Cs^+ beam of <1% variation over 10 min and a maximum 10 kV Cs^+ beam current > 600 nA.

Also during Part B, the charge compensating electron gun must be demonstrated to meet the specifications for electron beam intensity ≥80 µA, and the electron beam size of up to 100 µm for negative secondary ions, centered into the field aperture. The electron beam diameter is to be evaluated from the total secondary ion direct image induced by the electron beam @ 20 eV energy. Observed with a 400-µm-diameter contrast aperture, the total ion image must cover 4 meshes of a Cu grid deposited on an Al test sample, corresponding to about 100 µm in diameter.

Routine procedures and specifications must also be demonstrated under Part B, such as attainment of the stipulated vacuum conditions, complete operation of all supplied mechanical, optical, and electronic components, and robust operation of all supplied computer hardware, firmware, and software. These tests are also to be completed to the satisfaction of a representative from NRL during the one-week-long site visit, or by other means agreeable to NRL.

Once all these tests are satisfied, the modified SIMS instrument will be delivered to NRL. Upon arrival at NRL, the modified SIMS instrument will be installed and subjected once again to the Part B acceptance test, to confirm the instrument was not damaged during shipment or installation. Only after all tests described for Part B have been satisfied, can final acceptance of the modified SIMS occur. The offeror's expenses to perform all acceptance tests and the installation at NRL shall be included in the offeror's proposal. The installation will be performed with the assistance of an engineer from the offeror. The installation service shall include any required supplies such as bottled gases. The offeror shall also provide to NRL one set of spare parts for the first year of operation, and a maintenance kit with tools. The installer will complete all required mechanical assemblies and all connections to utilities such air, water, and electricity. The offeror will provide all installation requirements (e.g., utilities, space, and environmental conditions) as described in the documentation specification section. NRL shall be responsible for meeting these requirements, by the delivery date projected at the time of award.

The offeror must provide operator training by an instrument engineer for up to three users at NRL, Washington DC, for a minimum of five days during the period of instrument installation and acceptance testing. The offeror shall also provide additional training utilizing the complete SIMS instrument for a minimum of five days for up to two people. This training may be conducted at the contractor's facility.

Specifications for Optional Items

High Resolution CCD Camera

The successful offeror shall provide a high resolution (at least 420 horizontal lines) color CCD camera with zoom optical system for sample viewing, providing a variable field of view from 0.56 mm to 1.7 mm in diameter.

Z Axis Manual Movement

The successful offeror shall provide an option for Z-axis manual movement of sample with a range of 3 mm, reproducibility of 5 μm, and position reading accuracy of 1 μm.

Oxygen Flooding Attachment

The successful offeror shall provide an oxygen flooding attachment for controlled ultra high vacuum leak of oxygen to enhance sensitivity for certain elements.

Chiller

The successful offeror shall provide a closed loop water-cooled chiller for use with the primary beam mass filter and other modified SIMS components.

Step up Transformer and Line Conditioner

The successful offeror shall provide a step up transformer and 3-phase line conditioner, 15kVA