FINAL STATUS REPORT

For the Period

June, 1966 to December, 1969

DEFECT PRODUCTION IN SINGLE CRYSTALS RESULTING FROM ION BOMBARDMENT

by

Lawrence B. Shaffer

Prepared for

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

22 May 1970

GRANT NGR 36-019-001

X RAY PHYSICS LABORATORY
Hiram College
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INTRODUCTION AND SUMMARY

Contained in this report is a summary of the progress made during the period of time from June, 1966, through December, 1969, on work supported through the Lewis Research Center of NASA. This work, begun in June, 1966, constitutes an area of surface physics involving a study of the damage produced on the surface of metal single crystals during ion bombardment using x-ray methods. The technique to be developed as part of this study will be useful in supporting the investigation of metal single crystals using low-energy electron diffraction methods as well as other areas of investigation where information as to the dislocation density and interstitial content of a surface under ultra high vacuum is useful. Other such areas might be ion implantation studies, rolling contact studies,[1] etc.

Another area of interest is that of applying standard x-ray techniques to the determination of certain thermodynamic properties of cesium gas, e.g., isothermal compressibility, and possibly the ion - ion interaction potential. The methods to be used are fairly direct and straightforward,[2,3] however the latter parameter may be quite sensitive to experimental conditions.

PART I

CESIUM GAS X-RAY SCATTERING STUDIES

The study of the structure of liquids and gasses by radial distribution function analysis is a standard technique in x-ray diffraction.[4] From an analysis of the radial distribution function information about the distance and number of neighboring atoms can be obtained. One can also transform the equations of the above type which are concerned only with the geometry of a group of particles into thermodynamic equations which depend upon the temperature and the potential energy of a pair of particles. Then measurements of the interparticle potential function may, in principle, be made as a function of temperature and pressure, i.e. concentration. Parameters such as size (radius of gyration), shape, molecular weight, density, and isothermal compressibility may also be determined.[5]

Thus x-ray scattering can be used to determine the interaction potential of a pair of cesium atoms and to make certain absolute measurements of other thermodynamic properties of cesium vapor. Calculations on the interaction potential require that the number density of the vapor be known. This can be determined by measurement of the small angle scattering using x-ray beam of known absolute power.

The feasibility study of the cesium gas problem suggested in the grant proposal was divided into several tasks, each of which is discussed below.

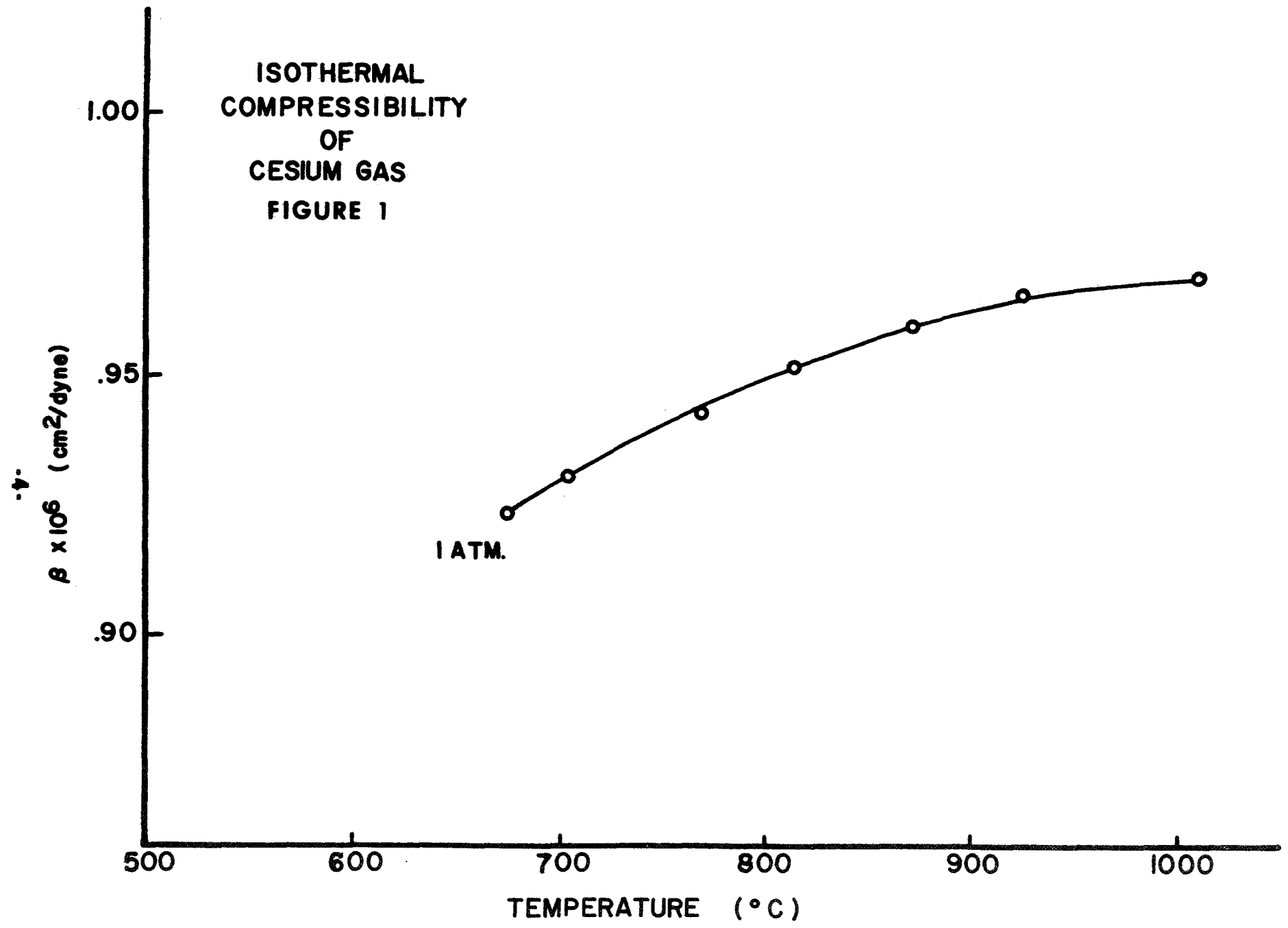
THERMODYNAMIC PROPERTIES OF CESIUM VAPOR

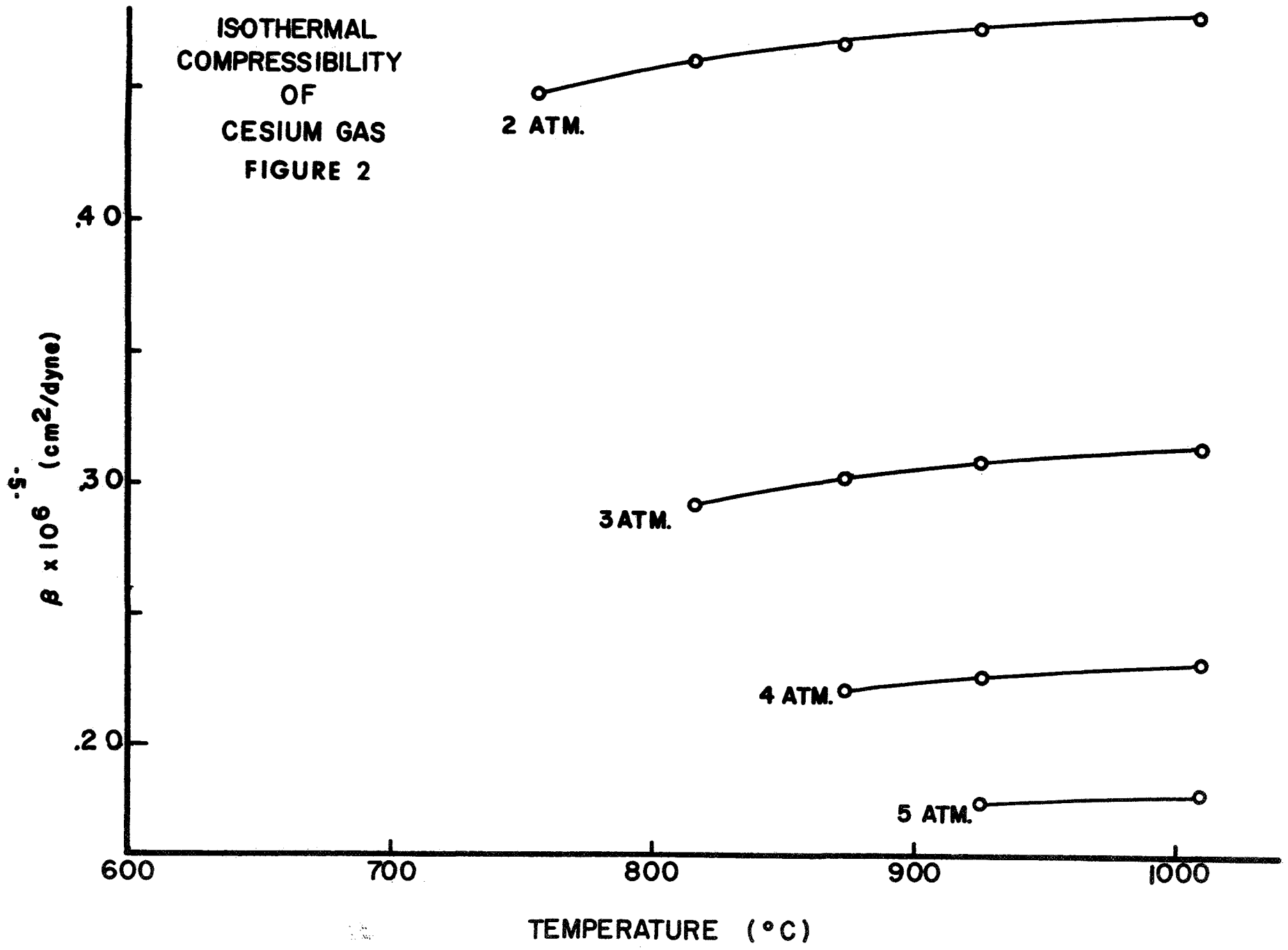
Employing the data of Heimel[6] and Ewing[7], et al., calculations have been made on the value of the isothermal compressibility and density of cesium vapor in the temperature range from 676°C to 1010°C and in the pressure range from 1 to 5 atmospheres as shown in Figures 1 to 3. This value combined with the number of atoms per cc (number density), the Boltzman Constant k and the Kelvin Temperature t, yields the product $ckt\beta$, the interparticle interference function at zero scattering angle.[8]

In all calculations in the above range, however, this product is essentially unity which indicates a nearly ideal gas with zero interaction potential. Since it seems reasonably certain from the properties of other vapors that at temperatures near the condensation point such an interaction should become evident, observations should be made at temperatures and pressures very close to the saturation curve of cesium vapor. Since this product contains c, then for a constant c and T the value of $ckT\beta$ should follow β and should the product depart significantly from unity, it would indicate that β does not follow the value predicted for an ideal gas, namely,

$$\beta = \left(\frac{1}{P}\right)_V \propto \left(\frac{1}{T}\right)_V$$

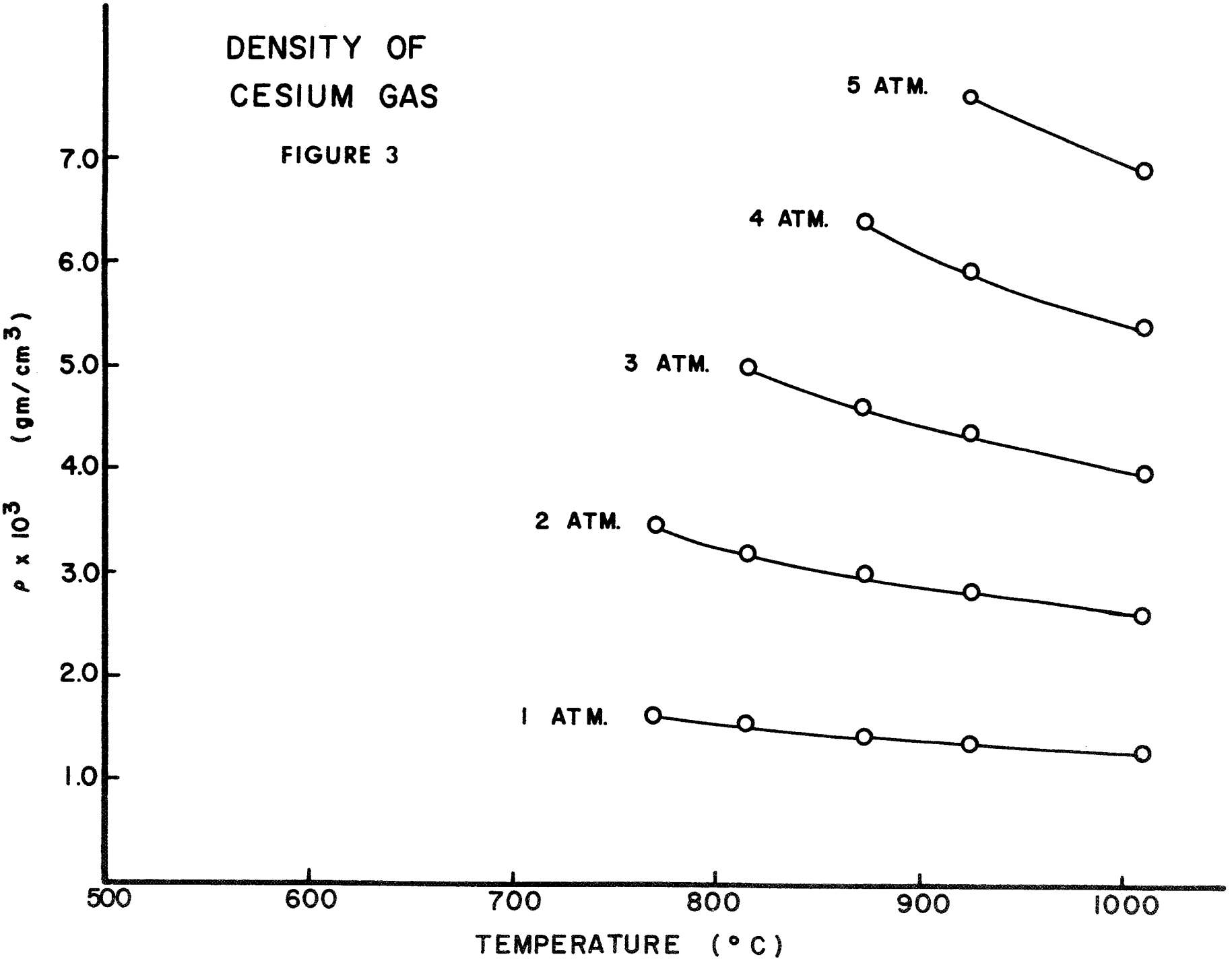
The above consideration assumes a constant c. However, if in the process of heating, cooling, and reheating the cesium vapor vessels, the adsorption of cesium atoms on the walls varies to any great extent the value of c would be uncertain. Calculations assuming a continuous monolayer of adsorbed cesium atoms in vessels whose dimensions are comparable to ours


ISOTHERMAL
COMPRESSIBILITY
OF
CESIUM GAS
FIGURE 1
1.00
.95
.90
β x 10^6 (cm^2/dyne)
1 ATM.
500
600
700
800
900
1000
TEMPERATURE (°C)


ISOTHERMAL
COMPRESSIBILITY
OF
CESIUM GAS
FIGURE 2
2 ATM.
3 ATM.
4 ATM.
5 ATM.
.40
.30
.20
600
700
800
900
1000
TEMPERATURE (°C)
β x 10^6 -S- (cm^2/dyne)

DENSITY OF
CESIUM GAS

FIGURE 3



indicate that a negligible variation in c could arise with departures from a continuous monolayer.[9] Further investigation of adsorption could be made by measuring the resistance across the length of the cesium vapor vessels. Such resistance should be influenced by the completeness of the adsorbed layer and its thickness, assuming more than a monolayer.

EXPERIMENTAL TECHNIQUES

Absolute Intensity Measurements

To measure the large ratio ($\sim 10^{-7}$) of the scattered to the incident x-ray beam special techniques must be employed.[10,11,12] The technique to be used primarily in this study will be that of scattering from a gas sample such as C_4F_8 or SF_6. Then with calculations from the thermodynamic data for three nearly ideal gasses, the predicted scattered intensity can be found and compared with the observed scattered intensity to find the absolute intensity calibration constant. The principal investigator has reported on this technique several times recently[13,14,15] and has submitted a paper for publication.[16] Additional work by a student involved in this task has also been reported.[17]

Sample Holder

Through the courtesy of H. R. Letner of the General Electric Company, a number of Lucalox (Al_2O_3) tubes were obtained with niobium-zirconium alloy end caps such as are being used in a recently designed lamp. These tubes, I.D. circa 7/32" = 0.555 cm - O.D. circa 16/32", length 3 3/4" plus end caps, can be filled with saturated cesium vapor at temperatures between 664°C and 1000°C. Our source of information on the Lucalox tubes states

that, "all of these materials-Al_2O_3, niobium, and the tungsten coils in the end caps including the sealant used to fasten the end caps to the ceramic should resist attack by cesium vapor for long periods of time at temperatures of at least up to 700°C to 800°C." Aside from attack by cesium, the structures will withstand several hundred more degrees if not subjected to severe thermal shock.

These tubes could be sealed off, brought to Hiram College, and reheated to their saturation temperature in an x-ray diffractometer where the small angle scattering they produce will be observed. From the curve of scattered intensity vs. scattering angle it is expected that effects of the atomic interaction potential would be observed and calculations made to evaluate this potential.

Although the Lucalox tubes appeared to be ideal in many ways, Cs insertion, seal off, high temperature capability, electric discharge capability, etc., two serious problems were found with their use. First, it was noted that indium oxidizes completely in a few minutes' exposure to air at, say 800°C. Hence, throughout this treatment at elevated temperatures, they must be surrounded with an inert atmosphere or vacuum. Second, and more serious, was that the x-ray transmission for CuK_α is very low ($< .005\%$). Even with the wall thickness of the tube reduced from 1 mm to 0.4 mm the measured transmission was only 0.013%. This represents a transmission far too low for the observation of any scattering by the cesium vapor even using the high intensity x-ray tube. The transmission can be increased significantly by using a shorter wavelength radiation, e.g., Mo K_α but the scattering probability is also lower. In addition, the optimum sample thickness, ie the $1/\mu$ thickness for cesium, where μ is the linear absorption coefficient, is much larger for Mo K_α than for Cuk_α which means a sample cell approximately 10 cm long. A sample cell somewhat less than this would

still prove adequate but would have necessitated design and construction of a new sample cell as the Lucalox tubes could not be used. Other materials which have lower x-ray absorption coefficients were considered for use in the sample cell and are listed in Table I along with their calculated transmission for the two radiations for the 1 mm wall thickness of Lucalox. The transmission, τ, is calculated from $100 \times e^{-\mu t}$ where μ is the linear absorption coefficient in reciprocal centimeters and t is the sample thickness (twice the wall thickness for a cylindrical tube). All of these materials except Lucalox and beryllium were rejected due to the expected reaction with cesium at high temperature.

TABLE I

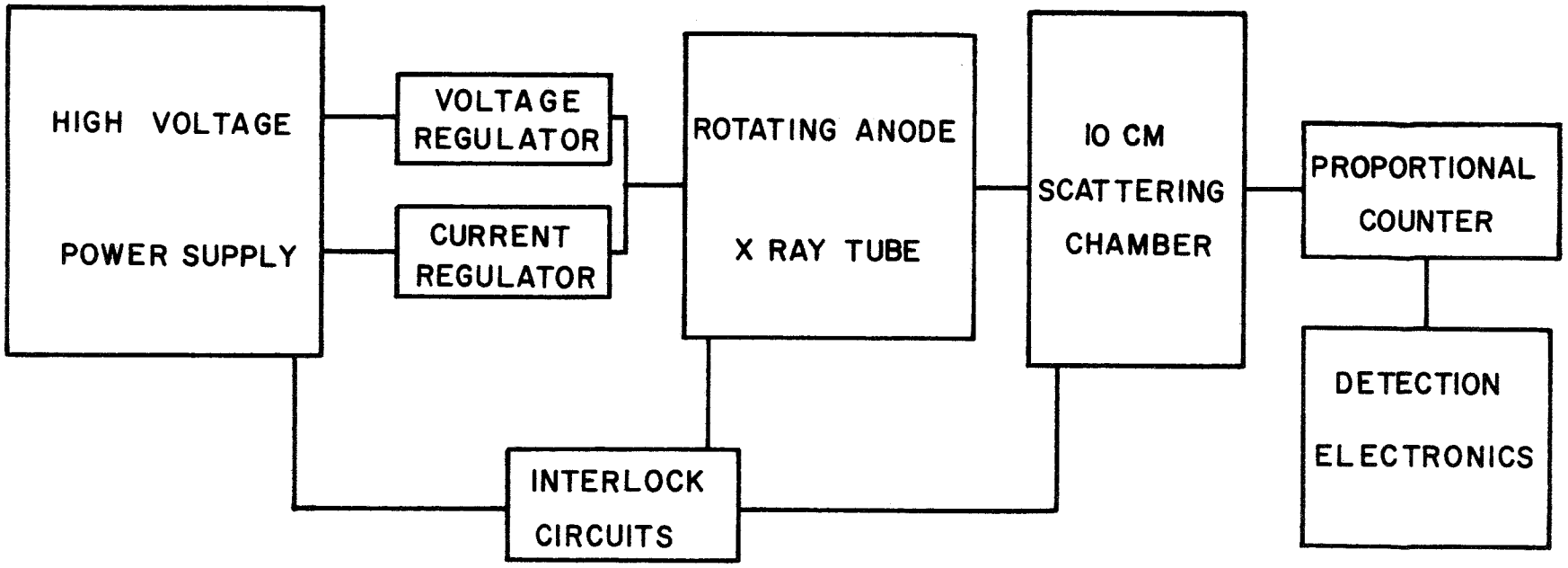
Calculated Transmissions for Various Cell Materials for Lucalox Wall Thickness (1mm)

	Cu K_α		Mo K_α	
Material	μ (Cm^{-1})	τ (%)	μ (Cm^{-1})	τ (%)
Lucalox	118	10^{-18}	12.5	8
Nickel	437	10^{-36}	420	10^{-34}
Quartz	91.9	10^{-6}	9.94	13.6
Beryillia	31.4	.18	3.80	46.7
Carbon	12.2	9	1.55	73.4
Beryllium	4.92	37.4	.55	89.6

IV. EQUIPMENT

X-Ray Diffractometer

A block diagram of the 10 cm x-ray diffractometer is shown in Figure 4. A detailed discussion of each major component of the diffractometer can be found in an earlier report.[18] The diffractometer was automated with the



BLOCK DIAGRAM OF
10 CM X RAY DIFFRACTOMETER
FIGURE 4

addition of a relay, stepping motor, and control unit[19] to the standard detection electronic circuit. This simple way of adding a step-scan mode to the diffractometer has been reported in the literature.[20]

Sample Holder Oven

A special oven and sample holder for the Lucalox tubes were designed and constructed so that the cesium sample holder was inside the 10 cm diffractometer scattering chamber. The design of the oven required that an upward extension be added to the steel scattering chamber. The oven was designed to go inside the steel chamber so that in case of accident all of the cesium would remain inside the steel chamber. Also, since it was possible to reduce the pressure in the steel chamber using a forepump, the problems of thermal isolation of the oven and the indium oxidation were solved.

A Foxboro potentiometer controller was incorporated into proper thermocouple and heater circuits to indicate and control the oven temperature.[21] Considerable time was spent testing the accuracy and reliability of this control because of the stringent temperature requirements. With this equipment it was possible to hold the thermocouple temperature to $\pm$ 1^{o}C at any desired value up to close to 1000^{o}C. However, there was some uncertainty as to the actual temperature of the Lucalox tubes.

These tubes were enclosed in a copper bar approximately 1" in diameter with an axial hole for the Lucalox tube. For irradiating the cesium vapor and for observing the scattered x-rays it was necessary to cut away the copper bar at the level of the x-ray beam. These entrance and exit apertures in the copper bar permit the Lucalox tubes to radiate energy to

the outside of the furnace where a lower temperature prevails at the very region where the scattering is to be observed whereas the remainder of the tubes are enclosed in the copper bar where the thermocouple is located. Attempts to position a thermocouple in the region of the apertures in the copper jacket were unsuccessful for a number of reasons. Thus there was an annoying uncertainty as to the exact temperature of the cesium vapor just at the region where that was most important.

CONCLUSIONS

This phase of the research project was included in the proposal as an exploratory project for two years and then if deemed feasible, the experiment would be performed. Due to equipment delays on the single crystal task more emphasis was placed on the cesium project during the second year of the project than originally planned and some equipment modifications were made. However, the technological problems discovered in the design of a suitable cesium gas sample cell along with special attention to the safety procedures involved with cesium and the difficulty of filling the tubes with appropriate amounts of cesium led to the conclusion that this project would not be pursued at Hiram College under the present grant.

PART II

SINGLE CRYSTAL DAMAGE STUDIES

The primary objective of the work proposed under this grant was to investigate the effect of ion bombardment on the surface structure of single crystal surfaces and in particular to use x-ray methods to measure the dislocation density before, during, and after ion bombardment as a function of bombardment time and ion mass. X-rays have been used extensively in the study of crystalline structure and perfection. Recent interest in the density and array of dislocations produced during plastic deformation in metal crystals has resulted in the study and application of three methods for observing these particular measurements of crystalline perfection: the Debye-Scherrer, microbeam Laue, and double crystal spectrometer rocking curve methods.[22,23,24]

The double crystal rocking curve method was chosen as the best for the proposed problem due not only to geometry considerations and to the use of counter rather than photographic x-ray detection, but also to the check it provides on the inter al consistency of the dislocation measurements. The half width of the double crystal x-ray spectrometer rocking curve can be resolved into components depending on lattice misorientations due to dislocation tilting, on the dislocation strain interaction, on the subgrain particle size, and on uniform lattice bending. Each broadening component can be related to the dislocation density thus providing some indication of internal consistency. The separation of these four effects

in the experimental rocking curve is accomplished by observing the rocking curve broadening as a function of Bragg angle for either four orders of the same line or by measurements on one order using four wavelengths.

Combining the double crystal spectrometer with an ultra high vacuum system was a difficult technological task. The crystal surface under investigation must be aligned in the double crystal spectrometer and also be available for the sputtering and annealing processes inside the vacuum chamber. Much effort was expended in designing and assembling a suitable system to perform the work.

EQUIPMENT - DESIGN

The design of a double crystal x-ray spectrometer with enough flexibility for all the vacuum requirements, crystal motion requirements, ion gun and electron gun geometry, etc. was quite a demanding task. Design ideas began with a "vacuum-oriented" system in which both crystals were mounted on precision rotary feedthroughs in the same 18" diameter vacuum chamber. This design had the advantage of future flexibility with the vacuum system and convenient interchange of ion bombardment, electron bombardment, and x-ray analysis procedures. However, not only was the placement of the x-ray exit and entrance windows a problem so as to have access to all the desired Bragg reflections, but also the requirements on the rotary motion feedthrough devices were quite stringent due to the crystal alignment requirements. The crystal faces must remain parallel to a given (usually vertical) line to within ± 5 seconds and for complete analysis must be rotated through angles as small as 1 second about a vertical (usually) axis.

Two bids were received on the above system each approximating $28,000, with one (Varian) being submitted only after a design contract to consider the precision rotary feedthrough requirements. A bid on a slightly modified system to that above was received from a third company, but their bid was quite high due to their limited capability. The receiving of bids was one of the hindrances to the grant program.

The next design idea was an "x-ray-oriented" system which featured and 8" vacuum chamber, which could be precisely located on a standard double crystal spectrometer for x-ray analysis of a crystal mounted inside and then could be attached to a vacuum system through a valve arrangement so that ion bombardment and rough pumping was accomplished through the valves. This design had the advantage that the necessary tilt and translation adjustments on the crystal could be made external to the vacuum chamber by positioning the entire chamber on a precision spindle in the air. However, the 8" vacuum chamber was limited to approximately 72 pounds due to load limitations of the precision spindle. Thus a vacuum system was necessary and provided space for the large ion pump, roughing pumps, ion gun, partial pressure gauge tube, etc. This meant that the ion bombardment took place with the ion beam traveling through the 2 straight-through valves. Also, the crystal had to be rotated 180° to face the ion beam and then returned to the same position under the requirements for crystal location given above for the x-ray analysis.

After considering the advantages and disadvantages of the two systems described above a contract was let on 29 December, 1966, to Varian Associates to construct the special chamber, provide a standard 12" vacuum system, and supply the partial pressure analyzer.

The double crystal x-ray spectrometer had to be designed to provide for the support and alignment of the 8" vacuum chamber above. Also a beryllium window had to be provided on the vacuum chamber to permit entrance and egress of the x-ray beam. Since the required design was similar to a spectrometer already available at the Ohio State University Physics Department and since the anticipated use of the spectrometer at Ohio State over the next few years was low, a request was made to Professor E. L. Jossem, Chariman of the Physics Department, to loan the spectrometer to Hiram College with the understanding that we could make certain modifications to the spectrometer for our use. This request was approved, and with the help of Professor Jossem, the spectrometer was transported to Hiram and reassembled. The two spindle axes were made parallel mechanically using a spirit level to less than 10 seconds of arc, the crystal faces were made parallel to the spindle axes to within a few seconds of arc, using a gauss eyepiece telescope, and each crystal face was made to contain the spindle axis to within 0.001 inch using a microscope. The appropriate crystal adjustments for the above are provided in the crystal holder design. Using two quartz crystals, the x-ray beam was reflected through the spectrometer and the final alignment was accomplished.

EQUIPMENT - FABRICATION AND DELIVERY

The construction of the vacuum system by Varian Associates involved many delays. From the first discussion it was known that the two difficult problems to be solved were the precision rotary feedthrough and the beryllium window. Almost everything else about the system was of standard design

and specifications. However, even after a Design Contract to work out the problems of the system design, Varian still had difficulty meeting specifications on the system. Shipment was promised on April 21, May 5, May 26, June 30, July 14, July 21, and July 28, 1967. On September 11, the Varian Service Engineer arrived to install the system and do the final check-out at Hiram. During the check out it was determined that the special rotary feedthrough device did not meet the specifications for either angular resettability or for axial alignment. In addition, during the field check-out of the feedthrough, the beryllium window on the vacuum chamber was accidently broken.

After the redesign of the feedthrough and the replacement of the beryllium window, the chamber was received at Hiram, on January 17, 1968. The Varian Service Engineer arrived January 23, Tests on the rotary feedthrough device at Varian Associates before shipment indicated that the specifications had been met.

Since the delivery delays and specification difficulties were of major importance in the performance of work on the grant, two letters from the principal investigator to Varian Associates are attached in the Appendix. The letter to Mr. McGarr of February 23, 1968, details the system deficiences and the letter to Mr. Dorney includes a listing of the contacts made between Hiram College and Varian Associates.

The ion gun, which was furnished by NASA-Lewis Research Center, also was a source of delay. After the vacuum system was accepted, tests were begun on the ion gun. A vacuum leak was discovered in one of the electrical feedthroughs in an octal socket. Repeated attempts to repair the leak with Torr Seal[25] were unsuccessful. The ion gun was returned to Lewis Research Center for replacement of the octal feedthrough. Due

to hard-to-obtain parts and a defective feedthrough this repair job took over six months to complete. After the ion gun was returned to Hiram, another leak was discovered around the leak tube feedthrough. Again, the attempts to repair this leak with Torr Seal were unsuccessful so the ion gun was disassembled and the leak was repaired using soft solder. The ion gun was put into service on April 25, 1969.

EQUIPMENT - FINAL CONFIGURATION

As described in the first Semi-Annual Status Report[26] the vacuum environment around the crystal during the ion bombardment and x-ray analysis is difficult to maintain at 10^{-10} Torr and still provide adequate adjustments for alignment of the crystal in the x-ray beam. Thus an "x-ray oriented" system was designed in which a special x-ray chamber containing the crystal is connected through two valves to a standard ultra-high vacuum system containing the ion gun.

The x-ray chamber is designed with a Beryllium window wrapped around the side of the 6" diameter chamber so that a 180^{o} view of the crystal face is possible with the x-ray beam as shown in Figure 5. A special rotary feedthrough device had to be designed so that the crystal could be turned 180^{o} to face the ion beam and then returned precisely (to within $\pm$ 10 sec on both axes) to face the x-ray window. The ion beam travels from the ion gun through the 12" chamber and both 1 3/16 valves into the x-ray chamber. Five electrical feedthroughs are provided into the vacuum for use in annealing the crystal, bias voltage, thermocouple wires, etc. Other ports on the chamber allow for a viewport

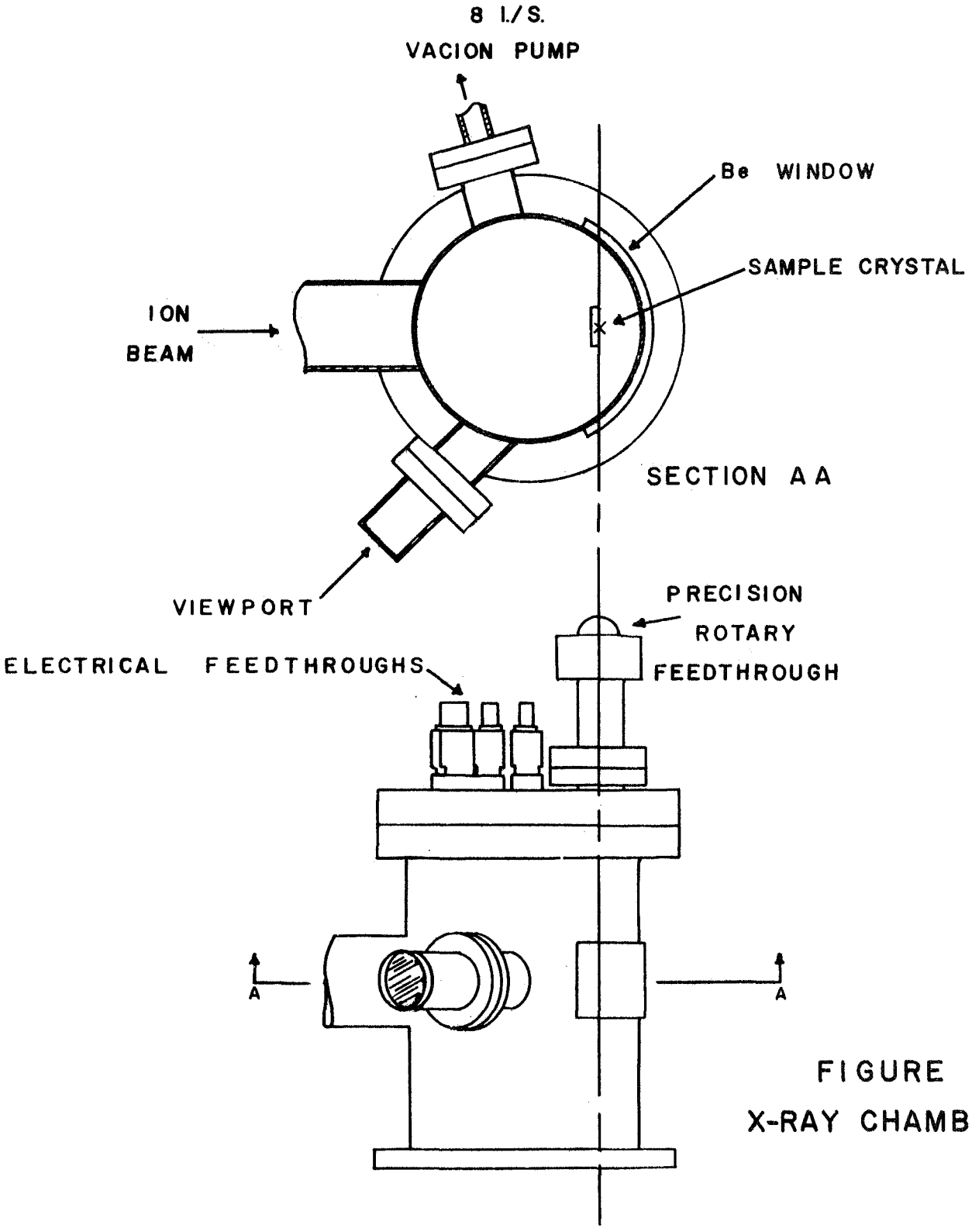
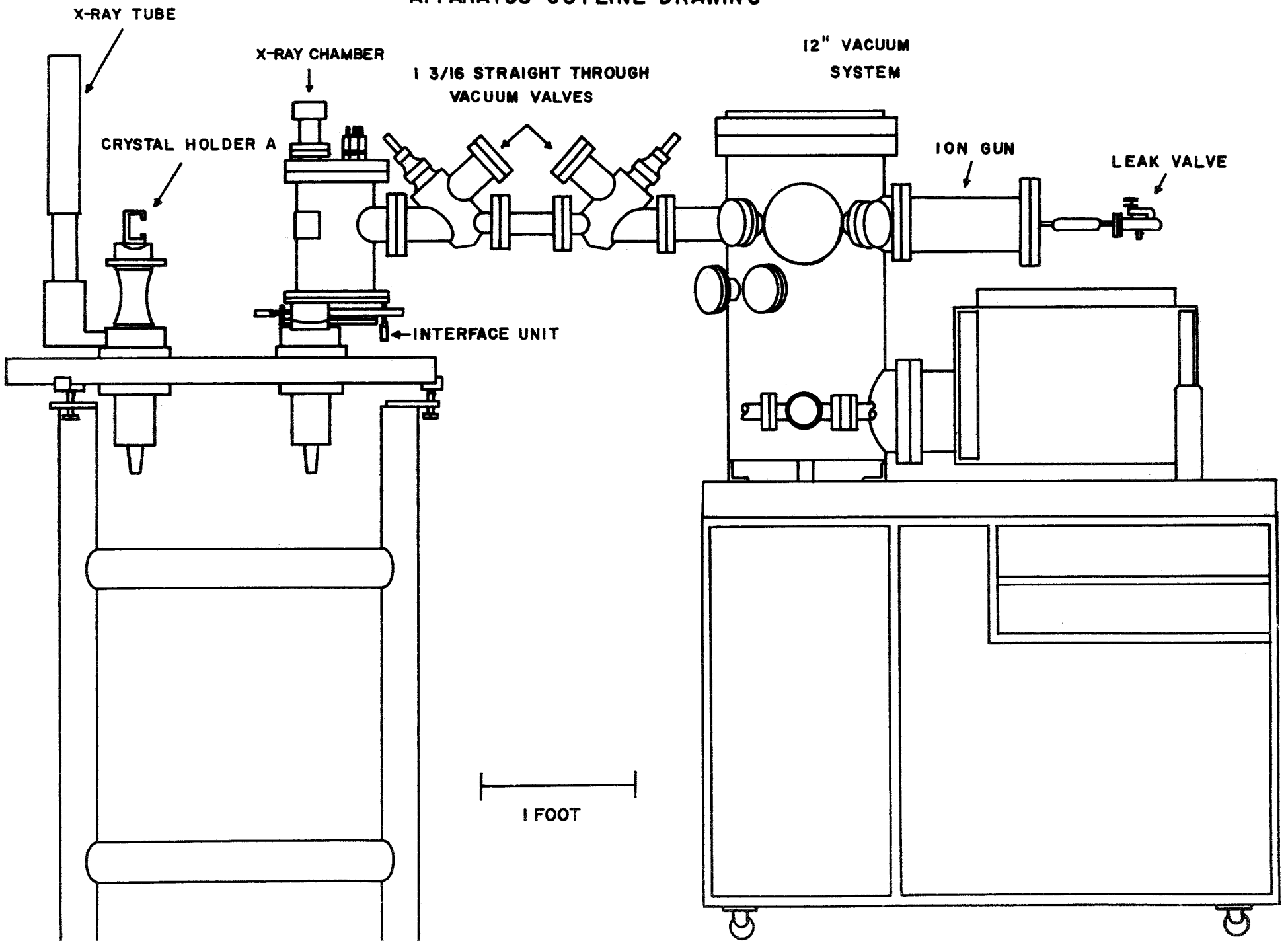
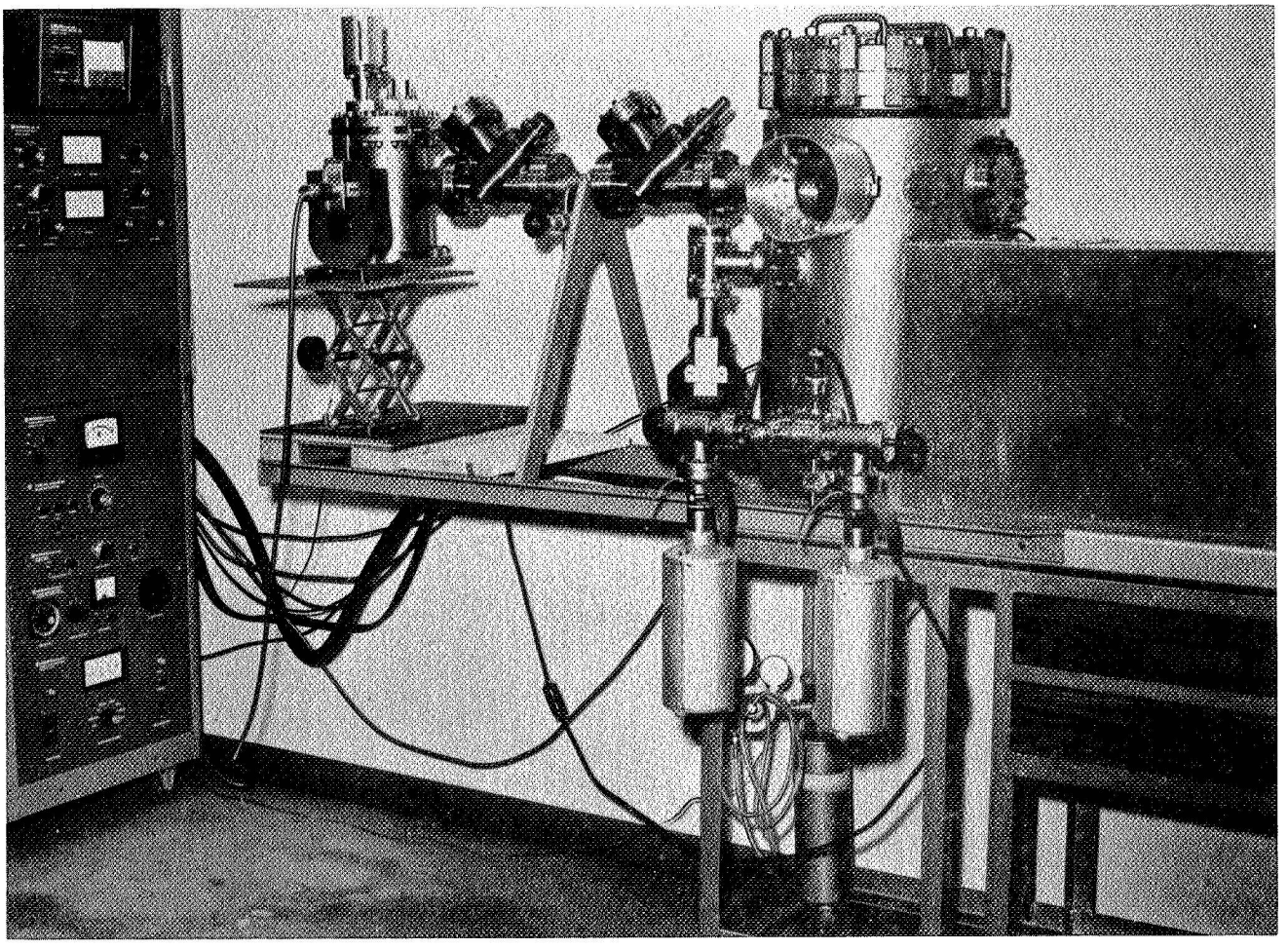


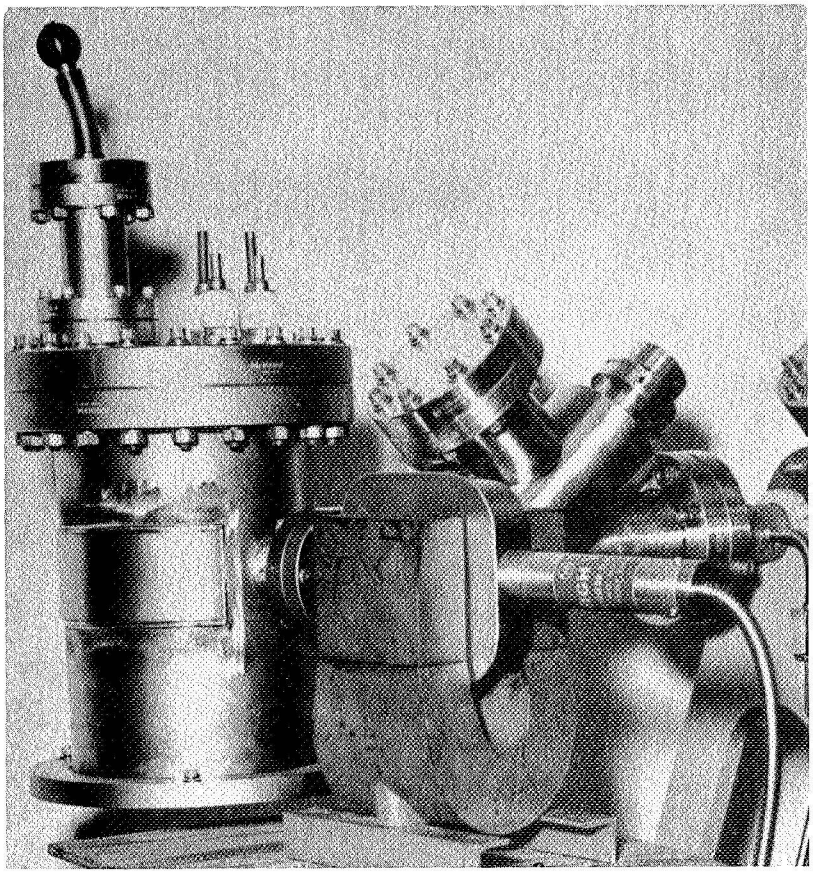
FIGURE 5
X-RAY CHAMBER

FIGURE 6

APPARATUS OUTLINE DRAWING

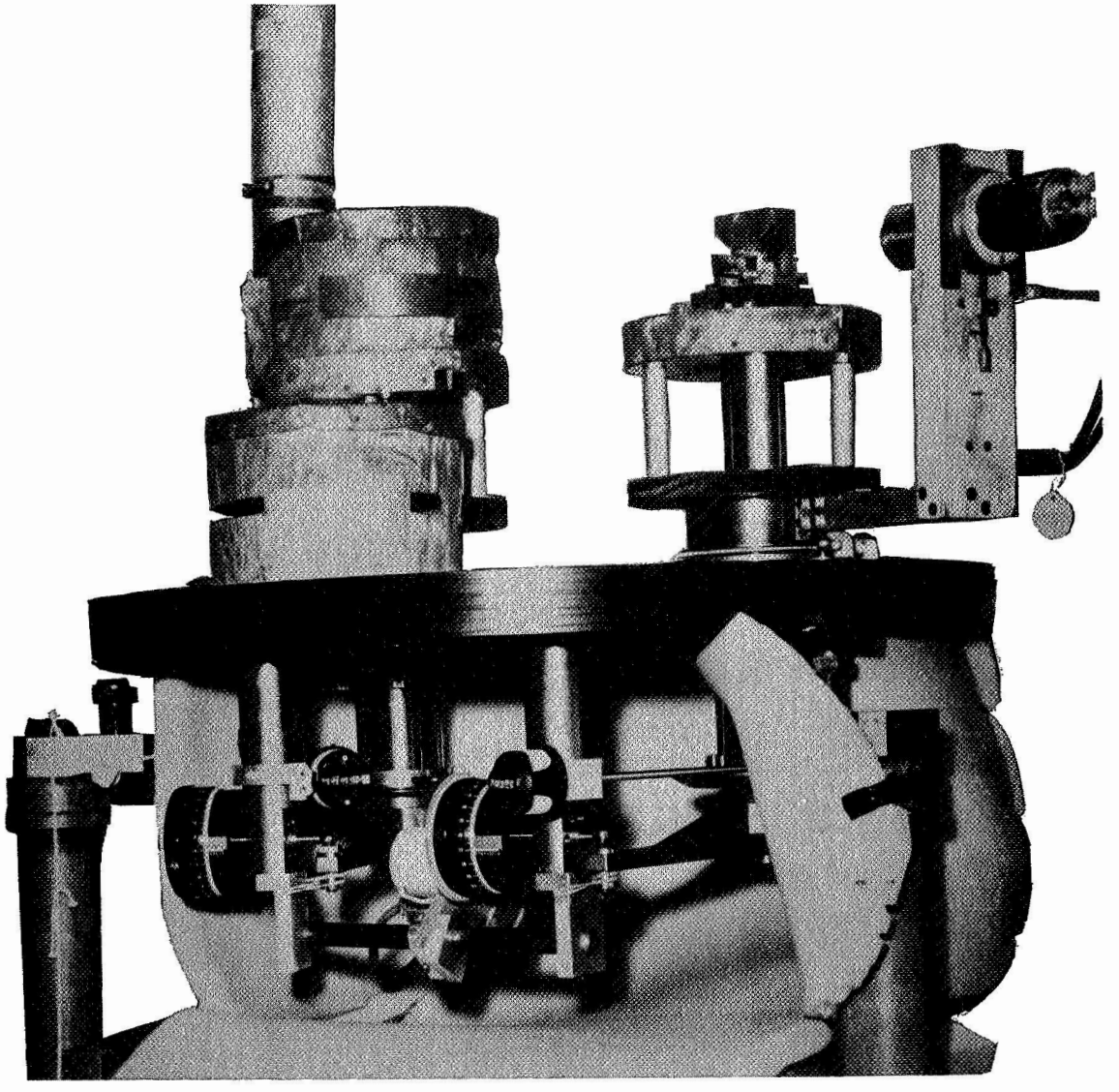






12" Vacuum System and X-Ray Chamber

Figure 7



Double Crystal X-Ray Spectrometer

Figure 8

facing the crystal axis, an 8 l/s Vac Ion Pump, and the 1 1/2" straight-through valve connection to the standard vacuum system. The weight of the special x-ray chamber had to be kept under 70 pounds due to the load limitations on the precision Whitnon[27] spindle on the double crystal spectrometer described later in this report. Normally the x-ray chamber will be disconnected from the standard vacuum system when positioned on the x-ray spectrometer and both systems will be maintained under vacuum conditions. An outline drawing of the standard vacuum system, ion gun, and double crystal spectrometer with the x-ray chamber is shown in Figure 6. Photographs of the standard vacuum system and x-ray chamber are shown in Figure 7. The standard vacuum system[28] is the Varian VI - 321; a bench-top, 12" diameter, ultra-high vacuum system. The pumping equipment consists of two aluminum sorption pumps for rough pumping, a 140 l/s diode sputter ion pump, and a titanium sublimation pump. All ports are sealed with conical sealing sexless flanges utilizing a captured OFHC copper gasket except for the 12" diameter Wheeler Flange at the top of the chamber which uses a captured OFHC copper wire gasket. The entire system is provided with strip heaters and aluminum shrouding for uniform and automatic system bakeout to 250°C. The total pressure of the system can be measured to 2.6×10^{-11} Torr with the nude ionization gauge[29] and the partial pressure of gases up to mass number 70 can be measured and recorded to a sensitivity of 2×10^{-11} Torr with the Partial Pressure Analyzer.[30]

The double crystal x-ray spectrometer is shown in Figure 8. As originally designed at Ohio State by Professor E. L. Jossem the spectrometer consisted of the special baseplate casting of minivar and two Whitnon spindles for providing the precision parallel axes. In addition, two

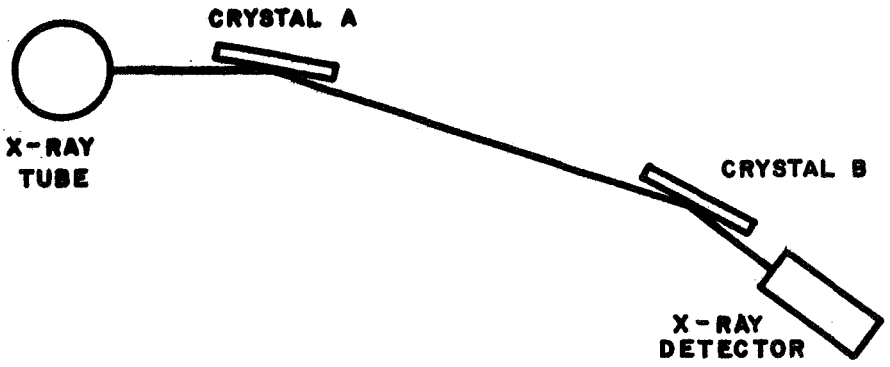
special crystal holders adjustable for tilt, translation, and crystal thickness were mounted on the Whitnon spindles. The precise angular motion was achieved with two Boeckeler[31] micrometers which gave angular resolution of 0.4 sec when used with the 10" tangent arms. Both the x-ray rube and x-ray detector can be set to 0.01^{o} around the axes determined by the Whitnon spindles.

Since obtaining the spectrometer for use at Hiram College the following modifications have been made: 1) the design and construction of an interface unit to mount on the Whitnon spindle which will translate and tilt the x-ray vacuum chamber described above; 2) the addition of two new Boeckeler micrometers increasing the angular resolution to 0.2 sec; 3) the addition of the appropriate gears and stepping motor to automate the data-taking process with a variable step size from 0.5 sec to 5000 sec.

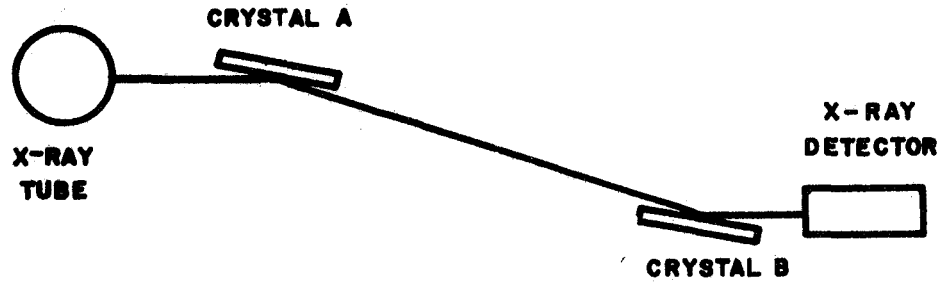
Figure 9 shows the x-ray path through the spectrometer with the crystals in the parallel and antiparallel positions. At present the x-ray tube is a Machlett Z-2L line focus diffraction tube with a copper target.[32] An earlier tube stopped operating after 30 hours and was replaced under warranty due to a cracked filament support. The x-ray detection system is similar to that described for the 10 cm x-ray Diffractometer[33] with the exception of the stepping motor electronics[34] and is shown in Figure 10.

The high voltage power supply used to supply anode voltage and filament current to the Machlett x-ray tube was constructed of an old surplus Westinghouse transformer and rectifier unit. This power supply operated somewhat successfully for awhile but the current regulation was not good. Also humidity caused severe leakage problems. Modifications

FIGURE 9



ANTIPARALLEL POSITION

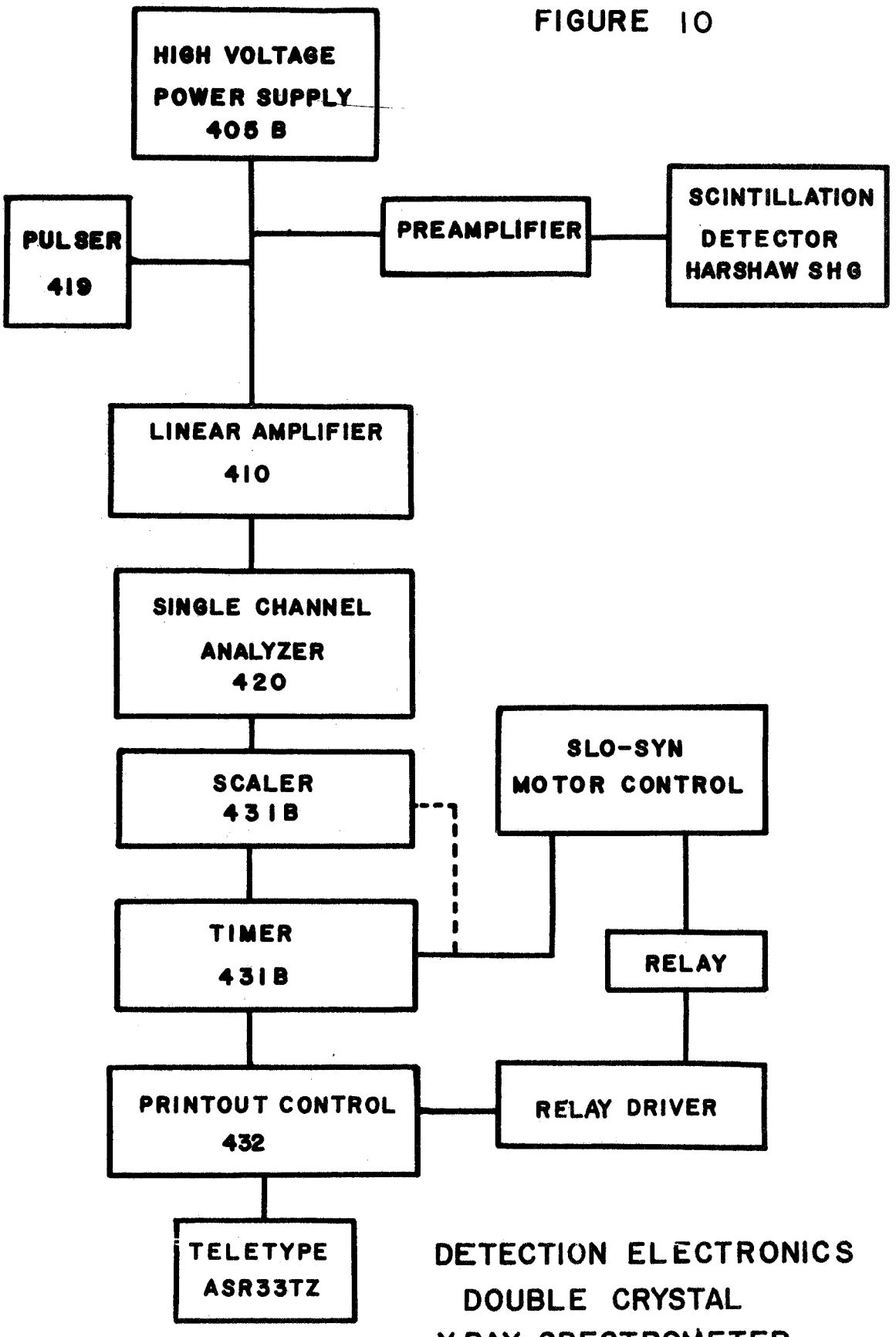


PARALLEL POSITION

X-RAY BEAM OPTICS

DOUBLE CRYSTAL X-RAY SPECTROMETER

FIGURE 10



DETECTION ELECTRONICS
DOUBLE CRYSTAL
X-RAY SPECTROMETER

were not successful in improving the operation of this power supply so it was replaced with a standard x-ray generator.[35]

Fortunately the spectrometer available from Ohio State was built on a large scale making the adaptation to use with the vacuum chamber fairly simple. With a spacing of 13.5 inches between crystals and the x-ray plane 9.5 inches above the baseplate, there was adequate room to design the interface unit to hold the vacuum chamber on the spindle. The base of the vacuum chamber is specially machined with a hole, groove, and slot to accurately reposition the vacuum chamber onto three steel balls on the interface unit. The entire vacuum chamber can then be tilted and translated so that the crystal planes are parallel to and contain spindle axis of rotation (see Figure 6).

The alignment of the two crystals in the spectrometer is quite critical. The preliminary alignment procedure has already been described. For the alignment using the x-ray beam the procedure of Schnopper[36] was adopted. If the crystals are not properly aligned, distortions are introduced into the rocking curves which affect the line shape, width, symmetry, and Bragg angle.[36] Thus it is imperative that the spectrometer be properly aligned and that the procedures be understood. During the alignment studies, two quartz crystals were used which had very narrow (1,-1) rocking curves as shown in Figures 11 and 12. These crystals had been previously studied and the present rocking curves agree with the earlier data.[37] However, the crystal closest to the x-ray tube (Position A) sustained considerable radiation damage as evidenced by a very dark discoloration streak across the face of the crystal. This damage produced no discernable change in the rocking curve shape or width.

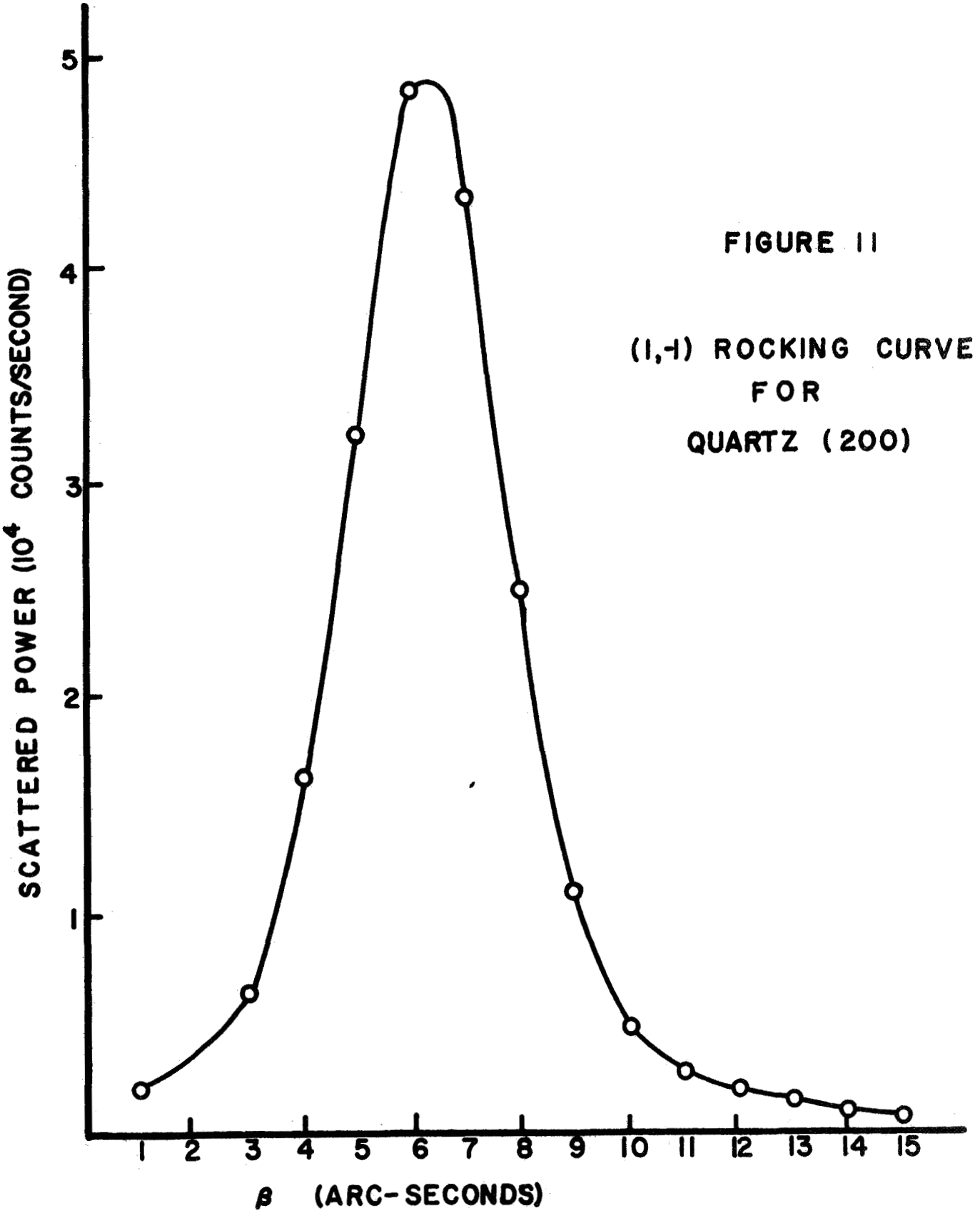
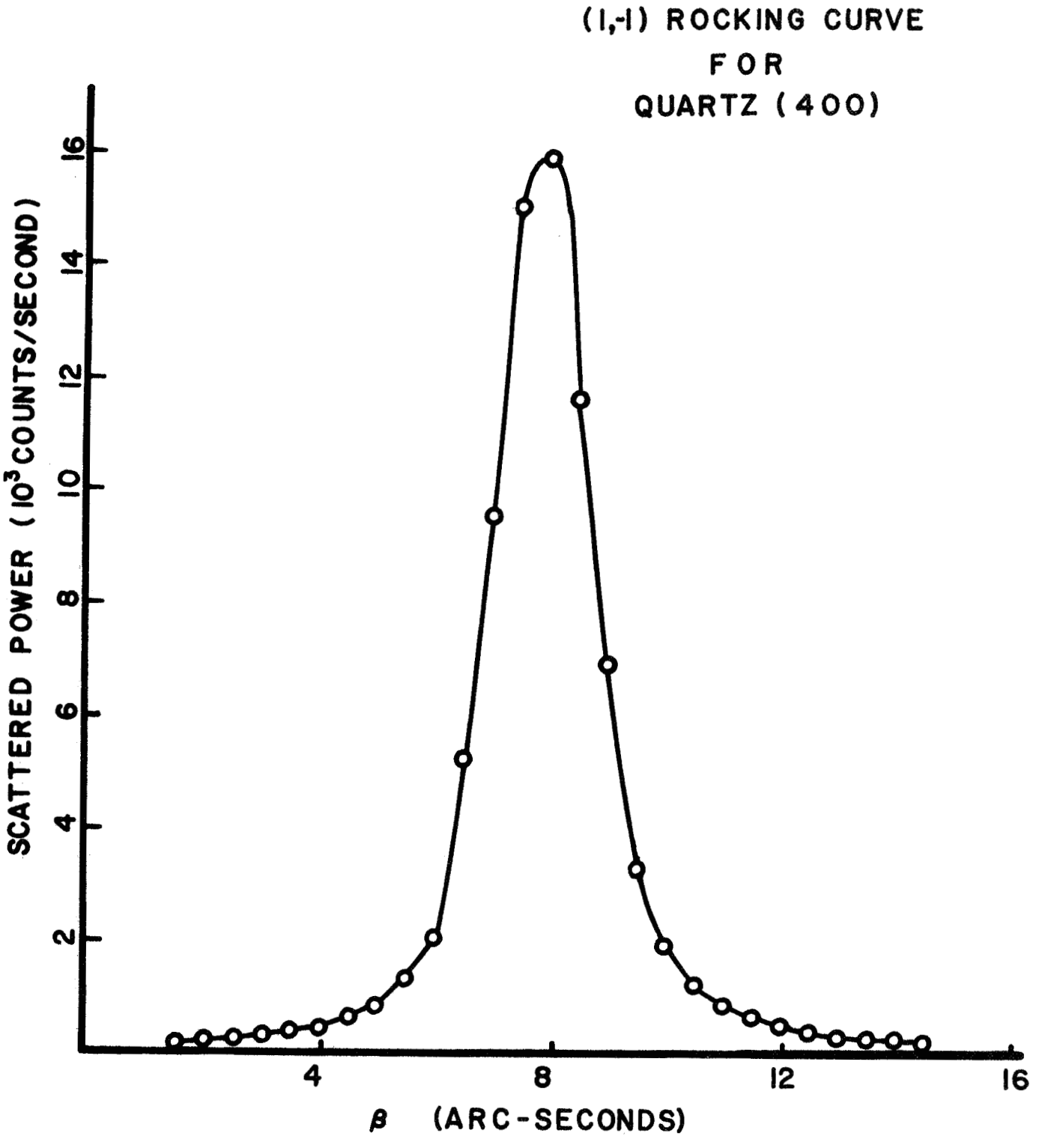


FIGURE 11

(1,-1) ROCKING CURVE FOR QUARTZ (200)

FIGURE 12


(1,-1) ROCKING CURVE
FOR
QUARTZ (400)
SCATTERED POWER (10^3 COUNTS/SECOND)
2
4
6
8
10
12
14
16
4
8
12
16
β (ARC-SECONDS)

EQUIPMENT - DEFICIENCIES

The vacuum system as outlined heretofore and constructed by Varian Associates did not meet the original specifications for the vacuum environment in the x-ray chamber of 5×10^{-10} Torr. The earlier deficiencies of the rotary feedthrough device and the Beryllium window have already been described. During the check-out of the vacuum system in January, 1968, by the Varian engineer it was observed and concluded that the x-ray chamber could not be maintained at 5×10^{-10} Torr with the valve closed, i.e. using only the holding pump. In addition it was decided that there was no way to measure reliably, the pressure in this chamber other than pump current. Due to the method by which Varian attached the Beryllium window to the stainless steel chamber the differential thermal expansion limits the bakeout to $120^{o}C$. Thus with the original 1 l/sec holding pump the chamber could be evacuated to 1×10^{-7} Torr and with an 8 l/s pump the chamber could be evacuated to 2×10^{-9} Torr. There has been no difficulty with the standard 12" Varian vacuum system only with the x-ray chamber. The standard system has several times "bottomed out" the ion gauge at 2.6×10^{-11} Torr.

Thus due to the system deficiences described above and the long delivery - original shipping date was April 21, 1967, the system was delivered the first time on August 19, and the second time on January 17, 1968, - Hiram College requested a renegotiation in the system price. See the Appendix for a summary of the contacts between Varian Associates and Hiram College.

EXPERIMENTAL TECHNIQUES

The procedure used to obtain the discharge in the ion gun is outlined in Table 2. The three hour warm up on the magnetic field coil was necessary to establish thermal and vacuum equilibrium conditions; otherwise continuous adjustments to the coil were required.

TABLE II

Turn Up Procedure for Ion Gun

1. Outgas magnetic field coil (@ 8A) and filament (@ 12A) for 3 hours prior to run.
2. Set anode voltage to 60 V.
3. Adjust leak rate of argon to 2-3 x 10^{-6} Torr.
4. Turn on high voltage power supply to 900 V. for screen voltage.
5. Acceleration voltage to 760 V.
6. Decrease magnetic field current to about 4 A to obtain discharge.
7. Then optimize the discharge by adjusting filament, magnetic field current, screen voltage, and leak rate.

Some typical operating parameters are filament current, 9.6 A; screen voltage, 800 V; acceleration electrode voltage, 760 V; magnetic field current, 5.2 A; anode current, 390 mA; anode voltage, 60 V; beam current, .48 mA; and crystal current, 125 μA. Figure 13 shows a schematic of the ion gun with all the parameter meters listed above. A Heathkit[38] strip chart recorder was used to continuously monitor the ion beam reaching the crystal. A full scale reading of 125 μA was obtained using

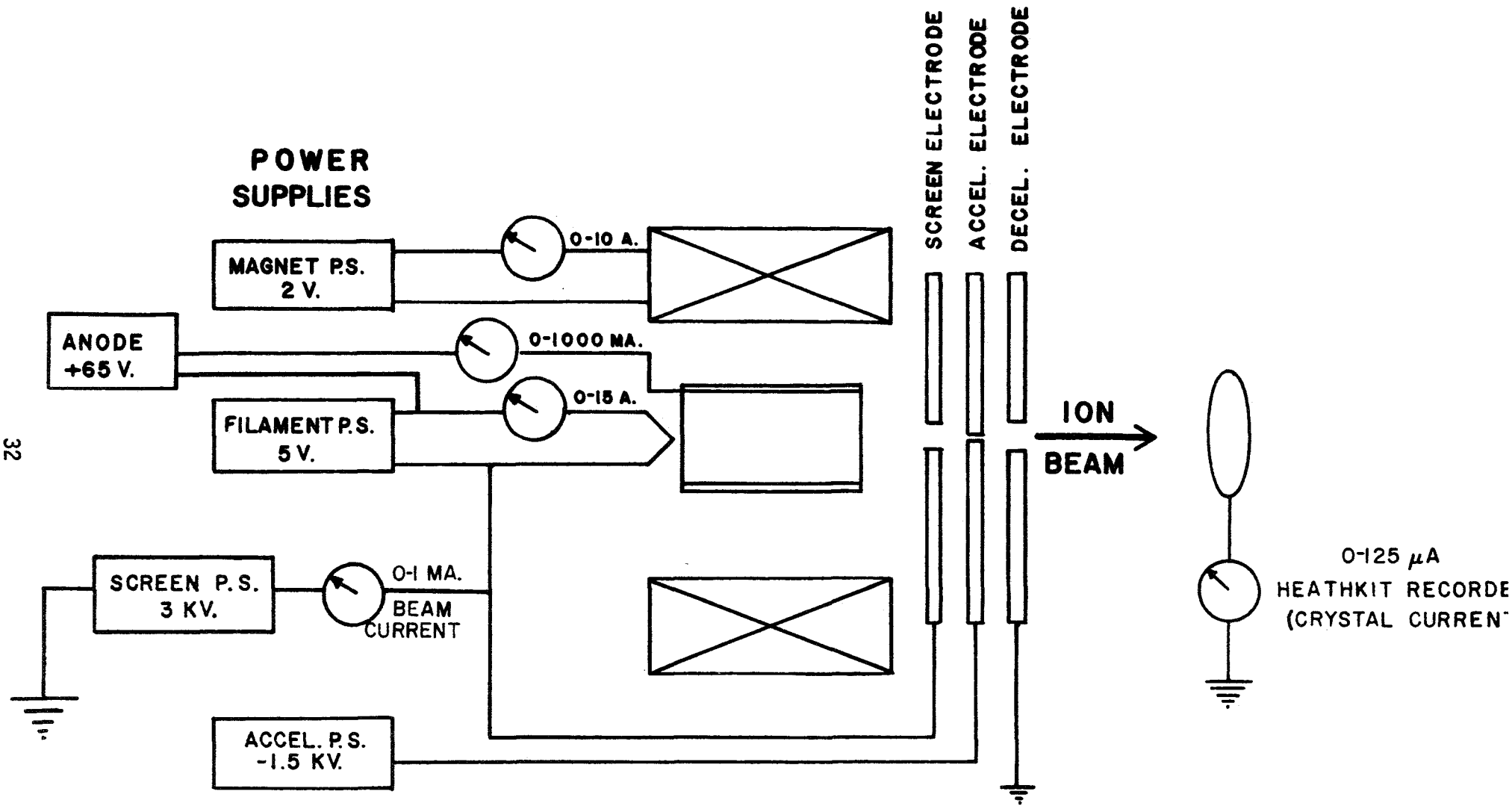


FIGURE 13

ION GUN SCHEMATIC

a 2 KΩ resistor in parallel with the recorder input. The total beam exposure on the crystal was ound by adding up the currents a 1 minute intervals and expressing the result in ion current x time or Coulombs. The crystal beam current stability depends critically upon the pressure range, filament current, and magnetic field current. Fluctuations from 110μA to 125μA were not uncommon.

The Varian ion pump apparently develops an "argon instability" in teh pressure range around 5 x 10^{-6} Torr and begins a pressure cycling up and down which usually results in the pump shutting off, i.e. the pressure rising above 1 x 10^{-4}, unless the argon leak rate is reduced. This cycling usually lasts from 5 to 30 minutes before shutdown occurs. Of course this instability causes problems with the ion beam. Attempts to hold the pressure using only the TSP, i.e. turning off the ion pump have not been successful. If the pressure is kept below 5 x 10^{-6} Torr, the pump instability usually does not occur.

The Argon gas is of a research grade purchased from Air Porducts and Chemicals, Inc.[39] The gas is delivered to the leak valve[40] at 2-3 psig.

The annealing circuit is shown in Figure 14. For these tests a copper single crystal[41] was mounted on the sample holder. This crystal was 1 inch in diameter and 1/4 inch thick. Thus an indirect heating method (electron bombardment) was used. At 1800 V and 50 mA the annealing temperature of 700^{o}C was reached in about five minutes with the vacuum staying at 1 x 10^{-7} Torr. Unfortunately during the several operations of bolting and unbolting the 8 inch flange on the x-ray chamber a small leak developed in the weld of the Beryllium window to the chamber walls. So far the leak has been repairable using Torr Seal.

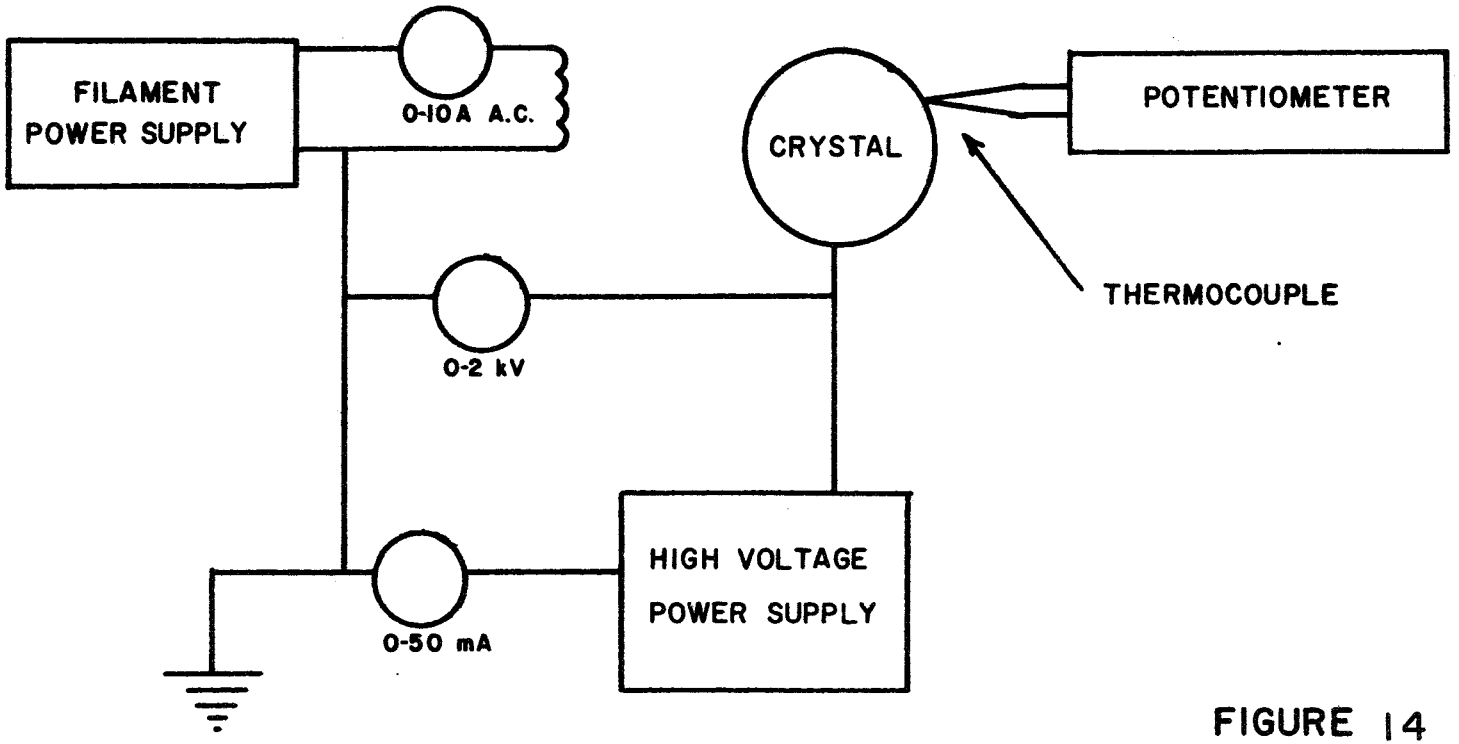


FIGURE 14

ELECTRON BOMBARDMENT
ANNEALING CIRCUIT

The double crystal spectrometer rocking curves are obtained by placing the crystal to be investigated in crystal position B (see Figure 9). A matched crystal technique similar to that described by Kaufman and Kulin was used.[42] The double crystal spectrometer theory requires two identical perfect crystals. By using the matched crystal technique one can approximate this condition but still examine fairly imperfect crystals by choosing a nearly perfect crystal.

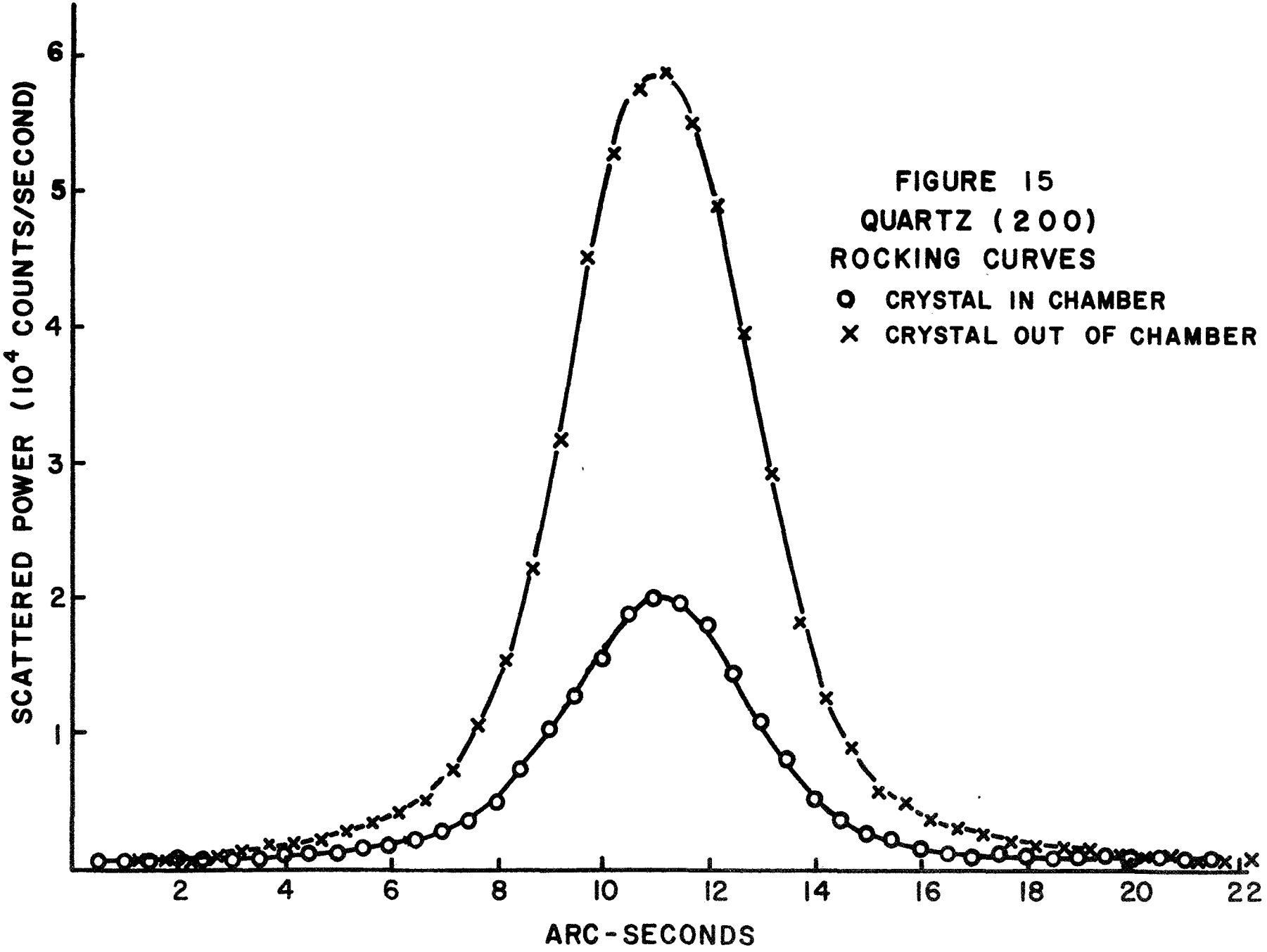
The planar spacings of crystal A were closely matched with the spacings of crystal B. The rocking curve was then recorded by step-scanning the angular drive through a series of predetermined angles close to the Bragg reflection of interest. The shape and half-width of this curve contain the information indicating the extent to which the crystal differs from a perfect crystal. Standard computer techniques were used to analyze the data as described below.

To permit rocking curve measurements on crystal B while under a vacuum environment the equipment described above is necessary. The placement of the x-ray chamber on the spectrometer is extremely critical. The following tests indicated that this phase of the work had been completed satisfactorily.

The check-out tests for the rotary feedthrough in the x-ray chamber and the interface unit for mounting the x-ray chamber on the double crystal spectrometer were performed in December, 1968. These tests consisted of determining the precision of the rotary feedthrough resettability after an 180^{0} rotation and determining the precision of repositioning the x-ray chamber on the interface unit. To position the x-ray chamber on the double crystal spectrometer so that the quartz crystal was in proper x-ray alignment it was necessary to

remove the 8 l/s ion pump, the viewport, and the 1 3/16 straight through valve. Then by mounting a mirror on the face of the crystal so that it extended beyond one end of the crystal it was possible to use a Gauss eyepiece and telescope to adjust the crystal orientation quite closely to the x-ray position. The translation adjustment was made using a dial gauge contacting the mirror. Then the mirror was removed and the final orientation was determined using x-rays. Once the peak position was found and the appropriate tilt adjustments[36] were made the chamber was removed and replaced a total of seven times. The change in the position of the peak of the quartz (200) rocking curve was less than $\pm$ 2 seconds of arc. The rotary feedthrough was then tested by rotating the crystal through 180^{0} and then back against the stop a total of seven times. The test performed with the crystal in air indicated a precision of $\pm$ 11.5 seconds of arc and with the chamber evacuated the resettability was $\pm$ 8 seconds. With the chamber reassembled for evacuation a counterweight of 6 kg located about 16 inches from the chamber was necessary. Thus the design of both the rotary feedthrough and the interface unit appears to be within the original specification.

The quartz crystal was used in the chamber for the above check-out since it has an extremely narrow rocking curve and thus was quite sensitive to small changes in the mechanical mounting and crystal rotation. Figure 15 shows a quartz (200) rocking curve for the crystal both in and out of the chamber to illustrate the effect of the Beryllium window on the diffracted x-ray beam.


SCATTERED POWER (10^4 COUNTS/SECOND)
1
2
3
4
5
6
2
4
6
8
10
12
14
16
18
20
22
ARC-SECONDS
FIGURE 15
QUARTZ (200)
ROCKING CURVES
O CRYSTAL IN CHAMBER
× CRYSTAL OUT OF CHAMBER

The automation of the data taking process on the double crystal spectrometer was similar to that described for the 10 cm diffractometer and has been reported in the literature.[20] A recent improvement has been the use of the PIP-400 in the multiscale mode to record the rocking curve data so that easy comparisons of successive rocking curves can be made. The PIP-400 has three data output options: photograph of cathode ray display, x-y recorder output, and digital readout into a 33ASR Teletype page printer and paper tape punch. For this purpose up to four rocking curves can be stored and overlapped for easy comparison before readout, usually by photograph. The PIP-400 is an option in addition to the regular readout of data through the ORTEC 432 and the other Teletype unit.

The data taking process for the ion bombardment damage and annealing studies consisted of the following procedure:

1. sputter crystal with ion beam - x-ray chamber attached to vacuum system
2. take rocking curve - x-ray chamber positioned on double crystal spectrometer - repeat 1. and 2. until adequate sputtering has occurred.
3. anneal crystal - x-ray chamber attached to vacuum system
4. take rocking curve - x-ray chamber positioned on double crystal spectrometer

The rocking curve information was recorded on punched paper tape and then taken to the Computer Center. A data reduction routine has been programmed for the IBM 1130 system to analyze the rocking curves from the double crystal spectrometer. Input was the tape from the teletype, containing power and time from the counting electronics and the

step size of the abscissa. The abscissa (2θ in seconds) could contain up to 400 points. The routine found the centroid, the maximum, and width at half maximum using a least squares polynomial fit in appropriate regions for interpolation. It also found the Fourier coefficients for the normalized curve and evaluated the series to check the accuracy of the coefficients. Vectors of the power (input, normalized, and recomposed), the square root of the input count, and the corresponding time and vectors of Fourier coefficients were output in tabulated where appropriate, graphical form. The original data was then punched onto cards for storage. The output of the program also included ample parameters for the evaluation of the fitting and interpolation process.

After the above job had been executed the vectors and parameters remained available in disk storage for a second routine that allowed the programmer to vary parameters on either a Gaussian or Cauchy function to get the best least squares fit on each function.

RESULTS OF THE CRYSTAL INVESTIGATION

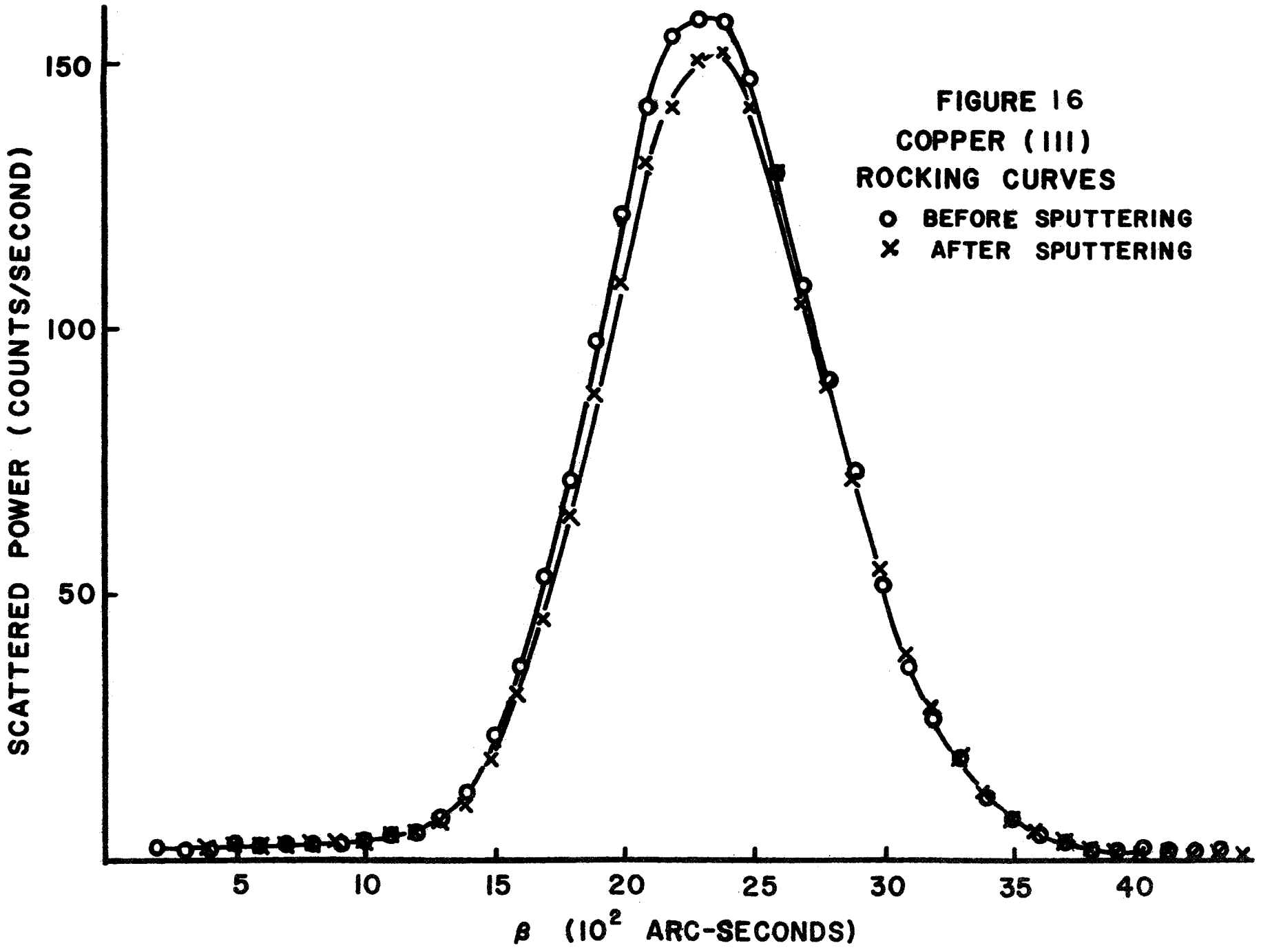
These crystals have been through a sputtering and annealing sequence along with the appropriate rocking curves. The crystal was placed in the twelve inch vacuum chamber approximately four inches away from the ion gun. After sputtering the crystal was removed from the vacuum chamber and placed in the standard sample holder on the double crystal spectrometer. The crystal orientation in the sample was carefully preserved from one rocking curve to the next by using lines on the crystal edge and surface gauge pointers.

Silicon

The silicon crystals were furnished by NASA-Lewis Research Center. For the silicon (111) reflection the half width of the rocking curve was measured to be 80 sec. This width is much broader than that for a good silicon crystal indicating that these crystals were quite imperfect. The half width for a perfect silicon crystal can be calculated to be 0.88 sec and silicon crystals have been measured with a half width as low as 1.0 sec.[43] In addition the crystal planes were not parallel with the crystal surface (off by approximately 1^{o}) which made the alignment in the spectrometer quite difficult. A definite change in the crystals occurred during the x-ray experiments as eventually no Bragg reflection could be observed. The crystals were returned to Lewis Research Center for examination. Back reflection x-ray photographs of these silicon crystals did confirm that a change had occurred. To date no adequate conclusion has been reached as to what caused these fairly imperfect crystals to suddenly exhibit much broader Bragg relection spots than before.

Copper

The copper single crystals[44] were grown by the Czochralski technique, polished, strain annealed, and oriented to (111). Crystal A, the crystal nearest the x-ray tube, is selected so that its lattice planes are as parallel as possible to the planes of crystal B, the crystal to be analyzed. For copper (111) in position B, a quartz crystal in the (200) reflection is used in position A. This combination not only has a close match on lattice spacings (2.081Å for copper vs 2.128Å for quartz) but also the width of the quartz rocking curve ($\sim$ 4 sec) is much narrower than the copper rocking curve (1100 sec) which makes the curve separation problem much simpler.[19]


150
100
50
SCATTERED POWER (COUNTS/SECOND)
5
10
15
20
25
30
35
40
β (10² ARC-SECONDS)
FIGURE 16
COPPER (111)
ROCKING CURVES
BEFORE SPUTTERING
AFTER SPUTTERING

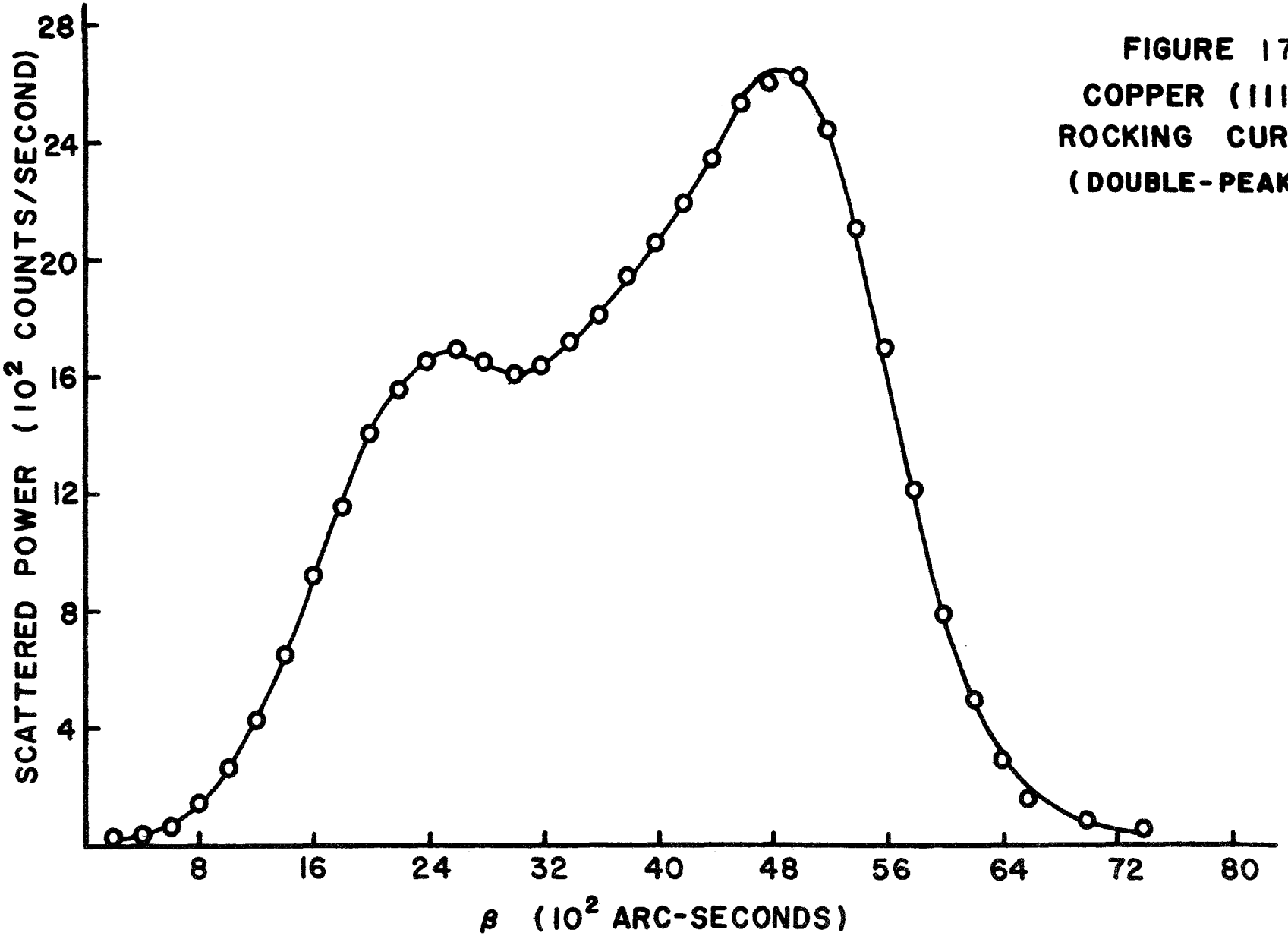


FIGURE 17
COPPER (111)
ROCKING CURVE
(DOUBLE-PEAKED)

Figure 16 shows the copper (111) rocking curve before and after sputtering with 1500 V Argon ions for 15 hours at 50μA, a dose of about 3 Coulombs for the one inch diameter crystal. Only part of the crystal face was in the ion beam but rocking curves in the two regions were the same. During this rocking curve and sputtering sequence, the copper crystal was observed to take on a double-peaked appearance as shown in Figure 17. Many tests were performed to check the spectrometer and crystals to determine the cause of the double peak. When it was finally determined that the crystals were producing the effect and not the spectrometer the crystal supplier was contacted. Apparently crystals grown with this technique sometimes undergo slippage. The amount of slippage for the copper crystal was found to be about 40 minutes of arc. Venetron now offers crystals grown by the Bridgeman technique and four new copper crystals were obtained.

By using a narrow beam so that only a very small region of the copper crystals were examined it was possible to continue using the old copper crystals during delivery of the new ones. Annealing the copper crystal at 750^{o}C for thirty minutes produced no change in the rocking curve.

Rocking curves of the new copper crystals had a half width of approximately 5000 sec. This was a factor 5 wider than the previous copper crystals indicating that these crystals were even less perfect. Copper crystals have been observed with half widths of less than 300 sec.[42] One of the new crystals was sputtered for 9 hours with a total dose of 1.94 Coulombs but the half width data were not conclusive due to the extreme width. These data were obtained in late September, 1969.

Germanium

The germanium single crystals were also grown by the Czochralski technique.[44] They were one inch in diameter by one-quarter inch thick, polished, strain annealed, and oriented to (111). No quartz reflection spacing was found to be useful for use with the germanium crystal to utilize the matched crystal technique so a second germanium crystal was used as crystal A. The sputtering, annealing, and rocking curve sequence is summarized in Table III. There appears to be no apparent increase in half width of the rocking curve for germanium with a fairly severe dose of ion bombardment.

Recent work on low energy ion bombardment damage in germanium[45] indicates that with 1000 V Argon ions the damage effects saturated with an ion dose of 1×10^{17} ions/cm^2. In addition the damage should penetrate approximately 25 layers, i.e. 50 Å. The dosage attained at Hiram College was 2.5×10^{19} ions/cm^2 for 2.0 Coulomb dose over the face of the 1" diameter crystal assuming singly charged ions. Due to room temperature fluctuation the precision of the half width measurement is $\pm$ 0.3 sec as determined from taking a series of rocking curves without repositioning the crystal. Since the spectrometer is automated to minimum step sizes of 0.5 sec and the mechanical precision of the micrometer is only good to o.2 sec the above value of 0.3 sec is probably about as good as can be expected.

TABLE III

Damage Studies of Germanium

PROCESS	COMMENTS	HALF WIDTH
Rocking Curve		22.2 sec
Sputter	1.0 Coulombs	
Rocking Curve		21.6 sec
Anneal	100 minutes at above 450^{o}C	
Rocking Curve		22.4
Sputter	2.5 Coûlombs	
Rocking Curve		21.4
Sputter	1.2 Coulombs	
Rocking Curve		21.8

CONCLUSIONS

In spite of the difficulties, e.g. delayed delivery of custom equipment, lack of local machine shop facilities for repair, lack of good crystals, etc. a fairly effective x-ray and vacuum research program has been developped and established at Hiram College. The equipment is now all operational but the design and assembling of such complex and unique equipment at a small college short in support facilities has been extremely time consuming. As a result the data taking phase of the project was compressed into a fairly short period of time and was hampered by the lack of good crystals. Thus although this work has shown that it is possible to develop and obtain such unique equipment at a small college to do a specific task it is also necessary to consider that the time required for this work is greatly increased. The time is increased not only due to the necessity of doing most of the construction and repair work off campus but also due to the lack of time available to the investigators except for weekends, vacations, and summers. Although these problems were anticipated and solved as far as practicable by advanced planning and the overlap of certain tasks they were still probably more significant at Hiram College than they would have been at a more "research oriented" school with good support facilities.

On the other hand the development of this work has played a significant role in contributing to the improvement of the Physics Department

at Hiram College and in particular, has been an important influence on the students who have participated in this research program. Five physics majors and nine nonphysics majors have been involved in this research program along with four faculty members. The sense of accomplishment and the satisfaction of working on a significant research project has been especially rewarding to these people. One student paper[17] and several faculty papers[15,20] have been given as a result of this work.

Unfortunately the scientific conclusions of this project are not as promising as the conclusions above as to the feasibility of assembling the equipment and the valuable role of research involvement at a small undergraduate college. The results on the germanium crystal indicate no line width broadening within the precision of the measurement even with fairly severe bombardment. The data for the silicon and copper crystals were not good enough to permit a conclusion.

APPENDIX

X-RAY PHYSICS RESEARCH LABORATORY
Physics Department
HIRAM COLLEGE
Hiram, Ohio 44234
Telephone (216) 569-3211 Ext. 26

February 23, 1968

TO: Mr. McGarr

FROM: Dr. L. B. Shaffer

RE: Your telephone call of Monday, February 19, 1968 and Varian's performance in fulfillment of Hiram College contract #2900.

I am enclosing a summary of our problems to date with particular emphasis on the system delivered January 17, 1968 and its final acceptance. I am also sending a copy of the letter to Mr. Dorney in the hope of expediting this matter to a speedy conclusion one way or the other.

I. Differences between the system proposed and the system delivered

A. The original vacuum environment around the sample crystal of 1×10^{-10} Torr was modified to mid-range 10 scale when the two-chamber design was finalized due to the decade pressure differential between the 12" chamber (@ 5×10^{-11} Torr) and the 6" special chamber. Apparently the vacuum spec (mid-range 10 scale) is not now being achieved in the 6" chamber due primarily to the bakeout limitation resulting in wall outgassing.

1. Proposal AJL-1920 submitted in fulfillment of Hiram College Contract 2720 implied that a system with two 4" x 12" Be windows could be baked to 250° C and would attain 1×10^{-10} Torr in 20 hours. This proposal also implied that a suitable rotary feedthrough device had been designed - see B below
2. Mr. Cloyd's letter of August 8, 1967 stated that a limited bakeout of less than 80° C must be observed due to the Be window.
3. Mr. Rush's letter of January 10, 1968 stated that a limited bakeout of less than 120° C for the same reason.
4. Tests performed at Hiram College by Don Jonas of Varian and continued (at Don Jonas' request) by Hiram College personnel indicate that

   a. The 12" vacuum chamber will probably meet specs of 5×10^{-11} in 20 hours but not when connected to special chamber
   b. The special chamber will not reach mid-range 10 scale when after a total of 120 hours the ion pump current of the 8 l/s pump still indicates approx. 0.2 uA (corresponding to about 2×10^{-9} Torr assuming a linear extrapolation through the 9 scale
   c. In addition, if the ion pump current were to "bottom out" then the pressure in the special chamber could not be measured since the use of an ion guage apparently causes wall outgassing leading to higher pressures than with ion guage off (see data book p. 9-11)

Conclusion: The vacuum environment around the sample crystal in the special chamber cannot be maintained and/or measured in mid-range 10 scale.

B. The rotary feedthrough device supplied with the system January 17, 1968 apparently meets the specifications as indicated by Rush's letter of January 10 describing the tests in air. The final tests under vacuum conditions will necessarily be run at Hiram College not with mechanical and optical methods as attempted last fall but with x-ray methods. Hence the overall stability of the system with respect to maintaining the tolerances can be checked. I will assume that any deficiencies found here will be corrected under the system warranty.

1. Although the rotary feedthrough device problem has apparently been resolved based on data to date, I still am not happy with the way in which the repeated redesign of the rotary feedthrough has contributed to the delivery delays of the system, particularly in view of the fact that it was supposedly designed under Hiram College Contract 2720
2. My letter of June 15, 1967 still summarizes my feelings on this matter in spite of Lauer's reply of June 22 and Cloyd's letter of July 24, 1967.

C. For completeness in this letter and to indicate the ludicrous situation involving this system I will cite the promised shipping dates stated on various occasions by Varian officials.

April 21, 1967
May 5
May 26
June 30
July 14
July 21
July 28
Aug 19 - Shipment arrived
Sept 12 - Jonas arrived
Sept 14 - window broke, feedthrough not satisfactory, Jonas left, system returned to Varian

Nov 1 - (hopeful)
Nov 15
Nov 30
Jan 5
Jan 10
Jan 17 - chamber arrived
Jan 23 - Jonas arrived

D. Other equipment deficiencies (minor problems)

1. Quote AJL-1920-B Section B Part 3 states that " a temperature readout panel for monitoring temperature at four places on the system shall be provided." Assuming a thermocouple readout panel of some kind, this item has not been received.
2. Pump control unit no. 921-0014 was specified on drawing no. 625115-B. After several requests and a referral to Lauer's letter of December 28, 1966 I received pump control unit no. 921-0015. Although at this stage it is not of major importance, I am indicating yet another Varian error which I have had to accept.
3. Alignment of flange faces #49 and #4 on drawing no. 625373 were specified to be parallel to $\pm$ 0.1° and their symmetry axes to be colinear to $\pm$0.031" in order that the ion beam would travel through both valves and into the crystal chamber. Measurements on the system indicate that the symmetry axis through the valves is not parallel to the symmetry axis of flange #4 but makes an angle of not less than

one degree with it, i.e. the chamber is approximately 1/4" lower than it should be. What effect this will have on the ion beam traversing both valves to hit the crystal in the chamber is yet to be determined.

II. Contract renegotiations

A. The several attempts at contract renegotiation last summer and fall were delayed with a comment to the effect that "let's get your system 'on the line' and then we'll discuss the money."

B. On December 1, 1967 after discussion with NASA Lewis and Hiram Business Office I called you and suggested the following:

1. I expressed concern with the repeated delays on the shipment of the system.
2. I commented that at this late date neither of us could afford to cancel the contract (although effectively Varian had terminated the contract with the unreasonable delivery delay)
3. I suggested renegotiation of the contract considering:

   a. The unreasonable delay
   b. My cost of waiting
   c. Fixed time and money allotment on NASA grant

4. I also suggested that Varian should make a large reduction in the total price of the system or make the system a total gift since

   a. Varian costs are probably out of proportion to any profit in the system
   b. Bad public relations with Hiram College, NASA, and their friends
   c. A tax deductible gift might be more to Varian's benefit financially at this point

   Your comment was "this might be the best way to handle it" and that you would investigate.

C. Thus after waiting and delaying formal renegotiation in good faith I next hear from you to the effect that

1. Varian's commitment is fulfilled since apparently the experiment might be able to be performed in the system as delivered although the system does not meet specifications (as outlined above)
2. Your accounting people will be in contact (they called the next day, February 20, to schedule payment
3. No renegotiation is possible at this point since you authorized an additional $10,000 to redo the system after the first system delivery did not meet specifications

Conclusion:

1. Although Varian has been very slow on delivery, Hiram College is expected to immediately pay for a system which does not meet specifications.
2. The implied promise of renegotiation will not be kept without protest from Hiram College, hence this letter.

3. Your reason under C.3 for not renegotiating at this point was exactly my assumption under B.4.a and my response is that Varian has had ample opportunity to design and deliver a suitable system (remember discussions started on this system, particularly the rotary feedthrough and Be window, on March 14, 1966.)

III. Conclusions and recommendations

A. At this point you have my sympathy but no more time or patience. I am a reasonable man, but after my dealings with Varian Associates maybe I am too reasonable. The problems involved in constructing the system have been pinpointed from the beginning. I questioned your need for a design contract before bidding on the system (since none other of the four companies required such a contract) but reasoned that you were probably correct in examining your capability first. However the problems and delays since indicate that not only was the work done under the design contract incomplete and misleading but also that I am expected to accept a system less than that agreed upon, 11 months late, with no renegotiation.

B. Recommendations

1. In order to make one last attempt to "bottom out" the pump current in the special chamber, and if your vacuum engineer concurs, I would suggest that we mount a 15 l/s pump on the system to attempt to increase pumping speed.
2. If mounting a larger pump is not feasible or will not aid in pumping speed at this pressure, then I suggest that the system be accepted under reduced specifications and that in addition Hiram College be given consideration for the time and overhead expenses incurred during the delivery delay by Varian in the amount of $8000. This course of action seems called for after consultation with Jim Nicholson, Business Manager of Hiram College.

LBS:dsm

cc: Roger Johnson, Credit Coordinator
Credit Department, Varian Associates

Patrick E. Dorney, Vice President
Vacuum Division, Varian Associates

John Ferrante, Thermionics Branch M/S 302-1
NASA Lewis Research Center
21000 Brookpark Road, Cleveland, Ohio

Jim Nicholson, Business Manager
Hiram College

Art Shiver, Varian Associates

Appendix to letter

Purpose of the research program and its relationship to the system specifications. This section is included to clarify the intent of the research program and is for your information only since you have brought up the matter.

A. Ion sputtering methods are used as a standard technique in LEED studies for cleaning crystal surfaces. During the sputtering process the surface is apparently roughened and imperfections are produced, primarily interstitial atoms and dislocation lines. The purpose of this research program is to investigate damage produced by the ion sputtering process as well as the extent to which the crystal recovers during annealing.

B. Since x-rays have been used to investigate this type of damage in solids it was proposed that x-rays might also be sensitive to this damage produced by the sputtering. We are not examining bulk material effects but rather surface effects that produce strain fields extending into the bulk material. Surface adsorption of gases may change these strain fields and hence the surface condition of the crystal must be controlled. Thus it becomes necessary to maintain the same conditions as used in LEED studies since we are looking for the effects of surface damage extending into the material.

C. Thus we are investigating whether or not x-ray methods are sensitive to surface damage by sputtering methods and if they are then we will investigate

1. The effects of sputtering in producing crystal imperfections.
2. The effects of ion mass, energy, and bombardment time.
3. The effects of annealing.
4. The effects of surface adsorption of gases on the strain fields.

May 8, 1968

Mr. Patrick E. Dorney, Vice President
Vacuum Division, Varian Associates

Dr. Lawrence B. Shaffer

Your letter of April 9, 1968

Thank you for your review of the Ultra High Vacuum System purchased under Hiram College Purchase Order No. 2900. I appreciate your attention to this matter and, upon first reading, your proposal appeared to be equitable. However after reviewing the situation again I feel that several points must be cleared up:

1. The " creeping specifications" allegation as suggested in Mr. McGarr's letter of March 26 greatly bothers me as apparently Varian did not keep track of our early conversations. I am enclosing my summary of the discussions in hopes that it will clear up your communications problem. Also point 3 of McGarr's letter does not even refer to the appendage chamber, which was obviously part of the system (AJI 1920-B).

2. You have yet to respond to my suggestion that a 15 l/s pump will be of value in lowering the base pressure in the 6" chamber. If necessary I will accept a reconditioned 15 l/s pump if this approach seems feasible.

3. As the prospect for future research at Hiram College in this area depends upon successful completion of a major portion of this research contract and since with the year's delay it is unlikely that the major portion can be achieved in the time remaining (without an unsupported time extension), it would appear that the future credit offer suggested in your letter will not help me either now or later. If at all possible I request that the $5000 credit be applied against the present order so that the likelihood of completing the work and obtaining future work will be greatly increased.

If the $5000 is applied against the present order, Hiram College will acknowledge this amount or a charitable deduction for your tax purposes.

I trust that your reconsideration will be favorable to the above suggestion so that we can clear this matter quickly and I can get on with the work. Again, I appreciate your attention to this problem and feel that, by the tone of your letter, you understand my situation and reactions at this point. Thank you.

Lawrence B. Shaffer

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DEPARTMENT OF PHYSICS

Rev.: 11-30-67
1- 8-68
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SUMMARY OF CONTACTS WITH VARIAN ASSOCIATES OF PALO ALTO, CALIFORNIA

March 14, 1966. Meeting at NASA-Lewis with Yale Strausser, Don Dooley, Art Shivers, and myself. We discussed the preliminary specifications on the system.

May 31, 1966. Telephone call from A. J. Lauer to discuss parameters on rotary feed-through device.

June 17, 1966. I sent a letter to Lauer with the preliminary specifications for an Ultra High Vacuum Double Crystal X-Ray Spectrometer. The covering letter suggested first that Brush Beryllium of Cleveland say they can attach the beryllium window to either stainless steel or copper and second that I mentioned the shaft on the rotary feed-through device and the bearings could be machined from synthetic sapphire.

August 5, 1966. Lauer called and suggested total price of $27,500 with the delivery 120 days later for a system that meets specifications. I again questioned him about rotary feed-through device and their standard one is only good for one degree. Since I need $\pm$ 10 seconds, Lauer suggested that they needed preliminary design contract for five weeks for $700-$800 to design a better rotary feed-through device.

September 6, 1966. I received quotation AJL 1708 from Lauer and the fixed price for the design contract for five weeks was for $1200. First paragraph of the letter stated, " Enclosed is Varian Quotation AJL-1708 for a fixed price contract to determine the design parameters for a special rotary feed-through device that will provide precise crystal movements inside an ultra-high vacuum chamber."

November 2, 1966. Lauer called and said that the rotary feed-through device meets all the specifications.

November 3, 1966. Received letter containing Varian proposal AJL-1920 which was submitted in fulfillment of the design contract (Hiram College Purchase Order No. 2720). Two drawings were included with this proposal. Neither of them described the rotary feed-through device in any detail. Section F of the proposal only repeated my specifications of June 17 with no details on how to be accomplished. At this time the configuration of the vacuum chamber was determined to be too expensive in the 18-inch size and also did not allow for adequate flexibility for the alignment of the crystals for x-ray analysis purposes. The second design utilizing the same rotary feed-through device designed previously was suggested in which a small chamber containing the crystal would be mounted on a standard double crystal x-ray spectrometer with provision for aligning the whole chamber with the x-ray beam.

December 2, 1966. Letter from Lauer on special system. This proposal included drawings and specifications for the single chamber.

December 22, 1966. Meeting at NASA-Lewis including John Ferrante, Art Shivers, and myself to discuss final configuration. Comparison of the two proposals (AJL-1920

and AJL-1920 B) with regard to vacuum requirements, x-ray geometry flexibility, and construction feasibility led to the conclusion that the two chamber design seemed more suitable for the application. The only question remaining, assuming the design contract had been fulfilled with regard to the Be windows and rotary feed-through device, was the vacuum spectrometer in the 6" chamber when valved off. A small vacuum ion pump (1 l/s) was suggested as a "holding" pump and Art Shivers confirmed this configuration with the plant (phone call). It was stated with this holding pump that mid-range 10 scale vacuum was possible with the chamber valved off.

December 26, 1966. The invoice was ok'd for the design contract in the amount of $1200. Full information had not been supplied, particularly with reference to the rotary feed-through device. However, the assumption was made that this information was available and would be used in the completion of the system described in the Varian Proposal AJL 1920 B.

December 29, 1966. Hiram College Purchase Order No. 2900 was given to Varian Associates for constructing a system as described in Quote Number AJL 1920 B for the price of $18,495.

January 12, 1967. Received letter from Lauer sending three drawings needing my approval before fabrication began. These drawings were approved and returned.

February 10, 1967. I received letter from Lauer enclosing two complete sets of production drawings. Several questions with regard to straight-through valves versus gate valves and the beryllium window attachment to the stainless steel chamber were discussed.

February 15, 1967. I responded with some final questions on several of the drawings, including the location of the center of mass for the small chamber of the system.

February 23, 1967. I received a letter from Lauer stating that all drawings for my system had been released to their shop and also that they expected completion of the system by April 21. It was also stated that they had investigated the rotary feed-through device and they were confident that it meets my approval. Also they said they will add weights to the chamber to bring the center of mass within the desired radius.

March 13, 1967. My letter to Lauer asked one additional question about flange distance, asked also about the possibility of an earlier shipping date (three or four days) and asked about the best time to visit their facilities for a preliminary check-out of system.

March 17, 1967. Letter from A. J. Lauer said work is progressing on schedule, shipping date is still April 21 and their vendor has successfully welded 10 mil beryllium to piece of aluminum-clad stainless steel.

April 7, 1967. Lauer called. Shipment has been delayed until May 5. Try to arrange visit to be there on May 8. After some discussion it was still decided for my visit to be April 17 as the system would be complete enough to go over some of the final details. This phone call confirmed letter of April 5.

April 17-18, 1967. On my trip to California I found very little of the system completed. During the discussion with the chief engineer on the project he showed me a rotary feed-through device that seemed completely different from the ones previously discussed. He suggested certain modifications to the standard feed-through device so that it would meet my specifications. The chief engineer (Ed Berry?) also confirmed the "holding" pressure in the small chamber when valved off. It appeared that Varian did not have enough of the system put together to talk about details at all. Sometime during May the delivery was again changed from May 5 to May 26.

June 5, 1967. A. J. Lauer called. He stated a complete redesign of the feed-through device was necessary. Certain drawings had not been released to the shop as

previously stated. The beryllium window material had not been ordered (it was ordered on the first of June). He also stated that the project engineer had been fired and that delivery was now delayed until June 30.

June 15, 1967. My letter to Lauer summarized the problems and delays to date, expressed unhappiness at the employee's " goof" and requested additional information for the necessity of the redesign of the special rotary feed-through device by the new project engineer in view of the fact that Varian had been paid to design this rotary feed-through device the previous December.

June 22, 1967. Letter from Lauer in answer to my letter of June 15. The letter really didn't answer the questions at all. Shipment delayed to July 14.

July 12, 1967. Art Shivers called and said beryllium window was welded yesterday, the eleventh, and stated that A. J. Lauer had been transferred.

July 17, 1967. Shivers called. Leak in beryllium window, Shipment delayed to July 21 with delivery on August 4. After repeated requests to Varian via two long distance calls to John Shields, A. J. Lauer's boss, Varian supplied another letter from Willard C. Cloyd, Application Engineer, on July 24 listing a man-hour accounting for the design contract and three drawings which did not help explain the design of the rotary feed-through device.

August 19, 1967. System arrived at 9:10 p.m. with accompanying letter from Willard Cloyd stating restrictions on rotary feed-through device which were outside the limits of the original specifications.

September 11, 1967 (?). Don Jonas, Varian service engineer from Chicago, arrived to install system and do final checkout on system at Hiram. Vacuum specifications were met during this period in the large chamber. During checkout on precision of rotary feed-through device on September 14, Don Jonas broke beryllium window. Jonas left September 14.

September 16, 1967. Conference with Art Shivers, Mike McGarr and myself to discuss complete history and problems in the design of the vacuum system for Hiram College. Mike McGarr is in administration at Varian Associates. It was Shivers wish to acquaint this administrative person with one of his field problems. A complete discussion of what had been done and what should be done took place and Mr. McGarr promised he would get right on the job and solve the problem. No letters from Mr. McGarr.

September 27, 1967. Les Johnson, new design engineer from Varian, arrived at Hiram to discuss complete redesign of system. After six hours of conferences with him, a consensus was arrived at and minor changes were made in the concept of the system.

October 10, 1967. Les Johnson called and stated most of the problems seemed well solved. The beryllium window can be brazed to stainless steel by a firm in San Diego, California. We discussed the possibility of increasing the thickness of the window to 30 mils if necessary. Also Les Johnson seemed happy to report that he had found a source for standard ultra-high vacuum precision bearings from Barden Bearings in Connecticut. I commented that I had suggested this bearing to A. J. Lauer on May 31, 1966 in our first conversation. The rotary feed-through device now consists of a standard rotary feed-through device with two precision bearings, special coupling, and the position stops inside the ultra-high vacuum chamber.

October 19, 1967. I sent a letter to Les Johnson suggesting location of holes for adding weight to properly located center of mass of small chamber.

November 7, 1967. Received replacement control unit for one liter per second ion pump (still not as listed on drawing). On this packing slip it stated the special chamber will be shipped November 15.

November 14, 1967. Shivers called. Chamber will probably be shipped by end of November.

December 1, 1967. After consultation with Hiram Business Office, I called Mike McGarr at Varian and I discussed the following: I expressed concern with the delay on the shipment of the vacuum system and suggested that I didn't want to cancel the order at this late stage in the negotiations; I suggested renegotiation of contract considering the following: (1) the unreasonable delay, especially in view of the design contract (the NASA lawyer feels unreasonable delay plea is possible), (2) my cost of waiting, (3) fixed time and money allotment on NASA grant. A possible solution: Varian would make a large reduction in the total price of the system or make the system a total gift since (1) Varian costs are way out of proportion to the profits of the system, (2) bad public relations with both Hiram College and NASA-Lewis and their friends, and (3) possible that a tax-deductible gift might be more to Varian's benefit financially at this point. Mr. McGarr's response was that " this might be the best way to handle it."

December 4, 1967. Les Johnson of Varian called. The beryllium window is reinstalled in the chamber. He used an electron beam welding technique by a Palo Alto, California firm for $650 (brazing by a San Diego, California firm was bid at $2500 with no guarantee). The window was now holding less than 10^{-9} torr and is of 10 mil thickness. The rotary feed-through device should hold to less than 6350 A at a 1 inch radius. I also suggested that Don Jonas be present at Varian plant during checkout of feed-through if at all possible.

December 20, 1967. Les Johnson called. There was a leak in the beryllium window when the feed-through was attached to the chamber. This is now being repaired and should be returned tomorrow (Thursday). The rotary feed-through has been checked by the Varian quality control department in the following manner: by attaching a microscope slide to the axis of the rotary feed-through and using an electronic indicator to check the reproducibility of setting around a vertical axis which was 50 to 60 millionths of an inch as of last Friday. After preloading the bearings, the bearings, the reproducibility was cut down to less than 40 millionths of an inch. He also suggested using iron-constantin thermocouple on chamber to check uniformity of bakeout. The chamber must be heated uniformly due to possible window stresses. Heating tape is all right to use but be careful with placement of tape. Hiram will furnish variacs for these heater tapes. I asked Les Johnson to supply me with the following: (1) drawings of the rotary feed-through device, (2) complete instructions on the proper bakeout procedures to use on the chamber, and (3) special instructions on the handling of the rotary feed-through device, particularly when installing on chamber.

December 29, 1967. I called Art Shivers. He was out but was to return call.

January 3, 1968. I called Shivers, asked to be notified about method of shipment and asked him to check about possible variations from specifications with respect to rotations about the horizontal axis. He will call the plant and let me know.

January 5, 1968. Shivers called. Shipment is supposed to be today. I have top priority on Don Jonas so whenever system arrives he will be here. I also suggested that if anything in the shipment is not complete or any questions arise on checkout procedures on the system, all will come to a halt until the plant has been notified. Mike McGarr will be here at the end of January to discuss the system.

January 15, 1968. Art Shivers called. Chamber not shipped until January 10 (American Airlines Air Freight #001-4015992). Don Jonas is on way to Cleveland, somewhere in Indiana.

January 17, 1968. Varian system arrived! Shivers called. He and Jonas will come down tomorrow to check out shipment and then return on Tuesday to begin tests.

January 18, 1968. Preliminary check of shipment with Jonas. Repair of 140 l/s pump control unit. Discussion of test procedures and equipment shortages.

January 23-February 14, 1968. System check out. Conclusion: 6" chamber will not meet vacuum specifications when valved off, eg. the holding pressure with the 1 l/s pump is 2×10^{-7} and with the 8 l/s pump is 2×10^{-8} Torr. With extended bake of 120 hours on the 6" chamber the 8 l/s pump current was still 0.2 μA or approximately 2×10^{-9} Torr.

February 19, 1968. Mike McGarr called. Assumed that system met specifications, insisted on payment.

February 20, 1968. Varian accountant called Hiram Business Office.

February 23, 1968. Letter to McGarr summarizing Varian System deficiencies.

March 15, 1968. Jim Nickelson and I called Mike McGarr about progress on response to my letter of February 23. McGarr indicated letter would be out in several days. We also discussed a special anode sputtering project.

March 26, 1968. Nickelson called McGarr. McGarr said letter was mailed today.

March 28, 1968. Letter arrived in response to my letter of February 23 stating Varian's position.

April 4, 1968. Jim Nickelson and I called Mr. Pat Dorney to express our dissatisfaction with Mr. McGarr's letter and to ask for another review of the history. Mr. Dorney will do so.

April 9, 1968. Letter from Mr. Dorney.
January 16, 1969. Credit received from Varian.
February 11, 1969. System paid for.

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