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SYNCHROTRON TOPOGRAPHIC PROJECT

PROGRESS REPORT*

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ABSTRACT

The collaborators have participated in the Synchrotron Topography Project (STP) which has designed and developed instrumentation for an x-ray topography station at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL). The two principle instruments constructed consist of a White Beam Camera (WBC) and a Multiple Crystal Camera (MCC) with high planar collimation and wide area image coverage. It is possible to perform in-situ studies in a versatile environmental chamber equipped with a miniature mechanical testing stage for both the WBC and MCC systems. Real-time video imaging plus a rapid feed cassette holder for high resolution photographic plates are available for recording topographs. Provisions are made for other types of photon detection as well as spectroscopy. The facilities for the entire station have been designed for remote operation using a LSI-11/23 plus suitable interfacing. These instruments will be described briefly and the current status of the program will be reviewed. The Appendix of this report presents titles, authors and abstracts of other technical work associated with this project during the current period.

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I. INTRODUCTION

The Synchrotron Topography Project (STP) was established in 1979 as a Participating Research Team (PRT) with the goal of designing and constructing an x-ray topography station on the 2.7 GeV National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. This project has proceeded in cooperation with NSLS and the completed station, on beam-line X-19, will also serve as a general user facility for approximately 40% of the available time. The front-end of X-19 will intersect a 2.5 milli-radian beam with the radiation tight hutch located 20 m from the tangent point of the storage ring. Currently, this station is the only one at NSLS fully dedicated to synchrotron topography with general purpose peripheral equipment suitable for in-situ experiments. The configuration of the Synchrotron Topography Station (STS) is shown in Fig. 1. The layout of STS was planned for maximum utilization and experimental flexibility given a limited floor area. Two cameras, for different types of topography, will share the same beam-line and will have common components which has reduced both space and cost requirements. These units consist of a White Beam Camera (WBC) and a Multiple Crystal Camera (MCC) which will be described in detail below. Bean conditioning is accomplished with a double crystal monochromator following the design of Golovchenko et. al [1]. This method has the distinct advantage of wide-range wavelength scanning, using a single motor, coupled with a fixed output position of the exit beam for any given wavelength. In the present STS facility, either symmetric or asymmetric crystals will be used in the monochromator to adjust beam conditions for energy resolution, angular divergence and beam cross-section.

#This report includes substantial portions of a technical paper which will be submitted by the authors to Nuclear Instruments and Methods.

WHITE BEAM CAMERA (WBC)

The WBC assembly is shown in Fig. 2. The camera is based on a series of Hüber** components consisting of two goniometers (Huber model nos. 430 and 440) for θ and 2θ rotation on top of which is placed a no. 512 Eulerian cradle for χ rotation and a 360^0 rotation sample mount for ϕ rotation (Fig. 2). A fifth circle (no. 421) is added onto the detector arm so that it also can be swung in the vertical plane. This versatile five-circle goniometer enables diffraction experiment to be carried out in both vertical and horizontal modes. A nearly 360^0 rotation in the horizontal plane and greater than 180^0 rotation in the vertical plane can be achieved, thus allowing observations of all possible (hkl) reflections. Access will be limited in certain sample positions because the Eulerian cradle will interfere with the detector. This is easily overcome with the redundancy built into the rotating sample stage.

The Hüber ϕ-circle stage (Fig. 2) is capable of supporting peripheral equipment weighing up to 100 kg which permits one to attach a wide variety of in-situ stages for special experiments e.g. environmental chambers, mechanical testing, cryostats, furnaces, etc. The detector arm carries a direct imaging system plus a multi-plate auto-cassette for nuclear emulsion plates. All motions on the WBC are controlled by stepping motors which are interfaced to a local DEC LSI 11/23 minicomputer.

The WBC is positioned at a distance of 25 m from the tangent point. Source size is approximately 1.5 x 0.5 mm^2, which leads to a best geometric resolution of 1 μm for a 10 mm specimen to detector distance. This resolution is of the same order as that achieved at comparable synchrotron facilities elsewhere [2].

MULTIPLE CRYSTAL CAMERA (MCC)

A unique feature of the monochromatic topography camera is the adoption of a third crystal operating in the horizontal plane. This mode of operation permits one to satisfy the following design criteria: i) high angular resolution in both vertical and horizontal planes; and ii) large beam cross-section of an area at least 0.5 x 0.5 cm^{-2}. As a bonus all peripherals designed for the WBC are completely interchangeable with the MCC. Fig. 3 shows the operating principle of the MCC. In the edge view, Fig. 3a, the Golovchenko monochromator would have two asymmetric crystals in order to expand the beam in the vertical plane. The output from the monochromator is then reflected off the third asymmetric crystal which can be translated on a linear track in parallel with the incident beam direction to cover an angular range from 16.7^0 to 90^0 2θ, which is confined by

**Blake Industries, 660 Jerusalem Road, Scotch Plains, NJ 07076 the diameter of the MCC goniometer and the track length. This can readily be seen in the three-dimensional view of the MCC system shown schematically in Fig. 3b.

To determine the horizontal divergence it is important to understand the x-ray beam properties at the position of the third crystal. Since the energy selection is made in the vertical plane by a double-crystal monochromator, photons, with different energy (ΔE) will be separated in this plane also giving rise to a vertical divergence of γ_V [3]. Discussion will be limited to the horizontal divergence δ, which is defined either by a horizontal slit width or by the width of monochromator crystal, whichever is smaller. In view of the fact that a divergent (σ is typically of the order of milliradians) monochromatic beam strikes on the third crystal at an incidence angle θ_{in}, not all the rays can be deflected in the horizontal plane. The portion of the beam (δ_{eff}) acceptable for diffraction is in fact determined by either the acceptance angle $\Delta\theta_{in}$, of the crystal or by the projected crystal width $(\frac{W_3 \sin\theta_{in}}{L_3})$, whichever is smaller. These parameters are shown schematically in Fig. 4. For an asymmetric reflection $\Delta\theta_{in} = \sqrt{m_3}\ \omega_3$ where m_3 is the asymmetry factor and ω_3 is the natural rocking curve width for a pure π polarization, i.e.:

$\omega_3 = \frac{2d\ \lambda\ |F_{\bar{g}}|\ \cos 2\theta_B}{\pi V \cos\theta_B}$. The output horizontal divergence is thus given by

$\gamma_H = \delta_{eff}/\sqrt{m_3}$, and the output beam width is $W = m_3 L_3\ \delta_{eff}$, where subscript '3' refers to the third crystal. Figure 5 displays γ_H and W as a function of wavelength for a particular asymmetric crystal. Although the angular divergence is much reduced for a 2θ angle close to 90^o due to the polarization factor, the intensity is unfortunatley reduced by the same factor also.

Divergence in the vertical plane as well as beam height have been discussed in a paper by Chen, Hmelo and Bilello [3]. Basically, there are four contributing factors: (i) intrinsic vertical divergence of a Synchrotron Radiation source or the size of beam-limiting aperture before the monochromator; (ii) size of the crystal and the distance between it and the tangent point; (iii) asymmetry factor; and (iv) the intrinsic rocking curve width. Taking all these into account, one could determine the vertical divergence γ_V and thereby the beam height, $H = m_2 m_1 L_1 \gamma_V$. It should be mentioned that the vertical divergence is ultimately coupled to the energy resolution which in turn is dependent upon the size of the crystal under irradiation. Therefore in the vertical plane, the general notion about increasing beam size by employing a larger crystal is unavoidably accompanied with an increased divergence as well.

By careful selection of asymmetric crystals, the MCC camera can be designed with horizontal and vertical divergences in the order of one arc second at a designated wavelength. Crystal sets must be custom cut for each wavelength of interest. As examples, Table 1 lists several sets of crystals at 0.71 Å and 1.54 Å wavelength.

The above MCC camera is designed for horizontal plane operation with a sample crystal placed on a goniometer (Hüber no. 420). Projection topographs, or section topographs, can be taken with the third crystal placed in a magnifying, or demagnifying mode, respectively [4]. Angular resolution can be downgraded by oscillating the third crystal, or the sample crystal, such that topographs can be performed on crystals of less perfect quality.

If the third crystal is removed, or a portion of the monochromatic beam is allowed to reach the WBC, topography and diffraction can be operated in a vertical plane with the use of a Hüber five-circle goniometer. The θ rotation can be achieved by using either the ϕ-rotation axis at the $\chi = 90^{o}$ position or the χ-rotation axis at the $\omega = 90^{o}$ position whereas 2θ rotation is provided by an additional rotator (Hüber 421) mounted below the detector arm. In the vertical mode, however, one loses equal divergence and hence equal strain sensitivity in every direction of the sample, although no intensity reduction due to polarization factor is encountered.

ENVIRONMENTAL STAGE

For studies of the physical-chemical effects of structural imperfections in solids and the influence of mechanical deformation on these imperfections, an environmental chamber will be constructed for use with the white radiation camera. It will be constructed from stainless steel to UHV standards.

The basic vessel consists of two coupled chambers. The main cylindrical chamber consists of a stainless steel cylindrical nipple ∿ 8 in. O.D., 5 3/4 in. long fitted with rotatable Conflat flanges (∿ 10 in O.D.) at each end. The entrance and exit ports will be closed by 20 mil thick beryllium windows. These beryllium windows will withstand pressure differentials of up to 7 atms. Each window will be thermally annealed in air at ∿ $1300^{o}C$ to form a protective oxide coating for safe handling. Around the periphery of the main chamber are

Table 1

Crystal Sets for MCC for Particular Wavelengths

1. Hard wavelength set for output at sample position of $\lambda = 0.7$ Å

Crystal 1	Si (111)	Symmetrical
Crystal 2	Si (111)	Asymmetric, $\alpha = 5.05^{o} \pm 0.01^{o}$
Crystal 3	Si (220)	Asymmetric, $\alpha = 9.02^{o} \pm 0.01^{o}$

2. Soft wavelength set for $\lambda = 1.5418$ Å

Crystal 1	Si (111)	Symmetrical
Crystal 2	Si (111)	Asymmetrical, $\alpha = 13.50^{o} \pm 0.01^{o}$
Crystal 3	Si (220)	Asymmetrical, $\alpha = 20.50^{o}$ 0.01^{o}

12 axially arranged ports fitted with rotatable Conflat flanges which are: 2 x 3 3/8" O.D. at 90^0 to each other and 8 equally spaced 1 1/3" O.D. equiaxially between them. These ports are available for the insertion of electronic couplings, electrical feedthroughts, power leads, gas and liquid inlets, small sample stages, separate rotary and linear feedthroughs.

A second coupling chamber with a windowed face plate will be available to extend the volume of the system. This chamber which contains a 4 inch Be window mounted on a 6 in. O.D. flange has, in addition, 4 x 2 3/4" O.D. angled ports aligned with the sample positions at the chamber centre. These ports may be used for irradiation or observation. During experiments sapphire, beryllium and/or fused quarts windows will be provided. The entire system is illustrated in Figs. 6 (main nipple) and 7 (extension chamber), respectively.

For routine use experiments will be able to be performed at temperatures up to $\sim 1000^0$C and possibly higher with the use of heat shields. For this purpose both the main nipple and the coupling chamber will be double-walled with water cooling. The main chamber volume is large enough to take quite sizeable heating stages and with the additional chamber attached will be large enough to accommodate mechanical straining stages and other specialty stages. These can be attached to eight lugs provided round the internal perimeter of the stage or suspended from the radial ports. A general mechanical stage for the chamber is also under construction.

An additional main chamber will be constructed to accommodate dirty work ($\sim 10^{-6}$ Torr). The original main chamber and coupling chamber will be clean with background pressures $\sim 10^{-11}$ Torr. The entire system will be capable of suspension from 4 support eye-hooks located on the main chamber.

Evacuation of the system will be performed by 2, twenty-five litre/sec. differential ion pumps coupled directly via stainless steel tees to the environmental chamber. These pumps will be mounted together with the chamber directly on the goniometer stage. These pumps will be backed by an isolatable sorption pump via a UHV bakeable isolation valve.

MECHANICAL TESTING FACILITIES

The Topographic Beam line will be equipped with a number of instruments designed to apply stresses and plastic strains to specimens under conditions which are compatible with the requirements of both White Beam and Monochromatic Topography. This instrumentation will be used in the ambient and inside the environmental chambers which mount on the goniometers of the WBC and MCC cameras.

The design of the stressing apparatus will be compatible with many of the environments which can be used in the chambers, eg. vacuum, H_2, H_2S, O_2, etc. While the presently designed tensile apparatus, described below, will operate at room temperature, it is planned to construct a modification of this design which will be used at controlled themperatures down to $77^{o}K$. If the user demand develops, a somewhat modified design will be constructed for measurements at elevated temperatures.

The design of the tensile apparatus which is presently under construction is based on that proposed by Bowen and Miltat [5]. The major design parameters are:

a) the load range will be up to about 15 kg which should be sufficient to plastically deform crystals of a size suitable for transmission topography and most back reflection topography.

b) the displacement range will be of the order of 2.5 cm.

c) crystal sizes of dimensions 5 x 2 cm can be accommodated. The grips are of a clamp type, but experience has shown that the best results are obtained by attaching the specimens to flat sheets which are gripped by the clamps.

d) the load will be measured by a low compliance load cell with an accuracy of about 0.1% of the full scale range.

e) the displacement will be measured by an LVDT with a precision of about 5×10^{-6} cm.

f) the crosshead speed range will be about 10^{-5} - 10^{-1} cm/sec.

As shown in Fig. 8 the tensile stage design is essentially a fixed and a moveable frame between which the specimen is attached. The relative displacement of these two frames is controlled by a variable speed motor through suitable gearing, with the motor and gearing being outside the environmental chamber. The coupling to the moveable frame will be through a bellows. The load cell will be located inside the environmental chamber so that the differential pressures will not affect the load readings. While no provision is presently provided for feedback control of the load or strain this can be easily introduced.

One of the significant design conditions is that the two frames be aligned in a common plane and that they translate relative to each other in that plane. This is accomplished with the use of linear bearing to align the two frames. Using this arrangement, considerable deformation of the specimen can be carried out without introducing any bending or twisting since it is also constrained to undergo displacement in the plane of the frames. Use of this design has a number of significant advantages. The loading frame is relatively thin, allowing film to be placed close to the specimen and thus increasing the number of reflections which can be obtained. The loading apparatus is designed to have a low compliance relative to the specimen resulting in a hard machine. The resulting rapid decrease in load with specimen elongation makes it possible to stop propagating cracks in many materials if the specimen is designed properly. In addition, the apparatus can be easily modified by the addition of a high compliance member in a series with the specimen in those instances where this is desired.

REAL-TIME TV DETECTOR AND NUCLEAR PLATE CASSETTE

An electro-optical system has been constructed which couples a state-of-the-art microchannel plate image intensifier tube with a solid state CID television camera for real-time detection, display and recording of synchrotron x-ray topographs [6,7]. The particular design of this detector was based on several desirable features. Of primary concern was the need to accommodate to the relatively small space inside the synchrotron hutch as well as to make the detector as light weight as possible in order to permit it to be attached to a goniometer arm capable of rapid positioning. Also, an analog output format for display on a television monitor for remote viewing as well as a digital output format for computer image processing was important. Finally, a completely modular design was adhered to so that all components can be exchanged for other components optimally suited for any desired application and to permit replacement of components when future advances are made. The prototype miniature synchrotron x-ray television camera which has been constructed conforms to all of the above enumerated specifications. This system possesses a 50 mm x-ray input faceplate, is less than 10 in. long, and weighs less than 2 lbs.

The detector arm which supports the real-time television detector also supports a multi-plate cassette which permits remote automatic changing of 2 in. nuclear emulsion plates. As presently constructed, this cassette permits each nuclear plate, in turn, to be positioned directly in front of the electro-optical detector input faceplate. In this manner a low resolution real-time topographic image may be viewed directly on the television monitor for a variety of purposes such as checking the experimental set-up or monitoring a specific real-time test. Once the proper conditions are reached, the operator can remotely activate the automatic nuclear emulsion plate cassette, positon a nuclear emulsion plate in front of the electro-optical detector input window, and make an exposure resulting in a high-resolution topographic image. The Real-Time TV detector and nuclear plate cassette assembly is illustrated in Fig. 9.

AUTOMATIC CONTROL OF TOPOGRAPHY CAMERAS

The remote control of the goniometers, detectors, and the environments of the specimens are accomplished by means of a computer system consisting of a LSI-11 minicomputer and a stepping motor controller designed and manufactured by Brookhaven National Laboratory. In addition, microcomputers provided by the user to handle dedicated tasks can be linked to the main computer by standard serial or parallel interfaces. The main computer is supported by a Winchester disk storage, backed up by floppy disk drives. It communicates to the BNL motor controller by a serial line. This controller can also be activated manually by push button operation on a simple keyboard. Bi-directional Analogue and digital data are handled by the main computer by a sixteen channel A/D and two channel D/A converters, as well as a sixteen bit parallel interface. It is anticipated that most users will accomplish data acquisition and storage by microcomputer based auxiliary equipment. We are therefore linking to the main computer devices based on the Intel 8085 and 6502 microprocessors, for the purpose of sensing the x-ray intensity, temperature, stress, and other experimental parameters.

The software for the control of the White Beam Camera allows operation in a variety of modes. The commands for the BNL motor controller are entered either directly from the computer terminal or indirectly from a computer program. In the simplest mode of operation, the rotation of goniometer stages is achieved sequentially, allowing the recording of topographs any time during this sequence. The availability of a real-time TV detector, interchangeable remotely with a film detector, will facilitate the recording of topographs under this manual mode of operation. Alternatively, a series of topographs can be recorded by the commands executed in a computer program, allowing the activation of the detector at pre-determined intervals following the completion of each set of goniometer rotations to zero-in on a particular reflection. The attainment of certain predetermined value of a particular experimental parameter (e.g. x-ray intensity, temperature, stress, etc.) before an exposure is taken or a rotation is executed, allows the progress of an experiment totally controlled by the computer program.

The activities of all the components of the topography camera are recorded in computer files that can be retrieved and examined, thus forming a user's record. Furthermore, software limit switches are incorporated so that the allowed ranges of motion in all the stages will not be exceeded. This latter operation is facilitated by having all the stages returned to "zero" at the beginning of each user's session at the beam port.

At the present time, a number of computer programs have been successfully implemented, allowing the setting of a crystal specimen for Bragg reflection and the tracing of fine structures in reflection curves. Computer programs that maintain the setting of a crystal at a Bragg peak when an external parameter is continuously changing are currently being developed. These will be useful for the recording of topographs when the crystal specimen is in a fluctuating thermal or mechanical environment.

SUMMARY

This report has described the design and construction of a fully dedicated Synchrotron Topography station at the NSLS for both White Beam and Monochromatic applications. This facility will have a wide range of peripheral stages available for testing materials under dynamic and static load and for a variety of environmental conditions. The STP facility will join other dedicated synchrotron topography efforts already functioning in Britain [2] and West Germany [8]. It is anticipated that this facility will greatly expand the utilization of high energy x-rays for non-destructive studies on the defect structure of materials.

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FIGURE CAPTIONS

FIG. 1 Layout of Synchrotron Topography work station.

A) Steel paneled Experimental Hutch
B) White Beam Camera
C) Third Crystal track for varying wavelength on MCC
D) Golovchenko monochromator
E) Multiple Crystal Camera
F) Beryllium window on Beam Transport
G) Forward slit assembly
H) Safety monitoring cameras
I) Beam stop
J) Control room
K) Darkroom

FIG. 2 White Beam Camera. Consists of a five circle goniometer system for access to all (hkl) in hemisphere. Numbers on goniometers indicate Huber model designation.

FIG. 3a) Edge view of Golovchenko monochromator.

3b) Three-dimensional schematic of Multiple Crystal Camera.

FIG. 4) Defines quantities used for calculating beam divergence and energy resolution.

FIG. 5) γ_H, output beam divergence and $\overline{W}$, the output beam width, plotted as a function of wavelength, λ. Calculated for Si (220) with the asymmetric angle $\alpha = 9.02^{o}$, and Si crystal of length 3 cm.

FIG. 6 Front view of UHV environmental chamber.

A) 20 cm Beryllium windows
B) 25 l/s differential-ion pumps
C) Sorption pump
D) All shaded area is Huber camera components

FIG. 7 Top view of UHV environmental chamber. A)-D) as in Fig. 6.

FIG. 8 Exploded view of tensile stage.

A) Alignment Vee-grooves for upper and lower sample grips.
B) Upper grip
C) Lower grip
D) Mounting block for load transmitting rod.

FIG. 9 Schematic of real-time TV detector and multiple plate nuclear film dispenser.

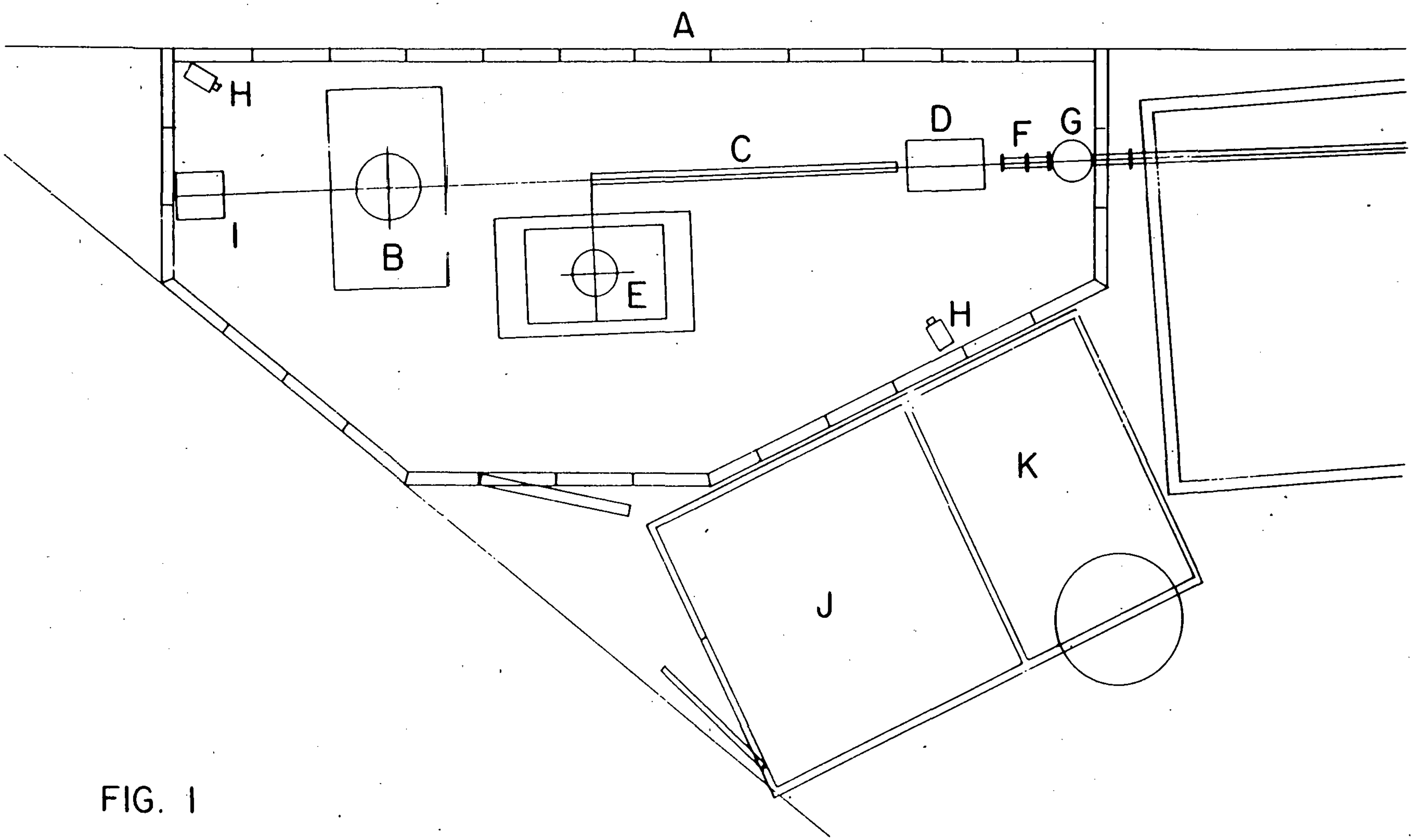


FIG. 1

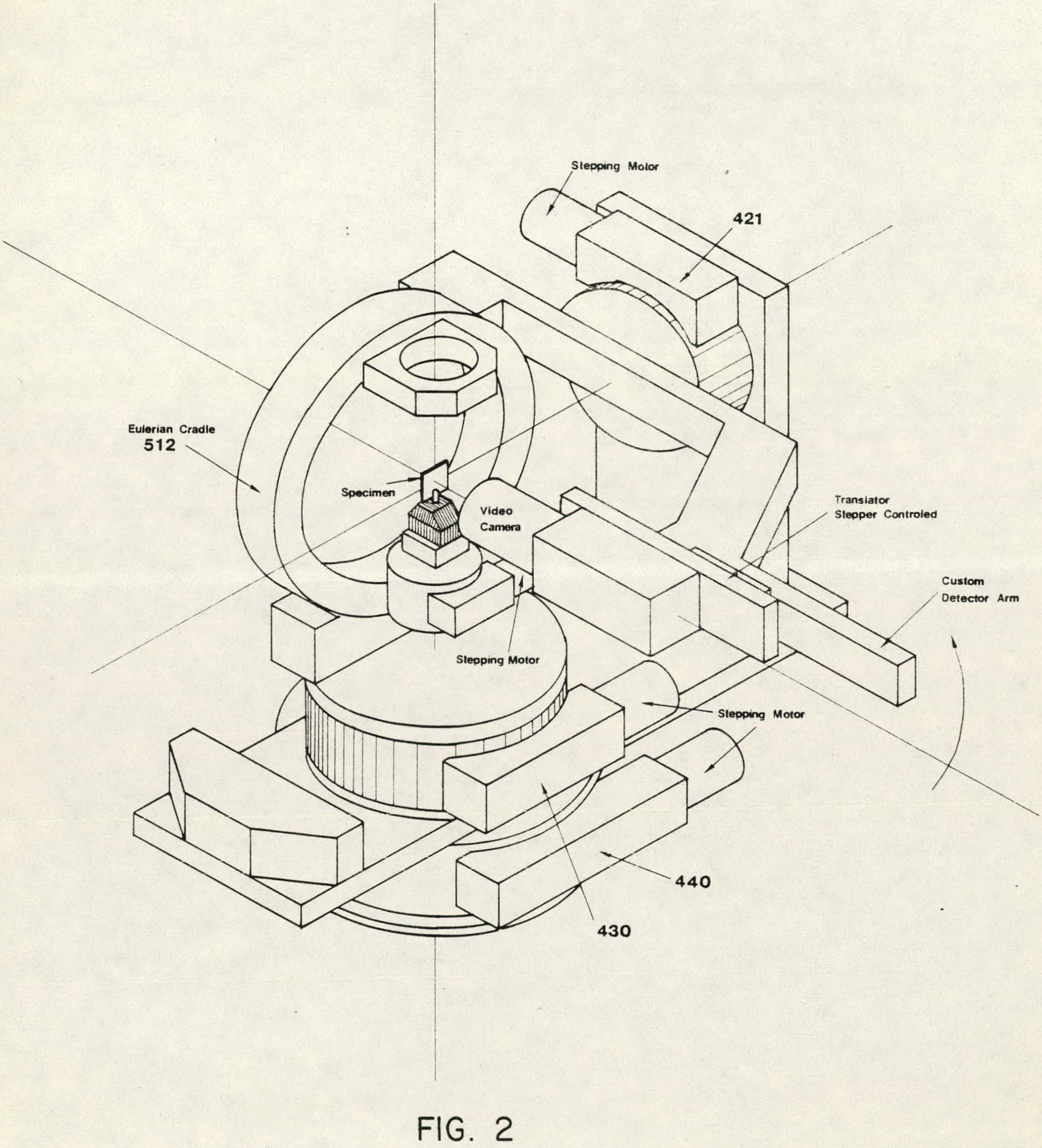

Stepping Motor
421
Eulerian Cradle
512
Specimen
Video
Camera
Translator
Stepper Controled
Custom
Detector Arm
Stepping Motor
Stepping Motor
440
430

FIG. 2

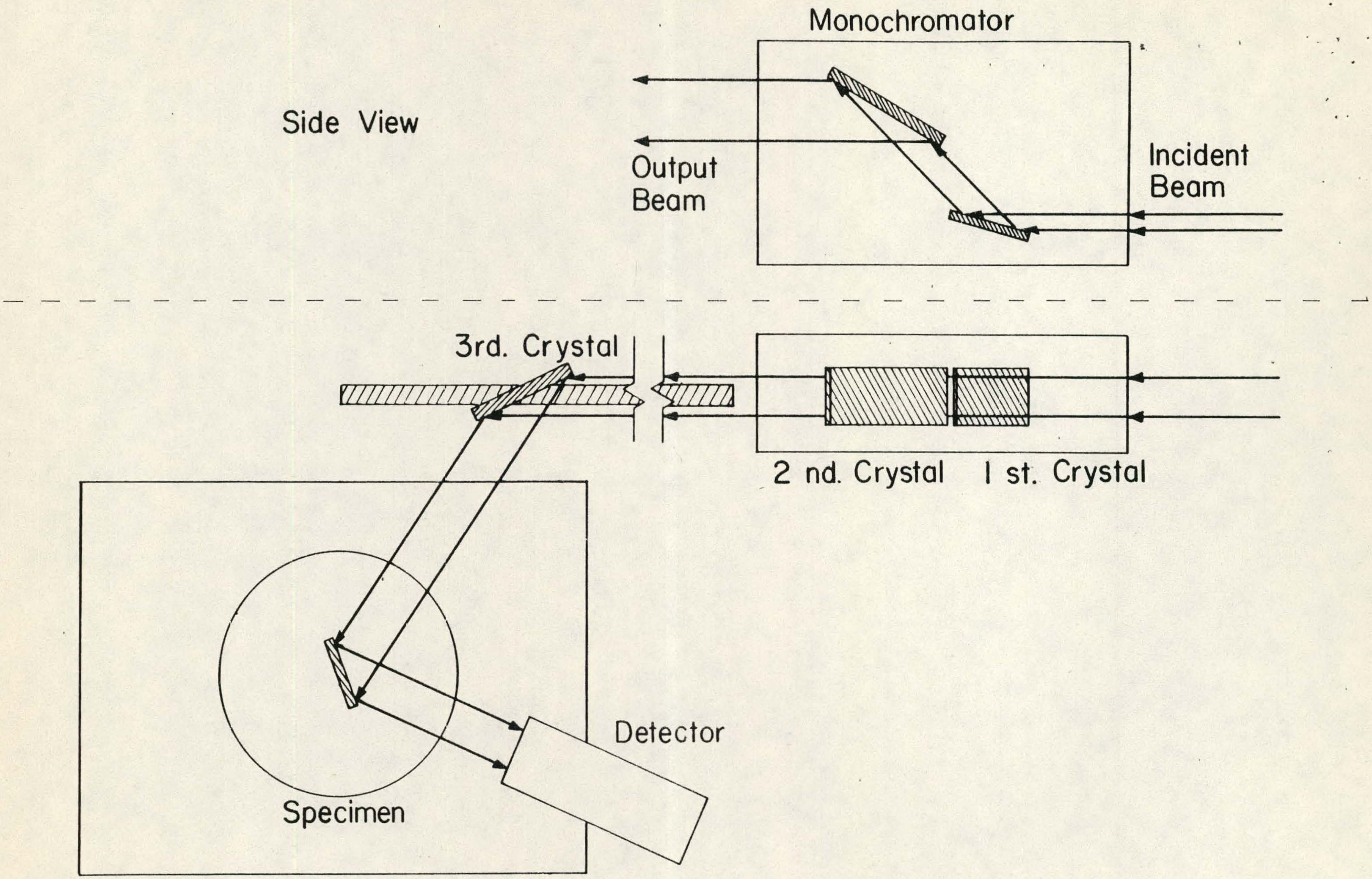

Monochromator
Side View
Output
Beam
Incident
Beam
3rd. Crystal
2 nd. Crystal
I st. Crystal
Detector
Specimen

FIG. 3 (a)

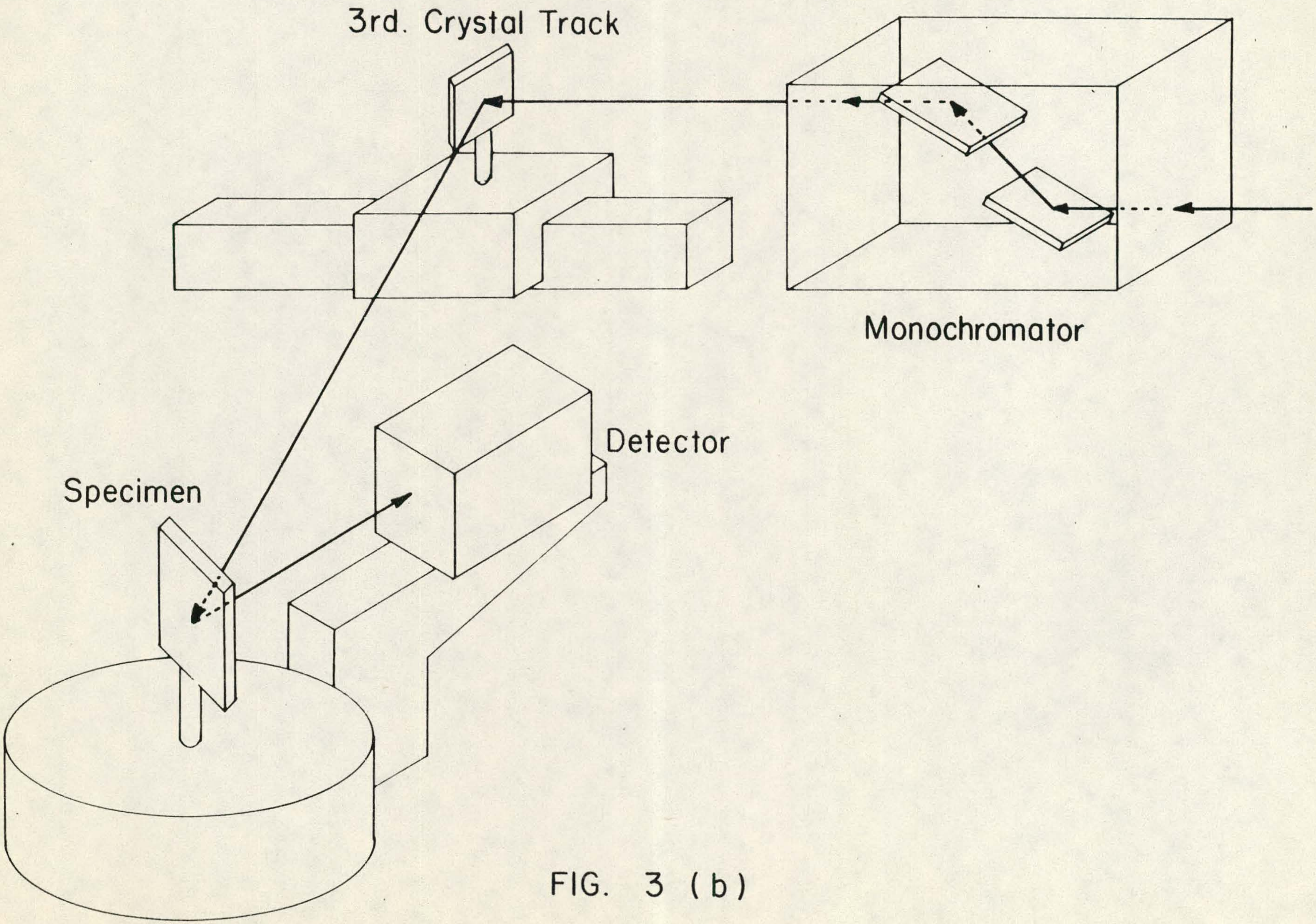
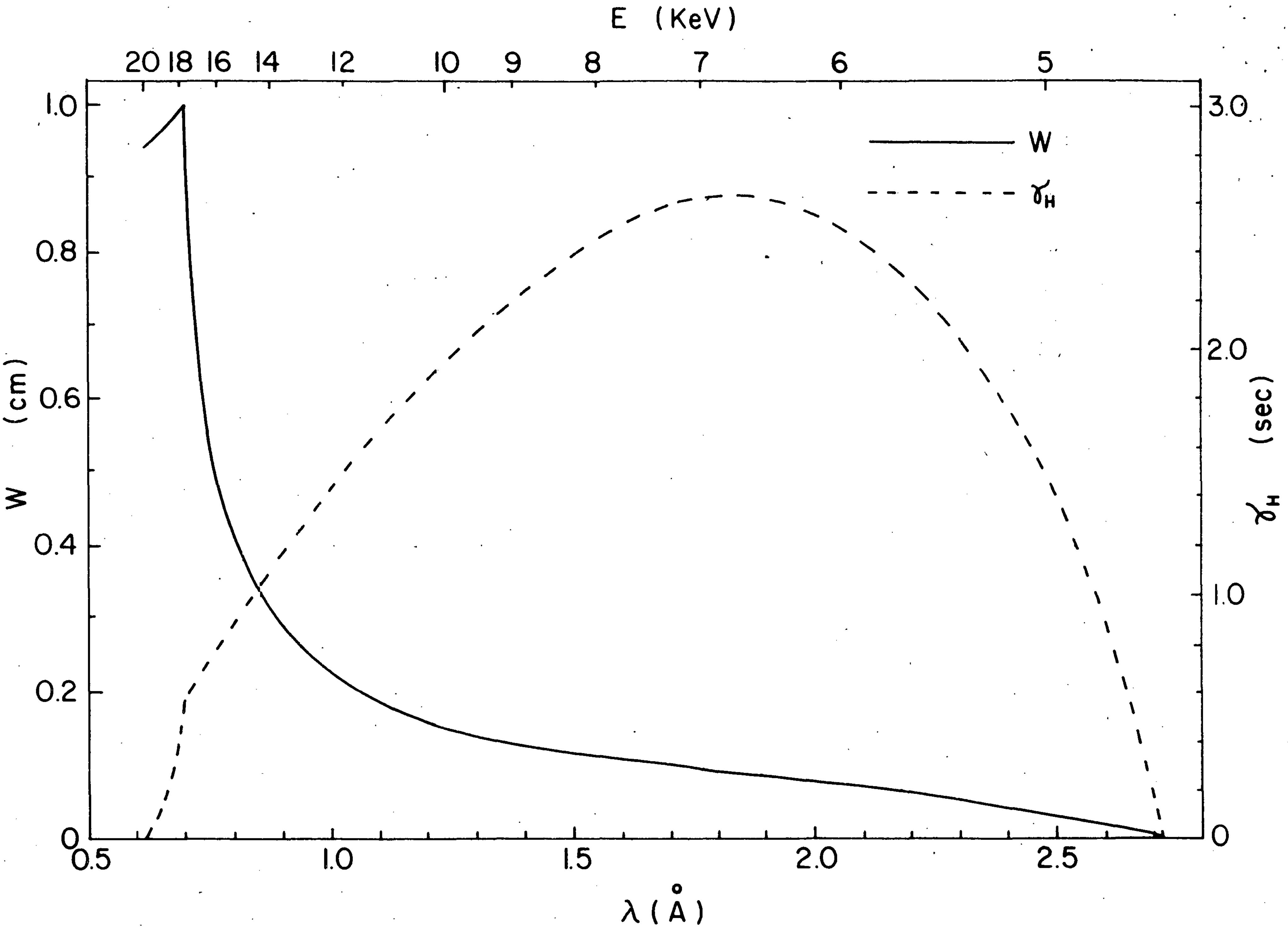
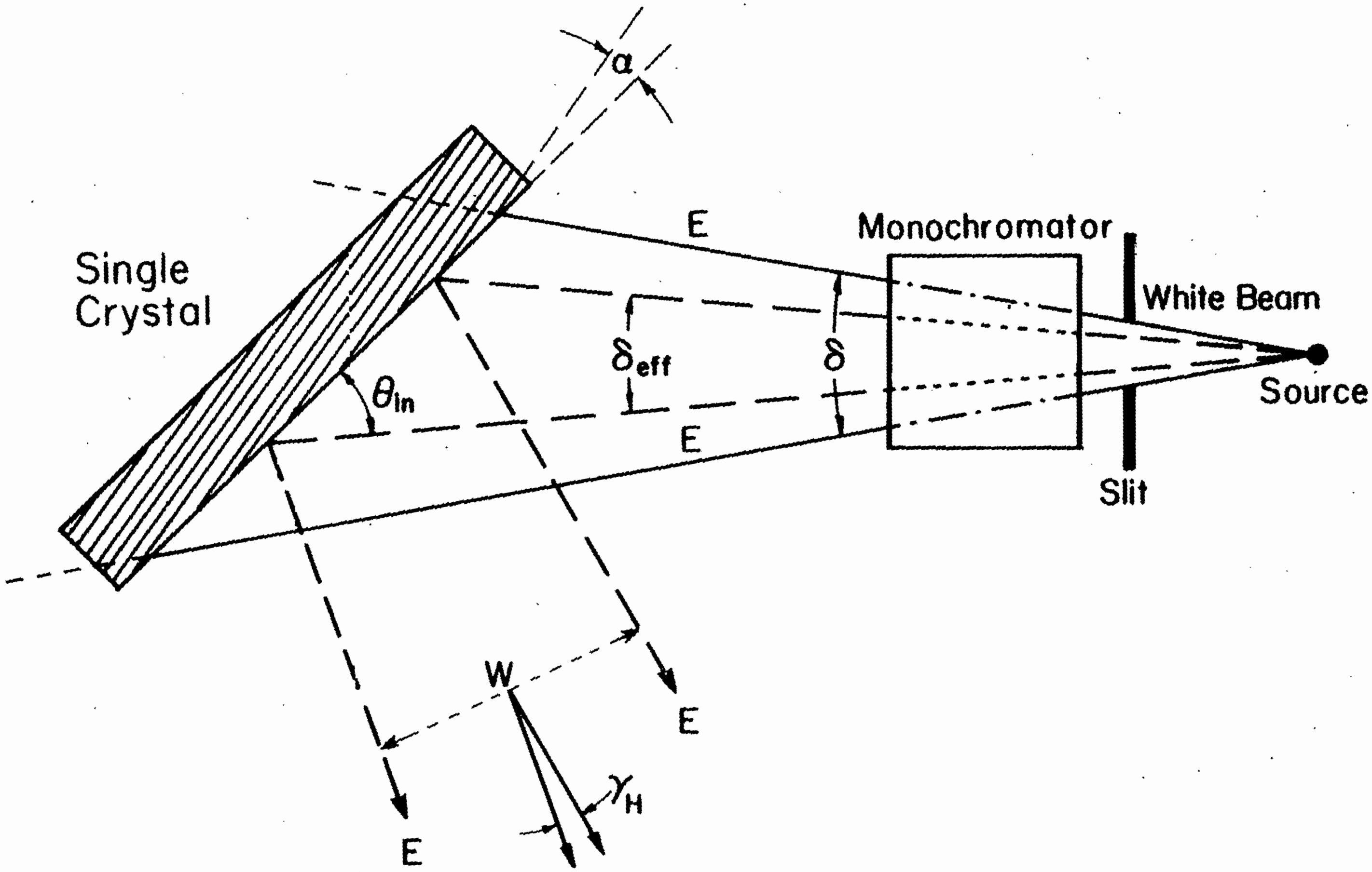


FIG. 3 (b)



E (KeV)
20 18 16 14 12 10 9 8 7 6 5
W (cm)
1.0
0.8
0.6
0.4
0.2
0
3.0
2.0
1.0
0
γ_H (sec)
W
γ_H
0.5
1.0
1.5
2.0
2.5
λ (Å)

FIG. 5



α
Single
Crystal
E
Monochromator
White Beam
δ_{eff}
δ
θ_{in}
Source
E
Slit
W
E
γ_H
E

FIG. 4

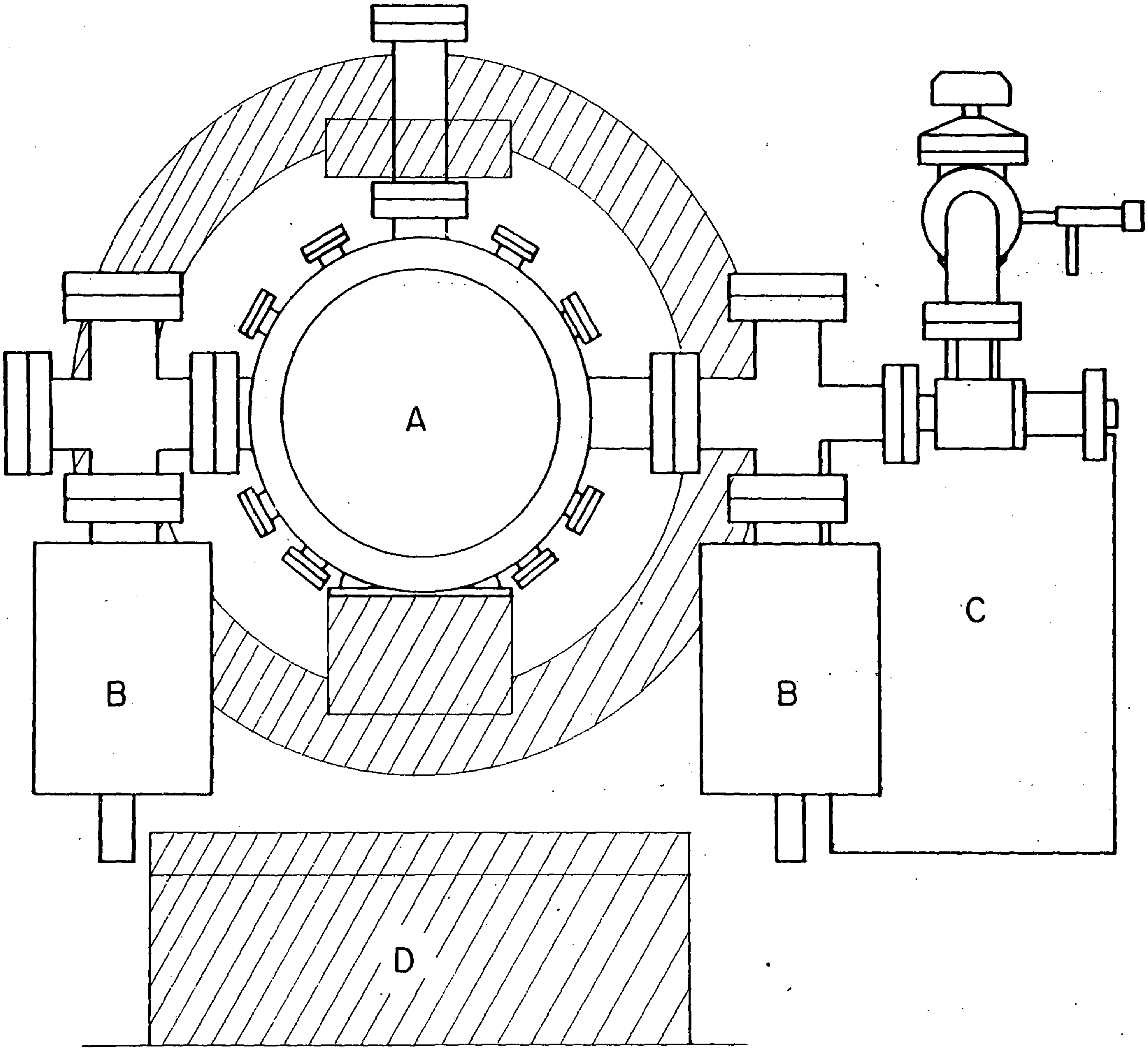


FIG. 6

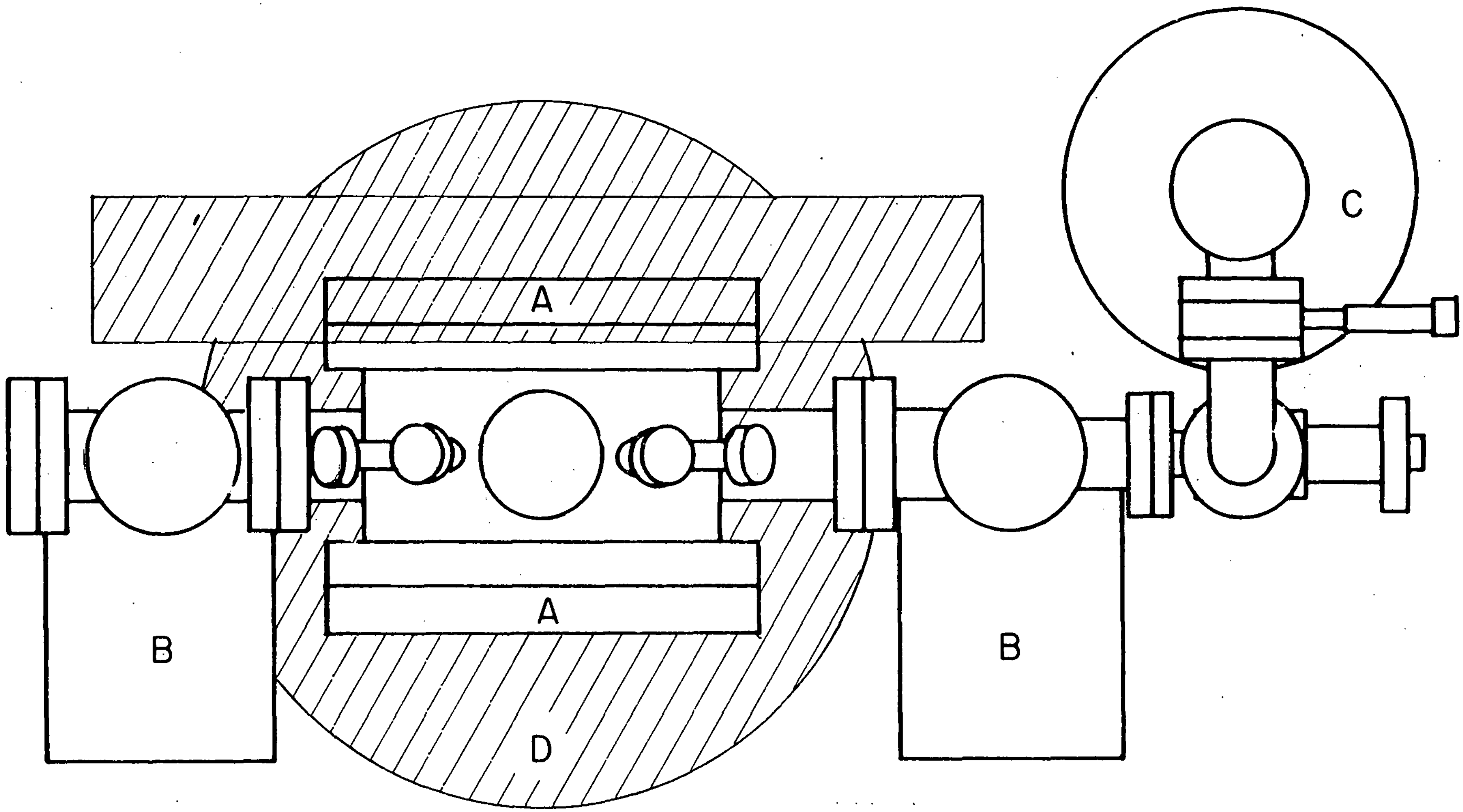


FIG. 7

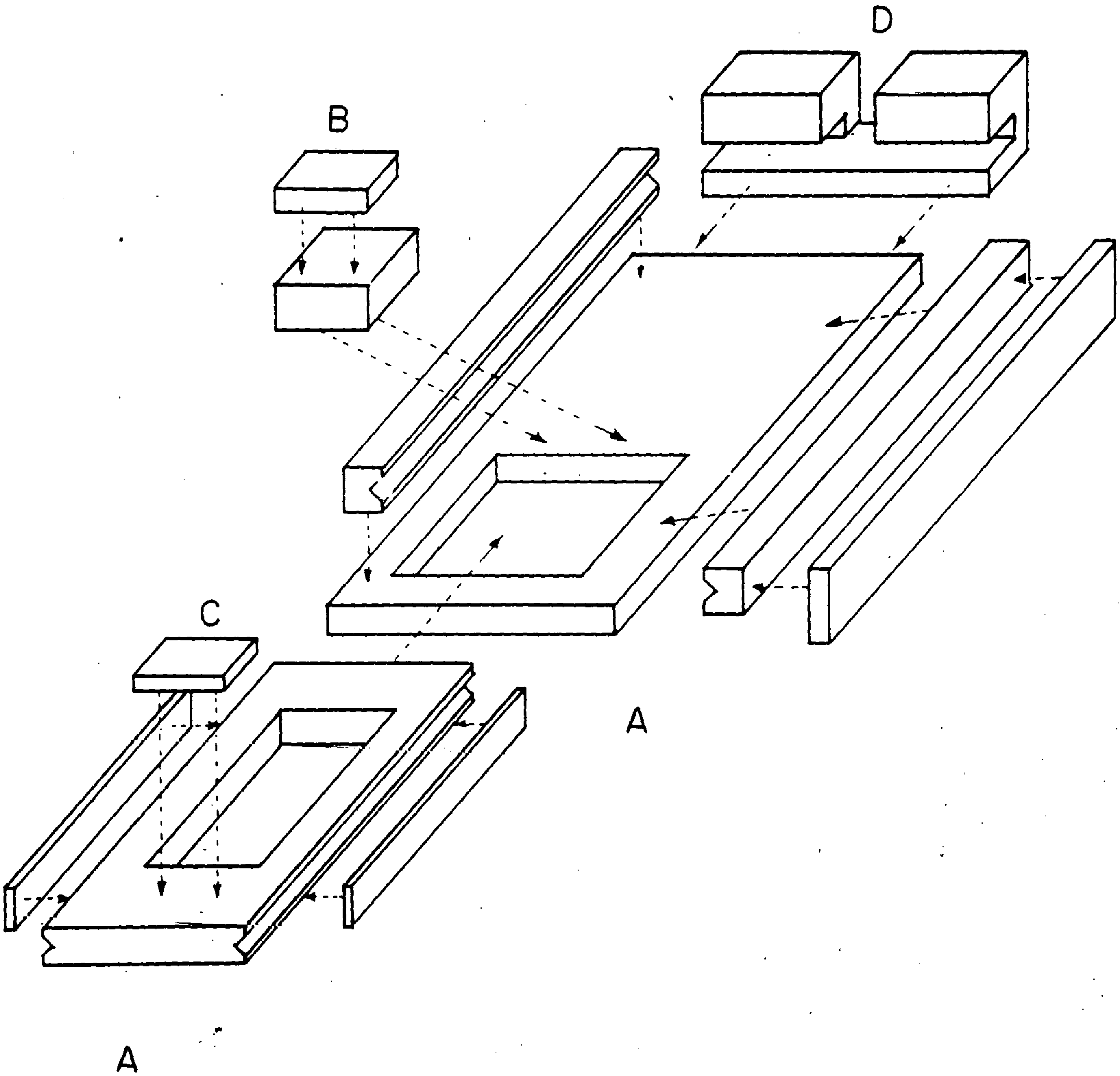


FIG. 8

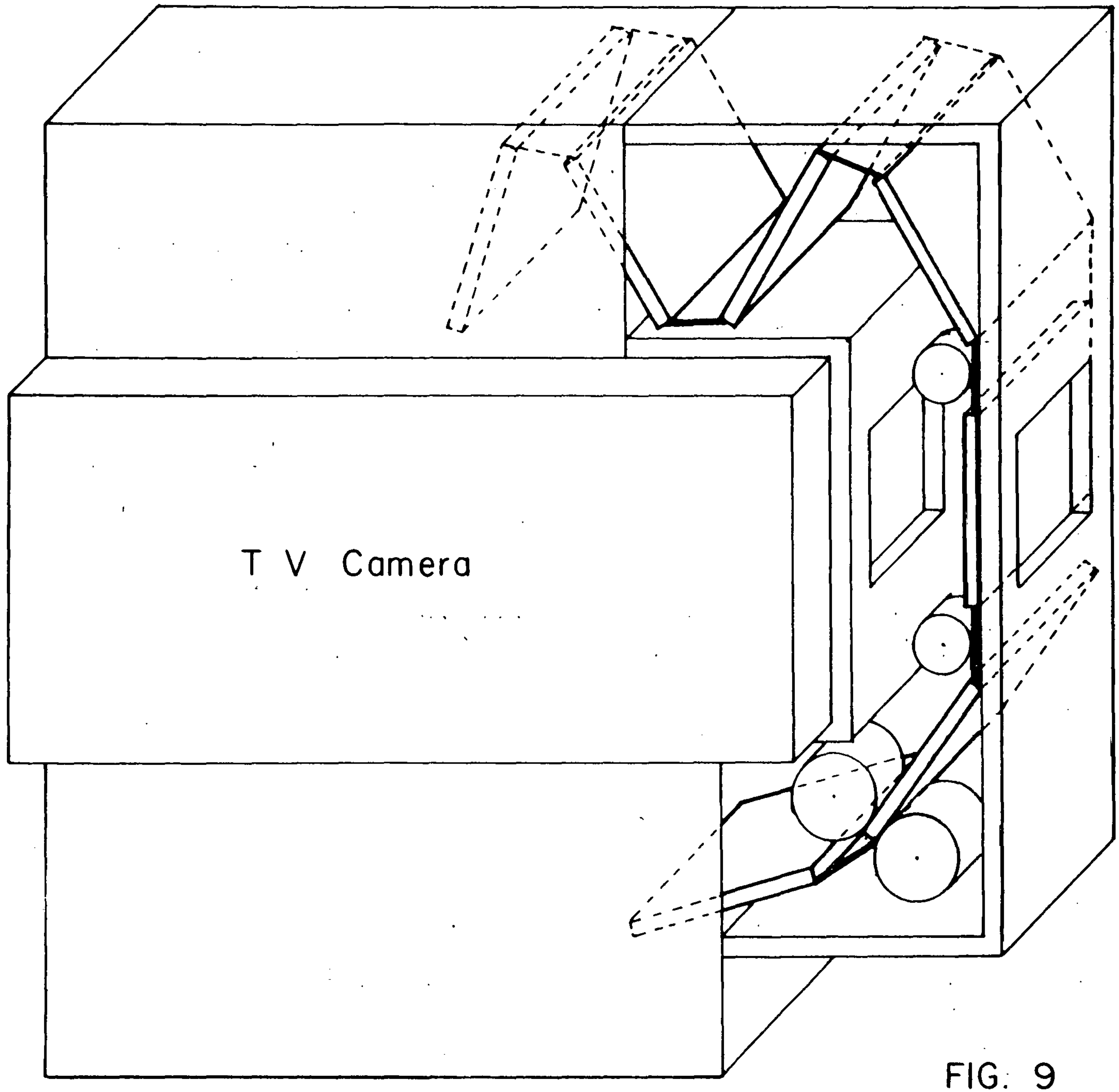


FIG. 9