Expanding Lab Capabilities with Customizable Analysis Tools

Michael R. Funk, Joel Alexa, Robert G. Bryant

Advanced Materials & Processing Branch, NASA Langley Research Center, Hampton, Virginia USA

Highly sensitive, specific surface analysis instrumentation is expensive, costing from ≈$10K to >$1 Million, depending on manufacturer and condition [1]. To stay current, a successful characterization laboratory is faced with frequent replacement of instrumentation. Additionally, when new technology or fabrication processes are introduced, new instrumentation may be required, especially where proprietary technology is concerned.

With the ever-decreasing costs of computers and the increasing reliability of open source code [2], it has become feasible for highly skilled personnel to build instrumentation that is on close par with a state-of-the-art system. Building a system allows the integration of several components and processes that may not be offered with a commercial system [3]. Ultra High Vacuum systems (UHV) are expensive to own and operate, so it makes good sense to include a wide range of surface analysis techniques [1].

A highly-instrumented system has several additional advantages. Sources and detectors that have the sensitivity and resolution desired can be selected, and columns and detector configurations can be changed out, giving greater flexibility. Technologies from different vendors can be integrated into one system, allowing selection of hardware for a variety of sources. The cost of purchasing the individual components and designing the system using different vendors is much lower over time because as new technology is developed, single components can be swapped or added instead of purchasing a new system [3]. Additionally, the in-house assembly of such a system tends to lend itself to in-house reparability, avoiding the cost of a service contract and decreasing downtime. Often, labs have spare parts, pumps, and flanges in storage; these can serve to offset costs as well [Fig 1].

A key is selecting chambers that will fit future as well as current needs.

The materials research group at NASA Langley Research Center (LaRC) must be on the leading edge of innovation, discovering new techniques for developing and processing materials that enable NASA missions. It was determined that a tool was needed in which accurate surface analysis could be performed, and in which additional steps could be inserted into that process “in-situ” for any materials class. These additional processes would require uninterrupted vacuum (regular and UHV) with the capability to switch out the components needed to perform specific analytical techniques. Personnel in the Advanced Material and Processing Branch (AMPB) at LaRC have designed a dual-chamber Auger/XPS/Ion/Electron/Microprobe/Thermo-gravimetric/Mass Spectrometer tool, with a nineteen port main chamber [Fig 2].

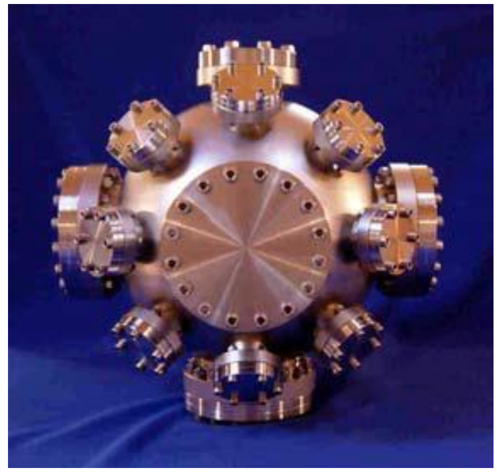
The cost of this LaRC-AMPB system is approximately $240K, whereas equivalent commercial tools, able to accomplish the range of techniques, would cost considerably more.

Custom Tool Combinations:

Technique	Expense New
Auger Electron Spectroscopy	≈ $70K-$80K
X-Ray Photoelectron Spectroscopy	≈ $40K
Ultraviolet Photoelectron Spectroscopy	≈ $20K
Electron Loss Spectroscopy	*$0
Electron Backscatter Diffraction	*$0
Secondary Electron Microscopy	≈ $100K
Energy Dispersive Spectroscopy	*$0
Wavelength Dispersive Spectroscopy	*$0
*use existing hardware	

Figure 1. List of Techniques and Associated Cost, Staib Inst.[4]

Figure 2. 19-Port UHV Chamber. Image courtesy of Staib Inst.



Vacuum and Stage

The UHV Analysis chamber base total pressure is currently 1 x 10^{-11} Torr. The stand-alone load chamber base total pressure is approximately 1 x 10^{-7} Torr. These chambers are coupled together via valve ports. The dry vacuum system has a piston pump backing turbo-molecular pumps backing ion getter pumps, and is designed for plasma cleaning and sample retrieval with minimal system disruption. With the two-chamber arrangement, samples within the loading ante-chamber can be thermally conditioned and their outgassing monitored. This prevents the time and expense of an unknown specimen outgassing under UHV and contaminating the instrumentation in the main chamber.

The stage purchased is as important as the analytical technique being used. Our stage is a motorized 5-axis cooled/heated stage for the main chamber [4]. It has a temperature range from -150°C to 800°C (flash to 900°C) for "in-situ" studies of materials.

The presentation will cover the specifications of add-on columns as well as the cost structures and current uses for this tool. Possible future uses will also be discussed.

References:

[1] Labx**. "**Analytical Instruments" http://www.labx.com, (Web Retrieved Feb 8, 2014).
[2] J. Pearce, "Open Source Lab", 1st ed., Michigan Technological University, Houghton, MI, USA.
[3] Pearce, Joshua M. 2012. "Building Research Equipment with Free, Open-Source Hardware." Science 337 (6100): 1303–1304.
[4] Fig 1 & cost structure, special thanks to Staib Instruments (figures collected 2/2014) Staib Instruments, Williamsburg, VA, 2014.
[5] Fabricated to spec. by Kurt J. Lesker Co.