

## ATTACHMENT NO. 1

### Technical Specifications

#### “Acquisition of a Atomic-Resolution Transmission Electron Microscope (TEM) for Cryo and In-Situ Microscopy at National Renewable Energy Laboratory (NREL)”

January 19, 2023

##### List of Acronyms:

BE – Back Scattered Electron

BF – Bright-Field

CMOS – Complementary Metal-Oxide-Semiconductor

Cold-FEG – Cold field-emission electron source

DF – Dark-Field

EDS – Energy Dispersive (X-ray) Spectroscopy

EELS – Electron Energy-Loss Spectroscopy

EF TEM – Energy Filtered Transmission Electron Microscopy

HAADF – High Angle Annular Dark Field

SDD – Si drift detector

STEM – Scanning Transmission Electron Microscope (or Microscopy)

TEM – Transmission Electron Microscope (or Microscopy)

##### OBJECTIVE:

The National Renewable Energy Laboratory (NREL) in Golden, CO seeks a readily available and demonstrable TEM/STEM instrument and related accessories. The following lists the required items that describe properties of the instrument and requirements of the vendors to meet our needs (all are **required** except where noted as **‘desired’**) and the instrument criteria which must be satisfied or demonstrated to NREL during the acquisition cycle and at certification/acceptance:

##### **I. Required items:**

1. Delivery and certification schedule
  - a. Must be delivered by March 22<sup>nd</sup>, 2024
  - b. Must be installed by the TEM vendor by June 21<sup>st</sup>, 2024
  - c. Must be certified by August 1<sup>st</sup>, 2024 (see the **certification** criteria III)
2. Combined TEM and probe-corrected STEM instrument
  - a. Operation at accelerating voltage of 200 kV
  - b. Operation at accelerating voltage of 80 kV
  - c. Factory alignments at 80 kV and 200 kV
  - d. Fully dry-pumped vacuum system (No oil-diffusion pump, etc.)
  - e. Instrument power requirements (Required voltage, frequency, phases, current, and vendor-provided transformer)
  - f. Compressor 115V, 50/60 Hz
  - g. Water cooling unit (water)
    - i. 0.1 degree C per min
  - h. Uninterruptable power system
  - i. Cold-FEG
    - i. Source brightness at 200 kV  $> 5 \times 10^8$  A/cm<sup>2</sup>/Sr, coherent current over 500 pA
    - ii. Energy spread/resolution  $< 0.4$  eV at 200 kV
    - iii. Frequency of flashing electron source less than once per 3 hours to maintain probe stability

- j. Aberration corrector on the probe-forming lenses at 200kV
  - i. 200kV and 80 kV column and corrector alignments included
  - ii. Auto tuning of aberration corrector software integrated into microscope operation software
  - iii. Wide gap polepiece of > 5 mm for objective lens
  - iv. STEM imaging resolution of 0.9 Å or better at 200 kV, 1.4 Å or better at 80 kV with > 80 pA beam current at both conditions
  - v. TEM point-to-point resolution 2.8 Å or better at 200 kV
  - vi. Magnification calibration package, using cross grating sample and oriented gold sample
  - vii. System stabilization time of less than 30 minutes for:
    - 1. Switching between the high- and low-operating voltages and alternatively low- to high-operating voltages
    - 2. Switching from TEM to STEM mode
    - 3. Switching between on and off the aberration corrector lenses
    - 4. After flashing of tip
- k. Automatic aperture control system
- l. TEM scripting
  - i. Ability to control beam settings using Python coding
- m. Piezo-stage
  - i. Step sizes down to 100 pm, 1 µm range in X and Y
- n. Included specimen holders
  - i. Low-background double tilt holder for EDS mapping
  - ii. Single-tilt Cryo-transfer TEM sample holder with turbo pumping station
- 3. In-Situ capabilities
  - a. Hardware and software control for pre-specimen fast beam blanking for controlling dose on the specimen
  - b. Microscope compatible with photoexcitation source, to simulate broadband light on the specimen other than through an TEM holder
  - c. CMOS camera with 4k x 4k pixels, with acquisition speeds better than 20 frames per second for 80 - 200 kV operation
- 4. Analytical accessories
  - a. STEM detectors
    - i. DF detector
    - ii. HAADF
    - iii. BF detector
    - iv. Segmented STEM detector for real time analysis of magnetic and electric fields, ADF
  - b. At least one > 100 mm<sup>2</sup> area windowless SDD EDS detector system, minimum solid angle of 0.7 sr, energy resolution 133 eV or better (at Fe K) or 136 eV or better at (Mn K), detection down to Be
- 5. Other requirements
  - a. Warranty term of at least 1 year with parts and labor included, with a quote provided for yearly service contract for subsequent 3 years after warranty term
  - b. Provide detailed operational manuals
  - c. User training of 5 days included with purchase to cover detailed operation of the microscope and all of its operational modes and accessories
  - d. Specify work breakdown details for meeting above requirements and delivery dates

- e. Needs to have NRTL electrical safety certification on base instruments and accessories, or needs to be certified with third party testing facility prior to delivery
- f. Transportation of the instrument will require all units as delivered to be under 5,000 lbs for use of building's freight elevator below its weight capacity
- g. No more than 5.5 L of liquid nitrogen can be required for tool operation in the defined lab space, confirm microscope/accessories can comply with this requirement
- h. A provided photoexcitation source must pass specifications to operate as a Class 1 laser
- i. A site survey of the room requirements will be required upon order

## **II. Desired items** in rank-order of importance

- a. Imaging filter for EELS and energy-filtered TEM hardware and software with energy resolution  $< 0.4$  eV with  $> 80$  pA beam current
- b. Factory alignment and operation at 300 kV accelerating voltage, with  $0.8 \text{ \AA}$  or better spatial resolution
- c. Specimen photoexcitation system comprising of modulated laser, optical delivery system, inlet port and mirror for in-situ optical excitation experiments or similar capabilities
- d. Direct Electron Camera for 200 kV for low dose imaging
  - 1.  $4k \times 4k : 1 \text{ fps}$
- e. Retractable Cryo-box or fixed non-side entry cooling that does not interfere/shadow the EDS detectors (when retracted or as fixed)
- f. A second  $> 100 \text{ mm}^2$  area windowless SDD EDS detector system, minimum solid angle of  $0.7 \text{ sr}$ , energy resolution  $133 \text{ eV}$  or better (at Fe K) or  $136 \text{ eV}$  or better at (Mn K), detection down to Be
- g. Subframing system to improve frame rates on CMOS camera
- h. BE detector or low-magnification STEM mode to see sample on mm length scale
- i. Preference for full integration of accessories with the microscope control for automation and scripting purposes
- j. Remote microscope operation from adjacent room or farther and appropriate digital camera for such operation

### III. Certification criteria

1. Instrumentation specifications that can be defined by measurable parameters in the required item I must be measured at the time of certification/acceptance with NREL staff present by using the methods specified below:
  - a. Energy-spread of the electron source of  $< 0.4$  eV at 200 kV – measurement of EELS zero-loss peak under gun operational parameters used for analytical work at 10 milliseconds and 1 second integration times for  $> 80$  pA beam current
  - b. TEM point-to-point resolution of  $2.4$  Å or better at 200 kV – using standard Young fringe pattern method and a suitable vendor provided specimen
  - c. STEM HAADF resolution of  $< 0.9$  Å at 200 kV using  $<211>$  GaN standard sample, and  $< 1.4$  Å at 80 kV both at  $>80$  pA beam current
  - d. Demonstration of atomic resolution ( $< 1.4$  Å) STEM EDS and EELS spectrum images, e.g., on  $<001>$  STO or  $<211>$  GaN standard samples
  - e. All detectors/cameras operation to values provided in quote document
  - f. Demonstrated nanosecond beam blanking for dose control on beam sensitive specimens, perovskites will be used for damage measurement over a 10s period
  - g. Demonstrated stage and imaging stability over a 30 min period
  - h. X-ray measurement to ensure local safety requirements are met,  $< 1$   $\mu$ S/hr at 10 cm from surfaces
2. Instrument specifications that can't be defined by the measurable parameters such as: integrated computer control; corrector alignment; and overall system integration must be demonstrated to the National Renewable Energy Laboratory, in person, on site at the time of certification.