

Technical specification for Procurement of Atomic Force Microscope with piezoforce and electrochemical imaging capability.

IIT-Madras is looking for Atomic Force Microscope with piezoforce and electrochemical imaging option for research on materials and electrochemistry. The following technical specifications should be met, to qualify the technical bid. A detailed compliance statement should be provided and it should be supported by the manufacturer's product brochure or website. Information provided in the supplier/redistributor/vendor/reseller's brochure or website cannot be considered as supporting document. Technical bids without proper compliance statement will not be considered. The bidder should be an Original Equipment Manufacturer or their authorized vendors/dealers/partners. If vendors/dealers/partners are participating in the bidding, an authorization certificate should be provided.

1. Modes of operation :

- a. The system should have the following imaging modes inbuilt with the system: Contact Mode, Force Curve Mode, Tapping mode (AC), Electric Force Microscopy(EFM), Lateral Force Microscope(LFM), Force Mapping Mode, Force Modulation, Frequency Modulation, Kelvin Probe Force Microscopy (KPFM), Phase Imaging, Piezoresponse Force Microscopy (PFM), Switching Spectroscopy PFM, Vector PFM and Electrochemical AFM.
- b. The AFM system must be able to image samples and perform measurements in air and in liquid and for all modes including EFM, PFM & KPFM using the same cantilever holder.
- c. The cantilever holder must be compatible with most commercial cantilevers.
- d. Nanoindentation measurements should be possible.

2. Scanner requirements:

- a. Single scanner for low- and high-resolution imaging
- b. System must automatically scan the sample in XY and the tip automatically in Z with appropriate computer control
- c. The XY & Z scanner should be decoupled.
- d. Each axis of motion is independently actuated using its own piezo stack and flexure stage. Piezo tube type scanner design will not be acceptable.
- e. Should have integrated LVDT/ equivalent position sensors in all three axes provide seamless closed loop operation.
- f. System must include a closed-loop XY scanner with a minimum range of 120 μm (closed loop) and with XY sensor noise $<0.6\text{nm } A_{\text{dev}} / \text{RMS}$ in a 0.1 Hz to 1 kHz bandwidth
- g. Scanner noise specifications and representative high resolution imaging examples must be available for inspection in publicly available brochures, datasheets or websites. The scanner must be compatible with all supplied scan modes and in both air and liquid environments. Closed loop sensor noise numbers should be demonstrated during installation at site.
- h. System must include a Z scanner with a minimum range of 15 μm that is capable of both open-loop and closed-loop operation. Noise on the Z sensor must be $<0.25\text{nm } A_{\text{dev}}$ in a 0.1Hz to 1 kHz bandwidth .
- i. The system should have Z height noise of $<0.01 - 0.02\text{nm}$ (off-surface – vertical noise floor – $A_{\text{dev}} / \text{RMS}$ in a 0.1Hz to 1kHz bandwidth)
- j. Scanner noise specifications and representative high resolution imaging examples must be available for inspection in publicly available brochures, datasheets or websites. The scanner must be compatible with all supplied scan modes and in both air and liquid environments. Scan head of higher Z range, 30 μm or more must be quoted as an optional item. Closed loop sensor Noise numbers should be mandatorily demonstrated during installation at site.

3. Details of piezoforce imaging:

- a. Our research requires piezoforce imaging option to visualize the ferroelectric domain walls in halide perovskite or oxide perovskite materials. IIT-Madras has a PE loop tracer that can polarize pellets/films of these perovskite materials upto 1000/10000V. On average, the voltage requires to polarize our samples lie between 200V and 300V (in both positive and negative bias conditions). So to supplement out existing PE loop tracer, we require a PFM imaging mode that can polarize sample at least at 200V. Even if the base PFM is not capable of switching domains at 200V, the upgradation option should be available to polarize sample at least at 200 V.
- b. The system must carrying out the following imaging modes; Vertical PFM (out-of-plane polarization), Lateral PFM (in-plane polarization), Vector PFM (real space reconstruction of the polarization orientation) and lithography for modification of the piezoelectric polarization.
- c. The system must be capable of simultaneous remnant and applied voltage hysteresis loops on ferroelectric materials and switching spectroscopy mapping while measuring contact resonance frequency, PFM drive amplitude, PFM phase and tip-sample dissipation/Q-factor.
- d. The system must be capable of pulse-relaxation measurements while measuring contact resonance frequency, PFM drive amplitude, PFM phase and tip-sample dissipation/Qfactor.
- e. The instrument must exhibit extremely low crosstalk between the tip-drive voltage and the measured deflection. The measure of crosstalk is defined as follows:
 - i. Position the tip >1cm from the sample surface.
 - ii. Apply the bias voltage to the tip.
 - iii. with a tip bias voltage drive amplitude of 100V, sweep the drive frequency from 50kHz to 2MHz.
 - iv. The sum signal should be at least 7 volts and the deflection zeroed to provide the highest measurement sensitivity.
 - v. The measured amplitude at all frequencies should be less than 300 microvolts.
- f. A dedicated high voltage for +/- 200V (or better) module for tip or sample bias is required to enable measurements on materials with weak piezoelectric response. The high voltage module must provide necessary safety features for safe and easy operation. If the high voltage feature is an add-on to the base machine, quote this add-on accessory in the optional component.
- g. All measurement modes should include the ability to track the resonant frequency using signal enhancing contact resonance, which eliminates topography crosstalk and other artifacts arising from contact resonance frequency shifts. The PFM amplitude, phase, contact resonant frequency and contact resonance quality factor should be measured and returned.
- h. The system control must allow the user to route the AC bias either to the tip or the sample through software control.
 - i. The ability to perform PFM and Conductive AFM simultaneously is required.
 - j. The PFM control software must include a variety of PFM spectroscopy modes in various complexity levels to perform user defined bias curves.
- k. The software must include sufficient analysis routines within the data analysis environment to eliminate the need to export, re-import into third party software, or interpret the exported data scales.

4. Requirements on electrochemical imaging:

- a. The proposed machine should have capability to do surface AFM imaging of catalysts/photoabsorber materials in the film or bulk form during the electrochemical reaction.
- b. The microscope should be capable of imaging the surface while independent electrochemical characterization processes such as cyclic voltammetry, linear sweep voltammetry, chronoamperometry, and chronopotentiometry are being carried out.

- c. Electrochemical cell assembly should consist of a PEEK fluid cell assembly or better with working and reference electrodes along with an all PEEK cantilever holder. Should also include accessories like Electrical junction box with connections and programmable jumpers to interface with one of the potentiostats listed below, Carbon (graphite) counter electrode, A working electrode sample mount suitable for both conducting and insulating sample substrates. Spare O-rings and assembly hardware and a sealed Ag-AgCl reference electrode. Add the electrochemical accessory in the mandatory component.
- d. The electrochemical accessory quoted with AFM should be compatible at least with one of the following electrochemical workstations
 - i. SP-300 Biologic Electrochemical Workstation (Manufacturer: Biologic, France)
 - ii. Autolab PGSTAT128N Electrochemical Workstation (Manufacturer: Metrohm, Netherlands)
 - iii. Versastat 3 Electrochemical Workstation (Manufacturer: AMETEK, USA)

5. Controller requirements

- a. System must use at least 24-bit digital-to-analog converters (DACs) in order to generate the XY and Z piezo scan signals. At both 100-micron and 10-nm scan sizes, the corresponding bit resolution must be sub-Angstrom (<0.1nm). Note that this specification applies to the generation of the scanner drive signals, not the sampling of the scanner position sensors.
 - b. The system must provide thermal tunes of the cantilever up to at least 2 MHz.
 - c. The instrument must allow digital Q-control in the range 2 kHz – 2 MHz.
 - d. The instrument must include software controlled relays for the X, Y and Z high voltage supplies and the laser power.
 - e. The electronics must provide access to all major signals on BNC connectors on the controller front panel including deflection (A-B), sum (A+B), amplitude, phase, lateral force, X, Y and Z sensors, three user inputs, three user outputs, X,Y and Z piezo drive voltages, and user X, Y and Z modulation voltage inputs compatible with external hardware.
 - f. The instrument must include auto-configuration of external hardware and accessories. Device parameters must be stored in non-volatile RAM on the device itself and read into the software when the device is plugged in. This eliminates the need for parameter files.
 - g. The instrument must include a user programmable control knob that can be used to fine tune and adjust all scan parameters.
6. The AFM should be capable of characterizing opaque, transparent, insulating, and conducting samples. The instrument must accommodate samples sizes up to 80mm (diameter) and 10mm thick. There should be an option for samples of 25mm thick and above.
 7. A general purpose camera must be provided with the microscope
 8. Upright Microscope for sample observation with objectives – 10x, Kohler illumination and view of the cantilever and sample through a 10X 0.28 NA Mitutoyo objective. The base should contain a port for inserting fiber guide illumination and built-in cameras with differing magnifications (720 and 240 micron field of view for 1/4" CCD cameras).
 9. The instrument must use an infrared SLD (or equivalent) for the optical lever arm to eliminate optical crosstalk with epi- and transmission- fluorescence measurements.
 10. Force sensitivity < 30 pN or better. Experimental evidence on the force sensitivity should be provided.

11. Software requirements

- a. System must include the ability to track a changing contact resonant frequency during operating modes like piezoresponse force microscopy and contact resonance imaging. Phase locked loops (PLL) do not offer sufficient stability to satisfy this specification. System should use in-built lock-in amplifiers and same AFM software

for resonance tracking PFM mode. Use of external lock-in amplifiers & third party software for tracking resonance is not acceptable.

- b. System must be able to support multifrequency AC mode (tapping mode) operation where two specific frequencies ((both should be simple harmonic oscillations like 1st, 2nd or 3rd SHO) are driven simultaneously and detected simultaneously by lockin amplifiers to measure the amplitude and phase response at each frequency. Lockin detection alone at two frequencies is not sufficient, as both frequencies must be driven simultaneously with a mixed drive signal.
- c. Control and analysis must be user-programmable natively in an entirely open-source software programming language.
- d. The system's software must include a one-click configuration tool that sets up the software for standard and user-defined operation modes, such as AC imaging in air and liquid, contact mode, EFM, KPFM, PFM, and force measurements.
- e. The data acquisition system must be capable of recording individual image sizes of 8000x8000 pixels or greater.
- f. AFM control software environment must include 3D rendering technology for advanced image display. This feature must allow the user to generate, display and visualize 3 & 4D real-time scan images, as well as off-line processing.
- g. Must include drift compensation software. Software must allow a region of interest to be tracked in real time to within 1nm of precision while eliminating any scan distortion in the image. Drift compensation must be able to be applied to any imaging, spectroscopy or advanced characterization mode, and in conjunction with sample heating and cooling options.
- h. Software must include a feature that automatically optimizes the imaging gain and set point for tapping mode operation. The feature must use a predictive algorithm such that operation is stable and producing high quality data within the first few scan lines.
- i. System must include a feature that automatically calibrates the cantilever sensitivity (deflection sensitivity/INVOLS) and spring constant by simply selecting the probe type and clicking a button. To avoid tip damage, at no point during the calibration may the tip touch the sample. The feature must actually calibrate the probe. It must not use nominal tabulated values for the sensitivity and spring constant.
- j. Heads, scanners, probe holders and optional environmental control cells must be "plug and play", meaning that the software automatically recognizes them and configures the software appropriately (e.g. calibration parameters).

12. 3D Nanolithography Package

- a. Should have the lowest noise and highest precision with closed loop position control in all three axes.
 - b. Lithography should be operated in either vector based or bit-mapped modes.
 - c. All data types should be available for plotting during lithography, including deflection, lateral force, amplitude, phase and all user-available analog- to-digital converters (ADCs) and digital-to- analog converters (DACs).
 - d. Lithographic contrast to be controlled by modulating cantilever set-point (both contact and AC modes), cantilever drive voltage, cantilever potential and/or any other channel including the user-available DACs.
13. Probes/Tips (for all AFM modes) and other consumable items for running the equipment should be included. A listing of the type of and quantity of probes should be mentioned. Additional probes for these modes of operation should also be offered as optional.
14. Vibration isolation table: Suitable vibration isolation table/platform for the microscope system and appropriate tables for computer workstation /accessories should be provided.

15. Computing facility

- a. Latest computing systems with necessary operating system and dual 24" monitors should be provided
- b. The computing system should preferably have a current generation Intel processor
- c. 8 GB RAM or higher should be provided
- d. Internal hard drive space of 1 TB or higher capacity should be provided

16. General requirements for AFM supply/installation/warranty/training

- a. The quoted should be installed at the user specified location at IIT-Madras
- b. 3 years comprehensive warranty including service and support should be provided.
- c. 10 years spare parts, accessories, free software upgrade availability including necessary training and support.
- d. User list of the similar equipment supplied in India must be provided and atleast 2 Performance certificate from the previous supplier must be provided. . IIT-Madras shall inquire the bidders' customers about the quality of product/service. If the testimonial from their customers is not satisfactory, IIT-Madras reserves the right to reject the bid based on technical grounds.
- e. Demonstration of all the applications and facilities as per the demand of the users.
- f. Comprehensive training on all following speciality applications should be provided.
 - i. Demonstration for AFM, with non contact AFM, and AFM facilities
 - ii. Calibration, testing, and force measurement of standard and unknown samples.
 - iii. Training on the AFM software for operations.
 - iv. Demonstration of capabilities like for AFM nanolithography, LFM, MFM, PFM KPFM and Electrochemical AFM
 - v. Polymer imaging and characterization.
 - vi. The specified noise numbers should be demonstrated at the time of installation.
- g. The power requirement for the main facility and for the accessories is 220-240V and 50Hz. Required online UPS will be provided by the user.
- h. Requirements of space, electricity and other auxiliaries (e.g., gas lines, water, chiller, solvent sources, etc., if applicable) for the equipment should be specified.
- i. At least two similar instruments should have been supplied to IITs / IISC / IISER.

17. The following additional features which must be offered separately.

These will be purchased only if the price falls within the budget available. However, the vendors must state/certify that the AFM system provided will be compatible with these attachments in case these are purchased separately now or in the future and the priced offer should be made including the price of these optional modules / capabilities.

- a. System must include or optionally support (specify which) an accessory that allows application for Variable in-plane and out-of-plane magnetic fields, with field strength controlled from within the AFM software. The in-plane magnetic field should range from at least +/-7,000 G. And should be measured by an integrated Hall sensor and logged by the AFM software. The out-of-plane field strength should range from at least +/- 1200 Gauss (typically +/- 1500 Gauss), During steady state operation, the device must generate no heat, even at the maximum field strength.
- b. The AFM should have an option to upgrade and integrate an Inverted Optical Microscope and allow simultaneous AFM and optical measurements (i.e. brightfield, epifluorescence) and optional phase contrast illumination. This option

alone only needs a confirmation that compatibility is possible with specific mention of the brands of optical microscope/s with there is compatibility. No price is required at this time for an optical microscope.

- c. If the base AFM does not have a >200V PFM option, the high voltage accessory has to be quoted as an optional component.

18. Modes of operation for future upgradation:

The system should have possibility to upgrade to Scanning Tunneling Microscopy.