Recycling a Wavelength-dispersive Spectrometer for Synchrotron Microprobe EXAFS

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Introduction

The Microspec wavelength-dispersive spectrometer (WDS) is a compact in-vacuum Rowland circle designed for x-ray fluorescence (XRF) microanalysis in scanning electron microscopy (SEM) [1]. The 1- to 10 μm spatial resolution of the Microspec is well-matched to the beam footprint of a typical Kirkpatrick-Baez (KB) mirror-based synchrotron microprobe, and the Johansson analyzer crystals provide higher energy resolution than energy-dispersive solid-state detectors such as Ge or Si(Li). Although the solid-angle acceptance of the analyzer crystals is small, the reduction in background scattering results in a better signal-to-noise ratio than that of the solid-state detector in some cases. The WDS is most useful for measuring extended x-ray absorption fine structure (EXAFS) when there are closely spaced or overlapping fluorescence lines giving a large background signal — from trace amounts of Au in an As-rich matrix, for example [2].

Wavelength-dispersive spectrometers are readily available as surplus from SEM laboratories at a wide range of prices. In this activity report, we describe modifications made to a used WDS to integrate it into the PNC-CAT insertion device microprobe at the APS.

Methods and Materials

A Microspec WDX-2A without a control console was acquired on the eBay Internet auction site (http://www.ebay.com) for $1,200. The spectrometer, manufactured in 1984, had been removed from a SEM system and stored under vacuum for several years. It was received in excellent condition, with no degradation of the hygroscopic analyzer crystals.

Under normal operating conditions, the vacuum for the WDS is supplied by the SEM system through a valve at the entrance to the Rowland circle. For the synchrotron microprobe, samples are normally measured at atmospheric pressure. Since the bulky SEM mounting flange interferes with video cameras and other detectors, we first removed as much excess material as possible from the flange by milling. We then replaced the valve with a narrow, tapered flight tube sealed with a 0.2-in. Kapton® window to extend the case vacuum to 10 mm from the sample position, at the focal point of the Rowland circle. The Microspec has a 25-mm port on the bottom of the case sealed with an Al blank and an o-ring. To provide vacuum, which is necessary to prevent arcing in the proportional counters, we replaced the blank with a steel plate of the same size welded to an ISO NW25 quick flange adapter and attached an external roughing pump.

The standard analyzer crystals in the WDX-2A are LOD, PET, TAP, and LiF(200), covering the energy range from E1 to E1. For experiments at APS, a higher energy range is desirable. Several vendors will grind custom analyzer crystals for Microspec spectrometers.[1] We replaced the low-energy LOD and TAP crystals with LiF(420) and LiF(220) from SpexRay, extending the energy range from 3.5 to 24 keV. There is a slight advantage to using the older Microspec model WDX-2A over the WDX-600 currently available from Oxford Instruments: the analyzer crystals on the older four-crystal turret accept a larger solid angle than the crystals on the new six-crystal turret, giving count rates that are about 35% higher.

The Microspec control console provides high voltage to the two built-in proportional counters, power for the pre-amp and gas-flow solenoid, pulse processing electronics, and a sophisticated software package for data collection and analysis. A new Microspec console is a stand-alone personal computer with several input/output cards. A Microspec from about 1984, on the other hand, is likely to come with a vintage VAX computer that needs to be moved with a forklift. Fortunately, most of the electronics provided by the console are generic and commonly available at synchrotron beamlines, and the software package, which is designed for turnkey operation in a SEM, is not needed for microprobe EXAFS experiments. We list here the components used to replace the console on our WDS, along with additional support equipment needed to operate it with the synchrotron microprobe.

Table 1 gives prices for all new parts, although we either obtained most of ours as surplus or used existing equipment at PNC-CAT. The second half of Table 1 lists other support equipment and modifications that are not part of the Microspec package.

[1] Commercial suppliers of custom Johansson crystals for Microspec spectrometers include SPI Supplies (http://www.2spi.com); X-Ray Optics/AAT (904) 646-3069; Oxford Instruments (800) 447-4717, Erin Drake; and SpexRay (775) 853-0514, Richard Wolf.

TABLE 1. Components, Support Equipment, Modifications, and Prices.

Description	Brand	Price ($)
Components to replace Microspec control module		
NIM crate and power supply	Ortec 4001A/4002D	1800
Dual 0- to 2-kV power supply	Ortec 660	1900
Spectroscopy amplifier	Ortec	2300
Single-channel analyzer	Ortec 550A	650
Ratemeter	Ortec	900
Pre-amp power	Newark	80
Gas flow solenoid power	Newark	40
Breakout cable CEN-50 to Elko and BNC	Newark, misc. parts	50
Stepper motor control (4)	OMS, ACS	4400
Other support equipment and modifications for adapting Microspec to synchrotron microprobe		
Stepper motor control (3)	ACS	1800
P-10 gas bottle regulator	McMaster-Carr	250
Roughing vacuum pump	Varian	1200
Vacuum valve and lines	MDC	200
XYZ positioning stage	ADC	7200
Vacuum flight tube	Machine shop	180
Observation window	Machine shop	120
Mounting hardware, base	Machine shop	120
LiF(220) Johansson analyzer	SpexRay	2750
LiF(420) Johansson analyzer	SpexRay	2750

Calibrating the Detectors

There are two gas proportional counters built into the Microspec. The first is a flow proportional counter (FPC) the requires a source of P-10 gas. This detector has Mylar® windows on both front and back. Behind the FPC is a 1-atm. Xe sealed proportional counter (SPC). The output from both the FPC and SPC run through a single pre-amplifier. When they are operated simultaneously, the pulse heights need to be calibrated. This is done by looking at the output on an oscilloscope and adjusting the HV supply for each detector separately until the pulse heights are equal. Typically, the values of the HV for the two detectors differ by less than 10%. Depending on the fluorescence energy being detected, one detector may be unnecessary. For example, very little low-energy fluorescence will be transmitted through the FPC to reach the SPC.

Changing the Energy Range

The newer Microspec models have an in-vacuum motor mounted on the crystal turret for automatically changing the analyzer crystal in place. The WDX-2A turret needs to be moved to the low-energy limit to select a new crystal. Although it is not difficult to drive the stepper motor for this purpose, there are eight détente positions on the turret, which make it easy to reproducibly rotate it by hand. The crystal-changing process takes a couple of minutes, and we seldom change the crystal during a 1-week run.

Calibrating the Energy

For wavelength calibration, we use the position where two bolt holes (one in the case and one on the Rowland circle, normally used when the spectrometer is shipped) are aligned. We have bored a 7-in.-diameter hole in the top plate of the WDS and fitted it with a 1/2-in.-thick Plexiglas® window so that we can observe the motion of the Rowland circle. The values at the calibration point and the number of angstroms per motor step are tabulated in an on-line notebook at the beamline.

Focusing the Spectrometer

In addition, synchrotron users will need to purchase an XYZ positioning stage to bring the focal point of the WDS to the focal point of the KB mirrors. We use a stage from Advanced Design Concepts, driven by three stepper motor drivers. The WDS is mounted vertically on this stage to minimize the footprint and conserve valuable microprobe real estate.

Results

Figure 1 shows EXAFS data from a 250-Å Cr-doped film on a $LaAlO_3$ substrate, collected by using a 13-element Ge detector (red) and the WDS (blue). The WDS has removed the strong La background.

Discussion

Even though all of the support electronics were ordered new, the cost savings over the Oxford package

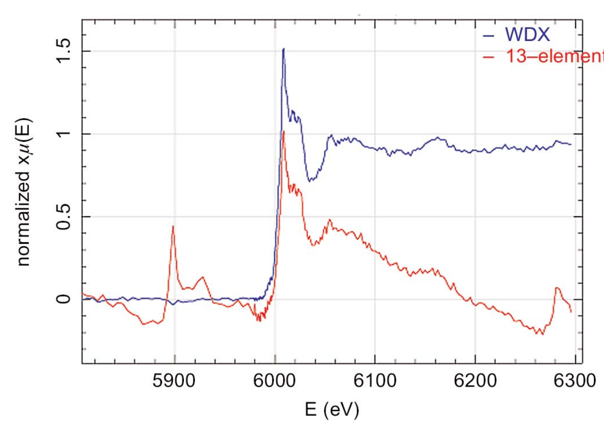

FIG. 1. EXAFS data from a 250-Å Cr-doped film on a $LaAlO_3$ substrate, collected by using a 13-element Ge detector (red) and WDS (blue). The WDS has removed the strong La background.

was significant, and the amount of time to set it up was balanced by the ease of using standard APS beamline software rather than the XRF analysis package, which is too sophisticated for our purposes. We estimate that we saved around $115,000 over purchasing a new Microspec, which would still have required purchasing the items from the second part of Table 1 separately.

The WDS is permanently installed as part of the PNC-CAT microprobe station at the APS and has been used for several experiments (e.g., see Ref. 2).

Acknowledgments

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