

ACTIVITY REPORT TO NATIONAL SCIENCE FOUNDATION

Period: May 1973 - December 1974

by

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Stanford
Synchrotron Radiation Project

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April 25, 1975

Howard W. Etzel, Associate Director
Dr. William Oosterhuis
Division of Materials Research
National Science Foundation
1800 "G" Street
Washington, D. C. 20550

Dear Howard and Bill:

It is a great pleasure to transmit to you this first activity report of SSRP.

Summarized here you find a brief account of the history of SSRP, the current status of facility plans (already out of date in view of the second beam line!) and reports from experimenters.

Of course, the real meat of SSRP lies in the new scientific results which are coming out. Things have gone so fast here that in many cases large amounts of data have accumulated and have not yet reached the publication stage. We anticipate that, with some time lag, publications will indeed grow as fast, if not faster, than the amount of beam time and user time which has gone into SSRP.

I am delighted to be able to claim that SSRP does indeed provide absolutely unique opportunities for research workers which have in fact been exploited. Specifically, the work on x-ray photoabsorption spectroscopy (EXAFS) has produced many "firsts" which could not have been done without the unique properties of SPEAR.

This package is sent as just a promise of things to come. The field looks healthy, the people engaged in it seem to be enjoying themselves. Your efforts on our behalf are much appreciated.

Sincerely,

A handwritten signature in cursive that reads "Seb".

Seb Doniach
Director, SSRP

SD:plm

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I. INTRODUCTION

This report summarizes activity at the Stanford Synchrotron Radiation Project (SSRP) from June 1973 to December 1974 under NSF Grant No. DMR73-07692 A02, nee No. 39525. The principal activity during this period was the design, construction and initial experimental operation of a national synchrotron radiation research facility utilizing synchrotron radiation from the 4 GeV storage ring SPEAR at the Stanford Linear Accelerator Center. Much of this work is described in a paper entitled "Design and Performance of the Stanford Synchrotron Radiation Project," presented to the 1975 National Accelerator Conference in Washington, D. C. A copy of this paper is included in an appendix to this report. The design of monochromators and other instrumentation on the 5 beam lines (sharing 1 port) along with descriptions of experiments in progress and completed is presented in other parts of this report.

The initial schedule for SSRP called for an 18 month design and construction period which would have ended in December 1974. However, the improvement program to upgrade SPEAR from 2.5 GeV to 4 GeV was scheduled to start in July, 1974, and it was uncertain when steady operation would resume. For this reason, it was decided on August 24, 1973, to accelerate the construction schedule of SSRP so that the facility and 5 initial beam runs and 5 initial experiments could begin operation during the May 12 - July 3, 1973 SPEAR cycle--the last one before the improvement program.

This effort was successful and much good experimental data was taken and extensive experience with facility operation was obtained. By December, 1974, the number of experiments and/or experimental programs had grown to 29 proposals representing work from 19 different experimental groups involving a total of 55 individual experimenters from government, private industry, and university research laboratories.

This rapid and continuing growth in the use of what might be considered a "minimum" synchrotron radiation research facility is evidence of the very considerable potential for research using photons in the range 20 eV up to 30 keV and higher for studies of condensed matter, atomic and molecular physics, surface properties, biological materials and many other research areas.

II. BRIEF CHRONOLOGICAL HISTORY OF THE CONCEPTION, DESIGN, CONSTRUCTION AND INITIAL OPERATION OF SSRP

The first of a series of regular bi-weekly meetings of SLAC, Hansen Laboratories and SSRP personnel and outside experimenters occurred on May 23, 1973. The minutes of these meetings provided a record of the development of the project. Highlights from these minutes and other sources (e.g., SSRP log books) are abstracted below to summarize this development.

Sometime in 1968

W. Spicer discussed possibility of applications of synchrotron radiation with W. Panofsky.

Summer 1970

A group of Stanford faculty was formed (Coordinator, S. Doniach) to work on a proposal for use of synchrotron radiation. Members of the group were S. J. Baldeschwieler, A. Bienenstock, S. Doniach, W. Marshall, W. Spicer, and H. Taube.

August 1970

SPEAR was authorized, synchrotron light pipe to be designed into vacuum chamber.

September 1970

Dan Pierce (Stanford Electronic Laboratory) started work as a part-time post-doctoral research associate on the design of a synchrotron facility.

February 1971

S. Doniach gave a talk at a Chicago one-day meeting on applications of synchrotron radiation. The main topic of the talk was the great potential of EXAFS as a tool for structural analysis using the synchrotron source.

December 1971

Proposal submitted to the National Science Foundation for a sum of \$500 K to build a synchrotron facility at SLAC for local users.

G. Fischer acted as SLAC representative and consultant.

March 1972

S. Doniach visited the National Science Foundation and talked to Ed Creutz, Howard Etzel and others about the need for a synchrotron radiation facility.

September 1972

S. Doniach talked at a Brookhaven two-day synchrotron meeting on comparisons between a synchrotron source and a laser source with frequency multiplication in the UV.

P. Pianetta joined the project.

December 1972

First NSF grant for \$60,000 received, S. Doniach and W. Spicer, principal investigators, for study of X-ray Photoemission using the x-rays at SLAC.

February 1973

Design and fabrication work started by Earl Hoyt of SLAC on beryllium window assembly for SPEAR. Funding is from Electrical Engineering Department and the Center for Materials Research.

Science Policy Board appointed: J. Baldeschwieler, Chairman, S. Buchsbaum, F. C. Brown and D. A. Shirley.

National Science Foundation's National Review Committee for Synchrotron Radiation visited Harvard University, the University of Wisconsin, and Stanford University to look for sites for a synchrotron laboratory.

National Science Foundation's Committee requested revised proposal to build a laboratory which would be for national use in the higher energy part of the spectrum. Proposal prepared in four days and delivered to committee meeting in Cambridge by "Captain's Mail."

April 1973

Decision reached to fund SSRP.

May 1973

First meeting of Science Policy Board, Washington, D. C.

June 1973

\$750 K arrived for SSRP.

July 1973

Ground was surveyed for SSRP building and beam line.

June 28 - July 3, 1973

Beam Study Layout Group Meeting held:

Attendees: A. Baer, F. Brown, L. Cooper, R. Gould, J. Harris, L. Lindau, P. Pianetta, V. Rehn, D. Sayers, W. Spicer, J. Stanford, N. Webb, M. Weissbluth and H. Winick.

Detailed discussions were held among experimenters, SLAC personnel and SSRP staff on the following:

A. Beam Requirements

1. Milliradians needed for different experiments.
2. Beam splitter mirrors need to be placed close to the source point.

3. Floor space requirements.
4. Independent steering of the SSRP beam.

B. Building

Need for:

1. Air conditioning for stability of aligned equipment.
2. A longer building than originally planned.
3. The importance of a thick, stable concrete floor.
4. Full services (He gas, water, air, etc.).

C. Second Level Experimental Area for 8° Rising Beam, XPS and Other Experiments

D. Desirable Facilities

1. Dark room.
2. Animal preparation facilities.
3. Chemistry facilities.

July 1, 1973

Fred J. Johnson joined SSRP and started to translate beam study group recommendations into detailed beam line drawings.

July 6, 1973

10:42 A.M. opened vertical mask and got synchrotron light out of the beryllium window on pilot project beam run.

July 11, 17, 23 and August 14, 1973

Meetings held on these dates refined many of the requirements for the building and beam run. Specifications for the initial shell and slab construction were put out for bid.

August 1973

Herman Winick joined the project as Associate Director for Technical Matters.

August 20, 1973

"Space Shot" philosophy adopted. Decision taken to have full facility and 5 experiments operational for May - June 1974 SPEAR cycle in order to obtain data and experience before SPEAR improvement program would begin.

August 22, 1973

Bids opened for building shell and slab.

September 4, 1973

Ray (Priss) Dannemiller joined SSRP as Project Secretary.

September 11, 1973

Decision made to continue operation of XPS experiment on the Pilot Project beam run until full SSRP facility will be ready. It was thought earlier that this experiment would have to be inoperative starting 11-19-73 because of incompatibilities with the full facility construction.

September 13, 1973

Specification of Vacuum Control System: Design will be by R. Melen and construction at SLAC.

September 20, 1973

First meeting held of SSRP staff with SLAC Radiation Committee to define SSRP radiation protection requirements.

September 27, 1973

Installation and first use of beam bump steering of SSRP beam for XPS Pilot Project experiment.

October 15, 1973

By this time, bid packages for mechanical and electrical contracts for the SSRP building were complete. Also, the design of special SPEAR roof blocks and wall blocks was complete. Detailed design of the beam line vacuum system by F. Johnson was proceeding.

October 16, 1973

Axel Golde joined SSRP as a mechanical engineer and Russel Matheson joined SSRP as an electrical engineer. A. D. (Don) Baer returned to other research activities after assisting with initial stages of SSRP planning. Preparations were made for relocating Pilot Project beam run and experiment so that SPEAR shielding modifications could be made as the first step in construction of SSRP facility during SPEAR shutdown starting November 19.

October 29, 1973

Primary beam line personnel protection system defined. System to be designed by J. Miljan and built at SLAC.

November 6, 1973

Sections for the building shell had begun to arrive--2 weeks behind schedule due to supplier problems. Bids opened for electrical contract for SSRP building. Detailed preparations made for relocating Pilot Project beam run and experiment so that SPEAR shielding modifications can be made as first step in construction of SSRP facility during SPEAR shutdown starting November 19.

November 8, 1973

Second meeting of SSRP Science Policy Board. Members: J. Baldeschwieler, F. Brown, S. Buchsbaum, D. Shirley.

November 10, 1973

4:13 P.M. first XPS spectra obtained of the 4f level of gold.

November 19, 1973

SPEAR shutdown began. Original Pilot Project equipment removed to permit SPEAR shielding modifications.

November 27, 1973

Parts of beam line vacuum system were in fabrication at Varian Associates, Hansen Laboratories and SLAC. SLAC Davis-Bacon Committee approved the writeup of SSRP jobs by J. Harris.

December 11, 1973

SPEAR shielding rebuilt with new blocks to form SSRP alcove. Concrete pad poured close to SPEAR. New He system designed to prevent further corrosion of Be window. Pilot Project beam run was re-installed. Detailed schedule was prepared for fabrication, cleaning, welding, assembly, testing and installation of all components of new SSRP beam line vacuum system.

January 3, 1974

Fast valve arrived from CERN.

January 17, 1974

EXAFS diffractometer was installed along side XPS equipment in new SSRP alcove. Spear was again in operation. New interlocked He system for Be window was installed and tested. W. Basinger joined SSRP for a few months to assist A. Golde on the design of secondary area beam stoppers, stands, etc. Description of secondary beam area protection system sent to SLAC Radiation Committee.

January 29, 1974

XPS and EXAFS experiments received beam. Additional shielding installed to reduce radiation levels in SSRP building during electron injection. Desorption diode tests started on lubricated bearings.

February 12, 1974

Work continued inside building on SSRP control room and office partitions, miscellaneous electrical and mechanical work. All vacuum beam line components were on hand. Cleaning and welding in progress. Tests completed successfully on lubricated bearings and on new China Lake seal design for mirrors.

February 26, 1974

R. Matheson left SSRP. Arrangements made with R. Larsen of SLAC to provide electrical engineering assistance to complete SSRP work--particularly on secondary beam hutch personnel protection system and overall electrical coordination.

Delays in arrival of components (750 MCM feed cable, hevimet shielding blocks, cable trays) were causing slippage of schedules.

March 12, 1974

Mirror support and mover system in fabrication. Plans were made to erect temporary plastic enclosed clean room in SSRP building for assembly of beam splitter mirrors and 4° and 8° monochromators.

B. Humphrey completed design of Hutch Protection System. Complex He system to enclose 3 X-ray experiments and shutters in design by A. Golde and R. Messimer.

March 25, 1974

SPEAR shutdown. Major activity had begun on installation, pump down and alignment of SSRP beam line vacuum system, mirrors, etc. Most of the work was done on 4-12 shift by SPEAR Vacuum Group. Jib crane installed in SSRP building. Shielding blocks for primary beam line enclosure being cast. 4° line monochromator was being assembled by F. Brown of Xerox and N. Lien of PSL, University of Wisconsin, the designer. 8° line McPherson monochromator in-house. Work continued on XPS, EXAFS and Cal Tech equipment.

April 16, 1974

B. Salsburg joined SSRP as electrical engineer. First meeting in new SSRP building (No. 120). All beam line components installed and pumped down and aligned. Electrical cabling installation was underway inside SSRP building and connecting to SPEAR and SLAC control rooms and SSRP alcove. Primary beam line shielding was being installed. First hutch protection system chassis completed. Delays in arrival of keys for this system posed a problem. The system fabrication completed and installation started. 4° and 8° monochromators assembled and under vacuum.

April 30, 1974

Vacuum Control System was operational. Primary beam line shielding completed. Strike of Stanford employees called for 5-13-74.

May 6 - 12, 1974

Most of remaining SSRP systems completed and checked (hutch protection system, He system, primary beam line personnel protection system, etc.).

Beam line vacuum system at $< 1 \times 10^{-8}$ torr.

May 13, 1974

Strike of Stanford Employees Union started. FIRST BEAM INTO SSRP BEAM LINE OBTAINED. Movable masks, Be foils and windows, etc. checked out. SPEAR beam dumped by jittery water interlock on SSRP line. Interlock repaired.

May 14, 1974

Primary beam line shutters opened to permit FIRST BEAM INTO SSRP BUILDING. Vertical orbit correction system checked out. Worked fine. XPS and EXAFS groups started checkout of experimental equipment with beam.

May 15, 1974

Pinhole camera pictures taken of SPEAR beam cross-section at source point. Vertical beam size (FWHM) of 1.5 mm during collision and 0.9 mm with separated beam measured at 2.4 GeV and 10 mA.

May 21, 1974

XPS monochromator operational-rocking curves obtained. EXAFS monochromator, detectors and data collection and control system operational. First Cu EXAFS spectrum obtained.

June 1, 1974

Doctor Ichiro Matsubara of the Department of Physiology, Faculty of Medicine, University of Tokyo, Japan, arrived and spent several weeks on the initial work on the experimental program on the Biology Beam Line with Dr. Nicholas Webb from the California Institute of Technology.

June 4, 1974

4° and 8° monochromators operational and some data taking had started. XPS spectra on Ag and Au had been obtained.

June 18, 1974

Cal Tech diffraction camera installation completed. Photoemission measurements made on 8° line.

June 19, 1974

SSRP flooded with 3" water and mud due to a broken 8" water main on hill above building. Massive cleanup restored operation in 3 hours.

June 20, 1974

By this time, diffraction pictures had been taken by Cal Tech, time of flight system was operational on 4° line, photoemission measurements obtained on the 8° line, and EXAFS measurements had been made on many samples including hemoglobin. Some single beam running had been made available to SSRP. A beam position monitor was designed.

July 3, 1974

SPEAR shut down. First SSRP running cycle ended. During the shut-down from July 3 to September 30, 1974, many facility improvement projects were initiated. These are separately summarized in Section IV of this report.

September 3, 1974

R. Gaxiola joined SSRP as vacuum engineer, replacing M. Baldwin who left to work at the University of Montana.

September 10, 1974

Pierre Lagarde of LURE Synchrotron Radiation Laboratory at Orsay and Michael Skibowski of DESY Synchrotron Radiation Laboratory at Hamburg arrived for 1 year of research at SSRP.

October 5, 1974

SSRP obtained first beam of new SPEAR running cycle. Initial tests were made on position monitors. Neither worked very well. Modifications were planned.

October 25, 1974

Serious deterioration noted on He system. Later measurements showed 5 per cent oxygen in system. During December shutdown, the problem was traced to air leaks in shutter and screen pneumatic systems. Problem solved by tightening all connections and use of He rather than air in these systems.

November 8, 1974

First sharp resonance discovered at SPEAR (ψ 3100). Beam energy required was 1.5 GeV. Much running at this energy followed. No X-ray experiments were possible at this beam energy. UV experiments continued.

November 11, 1974

SSRP was given a prime shift. Filled several bunches. Obtained stored beam currents up to 85 mA at 2.8 GeV. Experimenters were very happy.

November 19, 1974

EXAFS hutch had been modified so that scattering or fluorescence experiments could be done in addition to EXAFS.

November 20, 1974

Second sharp resonance observed (ψ 3700). Beam energy required was now 1.85 GeV. Better than (ψ 3100) but still not much X-ray flux above ~ 6 keV.

November 29, 1974

Removed in-vacuum position monitor for modifications. Installed 5 micron pyrolytic graphic foils for testing in vacuum beam line.

December 15, 1974

End of SPEAR cycle. During the SPEAR shutdown from 12-16-74 to 1-20-75, much work was accomplished on facility improvement projects. These are summarized in Section IV of this report.

III. BEAM TIME USAGE

MAY 12 - JULY 2, 1974: FIRST FULL FACILITY RUN

TOTAL BEAM TIME USED BY ALL SSRP EXPERIMENTS 1300 HRS

* STORED BEAM TIME AVAILABLE TO SSRP 610 HRS

Since all 5 groups could operate simultaneously, the total beam time used by all SSRP experimenters can exceed the stored beam time available. Any experimenter can come in during a colliding beam run, request that valves and masks be opened and proceed to use the synchrotron radiation. The following are estimates of the time used by each of the 5 beam lines that were operational during this period.

<u>XPS</u>	<u>EXAFS</u>	<u>4° LINE</u>	<u>8° LINE</u>	<u>BIOLOGY LINE</u>
~ 200 HRS	~ 350 HRS	~ 350 HRS	~ 200 HRS	~ 200 HRS

OCTOBER 6 - DECEMBER 15, 1974

TOTAL BEAM TIME USED BY ALL SSRP EXPERIMENTERS 1025 HRS

* STORED BEAM TIME AVAILABLE TO SSRP 510 HRS

During this period the Cal Tech monochromator was not used because it was being modified at Cal Tech. The other 4 beam lines all functioned, but energies were generally too low for data taking on the XPS or EXAFS lines because of runs at 1.5 GeV and 1.85 GeV to study newly discovered particles. Our record keeping was somewhat improved for the EXAFS line because many users shared this port. Estimates of hours used on the 4 beam ports operational during this period are as follows:

<u>XPS</u>	<u>EXAFS</u>	<u>4° LINE</u>	<u>8° LINE</u>
325 HRS	225 HRS	275 HRS	200 HRS

*Stored Beam Time Available to SSRP represents the time during which beams are stored at constant energy and available for use by SSRP experimenters. It does not include time spent on filling, ramping and adjusting the beam. Nor does it include machine physics time, downtime, or access time for high energy physics users.

IV. FACILITY IMPROVEMENT PROJECTS

A. The following facility improvement projects were initiated in 1974:

1. New 4° Beam Splitter Mirror

In cooperation with groups from Lawrence Berkeley Laboratory and the U.S. Navy Michelson Laboratory at China Lake, a new platinum-plated copper mirror is being fabricated for the 4° beam line. The new mirror should have an rms surface roughness of $\lesssim 25 \text{ \AA}$, thus reflecting photons up to $\sim 1.5 \text{ keV}$. The presently used mirror has 65 \AA rms surface roughness and cuts off at $\sim 650 \text{ eV}$.

2. Protein Crystallography Experiment

Plans were made in collaboration with K. Hodgson and J. Phillips of Stanford University to construct a focusing monochromator to be used with a precession camera for structural studies of protein crystals by X-ray diffraction. The apparatus would make use of the biology beam line temporarily vacated by the Cal Tech group. This apparatus would fit into the Helium system and be compatible with all other users.

3. New Beam Line Proposal by Oak Ridge National Laboratory Group

In collaboration with C. Sparks, J. Hastings and R. Hendricks of Oak Ridge, discussions were held and a preliminary plan made for a possible new beam line to be installed to be compatible with other experiments. A fixed energy beam and a tunable beam were discussed. This group also made plans to study X-ray

fluorescence in the main beam line with a temporary installation not requiring a separate hutch and personnel protection system.

4. National Bureau of Standards Self-Contained Fluorescence Spectroscopy Experiment

R. Delattes of NBS started the design of a monochromator and detector system that would be inserted in the main beam line and operated remotely requiring only infrequent access (during filling of SPEAR). No new shutters or secondary beam area is required.

B. The following improvements were completed during 1974

1. The temporary Be foil beam line chamber was removed and replaced with the modified original chamber. This enables TV observation of the in-vacuum foils.
2. Several improvements were made on the controls and leak tightness of the He system. Movable devices inside the helium system were changed from air to He operation.
3. New ZnS screens were installed at 10.8 m and 21 m from the source points. Both now have proper cutouts to eliminate filtration of X-ray beams. Because these fixed screens become severely darkened by radiation damage in a few days of operation, movable screens were installed alongside them. These screens are put in for a few seconds at a time to check vertical beam position.

4. Personnel protection shutters on the 3 X-ray secondary beams were provided with visible indicators.
5. Stay-clear aisles were marked on the floor as emergency escape routes.
6. Coaxial video cables were installed from the control room to each of 5 experimental areas for video signals and other signals.
7. A sump pump was installed on an electrical handhole and covers installed on 2 electrical handholes.
8. The original SSRP trailer was moved alongside the new SSRP building for use as office space and drafting table space for SSRP staff and experimenters.
9. The sewage ejector pump was repaired.
10. Two types of vertical beam position sensors were installed. One is a split ion chamber in the He system, the other is a pair of photoemitter surfaces in the vacuum system.
11. The XPS He box was modified to make it compatible with future installation of the Cal Tech focusing mirror.
12. The 4° beam line area was extensively modified to:
 - a. extend sample chamber space to accommodate 2 or more simultaneous chambers with a refocusing mirror system;
 - b. redo shield wall between 4° and 0° lines to increase space for 4° beam line area, and also to create additional experimental space in the 0° beam line area;
 - c. relocate facility rack for 4° area; and

d. improve ac power distribution system for computer, bakeout, etc.

13. A photoelectron collector was installed in the mirror box as possible mirror surface condition monitor.
14. Vacuum display panels were repaired. Emergency off switches which caused accidental dumps of SPEAR beam were insulated.
15. Beam splitter mirror drives were modified to improve controls and provide for automatic withdrawal in case of overheating.
16. Drainage around SSRP building was improved.
17. A portable staircase was installed to provide better access to the upper level experimental area.
18. Five-micron thick pyrolytic graphite foils were installed in the vacuum system to absorb the UV radiation before it strikes the Be window. If successful, these will replace 65 μ thick carbon foils now in use.

C. The following items have been discussed as highly desirable future facility improvement projects.

As of January 1, 1975, only planning or preliminary design work had been done on these projects--mostly because of limitations of funding. Many of these projects are expected to be initiated during 1975.

1. 8^o Mirror Mover Improvements

Two additional motions are necessary to enable remote adjustment of the fraction of the beam subtended by the mirror and the cant angle.

2. 8° Beam Line Alignment Modules

Movable screens and viewports are needed along the 8° beam line to facilitate alignment of the mirrors.

3. New Thinner Be Window

Preliminary analysis shows that it should be possible to make a Be window that is $\sim 100 \mu$ thick which would provide useful flux down to ~ 2 keV. The present window is 500μ thick and transmission cuts off sharply at ~ 3.5 keV.

4. XPS Crystal Holder Modifications

Changes to the first crystal holder of the XPS monochromator are necessary to adjust the amount of beam intercepted so that the experiment can operate compatibly with future downstream users.

5. Position Monitor Improvements

The signal from the position monitor will be used as a reference signal to control the vertical orbit steering, automatically keeping the beam centered.

6. Development of Multi-Wire Proportional Chambers

Many SSRP programs would benefit from the availability of one- and two-dimensional multi-wire proportional chamber X-ray detectors.

7. SPEAR Status Panels

Each experimental area should have a panel which displays the status of beams in SPEAR and permits rapid and convenient contact with SPEAR operator when necessary.

8. Expand AC Switchboard

Additional ac circuits are needed for new experimenters and to provide separate lines for computers, bakeout, etc.

9. Chemical Hood

A properly vented chemical hood is needed to safely prepare samples.

10. Thin-Windowed Gas Cells

The ability to use thin windowed gas cells in the SSRP vacuum lines would facilitate new types of studies. Devices to protect the monochromators and SPEAR in the event of thin window failure must be devised. Special automatic gate valves may also have to be developed for this application.

V. PROPOSALS RECEIVED BY 1-1-75

SSRP PROPOSAL SUMMARY SHEET JANUARY 1975

Proposal #	Date Received	Institution and Experimenters Names	Title
1	6/25/74	P. H. Citrin Bell Labs	Photoelectron Experiments with Multi keV Photons
2	9/9/74	University of Washington Group	Proposed EXAFS Experiments at SSRP 9/74 - 3/75
3	9/24/74	P. Eisenberger W. E. Blumberg Bell Labs	Proposed Study of Copper-Containing Proteins by X-Ray Scattering Techniques at SLAC
4	9/24/74	P. Eisenberger P. H. Citrin Bell Labs	Proposed EXAFS Experiments to Empirically Determine Phase Shifts and Test Their Transferability
5	9/24/74	P. Eisenberger R. G. Shulman Bell Labs	Proposed Study of Iron-Containing Proteins by X-Ray Scattering Techniques at SLAC

SSRP PROPOSALS (Cont'd)

Proposal #	Date Received	Institution and Experimenters Names	Title
6	9/24/74	M. Weissbluth Stanford	Structural Studies of Biological Molecules by the EXAFS Method
7	10/1/74	R. D. Deslattes National Bureau of Standards	Fluorescence Spectroscopy
8	10/4/74	P. Eisenberger P. H. Citrin Bell Labs	Revised EXAFS Proposal on Phase Shift Study
9	10/4/74	K. Hodgson S. Doniach Stanford	Investigation of the Coordination Geometry Metalloenzymes
10	10/7/74	A. I. Bienenstock Stanford	Study of As_2Se_3 Glasses using EXAFS
11	10/11/74	I. Lindau W. E. Spicer Stanford	Proposed Ultraviolet Photo- emission Experiments on the 8^0 and 4^0 Beam Lines at SSRP October - November 1974

SSRP PROPOSALS (Cont'd)

Proposal #	Date Received	Institution and Experimenters Names	Title
12	10/17/74	D. A. Shirley U. C. - Berkeley	Investigation of the Photo-emission Cross-Section Dependence on Excitation Energy in Solids: I. Gold
13	11/7/74	Farrel Lytle Dale Sayers Ed Stern Univ. of Washington Group	Addition to Proposed EXAFS Experiments at SSRP - November - December 1974
14	11/15/74	Farrel Lytle The Boeing Co.	Investigate the Structure of Supported Catalysts using the EXAFS Technique
15	11/15/74	W. E. Stutius B. A. Huberman R. Z. Bachrach Xerox	EXAFS on Superionic Conductors
16	11/13/74	P. Lagarde, SSRP	I. Study of Simple Alkali Halides with the same Halogen Element but Different Metals II. Study of Impurities in Insulating Materials III. Study of bidimensional Crystals

SSRP PROPOSALS (Cont'd)

Proposal #	Date Received	Institution and Experimenters Names	Title
17 and 17a	12/27/74	D. Crozier Simon Fraser Univ. Vancouver, Canada	EXAFS for Structural Studies of Liquid Binary Alloy Semiconductors
18	1/8/75	M. P. Klein Lawrence Berkeley Laboratory	EXAFS in Biomolecules
19	12/27/74	Seattle Group	L-edges
20	12/27/74	Seattle Group	Fourth Row Elements
21	12/27/74	Seattle Group	Excited Core Hole Interaction
22	12/27/74	Seattle Group	Anisotropic Effects
23	12/27/74	Seattle Group	Biological Systems
23a	1/27/75	Seattle Group	Biological Systems
24	12/27/74	Seattle Group	Aluminum Alloys

VI. SUMMARY OF BEAM LINE INSTRUMENTATION DEVELOPMENT AND EXPERIMENTS

A. The following reports summarize the development of beam line instrumentation at SSRP:

1. XPS - X-ray Photoemission Spectroscopy Beam Line (I. Lindau, Stanford University)

The XPS equipment was installed and ready to run for the cycle starting May 13, 1974. Basically, the same instrumental equipment was used as for the pilot project [described in Nature, Vol. 250, p. 214 (1974)]. The monochromator design was modified so that the first stage in the double-crystal arrangement was mounted on the first floor and the second stage on the second floor (slab). The environment up to the second crystal was enclosed in He. The crystal stages were provided with remote control. Flat Si (220) surfaces were used for both crystals. The monochromator was first aligned optically with a laser beam and then tuned to the rocking curve. Continuous monitoring of the alignment was accomplished with a transmission ionization chamber.

Extensive redesign and rebuilding of the first crystal mount was done in August - September, 1974, to accommodate the Cal Tech experiment. During the fall of 1974, further improvements were done in terms of the data acquisition equipment and "in situ" sample preparation techniques. The performance of the double crystal monochromator and the electron spectrometer has been

described in the literature [Nature, 250, p. 214 (1974)] and will not be repeated here.

2. 4° Beam Line - Grazing Incidence Experiment (F. Brown, Xerox Palo Alto Research Center)

The 4° beam line was initially designed to deflect ~ 2 milliradians of synchrotron radiation into an experimental area on the main floor. Simultaneous operation with the other SSRP beam lines is possible. The wavelength range covered by the line as presently instrumented is from 400 \AA to below 10 \AA (30 to 1200 eV). The line has been implemented by the Xerox Palo Alto Research Center together with the SSRP. Special mirrors were developed and manufactured by the Michelson Laboratories, China Lake, CA.

In this line the vacuum ultraviolet and soft X-ray radiation is focussed at two degree grazing incidence. Total external reflection is employed from smooth clean metallic surfaces with high atomic number (platinum and gold). In this way an upper limit to photon energy is achieved which lies in the kilovolt range. It should be stressed that state-of-art optics is involved and a number of significant advances have been made.

A very brief description of the optical system would be that it consists of a cooled deflecting mirror M_0 (6.4m from orbit), a novel grazing incidence ultrahigh vacuum monochromator (16m from orbit) plus sample chamber optics. The entire line is continuously

evacuated with ion pumps and is compatible with the storage ring vacuum. In general, components and materials are employed which have been closely specified by the SPEAR vacuum shop. Special materials such as dry lubricated bearings, thin film filters etc. have been carefully tested by residual gas analysis or by diode desorption tests. Basically, the line is divided into regions A (SPEAR vacuum) B (monochromator and grating - separately pumped and isolated) and C (sample chamber - beyond ten micron exit slits). Vacuum requirements in region C are considerably less strict than region A allowing ease of operation from a user's point of view.

The first mirror M_0 (copper-platinum coated) has a spherical figure with 210 m radius in order to focus the point in orbit onto the horizontal entrance slit of the monochromator located 16 m from orbit. It is maintained at about 30° C by either heating or cooling using thermoelectric devices. Cooling capacity to handle the full unfocused power of synchrotron radiation from SPEAR-2 has been provided (200 watts/cm^2). This mirror is inside the primary shielding wall but can be rotated and retracted under remote control.

The monochromator has been described by Brown, et al. in the Proceedings of the IV International Conference on Vacuum Ultraviolet Physics, Hamburg, July 1974. Its novel features include

(1) scanning over an extreme range accomplished by means of toggle mechanisms and the use of a precision air bearing
(2) fixed entrance and exit beams achieved by combining a Rowland circle-Vodar principle with a rotating mirror-entrance slit combination and
(3) all metal gasket ultrahigh vacuum bellows construction. A vacuum of 2×10^{-10} Torr was achieved before finally installing the grating. It customarily operates in the mid 10^{-9} range. High resolution and reproducibility has been achieved and scans taken out to 500 eV. Moreover the instrument is of rugged and durable construction.

A special valve has been developed in order to close off the exit slit of the monochromator when changing samples or accommodating different users. This is part of an ultrahigh vacuum sampler chamber designed and built in the Physics Department at the University of Illinois. When the valve is open a number of small samples or filters can be inserted in an intense monochromatic beam right at the exit slit of the monochromator. A mirror box with sublimator and ion pump is situated just downstream of this region. The exit beam can be highly focussed at a point one meter from the exit slit by means of an adjustable toroidal mirror kinematically mounted in the mirror box. This will facilitate a variety of experiments, especially photoemission.

The four degree line was tested and successfully operated during the May and June runs of 1974. This includes the mirror box, vacuum beam line, hutch, protective circuits and monochromator with controller. Preliminary experiments were carried out. Since that time the storage ring has been down for modifications or running at very low current.

The sample chamber and mirror box are now installed (February 24, 1975) and tested. The new valve appears to work well so that this part of the system can be brought up to the atmosphere and turned around in less than 24 hours. When this is done the monochromator, separation chamber and line remain at ultrahigh vacuum.

In addition we have recently implemented a new radiation monitor which follows the decay of the storage ring beam and also produces fast pulses for trigger purposes. The small reflecting prism used in this monitor can be rotated so as to facilitate alignment of monochromator or sample chamber with a laser beam. The toroidal bent mirror is on hand for platinum evaporation and will be installed within the mirror box shortly.

3. 8° Beam Line - Normal Incidence Experiment (V. Rehn, U.S. Navy Michelson Laboratory, China Lake, CA.)

The photon flux available from the VUV beam line is determined by three factors: SPEAR emission intensity, beam line acceptance angles, and beam line transmission. The emission intensity is

essentially independent of SPEAR beam energy over the spectral range (4-70 eV) of interest here and is thus determined by the beam current. The solid angle of synchrotron radiation accepted is the product of the vertical (3.1 mr) and horizontal (3.5-5.25 mr., adjustable) acceptance angles subtended by the beam-splitting mirror M_0 (Fig. 1). The third factor, the transmission of the optical system, may be expressed as the product

$$T = R_0 R_1 R_2 R_G E_G S D_G W ,$$

where R_1 is the reflectance of the i^{th} platinum-coated optical surface, E_G is the grating blaze efficiency, S the entrance-slit function, D_G the linear dispersion of the grating, and W the exit slit width. From published values of platinum reflectance and typical values for the blaze efficiency ($E_G = 0.4$) and slit function ($S = 1$) we compute the transmission for $D_G = 8.33 \text{ \AA/mm}$ and $W = 0.2 \text{ mm}$ to be 1.8% at 10 eV. Photoemission measurements on clean copper by A. D. Baer in June 1974, assuming a photoyield of 10^{-2} , set the value at $T = 1.2\%$, the absolute flux being about 1.7×10^9 photons per second per milliamp of beam current with a 2 \AA bandpass and 4.25 mr of synchrotron radiation. The nonnormal-incidence reflections from the platinum-coated surfaces discriminate against the small fraction of light from

OPTICAL LAYOUT OF THE VUV BEAM LINE IN ELEVATION

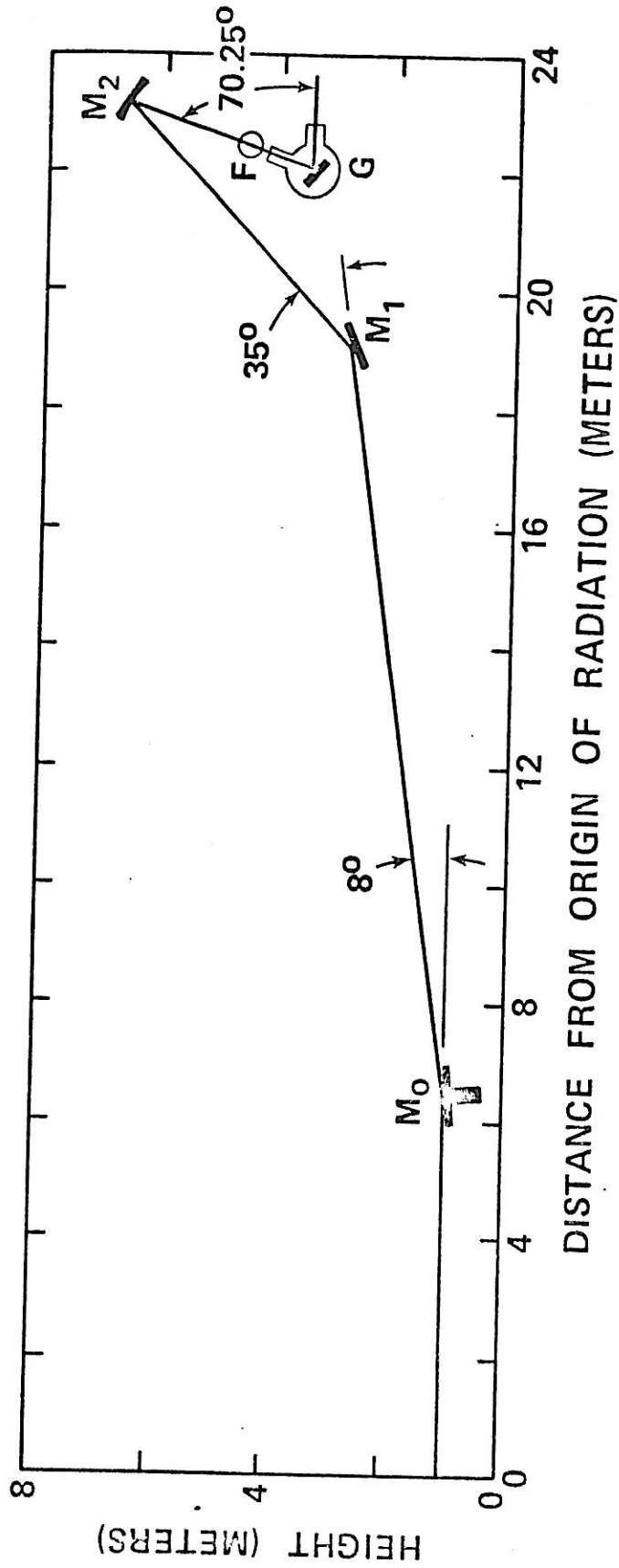


FIGURE 1.

SPEAR that is vertically polarized. This produces a polarization enhancement of a factor of 12 at the lower photon energies, resulting in a beam polarization of 97% or greater over the range of available energies. Preliminary reflectance data from platinum samples taken as a function of angle-of-incidence appear to confirm this enhancement.

Measurements of scattered light at the exit slit show that the fraction of unwanted light increases almost linearly from about 0.005 at 4 eV to about 0.02 at 20 eV. A sharp increase in scattered light occurs beyond this energy. The unwanted light results primarily from off-axis reflections of the zero order beam inside the exit arm of the monochromator. The 1/2-inch diameter light pipe detector in the reflectometer some 15 inches beyond the exit slit receives less than 1% of this scattered light.

The performance of the optical components in the line is quite close to theoretical expectations. We have measured the beam image size in the regions of the entrance slit and sample chamber where the cylindrical mirrors M_2 and M_1 focus the beam, respectively. The measured image height (Fig. 2a) of 0.18 mm at the entrance slit is less than a factor of two greater than the value predicted by the 0.097 magnification by M_2 of the vertical electron distribution in the SPEAR beam. Photographs

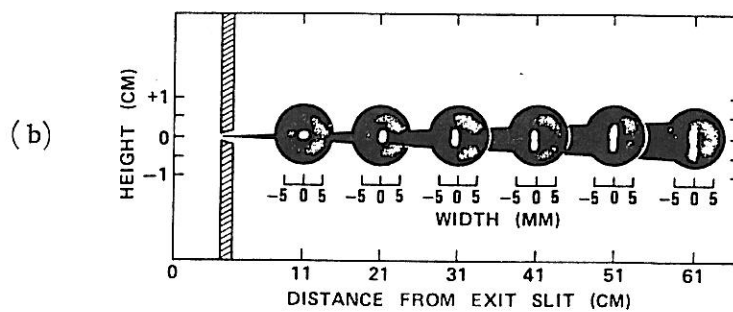
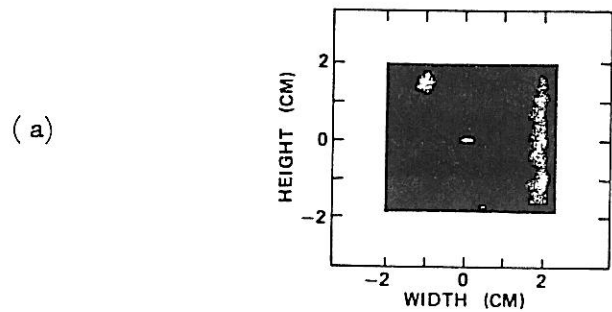


FIGURE 2.

of the beam at various distances from the exit slit (Fig. 2b) show the beam width to be in equally good agreement with values predicted from the design parameters of M_1 and the grating. The resolution of the monochromator with a grating having 1180 λ/mm and with 0.020 mm entrance and exit slit widths has been measured at the central image and at the 2536 \AA Hg line. The value of 0.20 \AA FWHM is in fortuitously good agreement with the calculated value of 0.198.

We have begun measurements of reflectance and scattering from platinum surfaces with varying microroughness and electro-reflectance from a variety of materials. Results of these measurements will be reported in the next status summary.

4. EXAFS - Extended X-ray Absorption Edge Fine Structure

Planning for an Extended X-ray Absorption Fine Structure (EXAFS) experiment began in November, 1973. After considering alternatives, the decision was made to employ a channel-cut single crystal as a monochromator. Computer-controlled stepping motors would rotate this crystal to provide for rapid variation of energy over a large range (3.5-30 keV). A small slit (1 mm high) ahead of the crystal restricts the angular spread of the incident beam and results in an energy resolution of approximately 1 eV at 10 keV.

An important feature of the design is the small displacement ($\sim 2 \text{ cm}$) of the monochromatized beam from the incident beam.

This permits the construction of a well shielded EXAFS experimental area around the monochromatic beam, with no direct view into the storage ring. Because this arrangement greatly reduces potential radiation hazards (compared to a direct beam) the secondary beam area hutch concept is possible, giving rapid and convenient experimenter-controlled access to samples and detectors.

Construction of the many parts of the apparatus took place from March, 1973, to May, 1974.

The first EXAFS spectrum was obtained within a few hours of first turning on the experiment. Then followed an intensive period of study of the properties of the detectors, the SPEAR beam, and of different methods of taking the data. For several weeks changes were made and a considerably different system evolved. Ion chambers were placed before and after a sample to simultaneously record incident and transmitted flux, permitting very accurate, low noise measurements of absorption to be made, essentially independent of the significant fluctuations in beam intensity.

An active experimental program was then followed for the remainder of the first running cycle (through July 3, 1974) and then again for the Fall SPEAR cycle. Several hundred EXAFS spectra were taken during 1974 by many groups. This includes spectra of

elements, simple compounds, amorphous materials and biological materials. Both K and L edges were measured in samples of solids, liquids and gases from room temperature down to liquid nitrogen temperature. These spectra have increased our knowledge of the EXAFS process (comparison with theory), tested usefulness for distance determination (test of transferability), demonstrated chemical and biological application, developed systematics on the chemistry of edge shifts and edge structure and made important studies in actual biological problems.

5. Biology Beam Line - Low Angle X-ray Diffraction Camera (N. Webb, California Institute of Technology, Pasadena)

Prior to constructing a low-angle X-ray diffraction camera, monochromator and focussing elements were tested for resistance to radiation damage. A fused quartz glass optical flat oriented perpendicular to the beam showed surface pits and a crack after approximately one months irradiation (40 SPEAR on-off cycles). The surface pits probably result from electric stress caused by photoelectrons, and the crack probably arose from a sharp thermal gradient in the glass caused by the occlusion of part of the beam. Gold, platinum, and nickel coated, and plain float glass mirrors were oriented at grazing angles to the beam and the reflected X-rays observed on a ZnS screen. Surface damage was apparent in a few days from the appearance of both the surface and the reflected X-ray beams.

A line-focus crystal monochromator with a logarithmic spiral curvature was then designed, installed and tested, and preliminary diffraction patterns have been obtained from specimens of muscle, collagen and nerve myelin. To eliminate harmonics of the fundamental wavelength, and to focus the line into a point image, a one meter long elliptically curved focussing mirror device has been designed and will be installed early in 1975.

Biological specimens can be situated close to the main X-ray beam line and can be manipulated safely owing to a carefully designed safety hatch system. Dark room facilities immediately next to the hutch allows X-ray photographic film development and the preparation of light sensitive specimens.

B. The following reports summarize activity on particular experiments at SSRP:

1. UPS - Ultraviolet Photoemission Experiment - 4° and 8° Beam Lines

(a) 4° Beam Line (I. Lindau, W. Spicer, Stanford University)

Experiments started on the 4° beam line Dec. 2, 1974, and proceeded up to the shutdown Dec. 16, 1974, in addition to the experimental program a series of diagnostic tests were made during this period of time.

(1) Position and direction of the light beam at the exit of the monochromator;

(2) Spectral distribution of the photo flux at the monochromator exit slit;

- (3) Yield measurements of gold to determine the throughput of the monochromator.
- a. It is crucial to know the position and direction of the light beam to align the sample relative to the energy analyzer. These parameters were determined by using a sodium salicylate layer to give fluorescence of the incident light and subsequently determine the beam positions with polaroid pictures taken outside the vacuum system.
 - b. The spectral distribution of the photo flux from the exit slit of the monochromator was determined by using a photo-multiplier tube. Since the quantum yield of sodium salicylate is constant over the entire photon energy of interest for us an accurate determination could be done in this way.
 - c. The photon flux accepted by the 4° beam line can be determined from known parameters. Intensity losses in the optical system, consisting of beam splitter and monochromator, was estimated by measurement of the photo yield from a gold sample prepared in situ (1×10^{-10} torr). With knowledge of the quantum yield of Au the photo flux at the exit slit of the monochromator was obtained. Furthermore, the energy distributions

were compared to these flux figures to ascertain that there were spurious losses in the electron spectrometer.

Photoemission measurements were performed on gold in the energy region 30-300 eV. It was thus possible to extend the photoemission measurements for the first time to the wavelength region above 100 eV. Particular emphasis was put on studying the relative cross-sections of the 5d, 5p and 4f subshells. Auger transitions involving the 5p levels and the valence band fell within the studied energy range as well. Furthermore, detailed studies of the valence band, 5d levels hybridized with a 6s, p band, were done with special emphasis on structure modulations for photon energies above 100 eV.

In addition to our proposed cross-section studies we started work on Ge. Preliminary measurements were performed for amorphous Ge, prepared in situ, and for changes in the distribution curves (valence band as well as core levels) when Ge is exposed to oxygen.

(b) 8° Beam Line (I. Lindau, W. Spicer, Stanford University)

As was the case for the 4° beam line, our work on the 8° beam line has of necessity been partially diagnostic. An estimate of the spectral distribution of the photon flux emerging from the exit slit of the monochromator was done by measuring the photo field from a Ni sample prepared in situ. There is a

drastic drop in the intensity for energies above 25 eV (about one order of magnitude) and therefore our photoemission measurements have been limited to 10-25 eV, but are planned to be extended up to 60 eV.

The performance of the beam line, including beam splitter and monochromator, was tested by photoemission from a thin Cu film prepared in situ. After satisfactory results were obtained, the measurements were continued on Nb and oxidized Nb surfaces. With the use of a signal averager, the intensity provides no problem up to 25 eV despite the fact that the storage ring was running at low beam currents at the time for the measurements.

At the end of 1974, our proposed work on the 8⁰ beam line was only in its initial stage. Part of our obtained results will be presented at the APS meeting in Denver, March - April 1975.

2. EXAFS (P. Eisenberger of Bell Laboratories with collaborators)

(a) Experiment Constructed (B. Kincaid, D. Sayers) - 1 publication

(b) Experiments Performed:

(1) Simple Molecular Gases - Test Theories (B. Kincaid) - 1 publication

(2) Demonstrations of Applications in Chemistry (Solutions) and in Biology (Porphyrin's) (B. Kincaid, K. Hodgson, S. Doniach) - 1 publication and 1 in progress

(3) Extensive Biological Measurements

a. Hemoglobin and Hemoglobin models (B. Shulman) publication in progress

b. Chemical Significance of Edge Shifts and Structures -

(B. Blumberg) data obtained; publication in progress

(4) Test of Transferability of Phase Shifts - Empirical
Determination of Distances - Model Molecular Systems
Measured (P. Citrin, B. Kincaid) - data obtained; publica-
tion in progress

(c) Experiments in Progress but Complete Data not Obtained

(1) Polarization Anisotropy Test (G. Brown)

(2) Fluorescence Method for Dilute Systems and Surface Studies
(G. Brown, M. Klein)

The development of a new fluorescent EXAFS techniques has begun
which is likely to be of great importance in studying very
dilute biological systems.

3. Resonant Raman (H. Winick, P. Platzman)

(a) Experiment Constructed

(b) Experiments Performed

(1) Low Resolution Study of Cu and Kr - 1 publication

(c) Experiments in Progress but Complete Data not Obtained

(1) High Resolution Study to Look in Resonant Region (Initial
Spectrum Successful Seen with 1eV Resolution)

4. Future Experimental Planning

(a) Compton Scattering and Simultaneous Detection of Recoiling
Electrons (B. Kincaid, P. Citrin). When Spear operates
regularly at 4 GeV this experiment should be feasible
(proposal in process of being submitted)

- (b) Some initial crude demonstration of timed Structure Studies
(B. Kincaid)

5. EXAFS (P. Lagarde, LURE at Orsay and SSRP)

- (a) Study of Simple Alkali Halides
- (b) Study of Impurities in Insulating Materials
- (c) Study of Bidimensional Crystals

Because of the few hours of available beam, I was not able to run many samples during the last session (November - December 1974). However, I took some data on KBr, NaBr, LiBr with a ratio signal/noise ratio good enough to start analysis. The EXAFS spectrum in such materials looks surprisingly very concentrated in a low energy range, compared to the one on bromine gas or copper. Moreover, the spectrum of LiBr is very similar to that of bromine ion in solution, and this may be an effect of the hydration of the sample. This must be checked by improving the way to prepare samples.

6. EXAFS - Study of As_2Se_3 Glasses Using EXAFS (S. Hunter and A. Bienenstock, Stanford University)

The ultimate goal of the research described below is to study the coordinations of network formers and modifiers in As_2Se_3 glasses using EXAFS at SSRP and classical x-ray diffraction methods. Thus far, effort has been concentrated on three areas. These have been assistance in the development of the experimental EXAFS facility at SSRP, sample preparation for EXAFS experiments and EXAFS data acquisition.

In the initial phases of the work, S. Hunter assisted in the establishment of the EXAFS measuring equipment. In particular, her help was in the assembly of the equipment as well as the development of computer programs to automate data acquisition. In that phase of the work, S. Hunter also assisted others in the acquisition of data and the testing of equipment. Some of those data complement the data that is listed below and will be used in structural analyses. Of particular importance in this regard are the runs on elemental, crystalline Cu, As and Se as well as amorphous As_2Se_3 .

In preparation for EXAFS runs, the following samples have been prepared:

CuAsSe_2 - crystalline
 Cu_3AsSe_4 - crystalline
 $\text{Cu}_{.05}(\text{As}_{.4}\text{Se}_{.6})_{.95}$ - amorphous
 $\text{Cu}_{.08}(\text{As}_{.4}\text{Se}_{.6})_{.92}$ - amorphous
 $\text{Cu}_{.10}(\text{As}_{.4}\text{Se}_{.6})_{.90}$ - amorphous
 $\text{Cu}_{.25}(\text{As}_{.4}\text{Se}_{.6})_{.75}$ - amorphous
 $\text{Zn}_{.018}(\text{As}_{.4}\text{Se}_{.6})_{.982}$ - amorphous
 $\text{Zn}_{.05}(\text{As}_{.4}\text{Se}_{.6})_{.95}$ - amorphous

EXAFS data have been obtained for the K-edges of all the constituents of the samples listed below. In general, data have been obtained with the samples at liquid nitrogen temperature. For crystalline

As_2Se_3 , data were obtained at both liquid nitrogen and room temperatures.

Samples

CuAsSe_2 - crystalline

CuAsSe_2 - amorphous (with small amount of crystallization)

Cu_3AsSe_4 - crystalline

As_2Se_3 - crystalline

$\text{Cu}_{.25}(\text{As}_{.4}\text{Se}_{.6})_{.75}$ - amorphous

In addition, there have been careful measurements of the edge positions in crystalline As, Se and Cu .

Data analysis began after the reporting period. It has been directed almost entirely towards a reanalysis of crystalline and amorphous As_2Se_3 systems. It is also our intention to focus on crystalline and amorphous Se before undertaking a ternary system.

7. XPS - Photoelectron Experiments with Multi keV Photons (P. Citrin, Bell Laboratories)

Diagnostic testing and evaluation for future development accounted for the bulk of activity during the period October 21 through November 11, 1974. Following the installation of higher transmission focusing grids, the electron energy analyzer performance was checked on campus with a He resonance lamp and was found to be satisfactory once the sample was properly aligned. A signal averager with automatic sweep control of the analyzer focusing voltage was interfaced with the apparatus. A rotating mask and quartz oscillator

assembly were designed for evaporation and monitoring inside the vacuum station. Attempts to analyze Auger and photoelectrons of widely different energies and widths using the 8 KeV photon beam proved unsuccessful. After extensive diagnosis it was concluded that the inability to align the sample was primarily responsible and that this, in turn, resulted from the lack of a suitably intense source of electrons from the sample. (Optimizing the inelastically scattered peak intensity was not sufficient). Areas for development involve improvement in analyzer detecting efficiency and sample alignment. Specifically, these include: 1) electron gun mounted along spectrometer axis, 2) more flexible sample manipulator, 3) He path up to analyzer window, 4) spherical pre-retarding grid surrounding sample (more uniform electric field), 5) larger acceptance apertures in analyser, 6) rotating analyzer axis 90° from present position (photoemission along \vec{E}), and 7) replacing double plane crystal spectrometer with a single curved crystal. Expected intensity improvements from steps 3), 5), 6), and 7) are factors of 3, 2, 2, and 20, respectively.

8. EXAFS - Investigation of the Coordination of Geometry of Metalloenzymes (S. Cramer, K. Hodgson, Stanford University)

We have been engaged in the synthesis and EXAFS study of model compounds for three different metalloproteins: hemoglobin, hemocyanin, and nitrogenase. For hemoglobin our models have been a series of Fe(II) and Fe(III) porphyrins, with and without axial

ligands. Our hemocyanin work has centered around two classes of compounds: first, copper-amine complexes of simple structure, and second, bridged copper complexes such as copper acetate. Finally, some simple molybdenum compounds have been prepared and will hopefully be run soon.

9. EXAFS - Structural Studies of Biological Molecules by the EXAFS Method (M. Weissbluth, C. Yuen, Stanford University)
 - (a) Sample holders for solid and liquid samples have been designed and constructed.
 - (b) Sample holder for operation at low temperatures have been designed and constructed.
 - (c) An analysis has been made of optimum sample thickness.
 - (d) An analysis has been made of optimum detector parameters.
 - (e) EXAFS data have been obtained on chlorohemin in powder form at room temperature.
 - (f) EXAFS data have been obtained on bovine hemoglobin in powder form at room temperature.
 - (g) An analysis has been made of the EXAFS spectrum of copper etioporphyrin (to be published in J. Chem. Phys.).
10. Fluorescence Spectroscopy (R. D. Deslattes, National Bureau of Standards)

Principal activities to date have been:

 - (a) Experiment planning and proposal generation
 - (b) Instrumentation design

(c) Negotiation about beam time and hardware ownership

Item (a) is complete, the rest are still in progress.

No utilization of radiation has taken place.

11. EXAFS - Experiments of the University of Washington Group:

The following report gives a brief summary of the experiments carried out by the University of Washington group at SSRP during 1974. Members of this group include Farrel Lytle, Dale Sayers and Edward Stern and beginning in the fall of 1974, a graduate student, Bruce Bunker. Other collaborators for specific experiments will be noted.

The experiments included:

- (a) K shell spectra of elements - K shell EXAFS were measured by us on Fe, Co, Ni, Cu, Zn, Ge, As, Se and Mo in order to understand systematic variations of EXAFS as a function of atomic number and to serve as standards for more complex samples. Further measurements of 4th row elements and lighter elements are planned.
- (b) L edge spectra of elements - L edge spectra were measured for Ta, Au and W. L edge spectra have many interesting features which have not been systematically studied until now. Understanding these spectra will also permit the study of more systems using EXAFS. Further measurements are planned for 1975 and some publications are in preparation.

- (c) K shell spectra of simple compounds - The EXAFS of the iso-electronic and isostructural series Ge, GaAs, ZnSe and CuBr was measured in order to study the effects of ionicity of the chemical bond on EXAFS.
- (d) Biological Materials.
- (1) Cu Etioporphyrin I (with M. Weissbluth, P. Pianetta)
Previous measurements on these systems were repeated at SSRP to determine the capabilities of EXAFS of measuring certain structural properties of metalloproteins. This work has been reported and further publications are in preparation.
 - (2) Other Fe metalloproteins (with J. Herriott, W. Parsons).
Preliminary measurements on some Fe compounds (FeS, ferrocene) were made in preparation for measuring some metalloproteins containing Fe which is bonding to S (e.g. rubedoxin, ferredoxin, etc.). Further measurements are planned.
- (e) Metal-Insulator Compounds (with F. Holtzberg) - Preliminary measurements of the Sm L edges and, where possible, the Y and As Kedges were made as a function of temperature for SmS, SmAs, SmAsS, SmYS. The quality of the data was poor and further measurements are planned. Other systems will also be measured (e.g. various oxides of vanadium).
- (f) Amorphous semiconductors (with S. Hunter, A. Bienenstock).
Measurements on crystalline and amorphous polymorphs of

GeSe and As_2Se_3 were made to measure the local structure (primarily first neighbor) in the amorphous materials. This data is currently being analyzed and some further measurements are planned.

12. XPS (I. Lindau, SSRP)

Measurements have been performed with high resolution the 4f levels of Au and Pt, and of the 3rd levels of Ag. These results were reported at the 4th International Conference on Radiation Physics in Hamburg in July, 1974, and are included in the Proceedings from that Conference. Since October 1974, no XPS experiments have been done due to low beam energies (< 2.4 GeV).

13. 4^0 Beam Line (D. Shirley, LBL, University of California, Berkeley)

The LBL Group under Professor David Shirley did preliminary photoemission experiments on the 4^0 line in October-November 1974. They studied photoemission from metallic gold.

VII. USERS GROUP MEETING

FIRST ANNUAL

SSRP USER GROUP MEETING

October 24-25, 1974

Stanford Linear Accelerator Center
Stanford, California 94305

PROGRAM
ABSTRACTS
LIST OF ATTENDEES

P R O G R A M

STANFORD SYNCHROTRON RADIATION PROJECT USERS GROUP MEETING

OCTOBER 24 - 25, 1974

SLAC AUDITORIUM

NOTE: Scheduled Times include 5 minutes of discussion.

Thursday, October 24

8:15 A.M. REGISTRATION

9:00 A.M. Opening: S. Doniach, Director, SSRP

9:15 A.M. Welcome: J. Ballam, Associate Director, SLAC

SESSION I - Chairman, W. Walker

9:30 A.M. "Soft X-ray Beam Line at SSRP," R. Z. Bachrach, F. C. Brown, S. B. Hagstrom.

10:00 A.M. "The VUV Beam Line at SSRP: Design, Performance and the Experimental Program,"
A.D. Baer, T. Donovan, V.O. Jones, D.S. Kyser, V. Rehn, J.L. Stanford.

10:30 A.M. COFFEE BREAK

10:45 A.M. "Prospects for Surface Studies Using Synchrotron Radiation," W. Spicer.

11:10 A.M. "X-ray and UV-Photo-electron Spectroscopy at SSRP," E. I. Lindau, P. Pianetta,
W. Spicer.

11:35 A.M. TOUR OF SSRP

12:45 P.M. LUNCHEON - SLAC Cafeteria

SESSION II - Chairman: D. Shirley

2:00 P.M. "SPEAR Operation and Improvement Program," J. M. Paterson.

2:20 P.M. "Future Plans at SSRP," S. Doniach, H. Winick.

2:40 P.M. "University of Wisconsin Synchrotron Radiation Center - The First Six Years,"
E. Rowe.

3:05 P.M. "Photo-emission Spectroscopy Studies of Bulk and Surface States Using Synchrotron Radiation," D. Eastman.

3:30 P.M. COFFEE BREAK

3:45 P.M. "Review of Synchrotron Radiation Research at DESY," C. Kunz.

4:10 P.M. "Review of Synchrotron Radiation Research at Orsay," P. Dhez.

4:35 P.M. "Biological X-ray Diffraction at SSRP," N. Webb.

5:15 P.M. TOUR OF SLAC

7:00 P.M. CONFERENCE DINNER - MING'S - Private Dining Room - No-Host Bar

7:45 P.M. Dinner Served

Friday, October 25

SESSION III - Chairman: P. Platzman

9:00 A.M. Extended X-ray Absorption Fine Structure (EXAFS):

- A. "X-ray Absorption Measurements at SSRP," P. Eisenberger and B. Kincaid.
- B. "Analysis of EXAFS Data," F. Lytle, D. Sayers, E. Stern.
- C. "Studies of Amorphous Materials Using EXAFS," A. Bienenstock, S. Hunter.
- D. "Structural Studies of Biological Molecules by the EXAFS Method," M. Weissbluth.

10:40 A.M. COFFEE BREAK

11:00 A.M. "Daresbury Laboratory - The Present Programme Using the Synchrotron and Progress with the Dedicated Storage Ring Project," I. Munro, V. Suller.

11:25 A.M. "Single Crystal Protein Structure Studies Using Synchrotron Radiation," K. Hodgson, L. Jensen, J. Phillips, A. Wlodawer.

11:50 A.M. "Mossbauer Measurements Using Synchrotron Radiation," S. Ruby.

12:15 A.M. "Design of an X-ray Light Pipe at 6 keV," R. Pound and W. Vetterling.

12:45 P.M. LUNCHEON - SLAC Cafeteria

SESSION IV - Chairman: S. Doniach

2:00 P.M. The Possibilities for Synchrotron Radiation Research With PEP - The Proposed LBL/SLAC 15 GeV Storage Ring:

- A. "PEP Description and Schedule," J. Rees.
- B. "A PEP Synchrotron Radiation Facility," H. Winick.
- C. "Use of High Energy Synchrotron Radiation at the Cornell 12 GeV Synchrotron," V. Kostroun.
- D. Panel Discussion on Scientific Uses of PEP Synchrotron Radiation:
Panel: R. Deslattes, P. Eisenberger, K. Hodgson, E. Rowe, H. Winick.

3:45 P.M. COFFEE BREAK

ABSTRACTS

- J. Ballam, "Summary for Talk to SSRP User Conference"
- R. Z. Bachrach, et al., "Soft X-ray Beam Line at SSRP"
- V. Rehn, et al., "The VUV Beam Line at SSRP: Design, Performance and the Experimental Program"
- W. Spicer, "Prospects for Surface Studies Using Synchrotron Radiation"
- E. I. Lindau, et al., "X-ray and UV-Photo-electron Spectroscopy at SSRP"
- J. M. Paterson, "SPEAR Operation and Improvement Program"
- S. Doniach, et al., "Experimental Program at SSRP and Future Plans"
- E. Rowe, "University of Wisconsin Synchrotron Radiation Center - The First Six Years"
- D. Eastman, "Photo-emission Spectroscopy Studies of Bulk and Surface States Using Synchrotron Radiation"
- C. Kunz, "Review of Synchrotron Radiation Research at DESY"
- P. Dhez, "Review of Synchrotron Radiation Research at Orsay"
- N. Webb, "Biological X-ray Diffraction at SSRP"
- B. Kincaid, et al., "X-ray Absorption Measurements at SSRP"
- D. Sayers, et al., "Analysis of EXAFS Data"
- A. Bienenstock, et al., "Studies of Amorphous Materials Using EXAFS"
- M. Weissbluth, "Structural Studies of Biological Molecules by the EXAFS Method"
- I. Munro, et al., "Daresbury Laboratory - The Present Programme Using the Synchrotron and Progress with the Dedicated Storage Ring Project"
- K. Hodgson, et al., "Single Crystal Protein Structure Studies Using Synchrotron Radiation"
- S. Ruby, "Mossbauer Measurements Using Synchrotron Radiation"
- R. Pound, et al., "Design of an X-ray Light Pipe at 6 keV"
- J. Rees, "PEP Description and Schedule"
- H. Winick, "A PEP Synchrotron Radiation Facility"
- V. Kostroun, "Use of High Energy Synchrotron Radiation at the Cornell 12 GeV Synchrotron"
- P. Pianetta, "Summary of PEP Panel Discussion"

LIST OF ATTENDEES - STANFORD SYNCHROTRON RADIATION PROJECT USERS GROUP MEETING OCT 24-25/74

<u>NAME</u>	<u>AFFILIATION</u>	<u>NAME</u>	<u>AFFILIATION</u>
R. Bachrach	Xerox Res Ctr	D. Judge	U. of S. Calif.
A.D. Baer	Michelson Lab	J. Jurów	SLAC
J. Ballam	SLAC	B. Kincaid	Stanford U
I.P. Batra	IBM-San Jose	W. Kirk	SLAC
B.W. Batterman	Cornell U	M.P. Klein	LBL-Berkeley
J. Berkowitz	Argonne Nat'l Lab	V. Kostroun	Cornell U
W. Blumberg	Bell Telephone Lab	S. Kowalczyk	LBL-Berkeley
W.A. Brown	Lockheed Res Lab	C. Kunz	DESY-Hamburg
T.J. Chuang	IBM-San Jose	D. Kyser	Michelson Lab
P. Citrin	Bell Telephone Lab	P. Lagarde	LURE-Orsay/SSRP
S. Cramer	Stanford U	L. Ley	UC Berkeley
B. Craseman	U of Oregon	I. Lindau	SSRP
C. Depautex	LURE-Orsay	F. Lytle	Boeing Aerospace Co.
R. Deslattes	Nat'l Bur of Standards	R. Martin	Xerox-San Jose
P. Dhez	LURE-Orsay	G. Mazenko	Stanford U
S. Doniach	Stanford U - SSRP	C.A. McDowell	U of British Columbia
T. Donovan	Michelson Lab	F. McFeely	UC Berkeley
D. Eastman	IBM-Yorktown Heights	I. Miller	IBM-San Jose
P. Eisenberger	Bell Telephone Lab	H. Morawitz	IBM-San Jose
E.L. Garwin	SLAC	F. Mueller	Argonne Nat'l Lab
D.C. Gates	Stanford Res Inst.	I.H. Munro	Daresbury - UK.
R. Gaxiola	SSRP	W. Oosterhuis	Nat'l Sci Foundation
T. Geballe	Stanford U	J. Paterson	SLAC
R. Godwin	Los Alamos Sci Lab	M. Perlman	Brookhaven Nat'l Lab
A. Golde	SSRP	J. Phillips	Stanford U
P. Grant	IBM-San Jose	P. Pianetta	Stanford U - SSRP
R. Greene	IBM-San Jose	P. Platzman	Bell Telephone Lab
J. Harris	SLAC	R.V. Pound	Harvard U
T. Hayes	Xerox Res Ctr	J. Rees	SLAC
R. Hazard	Pfizer Chem Co.	V. Rehn	Michelson Lab
F. Herman	IBM-San Jose	E. Rowe	Phys Sci Lab/Wisconsin
E. Hoyt	SLAC	S. Ruby	Argonne Nat'l Lab
S. Hunter	Stanford U	A. Salop	Stanford Res Inst
F. Johnson	SSRP	B. Salsburg	SSRP
V. Jones	Michelson Lab	J. Samson	U of Nebraska

<u>NAME</u>	<u>AFFILIATION</u>
D. Sayers	U of Washington
P. Sen	Xerox Res Ctr
T. Sharp	Lockheed Res Lab
S. Sheng	Stanford U
D. Shirley	UC Berkeley
R. Shulman	Bell Telephone Lab
M. Skibowski	DESY-Hamburg/SSRP
M. Sogard	Cornell U/SLAC
W. Spicer	Stanford U - SSRP
J. Stanford	Michelson Lab
E. Stern	U. of Washington
J. Stevenson	Georgia Inst of Tech
V. Suller	Daresbury - UK
P. Temple	Michelson Lab
Y. Tsai	SLAC
W. Walker	UC Santa Barbara
J. Weaver	Phys. Sci Lab/Wisconsin
N. Webb	Calif Inst of Tech
M. Weissbluth	Stanford U
H. Winick	SSRP
A. Wlodawer	Stanford U
C. Worthington	Carnegie-Mellon U
C. Yuen	Stanford U
G. Zimmerer	DESY-Hamburg

VIII. VISITORS TO SSRP:

In addition to the attendees of the Users Group Meeting, the following persons, among others, have visited SSRP:

<u>Name</u>	<u>Affiliation</u>
S. Ruby	Argonne National Laboratory, Argonne, Illinois
S. Wolff	Atomic Energy Commission, Washington, D. C.
R. Chasman	Brookhaven National Laboratory, Upton, New York
H. Wegner	Brookhaven National Laboratory, Upton, New York
J. Wolff	National Institutes of Health, Bethesda, Maryland
A. Salop	Stanford Research Institute, Menlo Park, California
Physics Class	John Woolman School, Nevada City, California
M. Klein	U of California, Lawrence Berkeley Laboratory Berkeley, California
Y. Petroff	U of California, Berkeley, California
D. Eisenberg	U of California, Los Angeles, California
T. Schuster	U of Connecticut, Storrs, Connecticut
P. Coppens	The State University of New York at Buffalo, New York
P. Csonka	U of Oregon, Eugene Oregon
M. Boudart	Stanford University - Chemical Engineering
Executive Committee, Center for Materials Research	Stanford University
E. Shooter	Stanford University Medical Center
L. Jensen	U of Washington, Seattle, Washington

<u>Name</u>	<u>Affiliation</u>
M. Berry	U of Wisconsin, Physical Sciences Laboratory, Stoughton
A. Ashmore	Daresbury Synch. Rad. Lab., Daresbury, England
W. Hayes	Oxford University, Oxford, England
C. Depautex	LURE, Orsay, France
Y. Farges	LURE, Orsay, France
R. Haensel	DESY, Hamburg, Germany
M. Iannuzzi	Lab. Nazionali di Frascati, Italy
G. Torelli	Inst. Naz. di Fisica Nucl., Pisa, Italy
A. Luccio	Univ. di Pisa, Italy
L. Borooskii	Baikon Inst. of Metallurgy, Moscow, USSR
I. Kalechitz	USSR
V. Kazanski	USSR
O. Krylov	USSR
Y. Yermakov	USSR

IX. PUBLICATIONS BASED ON WORK DONE AT SSRP

1. "Beryllium vs Carbon for Low Energy Synchrotron Radiation Attenuation," P. Pianetta, Stanford University, with E. W. Hoyt, SLAC, Stanford University, Stanford, CA, SLAC-TN-73-10, September, 1973.
2. "X-ray Photoemission Spectroscopy at the SPEAR Storage Ring," I. Lindau with P. Pianetta and S. Doniach, Stanford University, Stanford, CA, Proc. Thirty-fourth Annual Conf. on Phys. Electronics, Murray Hill, N. J., Feb. 25-27, 1974.
3. "Extended X-ray Absorption Fine Structure," C. A. Ashley, SSRP Report No. 74/01, March 1974.
4. "The Stanford Synchrotron Radiation Project (SSRP)," H. Winick, Stanford University, Stanford, CA, Proc. of International Accel. Conf., SLAC, May 1974; also, SLAC-PUB-1439, June 1974.
5. "Theory of Extended X-ray Absorption Edge Fine Structure (EXAFS) in Crystalline Solids," C. A. Ashley and S. Doniach, Dept. of App. Phys. and Stanford Synchrotron Radiation Project, W. W. Hansen Laboratories of Physics, Stanford University, Stanford CA, June 1974; also, Phys. Rev. B, 11, No. 4, 15 February 1975.
6. "X-ray Photoemission Spectroscopy at the Stanford Synchrotron Radiation Project," I. Lindau with P. Pianetta, S. Doniach and W. E. Spicer, Nature 250, 214, July 19, 1974.
7. "Electron Spectroscopy Using Synchrotron Radiation," I. Lindau, S. B. M. Hagstrom, Stanford University and Xerox Palo Alto Research Center, resp., Proc. 1974 Pac. Conf. on Chem. and Spectroscopy, San Francisco, CA (1974).

8. "Theory of High Energy Excitations in Solids," S. Doniach, Proc. 24th Nobel Symposium, 223 (1974).
9. "Core Excitons in the Alkali Halides," W. Schiefley and F. C. Brown, Dept. of Phys. and Mat. Res. Lab., University of Illinois, Urbana, and S. T. Pantelides, App. Phys. Dept. and Stanford Synchrotron Radiation Project, Stanford University, Proc. of the IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 396.
10. "The Stanford Synchrotron Radiation Project (SSRP)," H. Winick, SSRP, Stanford University, Proc. of the IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 776.
11. "Design of the Ultraviolet Synchrotron Radiation Beam Line at SPEAR," V. Rehn, A. D. Baer, J. L. Stanford, D. S. Kyser and V. O. Jones, Michelson Laboratory, Proc. of the IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 780.
12. "Low-Scatter Metal Mirrors for High-Intensity Synchrotron Radiation at SPEAR," J. L. Stanford, V. Rehn, D. S. Kyser and V. O. Jones, Michelson Laboratory, and A. Klugman, Northrop Res. and Tech. Ctr., Hawthorne, CA, Proc. of the IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 783.
13. "An Ultrahigh Vacuum Monochromator for Synchrotron Radiation," F. C. Brown, R. Z. Bachrach, S. B. M. Hagstrom, Xerox Palo Alto Res. Ctr., Proc. of IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 785.
14. "Time of Flight Angularly Resolved Photoelectron Energy Spectroscopy Using Synchrotron Radiation," R. Z. Bachrach, S. B. M. Hagstrom and F. C. Brown, Xerox Palo Alto Res. Ctr., Proc. IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 795.

15. "X-ray Photoemission with Use of Synchrotron Radiation," I. Lindau P. Pianetta, S. Doniach and W. E. Spicer, SSRP, Stanford University, Proc. IV International Conf. on Vac. Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 805. (Invited Paper)
16. "EXAFS Measurements at SPEAR," P. Eisenberger, Bell Laboratories, B. Kincaid, S. Hunter, Stanford University, D. Sayers, E. A. Stern, University of Washington, and F. Lytle of Boeing Aerospace Co., Proc. IV International Conf. on Ultraviolet Rad. Phys., Hamburg, July 22-26, 1974, p. 806.
17. "Synchrotron Radiation as a Probe of Condensed Matter," S. Doniach, Stanford University, Stanford, CA, Bull. of APS, Jan. (1975), p. 51.
18. "X-ray Experiments Using Synchrotron Radiation at a Source," P. Eisenberger, Bell Laboratories, Bull. of APS, Jan. (1975), p. 51.
19. "Fast X-ray Diffraction Studies on Biological Structures Using Synchrotron Radiation," N. Webb, Cal. Inst. of Tech., Pasadena, Bull. of APS, January (1975), p. 52.
20. "Extended X-ray Absorption Fine Structure of Iron in Solutions of Hemes and Heme Proteins," R. G. Shulman, P. Eisenberger, W. E. Blumberg and S. Ogawa, Bell Laboratories, and S. Doniach and B. Kincaid, SSRP, Stanford University, Abstracts, 19th Ann. Meeting of Biophysical Soc., Philadelphia, February 18-24, 1975, Vol. 15, p. 288a.
21. "Extended X-ray Absorption Fine Structure Analysis. A New Probe of Chemical and Electronic Structure of Copper Proteins," W. E. Blumberg and P. Eisenberger, Bell Laboratories, and S. Doniach and B. Kincaid, Stanford University, Abstracts, 19th Ann. Meeting of Biophysical Soc., Philadelphia, February 18-24, 1975, Vol. 15, p. 292a.

22. "Analysis of X-ray Photoabsorption Data for Molecules of Biological Interest," S. Doniach, K. O. Hodgson, B. Kincaid and P. Eisenberger, Stanford Synchrotron Radiation Project, Stanford University, Bull. Am. Phys. Soc., 20, No. 3, p. 317, March (1975).
23. "A Short Range Probe for Investigating Metalloprotein Structures: Fourier Analysis of the Extended X-ray Absorption Fine Structure," D. E. Sayers, University of Washington, F. W. Lytle, Boeing Aerospace Co., and M. Weissbluth and P. Pianetta, Stanford University," Bull. Am. Phys. Soc., 20, No. 3, p. 317 (1975).
24. "Ultraviolet Photoemission Studies of O_2 , CO, C_2H_2 and C_2H_4 Chemisorbed on Copper," K. Yu, I. Lindau, P. Pianetta and W. E. Spicer, Stanford University, Bull. Am. Phys. Soc. 20, No. 3, p. 359 (1975).
25. "Vacuum Ultraviolet Synchrotron Radiation From SPEAR," Victor Rehn, A. D. Baer, D. S. Kyser, J. L. Stanford, Michelson Laboratory, K. Yu, I. Lindau, P. Pianetta, W. E. Spicer, Stanford University, Bull. Am. Phys. Soc. 20, No. 3, p. 419 (1975).
26. "Photoemission Studies of Gold in the Wavelength Region 30-300 eV," I. Lindau, P. Pianetta, K. Yu and W. E. Spicer, Stanford University, Bull. Am. Phys. Soc. 20, No. 3, p. 475 (1975).
27. "Observation of s-d Final State Mixing in the $L_{II,III}$ EXAFS of Ta and Au," D. E. Sayers, E. A. Stern, University of Washington, and F. W. Lytle, Boeing Aerospace Co., Bull. Am. Phys. Soc., 20, No. 3, p. 488 (1975).
28. "High Resolution Soft X-ray Spectroscopy at the $L_{2,3}$ Edge of Doped Silicon," R. Z. Bachrach, F. C. Brown and M. Skibowski, Xerox Palo Alto Research Ctr., Bull. Am. Phys. Soc., 20, No. 3, p. 488 (1975).

29. "Correlation of X-ray K-edge Chemical Shift and Bond Ionicity," F. W. Lytle, Boeing Aerospace Co., Bull Am. Phys. Soc., 20, p. 488 (1975).
30. "K Edge X-ray Absorption Fine Structure of Br₂ and GeCl₄ Measured with Synchrotron Radiation," B. M. Kincaid and P. M. Eisenberger, Stanford Synchrotron Radiation Project, Stanford University, and Bell Laboratories, Bull., Am. Phys. Soc., 20, p. 489 (1975).
31. "Application of X-ray Photoabsorption Spectroscopy to the Study of Molecules of Biological Interest," S. Doniach, K. Hodgson, P. Eisenberger, B. Kincaid, Proc. of the National Acad. of Sci., 72, to be published (1975).
32. "Synchrotron Radiation Studies of the K-edge Photoabsorption Spectroscopy of Kr, Br₂, and GeCl₄: A Comparison of Theory and Experiment," B. Kincaid and P. Eisenberger, of Stanford University, Stanford, CA, and Bell Laboratories, Murray Hill, N. J., resp., accepted for publication by Phys. Rev. L.
33. "Synchrotron Radiation Measurements of K-edge Photoabsorption Spectroscopy of Copper, Nickel and Germanium," B. Kincaid, Stanford University, Stanford, CA, P. Eisenberger, Bell Laboratories, Murray Hill, N. J. and D. Sayers, Univ. of Washington, Seattle, WA, to be submitted to Phys. Rev.
34. "Photoelectron Spectroscopy by Time-of-Flight Technique Using Synchrotron Radiation," R. Z. Bachrach, F. C. Brown and S. B. M. Hagstrom, Xerox Palo Alto Research Center, J. Vac. Sci. Technol., 12, No. 1, Jan/Feb 1975.

Abstracts listed under Section VII, USERS GROUP MEETING, held at SLAC, Stanford University, October 24-25, 1974, are more fully set forth below:

1. "Soft X-Ray Beam Line at SSRP," R. Z. Bachrach, F. C. Brown and S. B. M. Hagstrom, Xerox Palo Alto Research Center, Palo Alto, CA 94304.
 2. "The XUV Beam Line at SSRP: Design, Performance and the Experimental Program," A. D. Baer, T. Donovan, V. O. Jones, D. S. Kyser, V. Rehn and J. L. Stanford, Michelson Laboratory, Naval Weapons Center, China Lake, CA 93555.
 3. "Prospects for Surfaces Studies Using Synchrotron Radiation," W. E. Spicer, Stanford University.
 4. "UV - and X-Ray Photoemission Spectroscopy at SSRP," I. Lindau, P. Pianetta, S. Doniach and W. E. Spicer, Stanford University.
 5. "Experimental Program at SSRP and Future Plans," S. Doniach and H. Winick, SSRP, Stanford University.
 6. "Biological X-Ray Diffraction at SSRP," N. Webb, J. Baldeschwieler, R. Stroud, S. Samson of California Institute of Technology, Pasadena, CA, and I. Matsubara, University of Tokyo, Japan.
 7. "X-Ray Absorption Measurements at SSRP," B. Kincaid, P. Eisenberger, D. Sayers, Stanford University, Bell Laboratories at Murray Hill, and University of Washington, Seattle, respectively.
 8. "Analysis of EXAFS Data," D. E. Sayers, E. A. Stern, F. W. Lytle, University of Washington at Seattle and Boeing Aerospace Company, Seattle, Washington.
- "Studies of Amorphous Materials Using EXAFS," A. Bienenstock and S. Hunter, Stanford University

9. "Structural Studies of Biological Molecules by EXAFS Method,"
M. Weissbluth, Stanford University.
10. "Protein Crystallography," K. Hodgson, L. Jensen, J. Phillips, A.
Wlodawer, all from Stanford University except L. Jensen from the
University of Washington, Seattle.
11. "A PEP Synchrotron Radiation Facility," H. Winick, SSRP.
"Summary of the PEP Panel Discussion," P. Pianetta, Stanford University.

APPENDIX

A. D. Baer,[‡] R. Gaxiola, A. Golde, F. Johnson, B. Salsburg, H. Winick,
W. W. Hansen Laboratories of Physics;
M. Baldwin,^{††} N. Dean, J. Harris, E. Hoyt, B. Humphrey, J. Jurow,
R. Melen, J. Miljan,^{‡‡} and G. Warren, SLAC
Stanford University
Stanford, California 94305

Summary

The Stanford Synchrotron Radiation Project (SSRP) is now in full operation as a national facility utilizing the intense ultraviolet and x-radiation from the storage ring SPEAR at the Stanford Linear Accelerator Center (SLAC). The experimenter-operated facility is designed to maximize access to, and utilization of the radiation by 5 or more simultaneous users, within the limits of parasitic operation on a high energy colliding beam storage ring. A novel experimenter-controlled personnel protection system permits independent access to each of 5 experimental areas. A vacuum monitoring and control system protects the storage ring vacuum from contamination, rising pressure, or catastrophic failure. The design and operation characteristics of these control systems and of the beam position monitoring and control system, vacuum system and thin beryllium windows are presented.

Introduction

The SSRP has been in operation since May, 1974, as a national facility for UV and X-ray research using synchrotron radiation from the storage ring SPEAR at SLAC. SSRP has been funded since June, 1973, by the National Science Foundation and is administered by the W. W. Hansen Laboratories of Physics at Stanford University. Contributions to the facility have also been made by the U. S. Navy Michelson Laboratory at China Lake, California, the Xerox Corporation and the Bell Telephone Laboratories. SLAC exercises control over radiation safety and sets vacuum standards for experiments which connect on-line to the SPEAR vacuum system.

The research program includes studies of UV and X-ray photo-emission, extended x-ray absorption edge fine structure, low angle x-ray diffraction, protein crystallography, UV reflectivity, and x-ray Raman scattering. Details are given in the 1974 SSRP Users Group Meeting, obtainable on request from R. Dannemiller at SSRP, and in the 1974 Hamburg Conference on VUV Radiation Physics. Professor S. Doniach of Stanford is the Project Director and Professor W. Spicer of Stanford is the Consulting Director.

The facility is built around a single beam port on the SPEAR vacuum system, accepting 11.5 mrad of synchrotron radiation which was initially split among 5 simultaneous users. As a secondary program on SPEAR, the facility was designed to permit operation of 5 or more simultaneous synchrotron radiation experiments during SPEAR colliding beam runs with maximum protection for

[†]Supported by National Science Foundation Grant Number DMR73-07692 A02, in cooperation with the Stanford Linear Accelerator Center and the Energy Research Development Agency.

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the SPEAR vacuum system and minimum involvement of SPEAR and SLAC operations personnel. Particular attention to 3 elements proved vital in achieving this goal. These are:

1. Vacuum system and vacuum interlocks.
2. Radiation shielding and personnel protection system.
3. Orbit monitoring and control.

In this report we present a general description of the facility with particular emphasis on the above 3 areas.

Plan of the Synchrotron Radiation Facility

A prefabricated steel building 12 m wide, 24 m long and 7.3 m high has been constructed adjacent to SPEAR as shown in Fig. 1 and 2. The building is well insulated and temperature controlled and has a thick (30 cm) concrete floor for stability. Vibration sources (such as compressors) are located outside the building and decoupled from the building and floor. A 6 m extension of the building is planned to accommodate a second beam run.

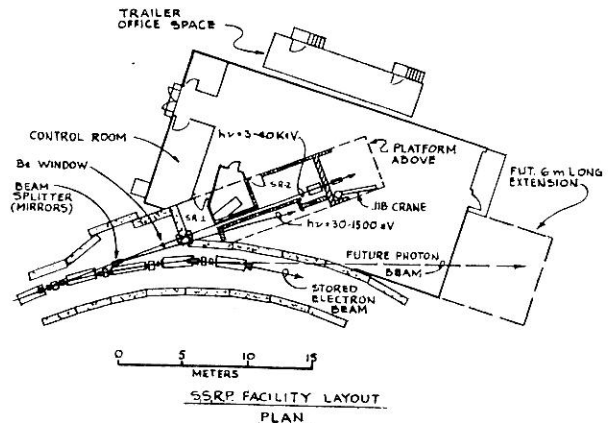


Fig. 1

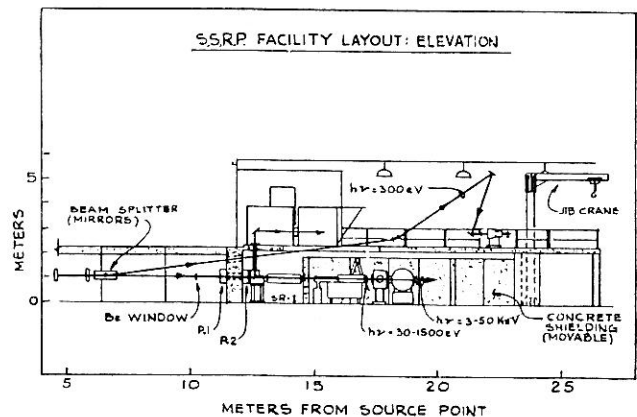


Fig. 2

About 11.5 mrad of synchrotron radiation, corresponding to 15 cm of curved path in a SPEAR bending magnet, emerges tangentially into a high vacuum pipe. The spectrum of this radiation corresponding to SPEAR stored beam energies of 1.5 to 4.5 GeV is shown in Fig. 3. Single beam currents of 50 mA at 3 GeV and 100 mA at 3.8 GeV are anticipated during single bunch colliding beam operation. These currents are limited by beam-beam interactions. In single beam multi-bunch mode of operation larger currents (250 mA and 3 GeV and 500 mA at 2.5 GeV) should be possible. The SPEAR RF frequency is 358 MHz which is the 280th harmonic of the orbital frequency. In single beam runs ~ 200 bunches have been filled. The one bunch mode offers unique timing capabilities since the pulse duration is $\approx .3$ nsec and repeats at 1.28 MHz. Other reports give more information about SPEAR¹ and the synchrotron radiation it produces.²

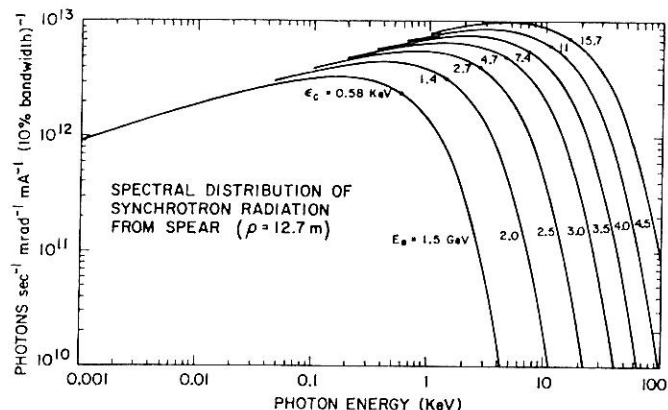


Fig. 3

The horizontal fan of radiation is split 3 ways by reflection at grazing incidence on 2 ultra smooth, platinum-plated copper blocks³ placed 6.5 m from the source point. These mirrors may be remotely inserted and adjusted by experimenters during operation. Five or more simultaneous experiments share the radiation as shown in Fig. 4.

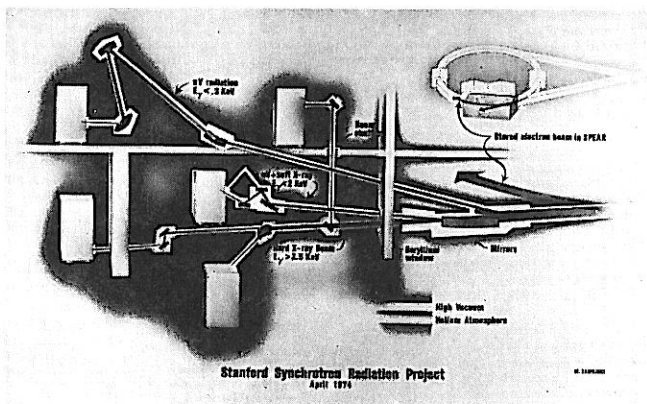


Fig. 4

One of the mirrors intercepts the outer 2 mrad of radiation at a horizontal grazing angle of incidence of 2° resulting in a horizontally focused 4° deflected beam. This mirror has an rms roughness of 65 \AA and reflects photons up to about 600 eV. A smoother mirror is now in fabrication which should extend this energy to ≈ 1500 eV.

A plane mirror with an rms surface roughness of 30 \AA intercepts the inner 3 to 6 mrad at a vertical grazing angle of incidence of 4° . The resulting beam rises at 8° and contains photons up to ~ 300 eV. Custom built high vacuum grating monochromators are connected to these lines. The mirrors are cooled thermo-electrically to enable operation with up to 25 W of synchrotron radiation per mrad.

The central part of the beam contains - to 10 mrad of radiation (depending on insertion of mirrors) which is not deflected by mirrors. This radiation proceeds down the high vacuum beam pipe and passes through a pair of $75 \mu\text{m}$ thick water-cooled carbon foils which absorb the UV and soft x-ray part of the spectrum. The radiation then leaves the vacuum system 10.5 m from the source point through a pair of $250 \mu\text{m}$ water-cooled beryllium windows.⁴ This foil and window system begins to transmit at about 3.5 keV and reaches 50% transmission at ~ 4.5 keV. It is planned to improve this transmission by replacing the $75 \mu\text{m}$ foils with $5 \mu\text{m}$ pyrolytic graphite foils. A pair of such foils is now undergoing test in the beam run. In addition, a new beryllium window is planned with a total thickness of $\approx 100 \mu\text{m}$. In combination with the pyrolytic graphite foils, this window should provide significant transmission down to 2 keV.

After emerging from the SPEAR vacuum system the x-rays travel in a helium atmosphere into a shielded area in which several crystal monochromators are installed. The helium system is carefully sealed and monitored to keep a high concentration of helium. For convenience the helium system is divided into several sections by 5μ thick kapton windows. Each section has an independent helium input flow meter and output bubbler.

An elevated concrete slab 4.5 m wide, 12 m long and 2.4 m above the floor serves as a second level for installing experimental apparatus. Its thickness (20 cm) is adequate to provide shielding from the main beam line.

Monochromatic x-ray beams and the rising 8° beam line vacuum system penetrate this slab as shown in Fig. 2 and 4. Electrical services, compressed air, and helium and water services are installed at several locations along the perimeter of the slab serving experimenters on both levels. A jib crane is used to bring heavy equipment to the upper level. Vacuum controls, radiation protection controls and signals to and from the SPEAR and SLAC control rooms are centralized in an adjacent control room.

Vacuum Systems and Vacuum Interlocks

The vacuum system is built to SLAC specifications⁵ and is all metal and bakeable. The central beam pipe extends to 10.5 m from the source, terminating at the beryllium window assembly within the SPEAR tunnel. The 4° and 8° beam runs continue in vacuum in the synchrotron radiation building and extend to 16 m and 23 m from the source point.

Four all metal, high vacuum gate valves isolate the beam runs from each other and from the SPEAR vacuum system. Water-cooled masks assure that synchrotron radiation strikes only water-cooled surfaces and 2 movable water-cooled absorbers may be remotely inserted to block the radiation. Four 110 l/sec triode ion pumps are used on the main beam line with additional pumps on the 4° and 8° beam lines.

All components of the vacuum system were chemically cleaned and baked to $\sim 200^\circ \text{C}$ prior to installation. Careful backfilling and purging with dry nitrogen is used during the assembly and servicing of the vacuum system. The system has not been baked since installation, but

it has been backfilled to dry nitrogen several times. The base pressure is 2×10^{-9} torr and rises to 8×10^{-9} torr with 50 mA of electrons stored at 3.0 GeV.

Ionization gauges and fast sensors⁶ are used to detect leaks and desorption diodes⁷ sense contamination. These devices are monitored by a vacuum control system which causes valves to close automatically in the event of vacuum problems. Fast isolation from SPEAR is provided by a vane which closes in 30 msec.⁸ Under certain conditions (e.g., water-cooling failure) the SPEAR beam is also dumped. Fig. 5 gives a block diagram of the vacuum control system.

The vacuum system and vacuum control system have functioned well since operation was started in May, 1974. No significant leaks or contamination problems have been observed.

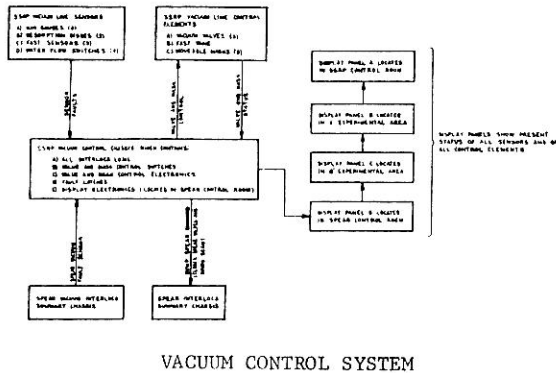


Fig. 5

Radiation Shielding and Personnel Protection System

To maximize accessibility to the radiation, shielding and a personnel protection interlock system were designed to permit access close to experimental equipment during all phases of SPEAR operation (filling, storing and dumping of beam). Concrete, lead and steel are used in sufficient thickness to guarantee that the highest possible radiation levels in occupied areas under worst case accident conditions are < 25 rad/h. Radiation monitors are set to dump stored beams and stop injection when radiation levels in occupied areas exceed 100 mrad/h.⁹ A permanent magnet at 5.5 m from the source deflects charged particles vertically so they cannot pass small vertical collimators and enter the SSRP building.

Protection against synchrotron radiation exposure is provided by thinner shielding (such as 1/8" steel sheets) adequate to attenuate the highest energy x-rays expected.

Access to the primary beam line area (along a tangent to the stored beam) is controlled by the SLAC operator in a standard manner used at SLAC. A pair of heavy shutters, each with redundant microswitches, must be inserted to block the beam before access is obtained. The SLAC operator logs the names of persons entering, observes the access on TV, and releases a key to open the door to the primary radiation area. All wiring to limit switches and key banks are in conduit used only for personnel protection circuits with the fewest possible cross connections within the system. Status panels are of the standard SLAC design. Telephone relays are used throughout and all circuits are hard wired.

Beam splitting mirrors and diffracting crystals placed in the primary beam generate secondary beams which enter areas from which there is no direct view of the

stored electron beam. Each of these secondary beams passes through a pair of shutters and into a secondary beam experimental area in which most experimental equipment is located. Wherever possible shutter operation is made directly visible to give experimenters confirmation of proper operation. Secondary beam experimental areas are called hutches. Free space inside a hutch is inadequate for a person to be inside and still close the door. The problem thus becomes that of assuring radiation safety while providing convenient access to the interior of the hutch for purposes of setup and adjustment of instruments.

Early in the planning of SSRP decisions were made in conjunction with the SLAC Radiation Committee to implement a self-monitored radiation area access control program. That is, each experimenter is permitted access to his hutch by means of his own control and interlock panel without requiring further permission from outside operators and independently of the condition of other synchrotron radiation secondary beam runs or of the SPEAR ring.

The Hutch Control and Personnel Protection Panel was designed on the basis of this requirement. This unit affords the experimenter the following control:

1. Beam Stopper Open/Close.
2. Ion Chamber Reset. Parallel-plate transmission ion chambers downstream of shutters are used in x-ray beams (which are not enclosed in vacuum systems) to insure that shutters have blocked the radiation.
3. Hutch Key Release (under safe conditions).

In addition, there is an On-Line/Off-Line keyswitch, the key to which is kept by SLAC Health Physics. In normal operation (On-Line) all interlocks are activated. When, from time to time, a hutch is disassembled this switch is turned to Off-Line after precautions are taken, and in this state only the Beam Stoppers remain as active interlocks.

The Hutch Panel receives inputs from the Stoppers (IN or OUT of beam) from the Hutch Doors (OPEN or CLOSED) and from the Ion Chamber inside the hutch (OK or RADIATION ALARM). In addition, it keeps track of whether or not the hutch key is retained in the Solenoid Key Release Unit (a part of the Hutch Control Panel).

With this assemblage of status information from these external and internal sources, the Hutch Panel forms 2 separate and redundant interlock chains which, when violated (from, for example, Hutch Door being OPEN with radiation present or from several other possibly hazardous configurations) dump the stored beam and stop injection.

Similarly, the input status information is processed into a control signal which allows the experimenter to obtain the key to open his hutch door under safe conditions. If any of a number of unsafe conditions occur after this key is released, the Dump SPEAR interlock is tripped and the source of radiation eliminated.

The philosophy of design of the Hutch Panel embodies safety through redundancy. Relay logic (24 V) has been used throughout. Fail-Safety has been achieved by creating interlock violations from any of the following: loss of power, uncabing, blowing of fuses, malfunctioning of single switches (e.g., Hutch Door switches). Also, connectors are recessed so that electrical bypassing ("buggering") of interlocks is difficult.

The accompanying figures portray the system in 2

hierarchical levels. Fig. 6 shows a schematized block diagram of the SSRP experimental areas and associated devices. Fig. 7 depicts the functional logic and status blocks which comprise the interlocking of the Hutch Control and Personnel Protection Panel, the heart of the self-monitored radiation access system. Fig. 8 shows the corresponding control block diagram.

This system has been in operation since May, 1974, and has performed as expected.

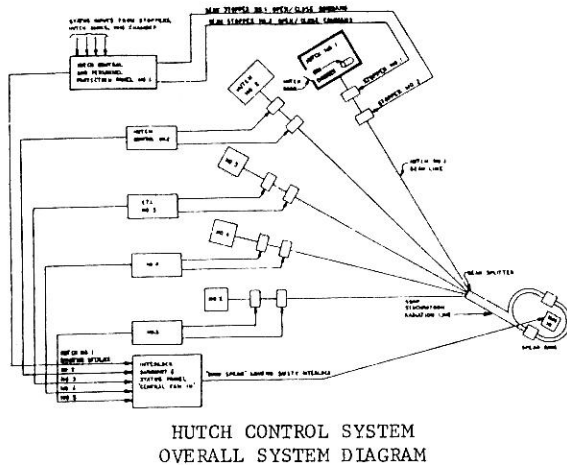


Fig. 6

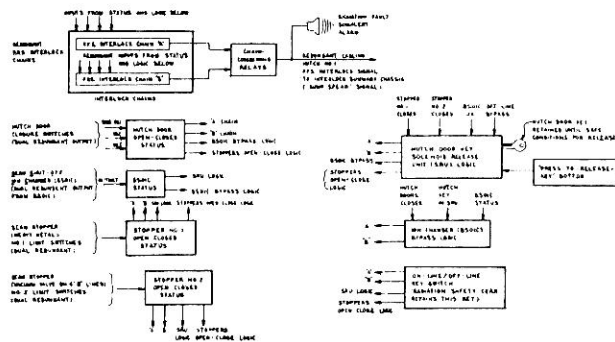
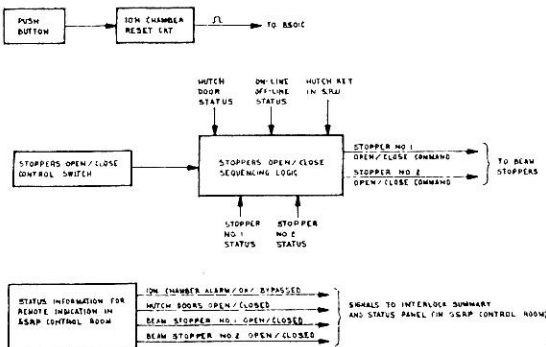


Fig. 7



HUTCH CONTROL SYSTEM SINGLE HUTCH CONTROL BLOCK DIAGRAM

Fig. 8

Orbit Monitoring and Control

The SPEAR beam is normally maintained within ± 3 mm of the nominal central orbit. In the radial direction this has been found to be adequate for position tolerance of the synchrotron radiation beam. In the vertical direction, however, only a small fraction of this is tolerable. The vertical opening angle of the synchrotron

radiation ($\sim \frac{mc^2}{E}$) is $\sim .2$ mrad. Some experiments collimate to 1 mm at 20 m from the source point. A vertical orbit distortion of a fraction of 1 mm can result in a displacement of several mm at the location of an experiment because of the angles associated with orbit motion. Thus it has been found necessary to reproduce the position of the synchrotron radiation source point to a fraction of 1 mm.

This is accomplished by powering a pair of trim coils which provide equal horizontal dipole fields. These coils are located in quadrupole magnets which are 6.55 m upstream and 8.75 m downstream of the synchrotron radiation source point. Since they are approximately 180° apart in the phase of the vertical betatron oscillation these coils produce a local beam bump with only a small residual ($\sim 5\%$ of the peak local distortion) around the rest of the ring.

At present the SPEAR operator centers the synchrotron radiation beam by TV observation of its location on an aligned screen located 21 m from the source point. Position monitors are now under development which will produce an electrical signal proportional to the vertical beam displacement. A feedback system on the power supply controlling the beam bump will then keep the beam centered automatically.

Since the synchrotron radiation beam is simply and accurately positioned, all experiments merely align their equipment to accept a beam at the height of the SPEAR median plane. No further adjustments are necessary.

Acknowledgments

The successful operation of SSRP and the speed with which it was designed and constructed is attributable in large measure to the excellent cooperation and support services provided by SLAC.

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