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MOLECULAR BEAM SCATTERING FROM SINGLE CRYSTAL SURFACES
UNDER ULTRA HIGH VACUUM CONDITIONS

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ABSTRACT

Studies of the angular distribution of scattered atomic and molecular beams from single crystal surfaces give detailed information about the gas-solid interaction. An ultra high vacuum system combining molecular beam scattering with in situ observation of the same crystal surface by low energy electron diffraction is described. A rotating quadrupole mass spectrometer and lock-in detection of the modulated (150 Hz) incident beam provide density profiles of the scattered gases. The scattering of thermal energy helium and oxygen beams from the (100) face of a platinum single crystal is given to demonstrate the types of surface studies that may be carried out in such an apparatus. Data are presented showing the effects of CO adsorption and surface roughness on the scattered beam intensity, and the scattering of various molecular beams from clean and contaminated surfaces.

I. INTRODUCTION

Studies of the interaction of gases with solid surfaces are usually conducted by introducing the desired gas into a vacuum chamber containing a previously prepared sample surface. Resultant changes in the physical-chemical properties of the surface (changes in work function, electrical, or thermal conductivity for example) upon the incidence or adsorption of gas atoms are then monitored to obtain information concerning energy accommodation or the nature of chemical reactions between the surface and the gas. Recent experiments^{1,2} have revealed, however, that the transfer of energy between a gas and a solid depends markedly upon the incident angle at which the gas atom strikes the surface, the gas velocity, surface roughness, and the atomic nature of the solid. It has thus become necessary to use directed atomic and molecular beams of well defined velocity and single crystal surfaces to probe the detailed mechanisms of various gas-solid interaction processes.

Theoretical investigations³⁻⁵ have shown that the angular distribution of the scattered gas beam and products may be used to determine (a) the partition of the interaction energy among translational, vibrational, and rotational modes, (b) the surface residence time, and (c) the reaction probability as a function of surface and gas temperatures.

To date, only a few of these determinations have been carried out and reported in the literature. Recent reviews by Smith and Saltsburg⁶ and Stickney⁷ discuss rare gas scattering from several surfaces, but the scattering of reactive gases has received much less attention. Also despite the recent use of single crystal targets, too little emphasis has been placed upon the attainment of well defined surface conditions. Detailed

information on the exact crystallographic nature of the surface structure is necessary if one hopes to provide a sound basis for a theoretical explanation of the observed scattering patterns. Consequently, such techniques as low energy electron diffraction (LEED) should provide additional important data in the study of gas-solid interactions. Finally, surface cleanliness is of vital importance to such experimental studies because contaminants only serve to obscure and complicate an already complex interaction. The two most popular solutions to this problem are to use either high sample temperatures to desorb contaminants,² or to continuously deposit the solid at a rate exceeding the adsorption rate of ambient background gases.⁸

The purpose of this paper is to describe an apparatus constructed specifically to study gas-solid interactions under nearly "ideal" conditions, i.e. in ultra high vacuum using atomic beams and single crystal surfaces combined with low energy electron diffraction, and then to demonstrate the type of experimental data that may be obtained by presenting the results of representative experimental studies with atomic and molecular beams of different reactive and non-reactive gases.

II. APPARATUS DESCRIPTION

A schematic diagram of the major components of the apparatus is shown in Fig. 1. The apparatus consists of three separate parts: (A) an ultra high vacuum scattering chamber, (b) a selector chamber to house either a beam chopper or rotating disk velocity selector, and (C) a source chamber which contains the molecular beam oven. Typical operating pressures in the three chambers are respectively 1×10^{-9} torr or lower, 3×10^{-7} torr and roughly 5×10^{-5} torr. The UHV portion, bakeable to 250°C , is pumped

entirely by sorption and ion pumps to minimize contamination while the other two chambers are pumped by conventional liquid nitrogen baffled oil diffusion pumps.

Using this apparatus, the scattering of molecular beams from the surface of a single crystal may be studied in an ultra high vacuum environment ($< 10^{-8}$ torr) at any incident angle and at any surface temperature between 77°K and the melting point of the solid. The crystallographic orientation of the surface is unrestricted and the choice of the solid is not confined to either refractory metals or to those materials that can be epitaxially grown in single crystal form as with more conventional systems. With the large number of ultra high purity metallic, semiconductor, and insulator single crystal surfaces commercially available today, an apparatus such as that shown in Fig. 1 may be used to investigate the gas-solid interaction at a wide variety of interfaces.

The target surface may be bombarded by either continuous (d.c.) or pulsed (a.c.) thermal atomic or molecular beams. LEED may be used to monitor surface conditions before, during, or after scattering studies. By employing the LEED optics to analyze inelastically back scattered electrons, Auger spectroscopy⁹ may be used to identify surface contaminants. These techniques in combination may be used to provide information on the transfer of energy between incident molecules and the surface during scattering or during chemical reactions.

A. Scattering Chamber

Figure 2 shows a cut-away drawing of the scattering chamber indicating the placement of the quadrupole mass spectrometer used to detect the beam and the LEED optics used to monitor surface structure. The chamber itself consists of a 4.8 mm thick sheet of 304 series stainless

steel welded into a cylinder 30 cm in diameter and 50 cm in height. Thick plates (25.4 mm) welded to the top and bottom of this cylinder prevent buckling due to the external and internal pressure differences. Various size copper sealed access ports (Fig. 2) are situated in convenient locations about the cylinder's circumference as well as in its top and bottom. Total pressures are measured with a nude ionization gauge mounted directly in the chamber. In the absence of a molecular beam, the gate valve between the scattering and selector chamber is kept closed, thus allowing the UHV chamber to be used independently for low energy electron diffraction and flash desorption experiments.

The molecular beam enters the scattering chamber along a fixed line intersecting the axis of the cylindrical chamber by traversing a bakeable gate valve (Whittaker Model SVS-3) which has been fitted with a (changeable) collimating orifice 1.5 mm in diameter. The target, an oriented single crystal disk of platinum (7 mm in diameter and 1 mm thick) is suspended from a Varian multipurpose electromechanical feed-through at the point where the LEED, ion bombardment, and molecular beams intersect the axis of the scattering chamber. This mounting allows the crystal to be rotated about an axis bisecting its scattering surface to permit variation of the incident angle, θ_i , of the beam during scattering experiments and to position the crystal with respect to the electron or ion beams. Both θ_i and the scattering angle θ_r are measured with respect to the surface normal. Sample temperatures above room temperature are attained through resistive heating while low temperatures may be reached by the use of a special liquid nitrogen cooled holder.¹⁰

In order to study molecular beam scattering from a surface in an ultra high vacuum ambient, differential pumping techniques must be used to avoid flooding the scattering chamber with non-directional gas molecules effusing through the collimating orifice. Even so, vigorous pumping of the scattering chamber is necessary to keep the ambient as gas free as possible. Ideally the pump should be located directly opposite the beam source so that the beam molecules pass directly into the pump unless scattered by the target surface. For this work, a 500 liter/second vacuum ionization pump (Varian Noble VacIon) was chosen. The large speed is desirable because, even for such "noble gas" pumps, the pumping speed falls to roughly 30% of the maximum rate observed for N_2 when one pumps He or Ar. A bakeable gate valve connects the pump to the UHV scattering chamber. This permits the pump to be isolated and left in operation when the main chamber is opened to change the sample, thus facilitating later pump-down. Figure 3 shows a typical mass spectrum of the residual gas background in the isolated scattering chamber (i.e. no parent beam present). The most prevalent background gases are hydrogen, water, and carbon monoxide.

Before, during, or after scattering by the molecular beam, the surface may be studied by low energy electron diffraction. The LEED optics are located perpendicular to both the beam line and the cylindrical axis of scattering chamber (Fig. 2). Back diffracted low energy electrons (energies of 5 to 500 eV yield wavelengths between 5.5 \AA and 0.5 \AA) are well suited to probe surface structures because their strong coulomb repulsion by the substrate permits penetration of only a few atomic layers.¹¹ LEED can also be used to identify the presence of contaminant surface structures. As we show later, the beam scattering properties of

the platinum surface are markedly changed in the presence such structures.

LEED techniques have demonstrated that merely heating a sample in vacuum is usually insufficient to prepare a clean surface. While some contaminants (i.e. carbon and hydrocarbons on Pt, W, or Ni) may be removed by heating in oxygen (followed by heating in hydrogen to reduce residual oxygen), cleaning is best accomplished in situ for most species by alternate cycles of sputtering with noble gas ions followed by high temperature annealing of the specimen. A Varian ion bombardment gun is used to provide low energy ions of Ar^+ or Xe^+ (140 to 375 volts) at current densities up to two microamps per square centimeter. These conditions have been found to yield a minimum of disordering of the surface structure coupled with an optimal removal of surface impurities.

The impingement of reactive gases (CO , hydrocarbons, etc) from either the ambient background or the beam may induce the formation of ordered or disordered surface structures due to the adsorption of incident gas molecules by the surface during the scattering experiment. Thus, one may observe a transient change in the properties of the surface as a function of time due to the formation of a new structure. This can be ascertained and studied by simultaneous beam scattering and LEED experiments as indicated in a subsequent example.

To detect the angular distribution of the scattered parent beam and the scattered products of a surface reaction, a quadrupole mass spectrometer (Electronic Associates Inc. Model 250-A) is used. Its versatile mounting, pictured in Fig. 4, is built from a commercial rotary motion feedthrough (Ion Dynamics Corp.) with the normal length 9.5 mm diameter shaft replaced by one extending 30 cm beyond the flange face. This feedthrough is attached to a specially built linear motion drive containing

the electrical connections necessary for the quadrupole's operation. Alignment of the rotating shaft is maintained by a ball bushing located on the axis of the scattering chamber. One advantage of this mounting system is that the mass spectrometer may be easily inserted or removed from the chamber with all its internal connections intact. The mounting allows study of the scattered beam both in and transverse to the incident plane and rotation from -10° to $+190^\circ$ relative to the parent beam line. A 7° acceptance angle at a distance of 2.5 cm from the target was obtained by adding collimation apertures to the standard Pierce gun ionizer.

B. Selector Chamber

This chamber acts primarily as a buffer between the source chamber containing the oven which generates the beam and the scattering chamber. During an experiment using an oven pressure of several torr, the pressure in the selector chamber is 3×10^{-7} torr. A tuning fork beam modulator (American Time Products Type L40 Light Chopper) with a frequency of 150 hertz is used to generate an a.c. molecular beam. Alternately, a rotating disk velocity selector¹² is placed in the chamber to prepare monochromatic beams for use in scattering and surface chemical reaction studies.

A 10" oil diffusion pump (MCF-1400 with Dow Corning - 704 fluid) trapped by a liquid nitrogen baffle and protected by an air operated gate valve (Temescal, Metallurgical Corp.) is mounted to one side of this chamber.

C. Source Chamber

A glass multichannel effusive source (Bendix Mosaic Fabrications) consisting of a 0.8 mm by 4.8 mm rectangular array of tubes (length to diameter ratio 100) is used to generate a thermal molecular beam of some

noncondensable gas. The capillary array is held between gold O-rings in a stainless steel oven that can be heated to 650°K and cooled to 77°K. A detailed description of this oven (shown in Fig. 5) is given elsewhere.¹³ These sources have been used by other authors for molecular beam studies¹⁴ and their characteristics have been fully investigated.¹⁵

The source chamber is separated from the selector by a collimating orifice having the same dimensions (0.8 mm by 4.8 mm) as the beam source. It is pumped by a diffusion pump which is identical to that used to pump the selector chamber. To handle the large gas load during operation of the beam, both pumps are backed by a rotary blower (Rootes model RS 120).

In order to insure that the crystal, gate valve collimator, and oven collimator were all collinear, a laser beam was used to align the last slit (oven collimator) with the first two positions. The main body of the oven chamber was then welded in place to the tube in which the oven resides. The laser beam can also be used to optically determine the angle of the mass spectrometer detector and the crystal surface with respect to path of the incident molecular beam.

The gas handling system for the oven is a glass line external to this chamber. Precise gas flow conditions from a high pressure reservoir to the oven and the ballast flask used to damp out pressure fluctuations are maintained by a variable sapphire leak valve (Varian). In order to minimize contamination of the beam gases and sapphire leak by stopcock grease, single O-ring sealed Teflon needle valves (Fisher and Porter Co.) have been used in place of high-vacuum stopcocks in this manifold.

III. EXPERIMENTAL RESULTS

Scattering distributions presented in this section represent density profiles as a function of angle in the incident plane and were obtained with a flow through ionizer on the quadrupole mass spectrometer. Modulated beam techniques are used to enhance the signal to noise ratio. The output from the electron multiplier of the detector is capacitively coupled into a lock-in amplifier (Princeton Applied Research, HR-8) and the d.c. component grounded through a 1 M Ω resistor. For these measurements, the phase of the scattered beam was assumed to be that of the unscattered modulated beam observed downstream from the target location (i.e. we assume elastic scattering) and the lock-in amplifier was used merely to discriminate against the background.

For the scattering experiments reported in this paper, the temperature of the beam source was maintained at $300^{\circ} \pm 5^{\circ}$ K and the angle of incidence was restricted to $\theta_i = 45^{\circ}$. Due to the physical size of the detector, measurements of the scattered signal cannot be made at angles closer to the incident beam than 45° . Consequently we were unable to obtain data at the surface normal, $\theta_r = 0^{\circ}$. We have normalized all scattered intensities to the incident beam strength to minimize any changes in the response characteristics of the detector as a function of time. Thus, the plotted intensities represent the per-cent of the incident beam scattered into the detector at a given angle.

In Fig. 6, we show the angular profile of an unscattered helium beam measured both upstream and downstream from the target locations to indicate the divergence of the beam as it crosses the scattering chamber.

Previous studies of the (100) face of platinum by low energy electron

diffraction^{16,17} and Auger spectroscopy¹⁸ have revealed that the clean surface is characterized by a surface structure having one dimension five times that of the bulk. Such a structure yields the (5×1) diffraction pattern shown in Fig. 7a. Extra spots occur along both the x and y axes due to surface domains rotated 90° to one another. The scattering pattern of a helium beam from this clean surface is shown in Fig. 7b. As has been observed by other authors on clean single crystals of Au,⁶ Ag,⁶ Ni,¹⁹ and W,²⁰ the maximum occurs at the specular angle, $\theta_i = \theta_r$.

Of somewhat more interest, however, is the helium scattering from an ion bombarded surface shown in Fig. 8. Note that the maximum intensity has fallen by almost one order of magnitude from that observed on the clean surface. Although this surface yielded no diffraction pattern, the helium scattering shows a definite peak. Surface disorder is apparent from the low intensity maximum and the peak breadth. For reference we show in the same curve a cosine scattering distribution. Such a curve would arise if the incident beam reached complete thermal equilibrium with the surface before being scattered.

Such a sharply peaked curve as that shown in Fig. 7b may only be obtained if one frees the platinum surface of residual adsorbed carbon monoxide. As mentioned earlier, beam scattering is strongly affected by and may be used to monitor surface contamination. To illustrate this fact, Fig. 9a shows the change in the scattered specular intensity ($\theta_i = 45^\circ = \theta_r$) for a helium beam as a function of time in an ambient atmosphere of 5×10^{-9} torr of CO. At time zero the sample was flashed to 1000°K to remove adsorbed gases. The scattered beam intensity decreases due to the gradual chemisorption of carbon monoxide on the surface. Changes in the LEED pattern were also apparent. The fifth order spots

were only faintly visible and the overall background intensity (diffuse scattering) had increased beyond that for a clean surface. Continued CO adsorption results in the diffraction pattern shown in Fig. 9b. Note the absence of the fractional order spots.

Finally, we show the effect of CO contamination on the scattering of an oxygen beam from the platinum (100) surface. The data in Fig. 10a were taken after flashing the crystal to just below 900°K between each point. Since CO has at least three binding states on the (100) face of platinum,¹⁶ and since the last of these is only removed above 900°K, the tightly bound β form of CO was presumably still present on the surface during the measurements. Energy exchange between the incident beam and the adsorbed gas may account for the angular shift in the scattered intensity maximum relative to the position of the scattering maximum from a clean surface shown in Fig. 10b. In this case the crystal was flashed to 1000°K between data points. Because the intensity maxima of the two curves are nearly identical, and because the LEED pattern observed during the course of the experiment depicted in Fig. 10a was virtually unchanged from a clean (5x1) it appears that the remaining strongly adsorbed CO represents less than 10% of a monolayer coverage.²¹

IV. SUMMARY

The results presented in this paper represent the first reported beam scattering of monatomic and diatomic gases from the (100) face of a platinum single crystal. Platinum was chosen for this work because it is a well known catalyst in many important chemical reactions and because the properties of its low index faces have been previously studied by LEED.¹⁷ Data presented herein are illustrative of the types of information available from combined molecular beam scattering and low energy

electron diffraction experiments. Measurements were made under ultra high vacuum conditions and a detailed description of the apparatus used in this work has been given.

ACKNOWLEDGEMENTS

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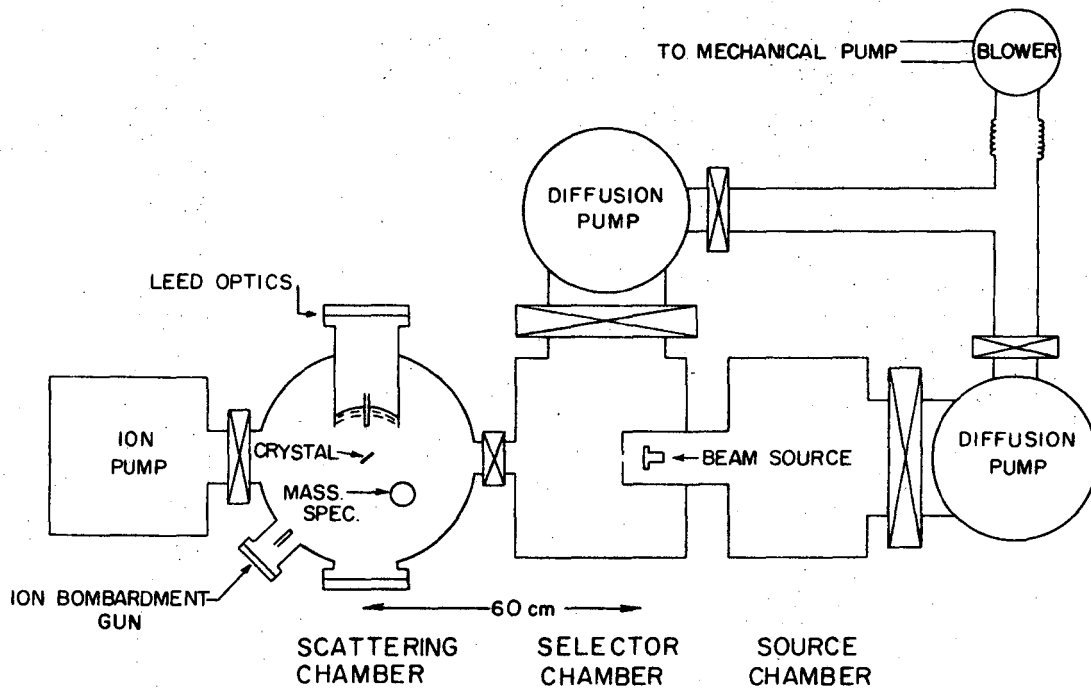
[†]National Science Foundation predoctoral fellow.

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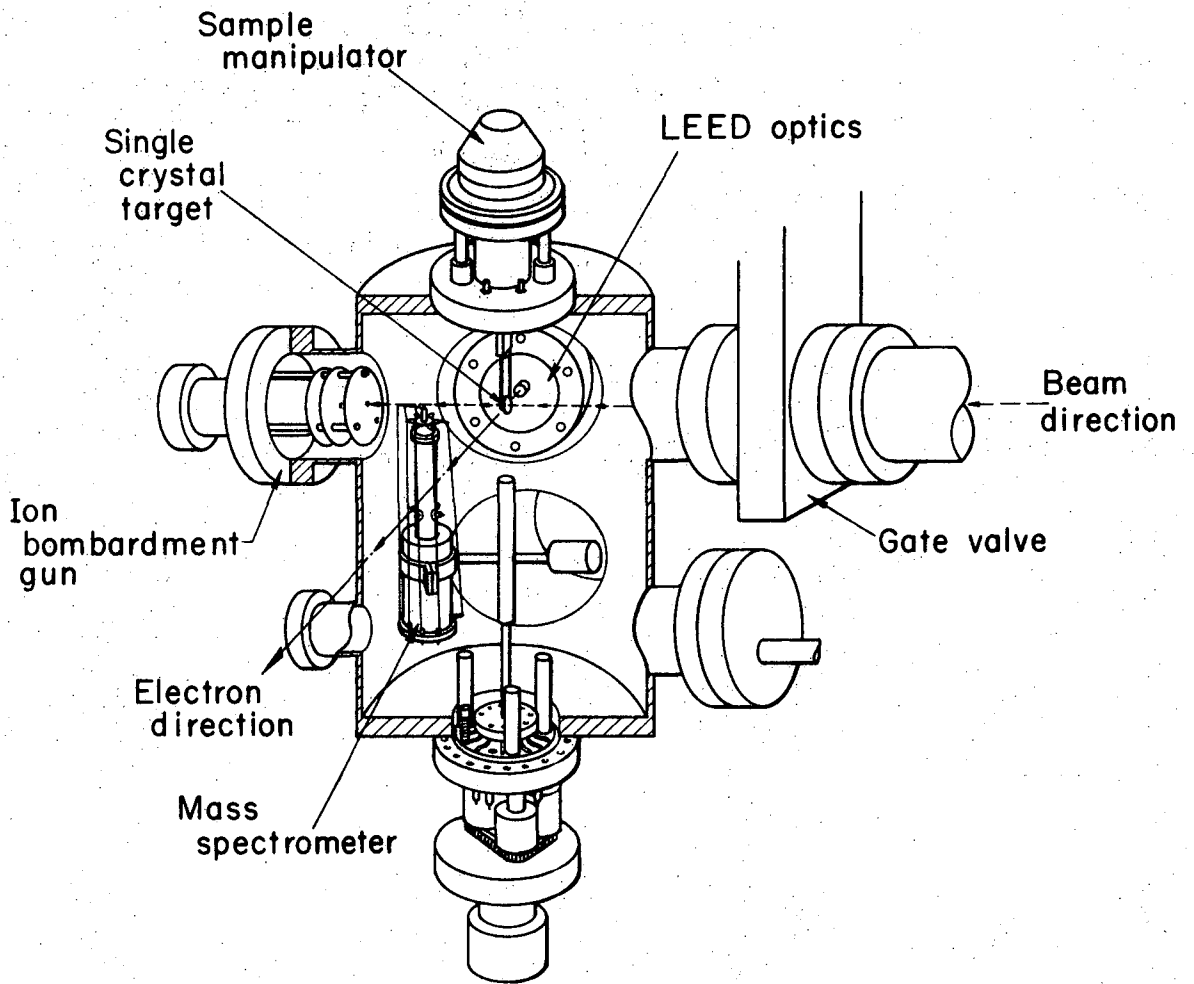
FIGURE CAPTIONS

1. Schematic diagram of molecular beam scattering apparatus.
2. Detailed view of the scattering chamber. Ion pump (not shown) located on port below LEED optics.
3. Mass spectrum of residual gases in scattering chamber in absence of molecular beam. Total pressure 9×10^{-10} torr.
4. Mass spectrometer and linear-rotary motion feedthrough.
5. Exploded view of multichannel molecular beam source.
6. Helium beam angular profiles upstream and downstream from target position.
- 7a. (5x1) diffraction pattern characteristic of a clean platinum (100) surface at $E = 63$ V.
- 7b. Helium scattering pattern from a surface yielding the (5x1).
8. Helium scattering pattern from an ion bombarded surface. The dotted line represents a cosine distribution.
- 9a. Decay of He beam specular intensity in a CO ambient after flashing the crystal to remove adsorbed gases.
- 9b. Diffraction pattern of a Pt(100) surface after exposure to 5×10^{-9} torr CO for several minutes, $E = 63$ V.
- 10a. Oxygen scattering from CO contaminated platinum.
- 10b. Oxygen scattering from clean platinum.



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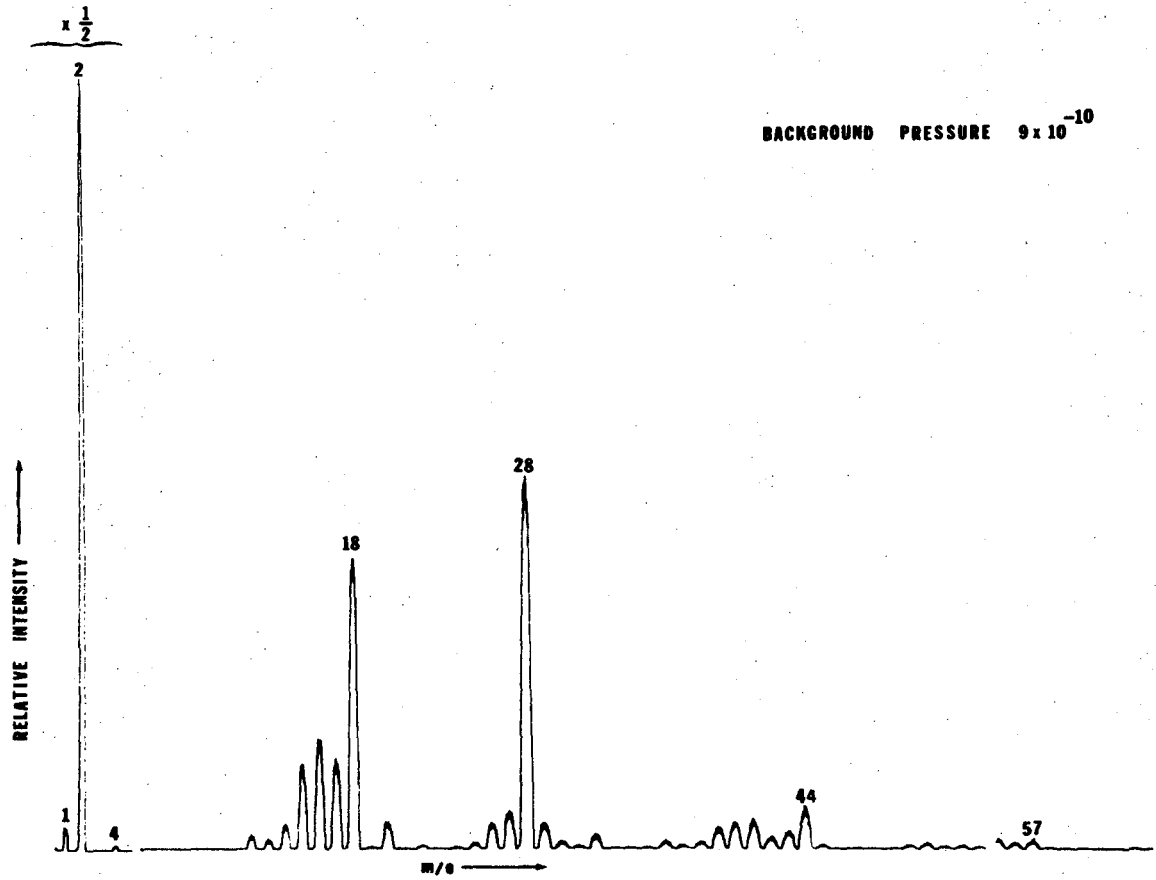
Figure 1



SCATTERING CHAMBER

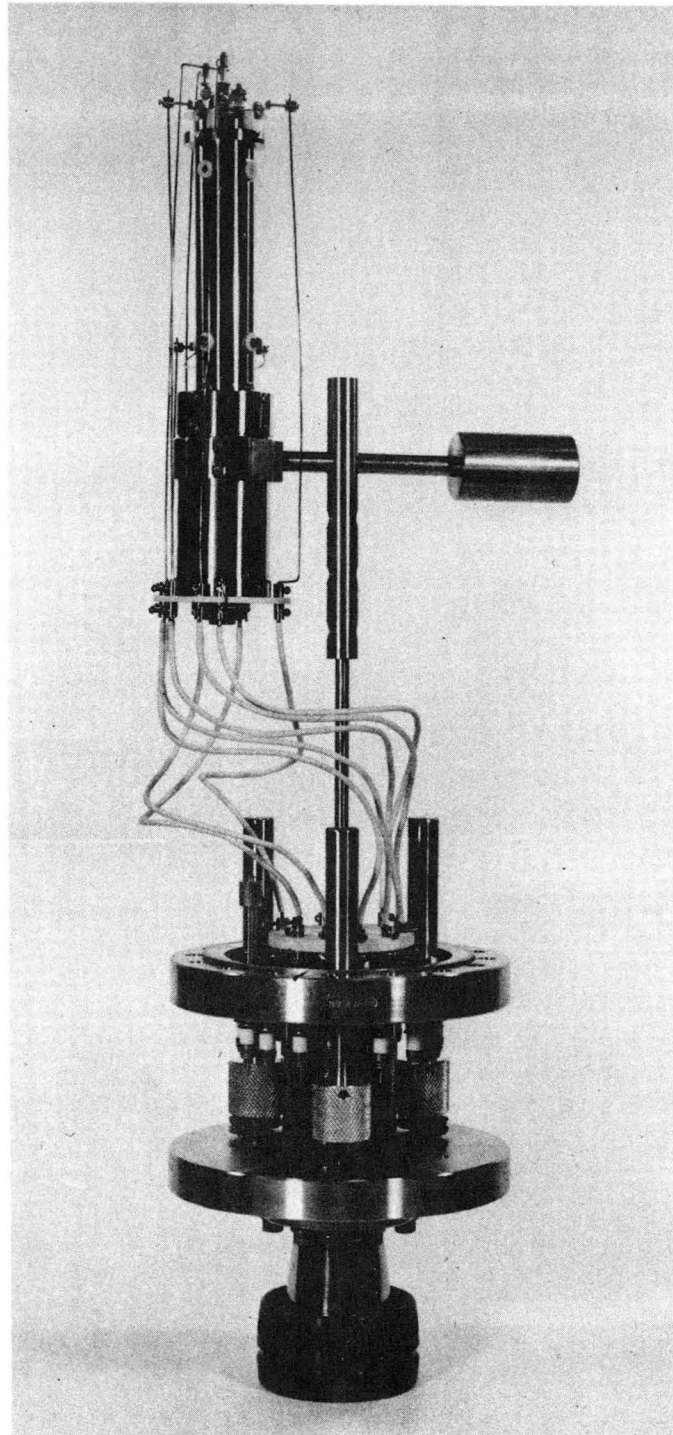
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Figure 2



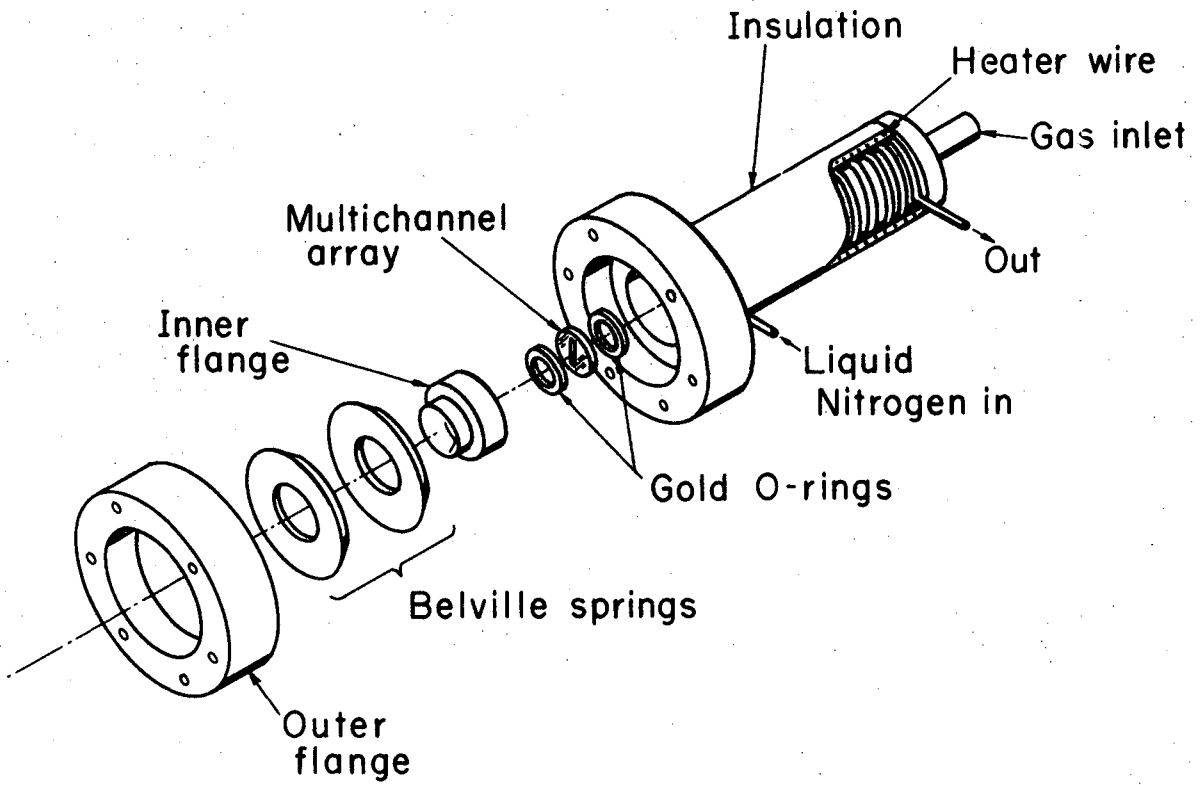
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Figure 3



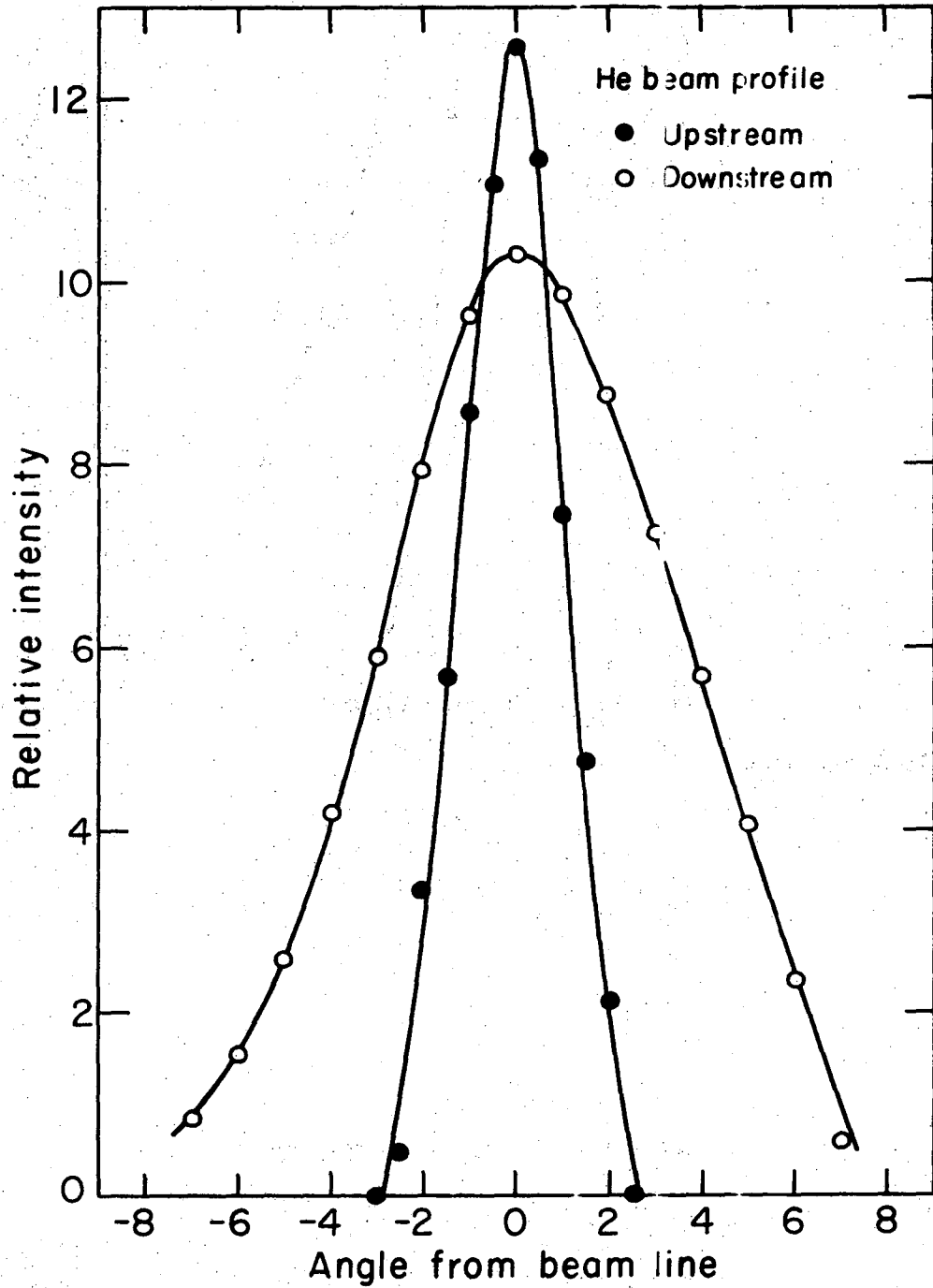
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Figure 4



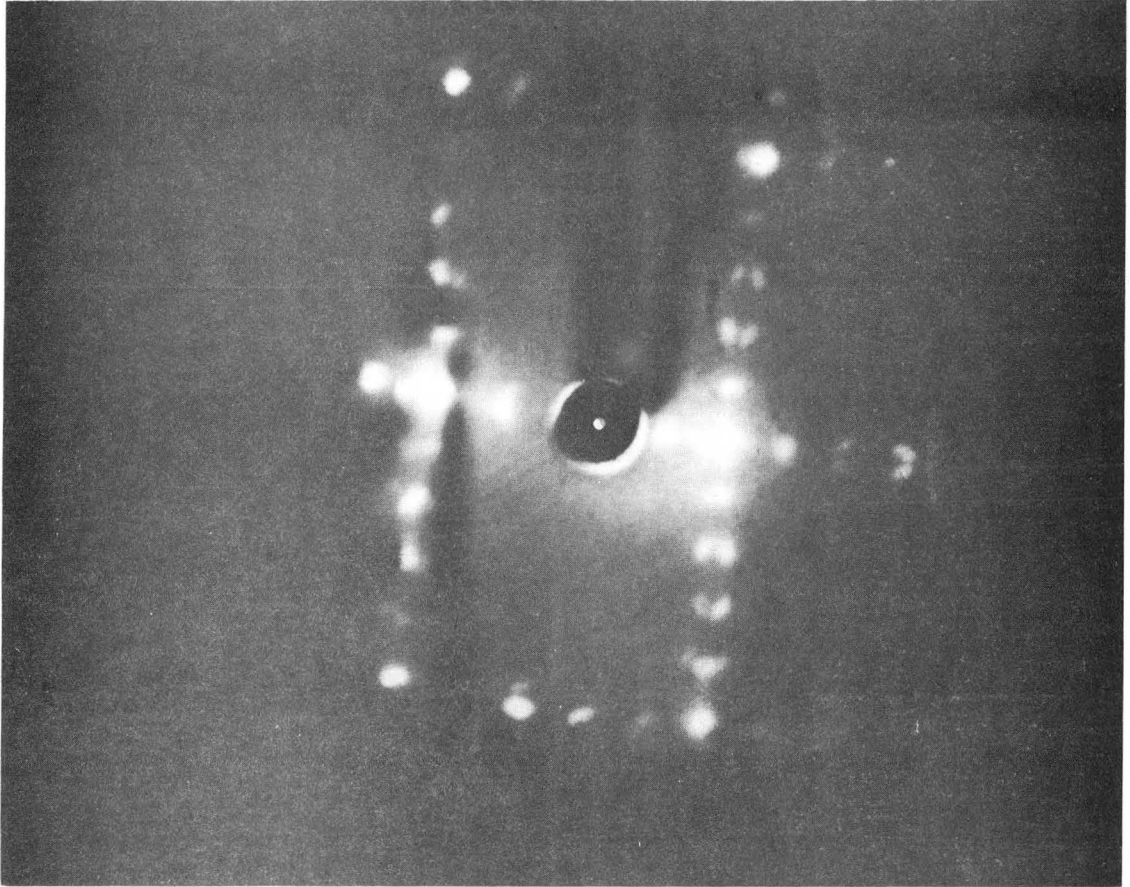
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Figure 5



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Figure 6



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Figure 7a

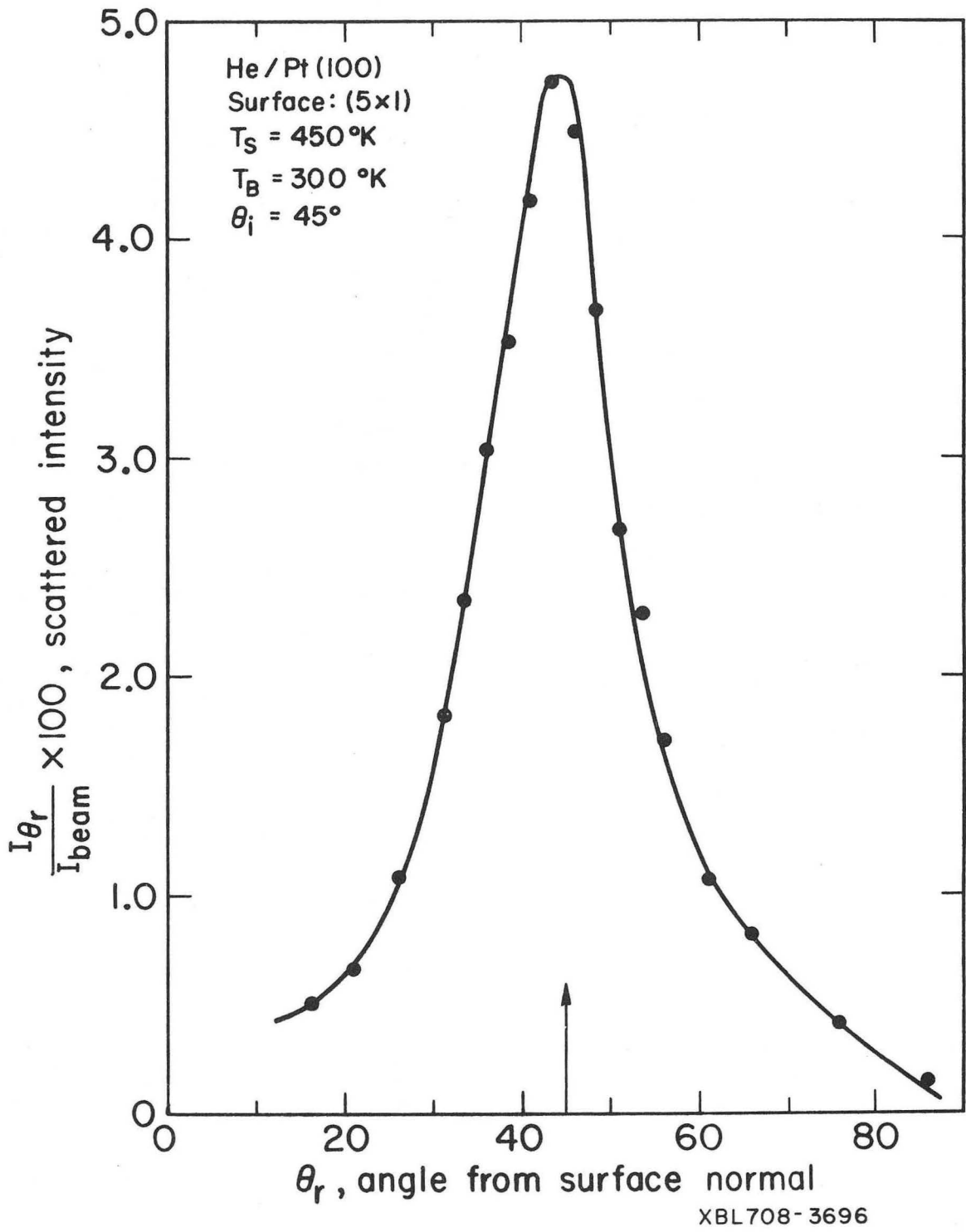
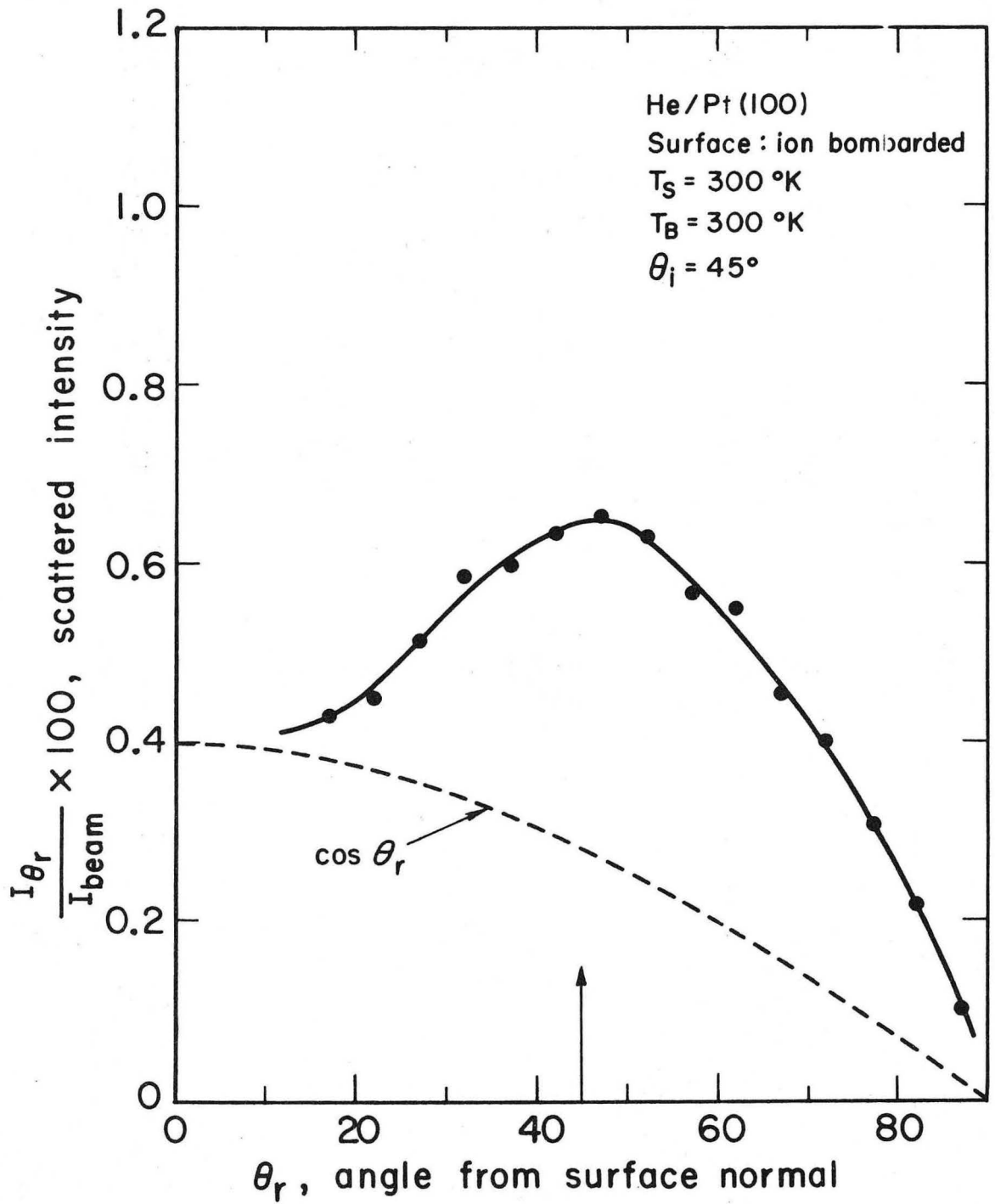
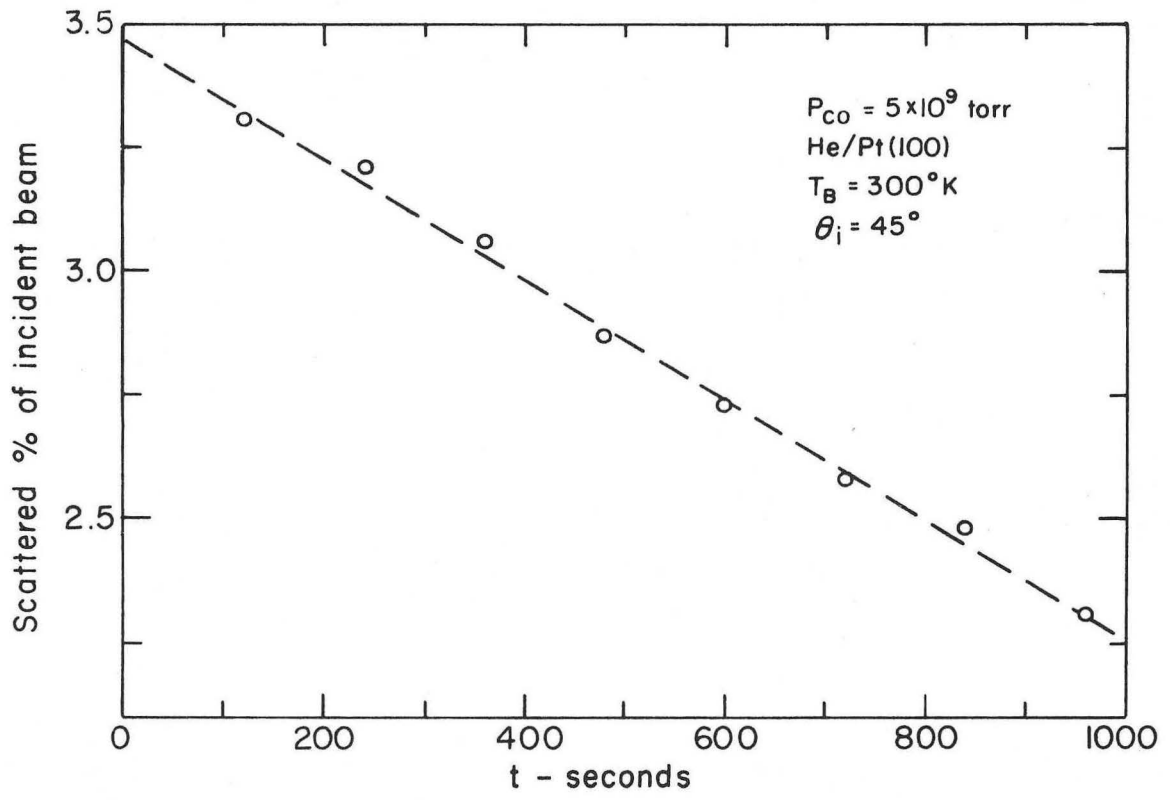


Figure 7b



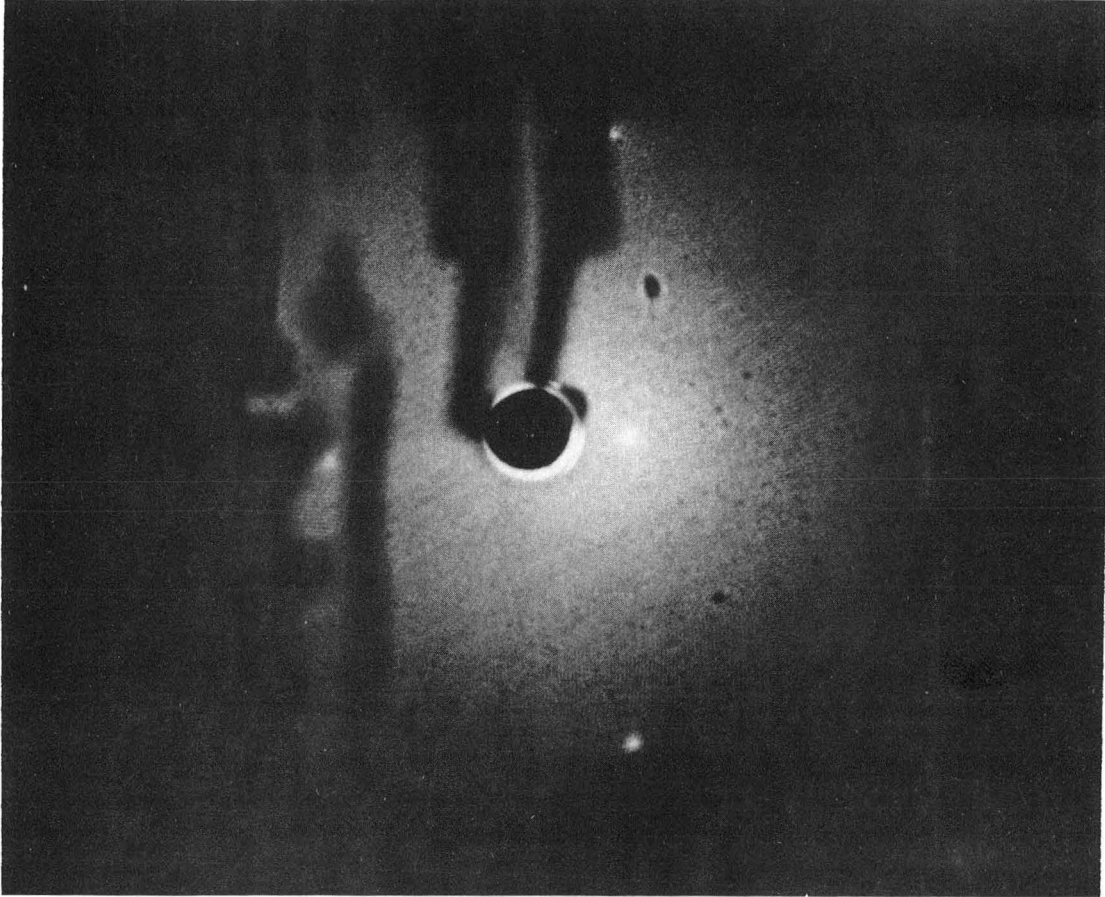
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Figure 8



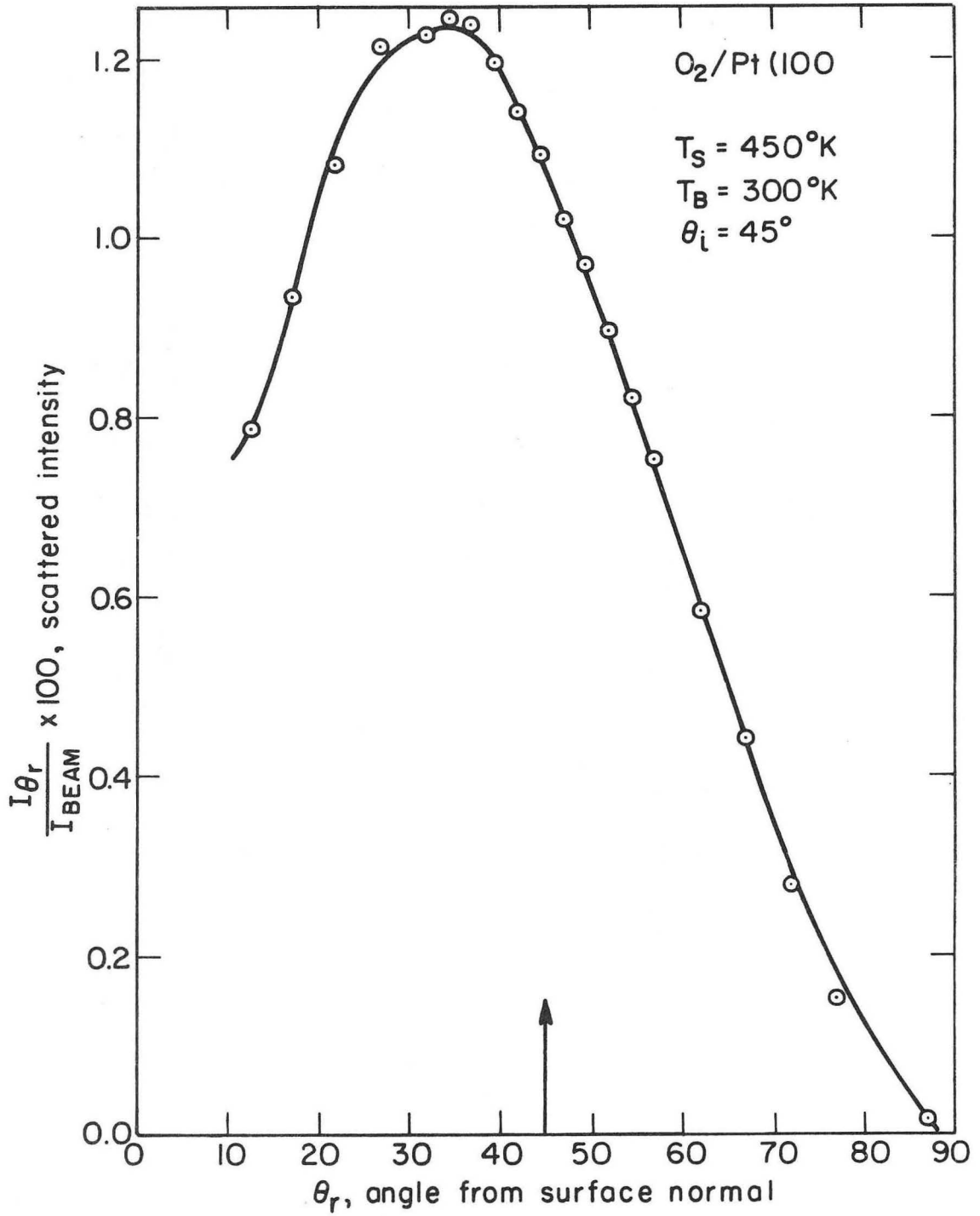
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Figure 9a



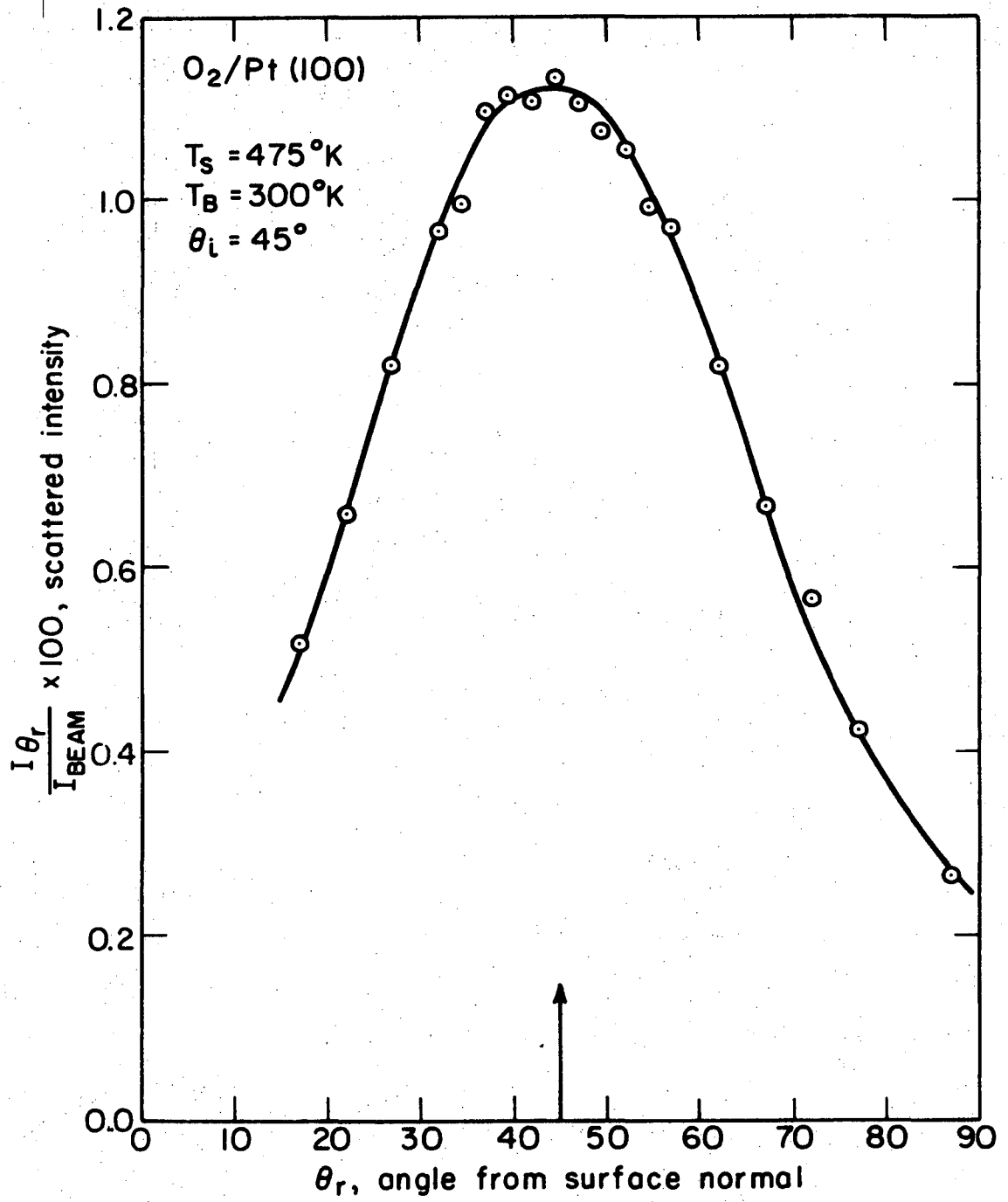
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Figure 9b



XBL 708-1967

Figure 10a



XBL 708-1966

Figure 10b

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