Appendix A: Technical Specification
X-Ray Photoelectron Spectrometer (XPS) including Ultra-Violet Raman Spectrometer (UPS), and combined Monoatomic Gas Cluster Ion Source with attached Recirculated Glovebox
RFP 609119
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Battelle Memorial Institute, Pacific Northwest Division, Management & Operating Contractor of the U.S. Department of Energy's Pacific Northwest National Laboratory (PNNL) for the U.S. Department of Energy (DOE)requires is a fully automated multi-technique capability including an X-Ray Photoelectron Spectrometer (XPS), Ultra-Violet Raman Spectrometer (UPS), Raman spectroscopy, including an Ar gas cluster source for high throughput research results. This system must include an XPS system capable of high-resolution, monochromated XPS, small area XPS, XPS linescans, XPS chemical mapping, depth profiling from conductors and insulating samples including integrated coincident Raman Spectroscopy. Maintaining the sample environment is vital for much of our work. This will require a sample introduction chamber with an attached recirculated inert atmosphere glove box for sample transfer under anaerobic conditions.

1.0 Overall System Specifications

1.1 The main vacuum chamber shall be a single piece machined from Ni-Fe alloy to combine optimum magnetic field shielding with precision component alignment. The analysis chamber base pressure after baking and cooling must be guaranteed at 5×10^{-9} mbar or better for ultra-high vacuum (UHV) surface analysis.

1.2 The analysis chamber shall be fitted with a turbomolecular pump with compatible oil-free backing pump and auxiliary titanium sublimation pump (TSP).

1.3 The main system shall be pumped by the combination of ion pump, Ti sublimation pump and turbo-molecular pumps backed by dry roughing pumps (no oil pumps).

1.4 The instrument shall have an automated specimen transfer mechanism controlled by an operating personal computer provided by the Offeror.

1.5 The sample introduction chamber shall be made of aluminum and pumped using a turbomolecular pump and a compatible backing pump. The sample introduction chamber shall be connected to the analysis chamber via an automated (low shock) gate valve.

1.6 Transfer of the sample holder into the analysis chamber shall be fully automated and integrated to the spectrometer's vacuum control software to preserve Ultra High Vacuum (UHV) conditions. A combination vacuum gauge shall be supplied to measure the chamber pressure and to control the automatic sample transfer via the data system. A typical metallic sample must be transferred from the sample introduction chamber to the analysis chamber in less than 10 minutes from the start of pumping.

1.7 The sample manipulator shall be fully automated and have at least 4 axes of movement, X, Y, Z and controllable continuous azimuthal sample rotation. The sample holder and system configuration shall allow for a minimum analysis area of 60 mm x 60 mm and a maximum sample thickness of 20 mm.

1.8 The instrument shall have an automatic baking function to control the vacuum system bakeout time and temperature. The bakeout control will be interlocked with the ion gauge pressure. There must be no requirement to remove cables for backout. Offeror shall provide suitable bakeout blankets or covers for system baking

1.9 The system shall include a high-performance color optical system for precise sample alignment and analysis position.

2. Al Kα Monochromatic X-ray Source

2.1 The x-ray source shall provide high-flux Al Kα monochromatic x-ray beam.

2.2 The instrument shall have an energy resolution of 0.50eV (FWHM) as measured using Ag $3d_{5/2}$ line.

Note: The energy resolution is defined as the full width at half maximum (FWHM) of the Ag $3d_{5/2}$ peak measured after removal of a linearly interpolated background. Ultimate energy resolution < 0.5 eV FWHM.

2.3 The X-ray spot size shall be variable in the range of ≤10μm to ≥400μm. The X ray spot size selection should be adjustable in 5 μm steps from the smallest to the largest spot size to facilitate matching the analysis area to the feature of interest.

Note: The X-ray spot size or spatial resolution is defined as the distance between the points at which the XPS peak intensity is 20% and 80% of the maximum value based on a linescan across a knife-edge or from a TEM grid.

2.4 The x-ray monochromator shall include an electron suppression device to reduce the number of unwanted high energy electrons reaching the sample analysis position.

2.5 The quartz crystal monochromator goniometer shall be motorized for automated or remote access X-ray spot alignment.

3. Electron Energy Analyzer

3.1 The electron energy analyzer shall be a spherical sector type fitted with a multi-channel detector with at least 128 channels for high sensitivity XPS.

3.2 The instrument shall be supplied with a large acceptance lens ($\geq 60^{o}$) and have at least the following XPS performance:

3.2.1 1.0eV (FWHM) of the Ag $3d_{5/2}$ with a peak height of ≥ 4,000,000 cps measured after removal of a linearly interpolated background using an X-ray spot size of 400 µm, and an X-ray power less than 75 W, on a horizontal silver specimen.

3.2.2 1.0eV (FWHM) of the Ag $3d_{5/2}$ with a peak height of ≥ 200,000 cps measured after removal of a linearly interpolated background using an X-ray spot size of 30 µm on a horizontal silver specimen.

3.2.3 1.0eV (FWHM) of the Ag $3d_{5/2}$ with a peak height of ≥ 35,000 cps measured after removal of a linearly interpolated background using an X-ray spot size of 10 µm on a horizontal silver specimen.

4. Specimen Charge Compensation

4.1 The system shall provide compensation on non-conductive samples even when analyzing small areas. A system based on a combination of low energy ions and electrons is required.

4.2 The flood control shall include automated Ar gas handling. The spectrometer data system shall include both automatic filament degas and conditioning routines.

4.3 The system shall have a charge compensation for carrying out measurements on insulators. The system must be capable of making measurements on PET (polyethylene terephthalate) with O-C=O peak resolution of ≤0.80 eV.

5. Raman Spectroscopy

5.1 Raman spectroscopy must co-incident with the XPS. The integrated system must be Class 1 Laser Safety compliant, including suitable laser filters or covers on viewports and hardware safety interlocks as required.

5.2 Raman and XPS data shall be able to be collected from the same point on a sample, without moving the sample.

5.3 Raman experiments must be able to be controlled by both the Raman and XPS data systems, and co-processing of Raman and XPS data must be possible. The Raman spectrometer shall include a capability to self-calibrate, including the capability to switch laser wavelengths.

6. Ultra-Violet Spectroscopy (UPS)

6.1 A gas discharge source capable of operating with noble gases shall be supplied.

6.2 All differential pumping, gas handling and source operation shall be automated.

6.3 The standard operation shall be configured for use with helium. The data system shall be capable of automatically starting the lamp and operate in He(I) or He(II) modes.

6.4 The UPS performance shall meet or exceed the rate of ≥2.2 Mcps using a clean Ag sample with the resolution at the Fermi edge of <120 meV.

6.5 UPS resolution is defined as the energy difference between the points at which the intensity is 20% and 80% of the maximum value below the Fermi level.

7. Dual Monoatomic and Gas Cluster Ion Source

7.1 A high-performance ion gun capable of operating in both monatomic and cluster ion modes must be included.

7.2 Ion gun shall provide surface cleaning capability and chemical depth profiling at normal or low ion impact energies.

7.3 In monatomic mode, this ion gun must offer high performance from ≥500 eV to ≥4 keV.

7.4 In cluster mode, the ion gun must offer acceleration voltages from 2 keV to 8 keV, and average cluster sizes of up to 2000 atoms, allowing energy per atom values suitable for depth profiling polymers of 1 eV per atom and above.

7.5 The data system must control the parameters of the ion gun and the gas handling, including switching from monatomic to cluster ion mode. The system shall include apertures and current measurement devices to support automated ion gun alignment routines. These apertures must be permanently mounted in the analysis chamber and always available to the user. The ion gun software shall also support both automatic degas and filament conditioning procedures.

7.6 Oil-free backing pumps are required to replace the standard rotary vane pumps.

8. Sample Stage

8.1 The sample navigation tool shall support several camera views to facilitate all sample navigation operations. To fulfill this requirement the following sample views are required:

8.1.1 A full sample platter view for sample to sample translation.

8.1.2 A real time magnified image of the sample giving a plan view of the sample. This view shall have a maximum field of view of 4.8mm x 3.5mm. This camera shall support digital zoom providing a magnification of x8.

8.1.3 The system shall include a microscope with camera for accurate sample height setting.

8.1.4 The system shall include mapping capability that can generate XPS images over areas of 0.1 mm^2 – ≥3 mm^2, which can be used to define analysis positions.

9. Computer Control

9.1 The data system must be capable of controlling and automatically recording the following:

9.1.1 X-ray spot size.

9.1.2 Analyzer settings.

9.1.3 Ion gin and flood gun settings.

9.1.4 The data system must have full control of the sample stage for multi-point analysis, azimuthal and compucentric depth profiling, lines-cans and maps.

9.1.5 Routines shall also be included for fully automatic spectrometer calibration, X-ray spot size measurement, ion gun alignment and source conditioning.

9.1.6 To complement these routines, position indexed standards (Cu, Ag, Au) shall be available in the analysis chamber. The routines shall have the capacity to align and prepare standard samples before acquiring data.

10. Data System

10.1 The data system shall include a comprehensive package of data acquisition and processing software for XPS, including depth profiling, line scans and maps.

10.2 Three (3) additional data processing licenses shall be included in addition to the one (1) application license included with the data system.

10.3 A built-in database of XPS information and spectra shall be included.

10.4 The Offeror shall include a workstation loaded with Microsoft Windows™ and the XPS data system software. It shall include a network card for easy networking, remote control of the XPS instrument and XPS data file transfer.

10.5 The data system shall include two ≥23” LCD high resolution widescreen video monitors.

10.6 The data system shall include routines for automatic data acquisition and reporting.

11. System Calibration

11.1 The following software controlled automated instrument calibration capability must include for the following functions:

11.1.1 Energy scale linearity.

11.1.2 Transmission function.

11.1.3 X-ray spot size calibration.

11.1.4 Ion gun modes tuning and alignment.
11.1.5 Flood gun alignment.

11.1.6 Electron lens optimization.

11.1.7 Detector optimization.

12. System Automation

12.1 The supplied system shall include automated features for sample handling, vacuum control and data acquisition to allow a spectrometer to be operated in a multi-user environment along with other analytical techniques. To meet these requirements the spectrometer shall include the following functions:

12.1.1 Automated sample transfer.

12.1.2 Automated vacuum control and gas handling.

12.1.3 Automatic sample height adjustment.

12.1.4 Automatic data acquisition for wide scan survey spectroscopy and high-resolution narrow scan data.

12.1.5 Automatic data interpretation and quantification.

12.1.6 Automatic data reporting.

13. System Footprint

13.1 The overall footprint of the entire system shall be less than 2.5 m x 1.5 m.

13.2 Electronic circuit boards shall be designed for modular replacement and therefore be easily removed.

13.3 All electronic boards shall be mounted in fan-cooled, shielded enclosures.

13.4 The bake-out shielding shall be integrated into the system housing without the need for additional baking tents or panels.

13.5 All cables connected to the main body of the system shall be designed to withstand the bake out temperatures (>120ºC) allowing them to remain in place during the system bake out.

14. Sample introduction Chamber and attached Glove box

14.1 The sample introduction chamber (load-lock) shall be attached to a glove box to allow for anaerobic sample transfer.

14.2 There shall be an access entry door to the sample introduction chamber through the glove box. The glovebox must be designed such that direct hand-loading of sample holder into the entry chamber is possible.

14.3 The glove box XPS adapter shall include a manual entry door, door open/close detection, and vent gas line connection to the glovebox environment.

14.4 Detection switches for XPS system load lock door, open or closed status must be linked into the XPS operating system software.

14.5 The glove box system shall include digital H_2O and O_2 monitors.

14.6 The glove box shall include a recirculating gas purification system capable of maintaining ≤ 10ppm H_2O and O_2.

14.7 The glove box gas circulation system blower shall be encapsulated in stainless steel.

14.8 The glove box shall include a quick sample entry while maintaining ≤ 10ppm H_2O and O_2.

14.9 The glove box shall be constructed of stainless steel.

15. Sample Vacuum Transport

15.1 Four sample holders capable of transferring samples from a glove box environment to the vacuum system without exposure to air shall be supplied:

15.1.1 Sample holders shall allow samples to be transferred under vacuum and into the system.

15.1.2 The vacuum transport sample holder shall be capable of allowing samples to be prepared in an inert environment, put into the holder, pumped down and then transferred into the spectrometer.

15.1.3 The sample holder must be able to transfer samples into the vacuum system without operator intervention once the transfer vessel has been introduced into the sample load-lock.

16. Sample Holders and Spares

16.1 The following samples holders and spare anodes, filaments and spares shall be supplied:

16.1.1 Six (6) each Standard sample platens or holders (≥60 mm x ≥60 mm)

16.1.2 Three (3) each Powder sample platen or holders (≥60 mm x ≥60 mm).

16.1.3 Two (2) each spare x-ray anodes.

16.1.4 Two (2) each spare x-ray filament assemblies.

16.1.5 Two (2) each flood gun filament assemblies.

16.1.6 Two (2) each ion gun filament assemblies.

16.1.7 One (1) each Channel plate kit.

16.1.8 One (1) each Sublimation pump filament kit.