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SPECTROMETER SYSTEM FOR INVESTIGATION OF BIOLOGICAL MOLECULES WITH SYNCHROTRON RADIATION AT WAVELENGTHS GREATER THAN 125 nm.*

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SPECTROMETER SYSTEM FOR INVESTIGATION OF BIOLOGICAL MOLECULES WITH SYNCHROTRON RADIATION AT WAVELENGTHS GREATER THAN 125 nm.*

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The SUPERB[1] experiment at the SURF II storage ring uses synchrotron radiation as a source of UV photons to measure circular dichroism (CD) of biological molecules. Conventional CD instruments are limited by the lack of stable laboratory continuum sources capable of providing large photon fluxes shortward of 200nm. Synchrotron radiation overcomes these limitations and enables one to extend CD measurements down to the limit of the window transmission cutoff, which is 125 nm for CaF_2 used in the present configuration. Some molecules of biophysical interest, DNA and proteins for example, exhibit large CD effects below 200 nm, which reflect specific molecular conformations. CD is a sensitive probe of molecular structure and enables us to learn how these molecules behave under various external conditions. In the future we plan to increase the measurement capabilities of SUPERB to include magnetic circular dichroism, fluorescence spectroscopy, and fluorescence lifetime measurements.

The SUPERB facility is presently operating on beamline 6 at SURF II, located at the National Bureau of Standards in Gaithersburg, Maryland. Its physical configuration (see Fig. 1) is determined by a number of constraints: (1) it must interface directly with the UHV conditions of the storage ring; (2) the entire optical path must be evacuated to eliminate absorption by atmospheric gases; (3) it uses a commercially available vacuum monochromator; and (4) it must fit into the available space on beamline 6 at SURF II, sandwiched in between beamline 5 and the ring magnet housing. To meet these requirements, we divided the vacuum chamber into three parts. The first chamber is connected directly to the storage ring. It is maintained at a clean UHV by a CTI cryopump system with a 1000 ℓ/sec pumping speed. The chamber contains a focusing mirror assembly described below. The UHV chamber is separated from the monochromator by a CaF_2 window. The monochromator comprises the middle vacuum chamber section, which is maintained at high

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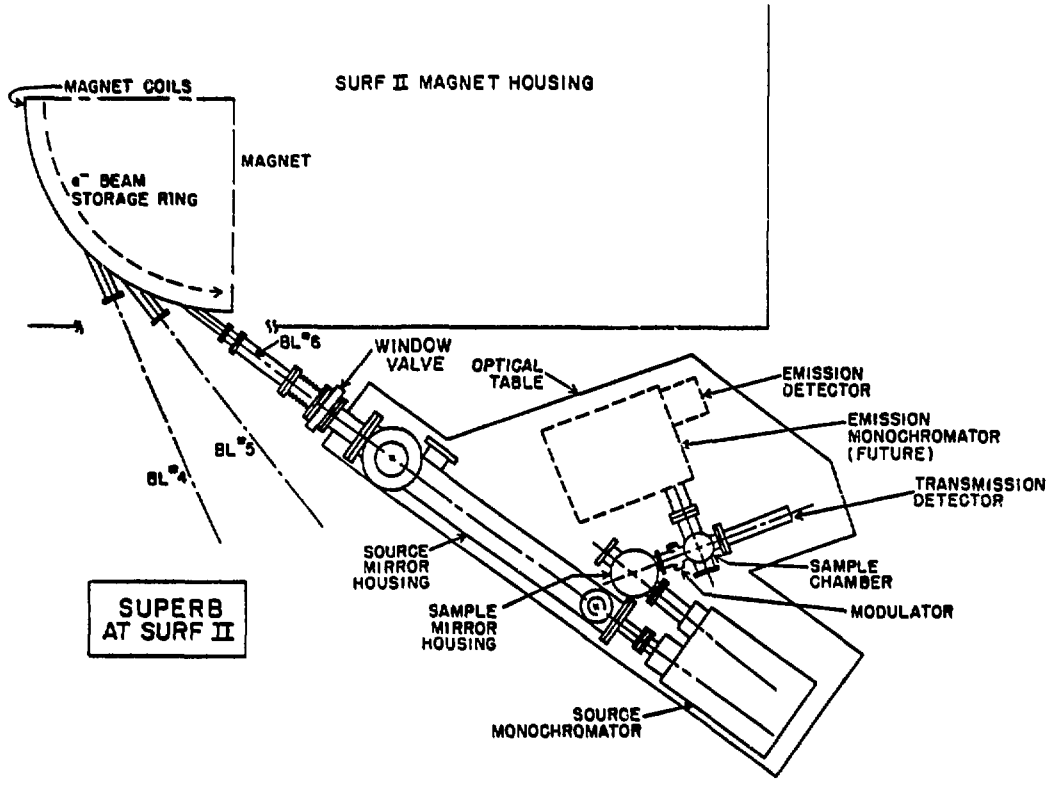

Fig. 1. Schematic plan view of SUPERB at the SURF II storage ring at the National Bureau of Standards, Gaithersburg, Maryland

vacuum by another cryopump. The monochromator is isolated from the sample chamber by another CaF_2 window, allowing the sample chamber volume to be cycled rapidly between atmosphere and roughing line vacuum. Only a modest vacuum (10^{-3} Torr) is necessary in the sample chamber to eliminate absorption by molecular oxygen.

The UHV chamber connected directly to the storage ring contains a focusing mirror assembly with two functions: if focusses a reduced image of the source onto the entrance aperture of the monochromator, and it intercepts the primary photon beam at normal incidence, effectively filtering out photons with energies greater than about 10 eV. The filtering action is necessary to prevent radiation-induced damage to the CaF_2 crystal window at the entrance to the monochromator, resulting in formation of color centers and reduction in UV transmission.[2,3] The mirror assembly is composed of two spherical mirror pieces (halves cut from a single 12.7 cm diameter, 150 cm focal length mirror) coated with Al + MgF_2. The mirrors are arranged so that the incident beam strikes the first mirror at near-normal incidence and is reflected back toward the source, where it is reflected from the second mirror and then focussed onto the monochromator. The total demagnification is a factor of 1.8, which is sufficient to collect most of the energy with

1 mm wide slits from a source that is 1.8 mm wide by 0.08 mm high. No external adjustment of the mirrors is possible through the vacuum chamber, other than by moving the entire chamber assembly within the freedom allowed by the front-end bellows. We have found that long-term image stability on the entrance aperture is excellent, no obvious movement noted over several backfill and pumpout cycles of the UHV chamber.

The monochromator is a Minutemen model 305MV with 0.5 meter focal length. It is mounted in its standard configuration with vertical slits, the plane of dispersion being horizontal. Most synchrotron monochromators are oriented with horizontal slits, but for ease of simple handling, for simplicity in mounting the cryopump, and because of our moderate resolution requirements (1-2 nm), we are able to use the monochromator in its normal configuration. A tuning fork chopper is mounted just outside the exit slit, but within the monochromator vacuum system, to modulate the signal for emission spectroscopy and to provide an AC signal for a photomultiplier gain control circuit.

The optical elements in the sample chamber vacuum section consist of a polarizer, a 30° angle-of-incidence toroidal mirror, a CaF_2 photoelastic modulator, a sample holder, and a detector. The toroidal mirror focusses the exit beam into the sample volume at unity magnification, while bending the beam away from the UHV chamber. This configuration allows adequate workspace around the sample chamber to add on future support equipment, such as fluorescence and scattering detectors, and a second monochromator to permit emission spectroscopy and lifetime measurements. The sample chamber vacuum system is designed for rapid sample changes and ease of operation. The limiting factor at present is the speed of the mechanical pump used to evacuate the chamber. It requires about ten minutes to backfill with dry nitrogen, change the sample, and reevacuate to the ten micron Hg range. We are changing to a sorption pump system for better vacuum and faster pumpdown.

We plan to operate SUPERB at SURF II until completion of the VUV ring at the NSLS at Brookhaven National Laboratory, scheduled for mid-1981. The NSLS will provide about two orders of magnitude greater flux than the SURF II facility. The physical plan of the NSLS installation will be similar to the existing plan, with the exception that the folding mirror optics will be modified to bring the beam out at a right angle from the initial direction and to erect the image, making it match the vertical orientation of the entrance slit. A precision adjustment mechanism will be necessary for the focusing mirror to allow repositioning of the image on the entrance slit to select for the time-resolved beam position.[4]

Preliminary results of CD spectra of DNA and selected proteins using the instrument at SURF II indicate that the quality of single-scan spectra compares favorably with spectra from conventional vacuum CD machines.

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1. SUPERB = Synchrotron Ultraviolet Project for Experimental Research in Biophysics.
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4. L. N. Blumberg, "Vertical Kicker for Fluorescent Decay Experiments in the NSLS VUV Ring", BNL Informal Report 26856, September, 1979.

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