Specifications for a Stand-Alone X-Ray Photoelectron Spectroscopy (XPS) System

1.0 Scope

This specification describes the minimum technical requirements and the minimum acceptable performance standards for a stand-alone X-Ray Photoelectron Spectroscopy (XPS) system to be installed by the contractor at the Naval Research Laboratory (NRL), Washington, DC. The XPS system will be placed in a multiple-user facility, therefore, it must be user-friendly for performing standard and advanced surface analysis, as well as provide ease of maintenance, troubleshooting, and calibration.

1.1 Installation Site

The system will be installed in Building 207, Chemistry Division, Naval Research Laboratory, 4555 Overlook Ave. SW, Washington, DC 20375.

1.2 Description and Primary System Components

The Naval Research Laboratory plans to purchase a stand-alone XPS system that will serve as a shared surface analytical facility for NRL users. The system will be used to analyze the surface chemistry and composition for a wide range of samples, including metals, semiconductors, ceramics and other insulators, polymers, powders, and nanoparticles.

A stand-alone XPS system must include the following primary components: an analysis ultra high vacuum (UHV) chamber equipped with an electron energy analyzer, an x-ray source, a motorized sample stage, an ion gun, a charge neutralization device, at least one digital camera for viewing the sample combined with at least one other means of viewing the sample; an introduction lock vacuum chamber connected to the analysis chamber; one or more vacuum pumps connected to the vacuum chambers; electrical and electronic equipment required for operation and control of the system components; a control computer.

2.0 Analysis Chamber and UHV Requirements

2.1 The analysis chamber must be constructed out of a metal or metal alloy that provides magnetic shielding for optimal XPS performance and is compatible with a UHV environment and with all the procedures required to maintain a UHV environment.

2.2 The analysis chamber base pressure after baking and cooling must be guaranteed at 5×10^{-9} mbar or below, thus providing a UHV environment for surface analysis. The specified base pressure may be achieved by an ion pump, a turbomolecular pump with dry scroll pump backing, or a combination of such pumps.

2.3 The analysis chamber must be fitted with an auxiliary titanium sublimation pump (TSP). The TSP must contain at least three (3) filaments and be capable of operating in automatic and manual modes.
2.4 The vacuum system must have an automatic baking function.
2.5 The vacuum system and control electronics must be designed to avoid venting the analysis chamber in an event of a power failure. Vacuum gauges on the system must be interlocked to the gate valves, and system components must be automatically and safely shutdown after a loss of power.

3.0 Introduction Chamber Requirements

3.1 The introduction chamber must be constructed out of a metal or metal alloy that is compatible with a UHV environment and with all the procedures required to maintain a UHV environment, such as baking the system.
3.2 The introduction chamber must be pumped using a turbomolecular pump.
3.3 The introduction chamber must be connected to the analysis chamber via an automated UHV-compatible gate valve.
3.4 The transfer pathway between the introduction and the analysis chambers must have clearance for sample holders or platens of at least 60 mm in diameter.

4.0 Electron Energy Analyzer Requirements

4.1 The electron energy analyzer must be capable of operating in both scanned and snapshot (parallel) acquisition modes using a multi-channel detector (MCD).
4.2 The MCD must have at least 16 channels.
4.3 The analyzer input lens system must be capable of efficiently collecting photoelectrons in both large (>100 μm) and small (<100 μm) analyzed area modes.
4.4 Any required adjustments of analyzer or input lens parameters for switching between different acquisition modes must be performed automatically by the control software.
4.5 The analyzer must achieve the following resolution and sensitivity specifications using a monochromated Al Kα x-ray source from a horizontal sputter-cleaned Ag sample.
- Ultimate energy resolution of <0.5 eV.
- The sensitivity of at least 1,000,000 cps at resolution of ≤1.0 eV when operated in a large area mode (measured from an analysis area with lateral dimensions >100 μm).
- The sensitivity of at least 90,000 cps at resolution of ≤1.0 eV when operated in a small area mode (measured from an analysis area ≤30 μm in diameter).

The energy resolution and sensitivity are defined, respectively, as full width at half maximum (FWHM) in eV and intensity in cps of the Ag $3d_{5/2}$ peak measured after removal of a linearly interpolated background.

5.0 X-ray Source Requirements

5.1 A monochromated Al Kα x-ray source is required for performing high-resolution chemical state analysis on this XPS system.

5.2 Using a horizontal sputter-cleaned Ag sample, the monochromated Al Kα x-ray source must be capable of producing the energy resolution of <0.5 eV, where the energy resolution is defined as FWHM in eV of the Ag $3d_{5/2}$ peak measured after removal of a linearly interpolated background.

5.3 The monochromated Al Kα x-ray source must be able to selectively illuminate an analysis area on the sample, for small area analysis the illuminated spot on the sample must be ≤30 μm in diameter.

6.0 Sample Stage Requirements

6.1 The sample stage must be motorized and fully automated.

6.2 The sample stage must have at least 4 axes of movement: X, Y, Z and continuous azimuthal rotation.

6.3 The instrument control software must be able to operate the sample stage automatically, including moving to predefined positions for sample analysis and performing continuous compucentric azimuthal rotation for sputter profiling.

6.4 The instrument control software must be able to operate the sample stage in order to automatically optimize the Z position of the sample for analysis, based on photoelectron signal intensity.

6.5 The sample stage must be capable of accommodating and positioning for analysis samples with heights up to 20 mm.

6.6 The sample stage must be capable of accommodating a sample holder or platen of at least 60 mm in diameter and of positioning all samples on such a holder for analysis.

6.7 A selection of sample holders must be provided with the system, including:
- 9 multi-sample holders or platens of at least 60 mm in diameter,
- 9 holders compatible with compucentric azimuthal rotation
- 6 holders for powder samples.

6.8 Each multi-sample holder must be provided with a full set of sample mounting hardware, such as mounting screws, removable spring clips and screens.

7.0 Ion Gun Requirements

7.1 An Ar ion gun capable of focusing down to a spot size of ≤500 μm in lateral dimensions is required for performing depth profiling analysis.

7.2 The ion gun must provide high performance for sample cleaning or depth profiling operating at the minimum voltage ≤200 V and maximum voltage ≥3000 V.

7.3 At operating voltage of 200 V, the ion gun must produce at least 0.5 μA of ion emission and a spot size ≤500 μm.

7.4 At operating voltage of 3000 V, the ion gun must produce at least 3 μA of ion emission and a spot size ≤500 μm.

7.5 All operating parameters, including the Ar gas pressure and effective beam sizes, must be under full control of the instrument control software.

7.6 The ion gun must provide continuous long-term stability and sputter rate reproducibility.

8.0 Sample Charge Neutralization Requirements

8.1 The sample charge neutralization system must provide reliable charge compensation on non-conductive samples for all the available sizes of analysis areas.
8.2 The sample charge neutralization system must be based on using a combination of electrons and low-energy positive Ar ions to flood the sample.
8.3 A single mode of operation must provide the optimal charge neutralization for all sample and experiment types. The charge neutralization system must be compatible with an "ON/OFF" mode of operation. Using the charge neutralization system must not routinely require adjustment of operating parameters by individual users.
8.4 The charge neutralization system must regulate the Ar pressure automatically.
8.5 On a fresh and clean specimen of polyethylene terephthalate (PET) illuminated by the monochromated Al Kα x-ray source, the charge neutralization system must be able to produce, within 100 sec of being turned on, a symmetric O–C=O component with intensity of 8000 cps and FWHM of ≤0.85 eV, after data processing that includes a Shirley background and symmetric peak fitting over the C 1s envelope.

9.0 Sample Viewing System Requirements

9.1 The sample viewing system must provide the main view digital images of the sample at the analysis position, with lateral resolution of at least 10 µm, in order to identify areas for analysis by small area spectroscopy.
9.2 The sample viewing system must provide digital images of an additional view at the sample analysis position, with lateral resolution comparable to the main view, in order to produce correct sample alignment for analysis by small area spectroscopy. The second view may be optical or produced by imaging secondary electron emission for the sample.
9.3 Precise alignment of the above two views must be performed during the system installation.
9.4 The sample viewing system must provide means for optimizing the viewing conditions based on the surface properties of the sample such as roughness and reflectivity.
9.5 The sample viewing system must be capable to record the digital images corresponding to the area being analyzed. The recorded images must be available for subsequent viewing, printing, and exporting.

10.0 Small Area XPS Analysis and Imaging Requirements

10.1 A lateral resolution of ≤30 µm is required for small area XPS analysis and for imaging on all types of samples, where the lateral resolution is defined as the distance between the points at which the Ag $3d_{5/2}$ peak intensity is 20% and 80% of the maximum value in a line scan across a silver coated knife-edge.

10.2 The XPS system and control software must be capable to produce chemical maps with lateral resolution of ≤30 μm, both as two-dimensional images and as one-dimensional line scans. A capability must be provided to acquire multiple spectral regions in a snapshot mode at each point of a chemical map.

11.00 System Control Computer and Software Requirements

11.1 The system must be controlled by a computer using Windows operating system (XP or later version) and software that operates the system, logs data, displays data, and processes data.

11.2 Keyboard- and mouse-controlled interface.

11.3 Commercial computer equipped with a CD/DVD writer and a network card.

11.4 Control software must be compatible with the system operation by multiple users (a minimum of 50 users), either via an internal multi-user interface or via being accessible by multiple Windows users on the control computer.

11.5 Control software must log and save system settings and parameters for each measurement. The saved settings must be available for subsequent viewing, printing, or exporting in text or tabulated format by the originating users.

11.6 The XPS data must be stored by the software, following the directory and filename conventions chosen by the users. The stored data must be available for subsequent viewing, printing, or exporting as formatted text.

11.7 The control software must provide measurement modes that are automated for system operation either in unsupervised mode following a user-determined set of instructions, or in a mode that requires minimal operator input for repetitive tasks such as sputter depth profiling, measuring multiple points on a sample, or acquiring chemical maps.

11.8 Analysis software must include features that either automate or assist users with routine analytical tasks, such as identification of elemental peaks in the spectrum and quantification of the nominal sample composition using predefined sensitivity factors.

11.9 Analysis software must include automated options for processing multi-level datasets, including depth profiles and multi-layers, line scans and chemical maps, angle-resolved datasets. The automation options should include the ability to repeat or batch analyze similar spectra without repeated entry of all analysis parameters.

11.10 At least one (1) additional copy (license) of the data analysis software must be provided with the system, for installation on an auxiliary computer workstation.

12.00 System Layout and Configuration Requirements

12.1 The XPS system will be installed in a standard laboratory module at the Naval Research Laboratory and will serve as a multi-user facility. Accordingly, the overall system layout and configuration has to be optimized to minimize the system footprint, while maintaining easy access for users and for servicing the instrument.

12.2 The combined footprint of the main system components—the vacuum chambers and components, power supplies, and control electronics—need to be less than 8 m^2.

Auxiliary items, such as computer desk and water chiller are not included in the footprint requirement.

12.3 The vacuum components must be configured in a manner that minimizes the number of cables and hoses that need to be disconnected for bake-out. Bake-out equipment such as fans and heater elements, need to be integrated with the vacuum chambers and the system enclosure.

12.4 The power supplies and control electronics must be designed, arranged, and integrated in modular fashion, which will allow the customer or the service engineers to easily remove and replace individual modules and boards.

13.00 Automation and Calibration Requirements

13.1 In order to be operated in a multi-user environment, the system and control software must include automated features for data acquisition, sample handling, vacuum control and gas handling, and for operating the x-ray source, spectrometer, ion gun, and charge neutralization.

13.2 The data acquisition automation must include the ability to navigate between predefined sample positions, to acquire user-specified spectra in scanned and snapshot modes, to acquire user-specified line scans and chemical maps, and to perform multiple sputter cycles followed by data acquisition.

13.3 The sample handling automation must include the ability to position the sample stage for sample transfer from or to the introduction lock, to automatically optimize the Z position of the sample based on photoelectron signal, and to perform compucentric sample rotation.

13.4 The control software must include the ability for troubleshooting and calibration of the instrument parameters, such as testing and altering the instrument transmission function, testing the binding energy scale, and optimizing the charge neutralization settings.

14.00 Accessories

14.1 All the power transformers required to allow normal operation at site of installation must be supplied with the XPS system.

14.2 A closed circuit water chiller must be supplied with the XPS system, if such a chiller is required for system operation.

14.3 The XPS system must include appropriate workstation table for the data system.

15.00 Spare Parts

15.1 In order to maximize the instrument availability after the installation, a set of recommended consumable parts for a minimum of one year operation must be supplied.

16.00 XPS Installation

16.1 The vendor must provide on-site installation of the XPS system in Building 207, Chemistry Division, Naval Research Laboratory, 4555 Overlook Ave. SW, Washington, DC 20375.

16.2 The vendor must specify the lead time for delivery and installation of the XPS system at the Naval Research Laboratory.

16.3 A factory-trained technician must perform initial setup, testing, and calibration of the XPS system, control electronics, and software.

16.4 The installed system must be able to meet the performance specifications as stated in sections 2–10 and in the vendor's proposal.

16.5 The operation and maintenance of the XPS system as well as the use of control and analysis software will be demonstrated over a period of several days to a dozen users by qualified vendor personnel..

16.6 The vendor must provide all documentation, drawings, and schematics that are necessary for the full operation, troubleshooting, servicing, and repairing of the system and its components.

17.00 Low Cost of Ownership

17.1 Systems that minimize or eliminate the need for expensive tools and consumables are preferred. Vendors are encouraged to provide evidence supporting low cost of ownership.

18.00 Warranty

18.1 A base manufacturer's warranty for at least one (1) year from first use must be provided for the XPS system and its components. The warranty must include technical support, a minimum of four (4) on-demand service calls per year (with response within 72 hours), and a minimum of one (1) planned preventive maintenance visit per year. Extending the warranty to three (3) years should be quoted as an option, see “19.1 Optional Items”.

18.2 The computer system and control software must be covered by the warranty, including the regular software updates during the warranty period.

19.00 Optional Items

19.1 Extended manufacturer's warranty for three (3) years from first use.