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A New Fast Photoelectron Spectrometer for Surface Studies*

W.R. Salaneck and H.R. Thomas

Xerox Webster Research Center
Webster, N.Y., 14580, USA

and

E.W. Plummer, B. Tonner and H.J. Freund

Physics Department
University of Pennsylvania
Philadelphia, PA 19104 USA

We have constructed a new photoelectron spectrometer which combines high resolution and high sensitivity with planned time resolution capabilities. The photon sources for this spectrometer are monocromatized AlK_α ($\hbar\omega = 1486.6$ eV) and monochromatized He II radiation (40.8 eV). The analyzer is a 36 cm mean radius spherical deflector working with a 100 channel spatial detector on the output and a three-lens optical system coupling the specimen chamber to the analyzer on the input side. The spatial detector is a stacked channel plate with 100 gold strip collectors coupled individually to 100 amp-discrimators and scalers. The data acquisition is controlled and facilitated by a DEC/1103 mini computer. This deletion system enables us to obtain fast readout times with low background signals. The specimen chamber is UHV with capabilities for low-temperature studies ($T = 10^\circ K$). This instrument is being applied to the following problems:

(1) Core and valence level spectra of physisorbed molecules in precursor states and the conversion to chemisorption. The system is fast enough to do the equivalent of a thermal desorption spectrum, i.e. a thermal conversion spectrum.

(2) Many-electron effects in the XPS and UPS line shapes in near core levels of adsorbed molecules, like CO, NO, N_2 and the equivalent transition metal complexes.

(3) Direct photoemission from excited states at molecular solid surfaces.

This contribution will serve as a status report on the instrument design, construction and implementation in a variety of studies as outlined above. Representative data on model systems will be presented with an aim at demonstrating the instrument capabilities.

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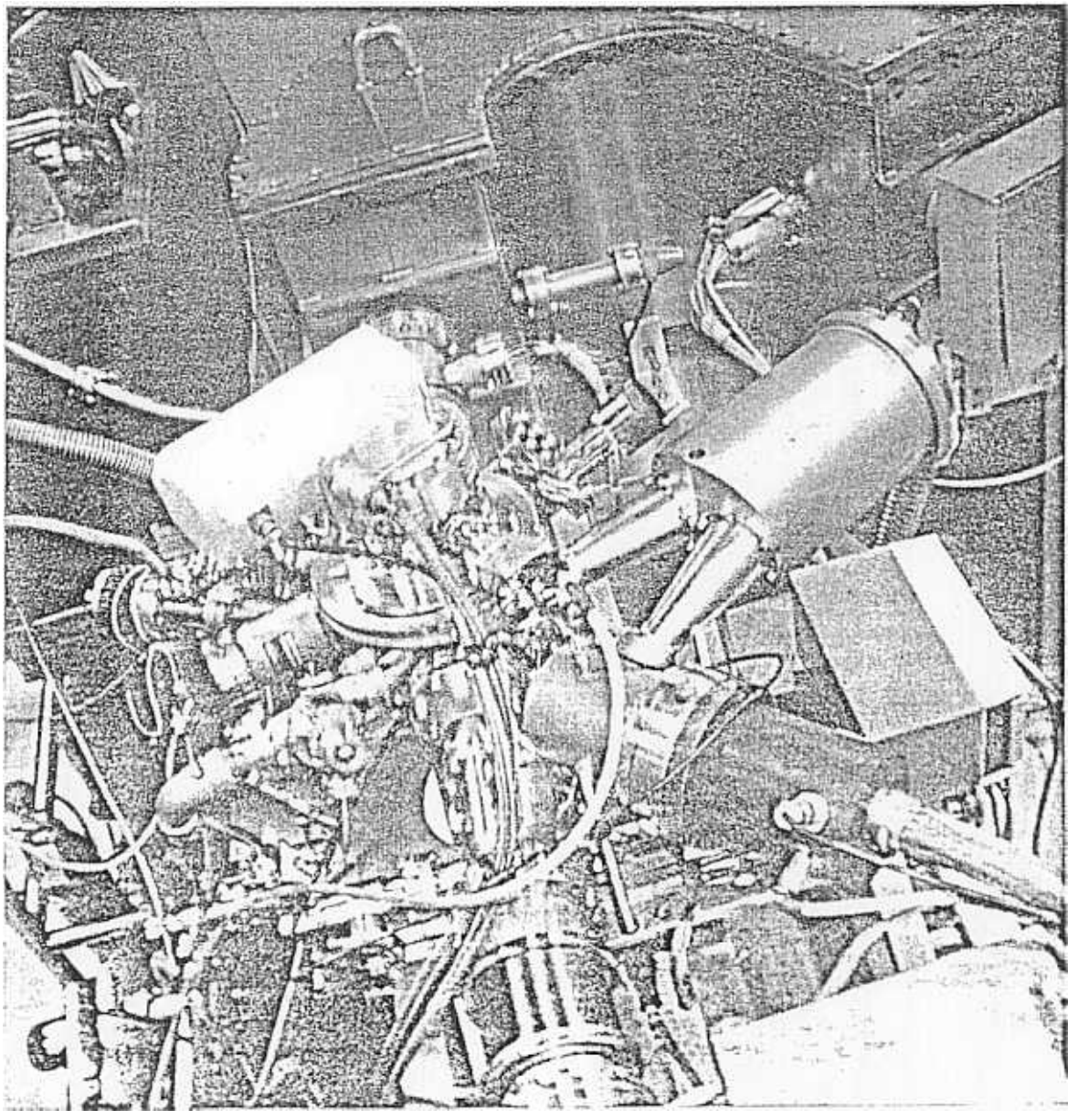
XEROX PENN PHOTOELECTRON SPECTROSCOPY PROGRAM

1980 YEAR END REPORT



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SUMMARY

The Xerox Webster Research Center (WRC) and the Laboratory for Research on the Structure of Matter (LRSM) at the University of Pennsylvania are involved in an ambitious program in molecular science. This program is based upon the design, construction and use of custom instrumentation for photoelectron and soft X-ray absorption spectroscopies. It is financed jointly by WRC, the LRSM and the U.S. National Science Foundation (NSF) in order to form a research program beyond the capability of either WRC or LRSM alone. A novel fast photoelectron spectrometer is under construction in WRC. This instrument has passed initial turn-on tests and is presently in the final stages of construction. A photograph of the instrument is shown on the following page. Additions to this instrument are planned for early 1982. Also, the planning and initial construction of equipment are underway to establish a research station at the National Synchrotron Light Source (NSLS) at the Brookhaven National Laboratory (BNL). Thus far Penn, Xerox (WRC), and the Physics Departments of BNL and SUNY at Stony Brook have been designated as a Principal Research Team (PRT) and allocated a beam line at the NSLS. Operations should commence in late 1981 or early 1982.

In this report we outline and indicate the progress and status of the Xerox-Penn program as of year-end 1980. We also document the program-related activities of the participants in the program during this period.

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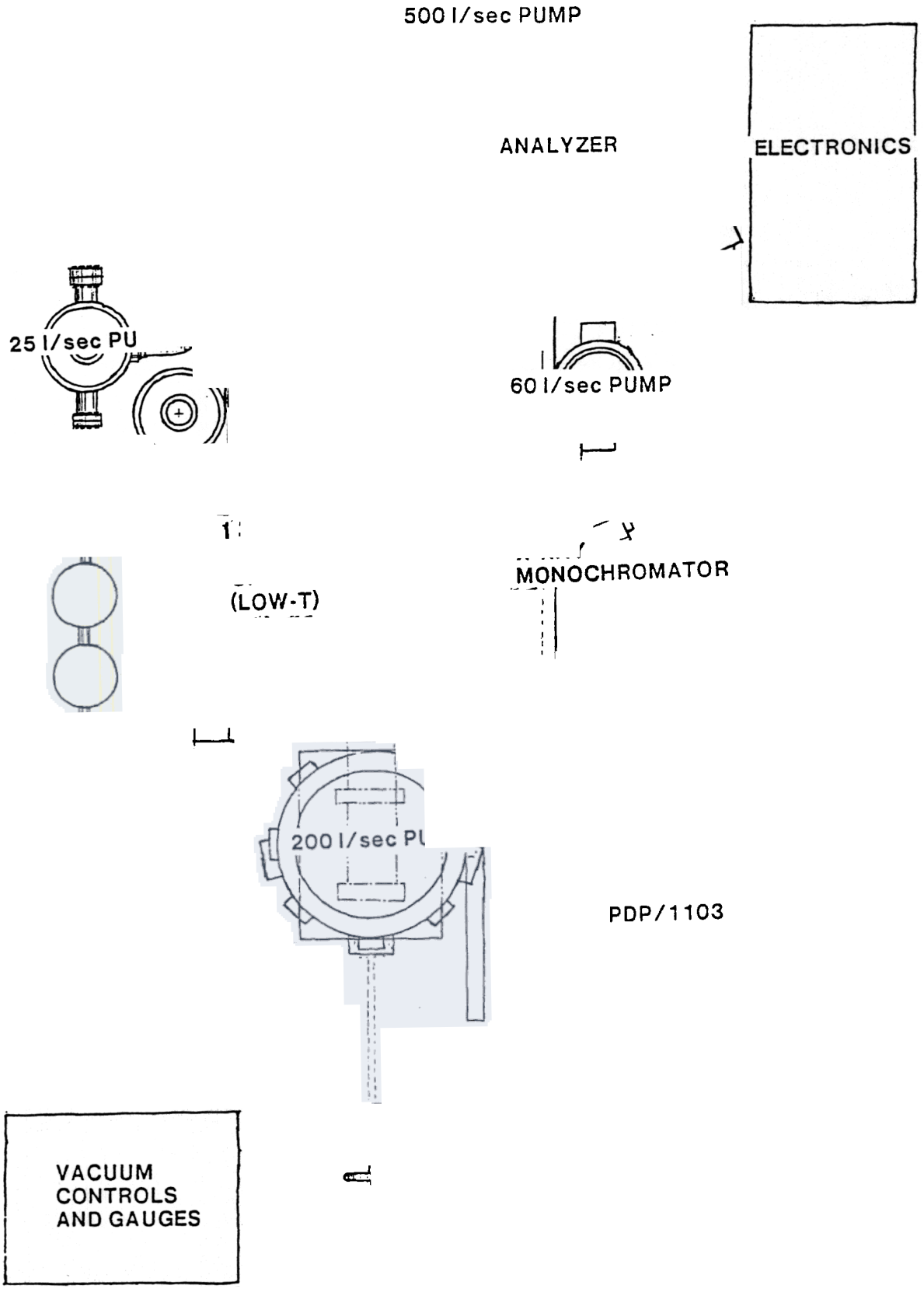
PERSONNEL

W.R. Salaneck	Xerox Principal Investigator
E.W. Plummer	Penn Principal Investigator
H.-J. Freund	Penn Post Doctoral Fellow
W.J. Greene	Xerox Associate Member of the Scientific Staff
C.B. Duke	Xerox Monitor

HISTORY

The Xerox-Penn program in molecular science is divided into phases. Phase I began in 1976, involved commercial instrumentation, is documented in a 1977 year-end report, and terminated with the beginning of Phase II in 1978. Phase II involves the design and construction of a novel fast photoelectron spectrometer (sometimes dubbed FRESCA for Fast Response Electron Spectroscopy for Chemical Analysis). A schematic diagram of the instrument is shown on the following page. Phase III overlaps with Phase II in time but is logically distinct in concept, since it involves soft X-ray absorption and threshold photoemission spectroscopy using synchrotron radiation. X-PPS-III began in late 1980, and will achieve full operation by early 1982. Phases II and III, both presently active, and are described individually in this report. Both are co-financed jointly by WRC, the LRSM and the NSF.



Fast Photoelectron Spectrometer: Schematic

DESCRIPTION: X-PPS-II

The objective of this program is to create a unique photoelectron spectroscopy facility for the investigation of the chemical and electronic structure of molecular materials in both the gaseous and condensed molecular solid phases using soft X-ray and far ultraviolet radiation. The facility is based upon the design and construction of novel instrumentation, the utilization of which will enable the investigation of phenomena and properties of molecular materials not presently accessible. Specifically, we have designed and have in the final stages of construction, a photoelectron spectrometer which should accumulate spectra about 10^3 times faster than commercial instrumentation, and about 10 times faster than any custom built instrumentation in the world today. The novel features of our spectrometer are (1) a new-design tube lens system to gather a large solid angle of photoemitted electrons; (2) a larger 36 cm mean radius 180° hemispherical electron energy analyzer with an 8 cm electrode gap; and (3) a unique 100-channel electron detector with 100 parallel amplifier-discriminator circuits to allow computer-controlled acquisition of 100 independent energy channels of information simultaneously. Item (1) allows a 10X increase in counting rate over commercial equipment; item (3) provides a 100X increase in counting rate over single channel detection (~ 10X increase over commercial sequentially read energy dispersive detectors); and item (2) allows for high currents and high resolution. Photographs of these three key components are found on the next three pages of this section.

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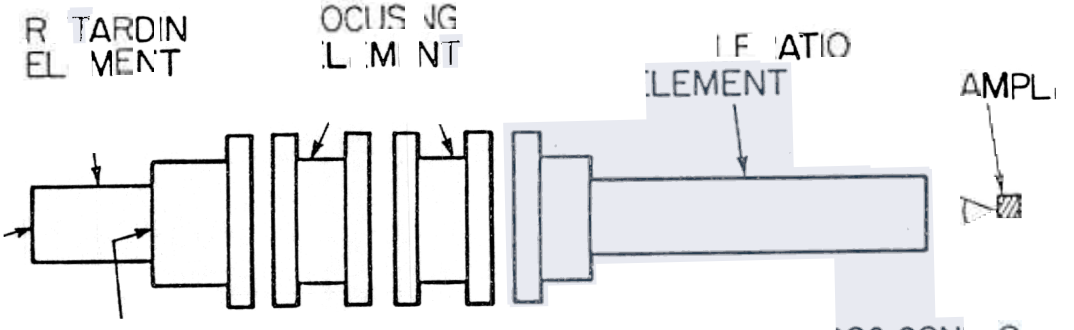

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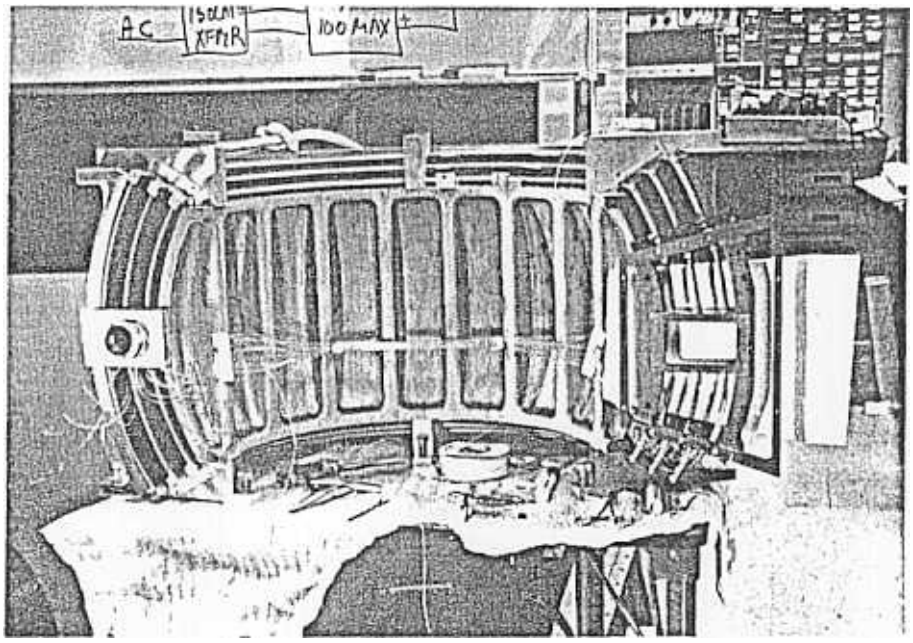
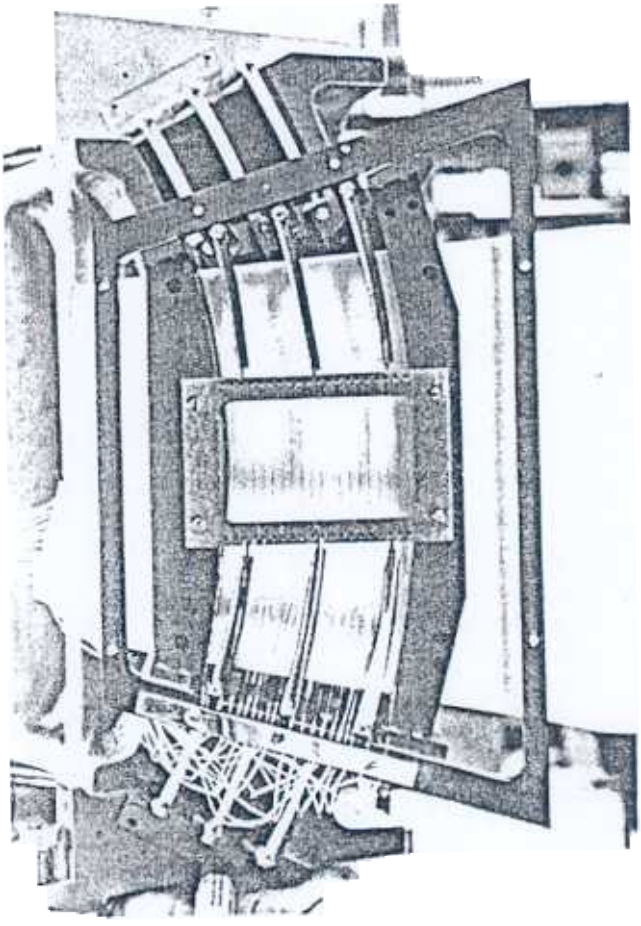
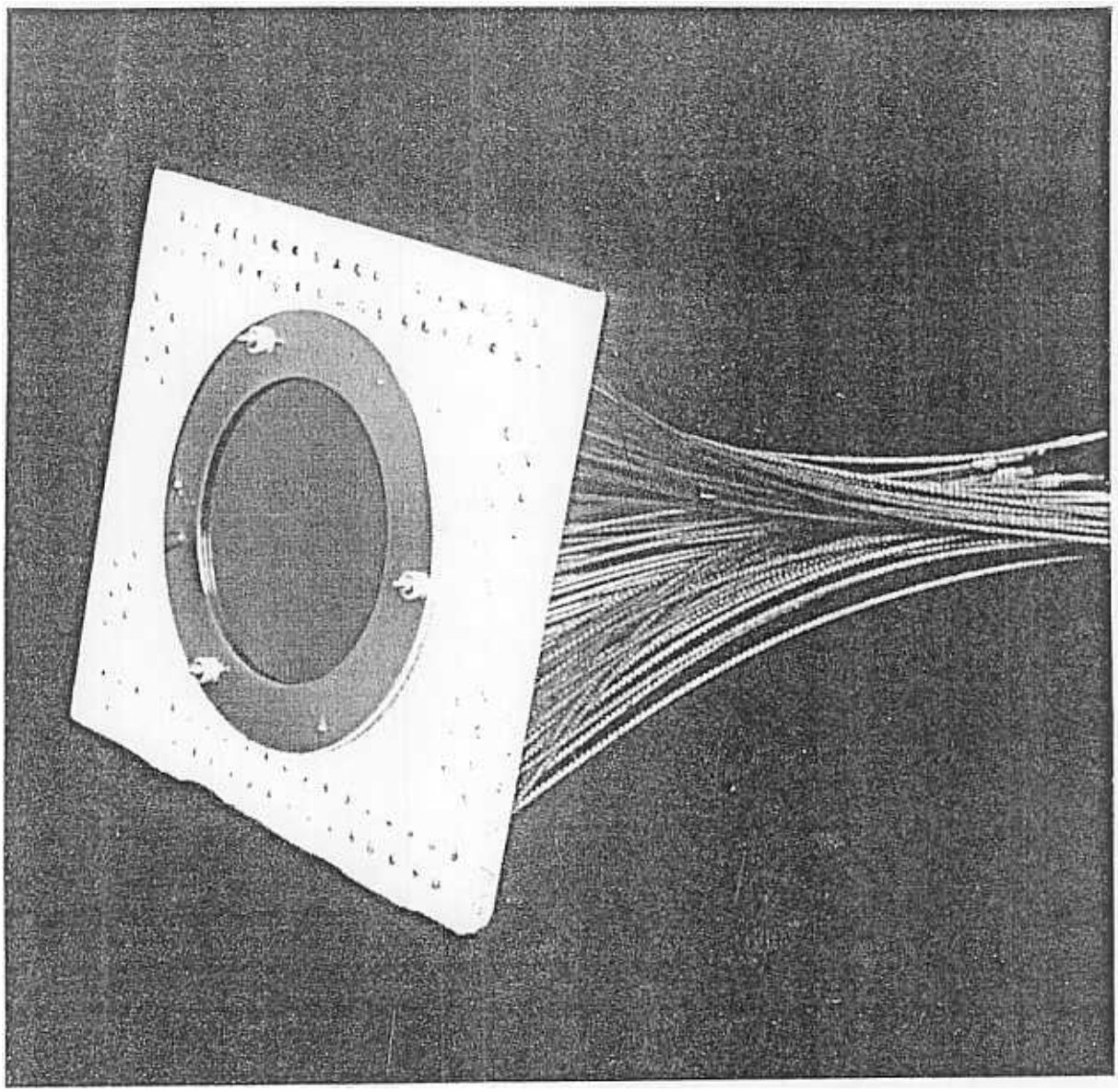




ABOVE: Analyzer with voltage-termination electrodes in place.

BELOW: Close-up of 20-wire field-termination at output of analyzer where the multichannel detector will be fixed.



The 100 -channel multichannel detector is shown prior to attachment to the electron energy analyzer. The 100 gold strips are hidden behind the large area (75 mm dia.) double channel plate electron multiplier (the large dark disc).

This instrument initially will have a time resolution capability of 10^{-3} sec and in 1982 a capability of 10^{-5} sec. The use of this spectrometer will allow the study of problems when the signal level is very low and/or where time domain information will be useful. Given the planned capabilities of the new spectrometer, we envision the following three generic types of experiments:

- Low signal and/or low temperature experiments, where the high sensitivity of the spectrometer will be used to obtain data on a laboratory time scale, and/or quickly enough that surface contamination does not become a problem at low-Temperatures ($10° K \leq T \leq 400° K$).
- Modest time-dependent spectroscopy (10^{-3} to 10^{-5} sec resolution) of reversible phenomena, initiated by a pulsed beam of, e.g., electrons, photons or ions, where good statistics are accumulated through many repetitions of a cyclic event.
- Modest time-dependent spectroscopy of irreversible phenomena, e.g., surface chemical reactions, where the time dependence of the phenomenon is slowed down to observable rates by going to low temperatures (a form of time-temperature trade off).

Some preliminary studies, necessary before pursuing specific examples of the 3 topics listed above on the new spectrometer, have been initiated and carried out elsewhere during the construction stage of the new spectrometer in WRC. These will be listed later in this report.

DESCRIPTION: X-PPS-III

Th object th progr to establish f cility the NSLS BN to utilize synchrotron radiation, from the infrared to the soft-X-ray region (i.e. out to abo V photon energy) in the study of the hem cal and electroni structure of molecular solids. The Uni ersity of Pennsylvania presently has elabora ersi of such cility the Synchrotron Radiation Center of the Uni rsi of Wisconsin W ha ilized th Penn equip Wisconsin to perform several studies er the past wo years. Penn will m their equipm to NSLS la in 1981. A tha tim Penn, Xerox WRC and the Physics Departm ts of NL and SUNY Stony Brook will oper so X-r y researc station NSLS based upon improved version of the Penn instrum tation fr Wisconsin. Because there are lo of photons each NSLS port, costs can be held down by sharing th facility There will be subs port gas phase molecular photoelectron spectroscopy (BNL and SUNY soli state hotoelectron spectroscopy (Penn-Xerox) and so X-ray absorp spectroscopy (Xerox-Penn); and infrared reflectance spectroscopy (Penn). W visi the Xerox ef orts focus main issues: (1) photoelectron spectroscopy mol cula ol ds ar pho oioniza thresholds the study of shake-up phenom in polymeric materials and (2) soft X-ray absorp ion spectroscopy near the carbon K-edge in organi molecular and polymeric erials. A proposal has bee subm ed NSF th Depart of En rgy or financing the remainder of the instrumentation.

STATUS

Funding for the fast photoelectron spectrometer (FRESCA) was allocated by WRC late in 1978, but NSF funding did not commence until July of 1979. Parts were ordered and serious construction began in October of 1979 (see Xerox-Penn Year End Report, 1979). In November of 1980, the spectrometer was successfully operated utilizing "test electronics", that is, with clip-lead wiring and a single channel electron multiplier. The tube lens system was tested and found to perform according to the design specifications. In December, the spectrometer was completely disassembled in order to install the multi-channel electron detector, to install permanent electrical cabling, and to repair leaks in two high vacuum components (delivered defective from the vendor). Because of the leaks, the vacuum was limited to about 10^{-8} Torr in the sample chamber under sputtering conditions, i.e., at room temperatures. We could not keep a clean surface long enough to make feasible any serious materials studies under poor vacuum. With the sample at 13°K, an ultimate pressure of 8×10^{-10} Torr was achieved, with a consequent undesirable condensation of contamination on the sample surface. At 8×10^{-10} Torr, however, the high vacuum rotatible seals worked perfectly. At 10^{-8} Torr, a UPS spectrum of contaminated ("dirty") polycrystalline copper was recorded, using only a simple single channel detection system. This spectrum, compared with one obtained by C. Brucker on clean copper, is shown on the following page.

Basically, all equipment has been received, assembled and is being installed. The multi-channel detector and associated 10^{-3} sec resolution electronics has been assembled and tested on the bench top. Only the final step of operation of the 100-channel detector in the vacuum system remains to be accomplished. Up-grading to 10^{-5} sec resolution awaits additional funding from NSF, a proposal for which is being written simultaneously with this report.

FIRST SPECTRUM FROM FAST PHOTOELECTRON SPECTROMETER
HYDROCARBON-CONTAMINATED COPPER, He I RADIATION
NOVEMBER 13, 1980