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UNIVERSAL SPECTROMETER I

ANDREW KEVEY

March 1964

BROOKHAVEN NATIONAL LABORATORY
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UPTON, NEW YORK**

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ABSTRACT

The Universal Spectrometer is a general purpose machine to be used for neutron diffraction experiments. Its versatility allows single-crystal, powder, or polarized beam studies, and it can be converted into a 3-crystal machine for inelastic scattering. This report describes both the operational and the design aspects of the instrument.

UNIVERSAL SPECTROMETER I

INTRODUCTION

As soon as the decision was made to build the Brookhaven High Flux Beam Reactor (HFBR), the need arose for several neutron spectrometers to be used at this new reactor. For experiments having similar requirements, a project was initiated in 1961, sponsored jointly by the Physics and Chemistry Departments, to design and build an instrument which could accommodate a wide variety of experiments with a minimum amount of modification. The result of this endeavor is the Universal Spectrometer I (US I).

It is hoped that the HFBR will furnish a flux of usable thermal neutrons at least 50 times as high

as that of the Brookhaven Graphite Research Reactor (BGRR). The experimenter can benefit from this increased flux in a variety of ways: by reducing the time required for an experiment by a factor of about 50, or by increasing the resolution, and/or by using smaller crystals without increasing the data collection time beyond that of the BGRR. The higher potentials of the HFBR and the increased flexibility of the US I will give the experimenter a powerful tool in the field of neutron diffraction studies.

Major consideration was given to the facilitation of fabrication, since BNL needed 6 machines. As a result castings were used rather extensively. At last count 15 machines of the US I design were

Figure 1. The Universal Spectrometer I as assembled in the shop.

either completed or under construction. Figure 1 shows the instrument as assembled in the shop, and Figure 2 gives two over-all views of the machine as it will appear at Hole No. 5 of the HFBR.

MONOCHROMATOR PLUG AND SHIELD

The purpose of the monochromator plug is to provide, by means of Bragg reflection from a suitable monochromator crystal, a monoenergetic neutron beam that has a known and variable wavelength over the greatest practical range, and that is accurately and reproducibly incident on the specimen. The most commonly used monochromator crystals are lead, copper, and germanium, the last having the advantage of eliminating the $\lambda/2$ component, which, though only a small percent of the total diffracted beam, may complicate the interpretation of the diffraction pattern. The crystal is held in a goniometer which allows two-dimensional orientation, i.e., rocking in a vertical plane and translation in the horizontal plane, by means of control rods operated from outside the monochromator plug. The goniometer is in an inner cylinder which can be rotated about the vertical axis with respect to an outer cylinder for initial alignment of the crystal and is locked when the position of the crystal is optimized. In order to maintain the 1:2 angular relationship between the crystal and the shield exit collimator movements, an angle divider is connected between the monochromator plug outer cylinder and the shield so that for any movement of the crystal the exit collimator will be automatically oriented to let the monochromatic beam through. The angle divider consists of a set of sturdy planetary gears with an accuracy of $\approx 0.01^\circ$ and can be operated through 360° of arc.

The cavity inside the monochromator plug is 12 in. in diameter to accommodate a magnet for polarized beam studies or a large single crystal in reflection. The cavity can be illuminated by 3 low voltage bulbs to allow visual inspection of the crystal through the exit collimator and to assist in the optical alignment. Nonmagnetic stainless steel is used for all monochromator plugs where the possibility of polarized beam experiments is likely.

When the incident beam of neutrons hits the monochromator crystal, only a small percent of this mixed radiation, namely thermal neutrons having a narrow wavelength spread, is reflected and subsequently used in the neutron diffraction

Figure 2. The US I as it will appear at Hole No. 5 of the HFBR.

measurements. The rest of the beam must be absorbed by a massive shield enveloping the monochromator cavity to protect both the operator and the detecting apparatus from the main beam of neutrons and γ rays. The shape of the shield approximates a sphere with a central cavity for the crystal. Proceeding outward radially, first there is a $\frac{1}{4}$ -in. boral layer to absorb most of the incident thermal neutrons. The next layer is lead to capture the power γ rays. The thickness of lead varies, with about 7 in. in the direct beam path, 3 in. on top and bottom, and 2 in. against back scattering. The rest of the sphere is filled with pure commercial paraffin in order to slow down neutrons above thermal velocities, which are subsequently absorbed by another $\frac{1}{4}$ -in.-thick boral layer just inside the outer shell. The 1-in.-thick iron shell serves both as a container for the paraffin and as a shield against capture γ rays, a by-product of captured thermal neutrons in the boral layer. The container was made of a one-piece Meehanite casting with both lead and paraffin poured into their respective compartments.

The shield has two openings in the horizontal plane. The larger, which is $2\frac{1}{2}$ in. high and covers an arc of 140° , is for the entrance of the beam from the reactor. About 100° of this opening is plugged by movable pie-shaped segments leaving a 40° opening for the beam to enter the monochromator cavity. The segments are made of Masonite, lead, and boral layers, the Masonite substituting for paraffin. They can easily be shifted in the shield in order to permit entry of the beam with the shield in any position within its operating range. The other horizontal opening is a $1\frac{3}{4} \times 2\frac{1}{2}$ -in. rectangular opening to let the monochromatic beam pass through the shield. This opening is suitable for housing a $1\frac{1}{4} \times 2 \times 24$ -in.-long collimator, or it may be enlarged to hold permanent magnets and rf coils for polarized beam studies.

The total angular range through which the monochromatic beam can be taken out is limited by the 140° opening in the shield. The relationship of this range to beam zero, however, is determined by the position of the exit collimator with respect to the 140° opening. Two options are of-

Figure 3. Simplified cross section of diffractometer.

ferred here. One gives an 80° to -60° , the other a 120° to -20° maximum take-off angle, to the left and right respectively from beam zero. If required, both exit openings can be provided; with one of them plugged by removable shielding. In this way a total range of 120° to -60° can be achieved by shifting the exit opening plug. Since the shield is symmetrical about its horizontal centerline, changing the maximum take-off angle from left to right or vice versa can be accomplished by turning the shield over. Simple rigs are provided to achieve this easily. This arrangement makes it possible to take out as long a wavelength as required, limited only by the cut-off point of the monochromator crystal, and also to reduce interference with neighboring experiments. The 2-ft-thick solid shielding in the direct beam path may easily be increased, if the need arises, by securing

Figure 4. Sample shapes and orientations.

Figure 5. Geometrical relationships for diffraction.

extra shielding blocks onto the machined outside wall of the shield. The present weight of shield and monochromator is ≈ 6 tons.

A stationary shield called the saddle envelops the shield over an arc of 180° and butts against the reactor face. It has in its center a rectangular opening large enough to let the beam through. This opening is lined with lead and boral, and the rest of the saddle is made of Masonite for good structural rigidity. The actual size and shape of each saddle varies, depending on the distance between reactor face and shield and on the geometry of the beam port opening and of the reactor face.

In order to orient the shield in the required position a sturdy rotary pedestal was required. For this purpose surplus 40-mm gunmounts were obtained from the Navy at very low cost to the Laboratory and incorporated into the design with minor changes. These gunmounts have a dead load capacity of 18 tons and a recoil capacity of 116,000 ft-lb. These are well above present requirements but serve as insurance for future expansions in terms of increased shield weight and cantilever arm moment, which, with the rapidly changing experimental requirements, may soon be needed. The pedestal and shield are rotated at a speed of $16^\circ/\text{min}$ by a $\frac{1}{2}$ -hp, 220/440-volt ac reduction motor. A $1\frac{1}{2}$ -in.-thick base plate and six adjusting screws are provided for leveling the whole assembly. A mechanical locking arrangement is used to secure the pedestal in any given orientation against accidental moving. The orientation of the shield can be determined by a dial and vernier arrangement, reading in minutes of arc. The dial is mounted on top of the shield, and the vernier is fastened to the stationary saddle.

THE DIFFRACTOMETER

The function of the diffractometer is to vary and measure the angular relationship between the monochromatic incident beam, the sample, and the detector. A simplified cross section of the unit is shown in Figure 3. The sample is mounted on an x-ray goniometer head on top of a vertical shaft. Figure 4 shows the various crystal shapes and orientations. The detector is mounted on a horizontal arm which can be rotated in a horizontal plane about the sample. The standard method of operation is by step scanning. In this operation the detector is fixed at an angle with respect to the monochromatic beam that satisfies Bragg's law,

$n\lambda = 2d \sin\theta$, and the diffracted neutrons are counted for a definite period. See Figure 5 for the basic relationships. The length of this period is usually the time required for a constant number of neutrons to strike the sample as determined by a monitor counting channel. After the count is completed and recorded the detector is moved by a small angular increment and the process is repeated. This operation is continued until the desired angular range has been covered.

In most cases of step scanning operations, the geometrical relation shown in Figure 5 has to be maintained. Thus, as the scanning proceeds and the detector advances by a certain increment, the sample must rotate by half this amount. This necessitates a 2:1 coupling between the detector and sample drives. The main drive consists of a 14-rpm Bodine motor with an optional 10:1 or 1:1 drive unit. A worm gear drive reduces this by a ratio of 360:1 for both the main table that supports the sample shaft and the detector arm. As indicated before there is a 2:1 step-up from the main table drive to the detector arm drive. This gives an optional 1.4°/min or 14°/min rotation for the main sample table and double that amount for the detector arm drive. These speeds are necessary for stepping and slewing when operated under automatic programmed control. For fast adjustment of the detector arm a separate 56-rpm Bodine motor drive is available to rotate the arm at 56°/min. Changing of drive speeds is achieved by means of electromagnetic clutches and brakes. The angular positions of the main crystal table and the detector arm can be read in minutes on their respective verniers, which rotate relative to a stationary main scale (see Figure 3). For readings in 0.01°, dials are mounted on the main drive. The continuous recording and repeating of any angular position is achieved by means of a Coleman digitizer coupled to the detector arm drive.

For step scanning operation cams are provided on the main drive shafts which actuate microswitches to stop the drive at every 0.1° change in detector position. By means of two adjustable limit switches the range of the arm movement can be conveniently determined. At times repeated fast scanning of an angular region is preferred to one slow scan for averaging the results of scans in the forward and reverse directions, or to average the effects of other time variables such as neutron flux (for constant time operation only) or temperature of sample crystal. This repeated scanning opera-

tion can be achieved by the use of two reversing switches, alternately moving the arm in a forward or reverse direction between the limits of the desired angular range.

In the study of a single-crystal sample, the crystal has to be oriented so that the set of scattering vectors to be examined lie in the horizontal plane. The geometrical relationship given in Figure 5 must also be satisfied so that the scattering vector bisects the angle between the incident beam and the detector. In the case of a powder sample, since it is composed of many small randomly oriented crystallites, some will have their scattering vectors in the proper direction for the detector.

The orientation of the single crystal, before step scanning can take place, is achieved by means of a separate crystal drive that can rotate the crystal shaft with respect to the main crystal table. A 9.3-rpm Bodine motor through an optional 10:1 or 1:1 reduction unit and a 100:1 worm gear drive rotates the crystal shaft at 33.5°/min or 3.35°/min. A Coleman digitizer is coupled to the worm shaft to record the angular position of the crystal. A cam and microswitch arrangement, similar to that of the main drive, can stop the drive at 1/20° intervals.

In single-crystal studies, the initial sample orientation does not follow the 2:1 relation between detector and sample movements; instead the crystal has to be moved in a non-uniform manner. The required positions vary with the type of crystal examined and with its vertical orientation. Plans are under way to utilize punched tape input for fully automatic programming of initial single-crystal orientation.

The accuracy of the angular settings for all drives was kept to a maximum by cutting down backlash between mating gears. All gears were held within 0.00025 in. of total composite error as measured by "True Blue Gear Tapes," and all gear centers were adjusted for minimum backlash without binding. As a result all angular adjustments can be repeated within <0.01°. Concentricity of the crystal shaft was held within ±0.0015 in. by close machining tolerances on the affected parts and by proper positioning of runouts on the main Timken bearings. The diffractometer arm is made of aluminum sand casting in all cases where it supports the detector. In the case of the 3-crystal machine the arm can be modified to support an analyser crystal with an arrangement similar to but simpler than that of the sample crystal. This additional load and torque on the diffrac-

tometer arm necessitates a cast steel construction for greater rigidity. Both aluminum and steel castings use a common pattern.

The diffractometer is equipped with a table some 11 in. below the sample centerline for the mounting of accessories such as a magnet or cryostat. The table can support a dead weight of 1500 lb and can be coupled to the crystal drive or to the main table drive, or, as a third alternative, may be made stationary with respect to either drive.

An alternative method of driving the diffractometer is by means of dc stepping motors, whereby the rate of advancement for arm and crystal table is determined by the time rate of dc pulses fed into the motor. This arrangement utilizes only one motor (Slo Syn No. 250) for both stepping and fast slewing and does not require cams to stop the drive for step scanning. It needs, however, a frequency source (logic pulses) and a translator to convert the logic pulses into motor pulses. The stepping motors can be installed by removing the Bodine motors and the 10:1 reduction unit, and mounting the stepping motor on the upper worm shaft by means of a special bracket. When the magnetic clutch is energized between the arm and table worms, both the arm and the table are driven; when de-energized, only the arm is driven. The separate crystal drive can also be converted by using a Slo Syn No. 50 motor.

The variation in wavelength of the monochromatic beam is achieved, for any given monochromator crystal, by the change of position where the neutron beam emerges from the monochromator shield. Since the sample to be investigated has to be in the center of the monochromatic beam, the diffractometer has to follow every move of the monochromator shield. For this reason the diffractometer is mounted on a cantilevered arm which is fixed directly under the exit collimator of the monochromator shield and rotates with it. "V" ways are provided for sliding adjustment of the diffractometer along the cantilever arm. In general it is preferred to keep the distance between the first crystal and the sample at a minimum. If the diffractometer is placed close to the monochromator shield, however, the maximum angle of the diffractometer arm is reduced by interference between the monochromator shield and the detector shield. Therefore, in case of high-angle detector operations (if $2\theta > 125^\circ$) the whole diffractometer is moved away from the monochromator to give a maximum 2θ of about 150° . Accurate leveling of

the unit is required to ensure chatter-free functioning of drives.

DETECTORS, CONTROLS, AND ELECTRONIC DEVICES

In general two neutron detectors are utilized in the neutron diffraction study. One is mounted on the diffractometer arm, as shown in Figures 1 and 3, and receives the neutrons diffracted by the sample. This detector is a medium pressure BF_3 proportional counter, 2.5 in. in diameter and ≈ 13 in. long to obtain adequate counting efficiency. The neutrons first pass through a collimator, then through a thin ceramic end window into the counter, whose full length is effective. To decrease background radiation, the detector is surrounded by a 14-in.-diameter cylindrical shield of boron carbide and paraffin construction, which absorbs almost all neutrons except those coming from the sample. The detector feeds its output into a pre-amplifier and an amplifier. The counts are then accumulated in a Beckman/Berkley 7060 Scalar. On a signal from the control unit the total on the Scalar is printed on a tape by a digital recorder.

The second detector is utilized as a monitor to insure that a constant but not necessarily known number of neutrons strikes the sample during each step of step scanning. This is necessitated by the fluctuation in reactor power level which has the result that the number of neutrons striking the sample is not constant in time. Since high efficiency is not required here, a smaller BF_3 counter is placed across the monochromatic beam (Figure 5). The signal from the monitor is amplified, as in the data collecting system, and the counts are accumulated in a preset counter which emits a signal at a desired count total. This signal is picked up by the control unit, ending the counting in the data collecting counter and printing the total on a tape, while simultaneously starting the drives for the next increment in sample and detector position.

IN-PILE COLLIMATOR

Although the in-pile collimator is not part of the US I, it is so essential to the spectrometer that a brief description of it is offered here.

One of the requirements of the monochromated beam is that it should be well collimated. It is preferable to achieve this collimation within the biological shield of the reactor, in order to utilize its

massive shielding to attenuate the scattering inevitably produced by a collimator. To obtain changing of resolution without having to remove the collimator, a rotary collimator arrangement was designed, whereby one of three collimators, each with a different resolution, can be selected by remote control. A fourth position acts as a shield by interposing a 3-ft-long solid steel slug in the beam path, thereby allowing work in the direct beam path for a short period of time without reactor shutdown. The rotary collimator consists of a 7-in.-diameter by 36-in.-long solid steel cylinder with three rectangular openings, 90° apart. Each opening houses a collimator, and the fourth quadrant is the shield. The center of rotation of the cylinder is in the horizontal plane and parallel to the beam centerline. Changing of positions is done by a motor and microswitch arrangement, while an indicator records at the pile face which of the four positions is in the beam path. The whole rotating assembly is in a heavy concrete plug with stainless steel walls, having its own water cooling coils and fitting accurately in the reactor port hole.

The three resolutions at present planned for powder work are 10, 20, and 40 min of arc in the horizontal plane. The useful area of the collimator is $1\frac{1}{4}$ in. wide by 2 in. high, in which 0.01-in.-thick tempered spring steel slats are positioned in the vertical plane by means of 0.012-in.-wide and 0.040-in.-deep grooves. For single-crystal work, the collimator has a tapered wall on all sides, tailoring the beam to the usually smaller size of the single crystal.

PROGRAMMING

The spectrometer will be operated as part of a complex of seven similar machines at the Brookhaven HFBR facility. Although the different research groups will be engaged in independent experimental studies, all the spectrometers will be under the control of a single stored program digital computer.

The computer will, on a time-shared basis, control the motion of all the axes of rotation and operate all the neutron detectors and monitors. In addition the computer will handle all the angle-specifying computations and will perform a certain amount of the data reduction in real-time.

Control of the individual spectrometers will be accomplished by switches, buttons, etc., at each

station and through typewritten commands and stored programs at the central computer console.

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DATA SHEET

MONOCHROMATOR

Crystal movements:

Bragg angle range $+40^\circ/-30^\circ$ or $+60^\circ/-10^\circ$

Translation range $+0.375/-0.375$

Tilt range $+40^\circ/-40^\circ$

Cavity size: 12 in. diameter

SHIELD

24 in. radial thickness in the horizontal plane

Exit collimator movement: $+80^\circ/-60^\circ$ or $+120^\circ/-20^\circ$

ANGLE DIVIDER

Range: 360°

Accuracy: 1 min of arc

GUNMOUNT PEDESTAL

Capacity: dead load 18 tons

Moment: 116,000 ft-lb

DIFFRACTOMETER

Counter arm range: $\approx 150^\circ$ in either direction

Sample crystal range: 360°

Counter arm load capacity: 40,000 in.-lb

Magnet table load capacity: 1500 lb

Arm speeds: $2.8^\circ/\text{min}$ and $56^\circ/\text{min}$, if Bodine motors are used

Sample speeds: $1.4^\circ/\text{min}$ or $14^\circ/\text{min}$ if coupled 2:1; $3.3^\circ/\text{min}$ or $33^\circ/\text{min}$ if driven independently with the crystal drive

COUNTER AND SHIELD

Medium pressure BF_3 counter, 6-in.-thick radial shield

BEAM HEIGHT

$46\frac{1}{2}$ in. (minimum)