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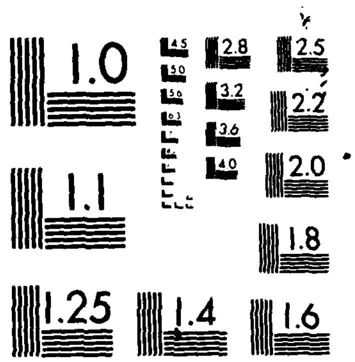

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During this one year period the installation of a single source ionized cluster beam (ICB) deposition system was completed, a three source ICB system was designed and purchased, the $LiNbO_3$ thin film and ellipsometry projects neared completion.

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1.0 INTRODUCTION

This interim report covers the period 9-1-84 to 11-30-85. During this time the program involved the initiation of a research effort on ionized cluster beam deposition (ICB), a continuation of an effort to synthesize $LiNbO_3$ films by sputter deposition, and the development of a high speed ellipsometer for in situ film deposition monitoring.

The work on ICB mainly involved the installation of a single source ICB system and the design and installation of a triple source ICB system. The purchase of the ICB systems were the result of successive 1983 and 1984 University Research Instrumentation Program (URIP) grants.

Sections 2.0 and 3.0 describe the Phase I and Phase II ICB efforts; Section 4.0 summarizes the $LiNbO_3$ work; and finally, section 5.0 summarizes the work on ellipsometry.

2.0 PHASE I - ICB

The 1983 DoD University Research Instrumentation Program Grant to ISU was for construction of the first phase of a major thin film research facility. Specifically, the grant was to provide funding for a single source ultrahigh vacuum (UHV) ionized cluster beam (ICB) deposition system which was to be coupled by a UHV transfer system to film diagnostics chambers and other deposition systems.

The overall long term plan for the thin film facility (TFF) is shown in Figure 1. The sputtering system was constructed previously as part of a research program on deposition of AlN thin films being supported by AFOSR. The electron energy loss spectrometer with LEED and UPS was constructed with partial support from DoE and ISU funds. The triple source ICB and the scanning Auger spectrometer were proposed for the 1984 University Research Instrumentation Program. The SIMS analysis chamber is proposed for the 1985 URIP.

A large room to house the TFF was provided at the Applied Sciences Center at ISU. The room was totally renovated with the addition of a laminar flow clean air system and a double door air lock. The floor, walls, and ceiling were sealed to minimize dust problems and new lighting was installed. A service chase was constructed down the center of the room below where the UHV transfer line would be constructed. This chase provides all the required power for operation of equipment, water cooling line, compressed gas for operation of the pneumatic gate valves, high purity gas line for backfilling chambers and an exhaust line for venting of the mechanical vacuum pumps used to back the turbomolecular pumps. Emergency power circuits were included for critical items in case of a general power failure. Figure 2 shows part of the service chase and the laminar flow bench where substrates will be loaded into the transfer line. In addition, each system is provided with a load lock for direct insertion of substrates.

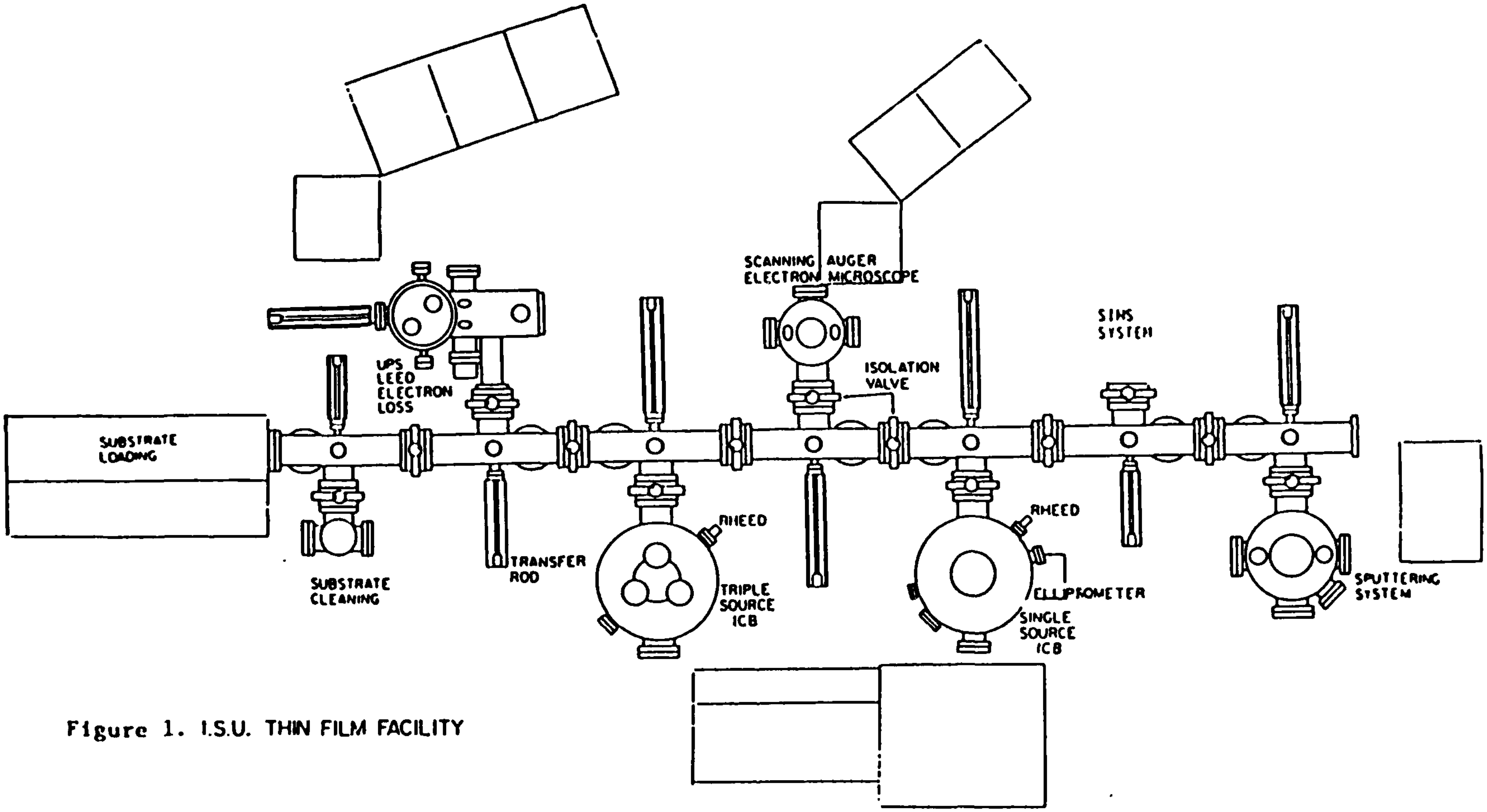

Figure 1. I.S.U. THIN FILM FACILITY

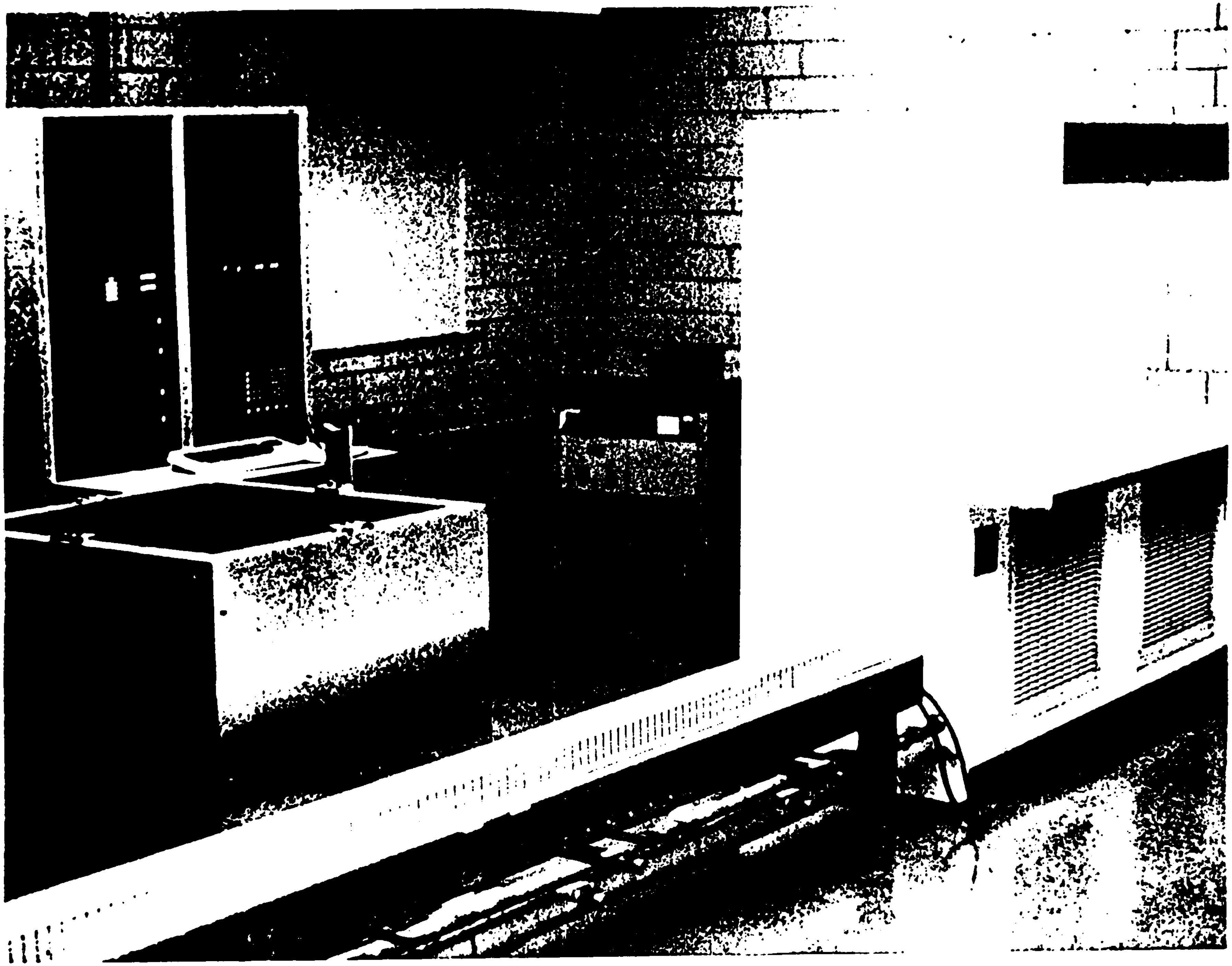

Figure 2. Service chase below transfer line in initial stages of construction.

For the single source ICB, the control system, power supplies, and the source were purchased from Eaton Corporation. The vacuum chamber was designed locally and custom fabricated. Figure 3 shows the vacuum chamber and Figure 4 shows the ICB source mounted on the base flange of the chamber. Figure 5 is an overview of the ICB system with source ready to install. The vacuum pumps for the chamber include a 2500ℓ/sec Ti sublimater (Figure 6) and a 500ℓ/sec turbomolecular pump with mechanical backing pump. Base pressure in the system is in the low 10^{-9} torr range.

The chamber has ports for RHEED and ellipsometry. A standard 10kV RHEED unit for MBE was purchased from Perkin-Elmer. This unit is used to monitor the surface structure of the growing film during deposition.

Ellipsometry is a technique just beginning to be used for monitoring thin film growth. It appears to be particularly useful for studies of island growth. A Gaertner ellipsometer with capability for obtaining a data point every 5 seconds can be attached to the ellipsometry ports on the ICB and monitor the film during deposition. For detailed studies of the initial growth stages of the film, however, a higher data rate is required. A new system with a 0.1 sec/data point capability which was designed and built locally as a M.S. thesis project is currently being tested. It will provide the detail needed for film nucleation studies.

Other diagnostics on the ICB chamber include a quartz crystal thickness monitor, a residual gas analyzer, and a Faraday cup for beam current measurements.

The transfer line, a section of which is shown in Figure 7, consists of seven sections isolated by gate valves. Each section can be individually pumped and contains a magnetically coupled push rod to move wafers from the main transfer line to the chambers. The line has a six-inch ID providing for use of three-inch diameter wafers. Ports are provided for viewing, vacuum gauging, and pumping as well as for the transfer drive mechanisms.

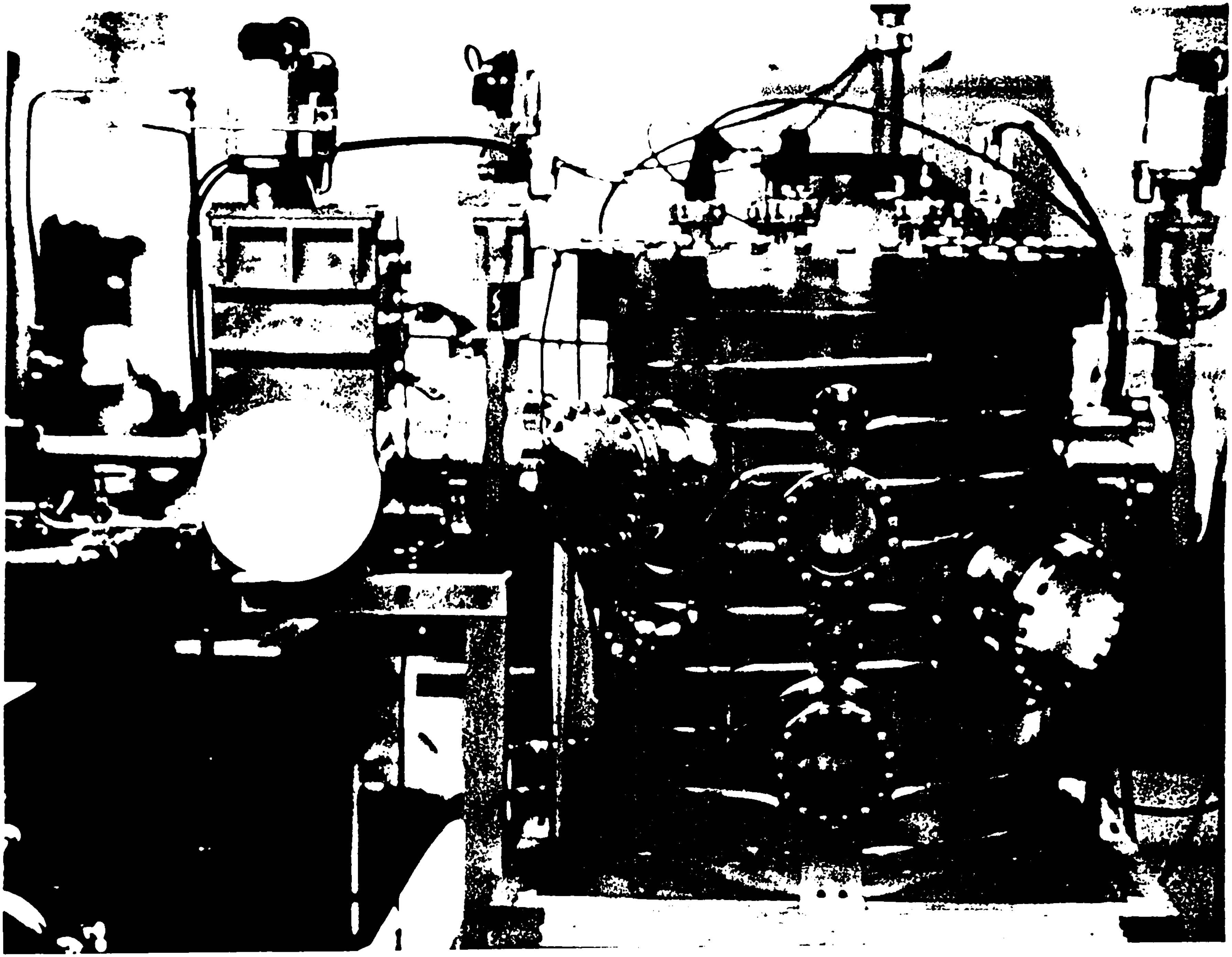

Figure 3. Ultrahigh vacuum chamber for ICB system.

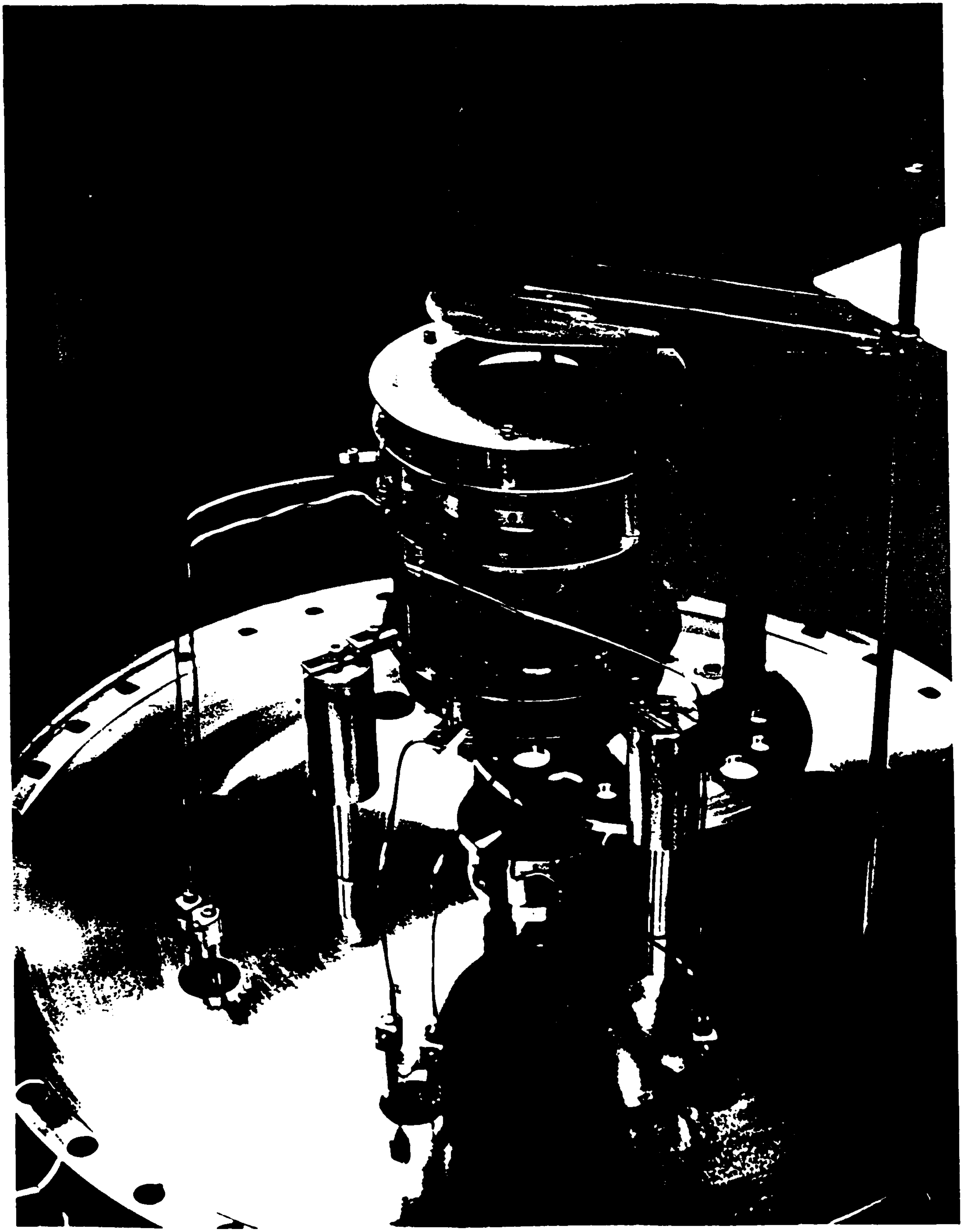

Figure [illegible]. Ionized cluster beam source mounted on UHV flange.

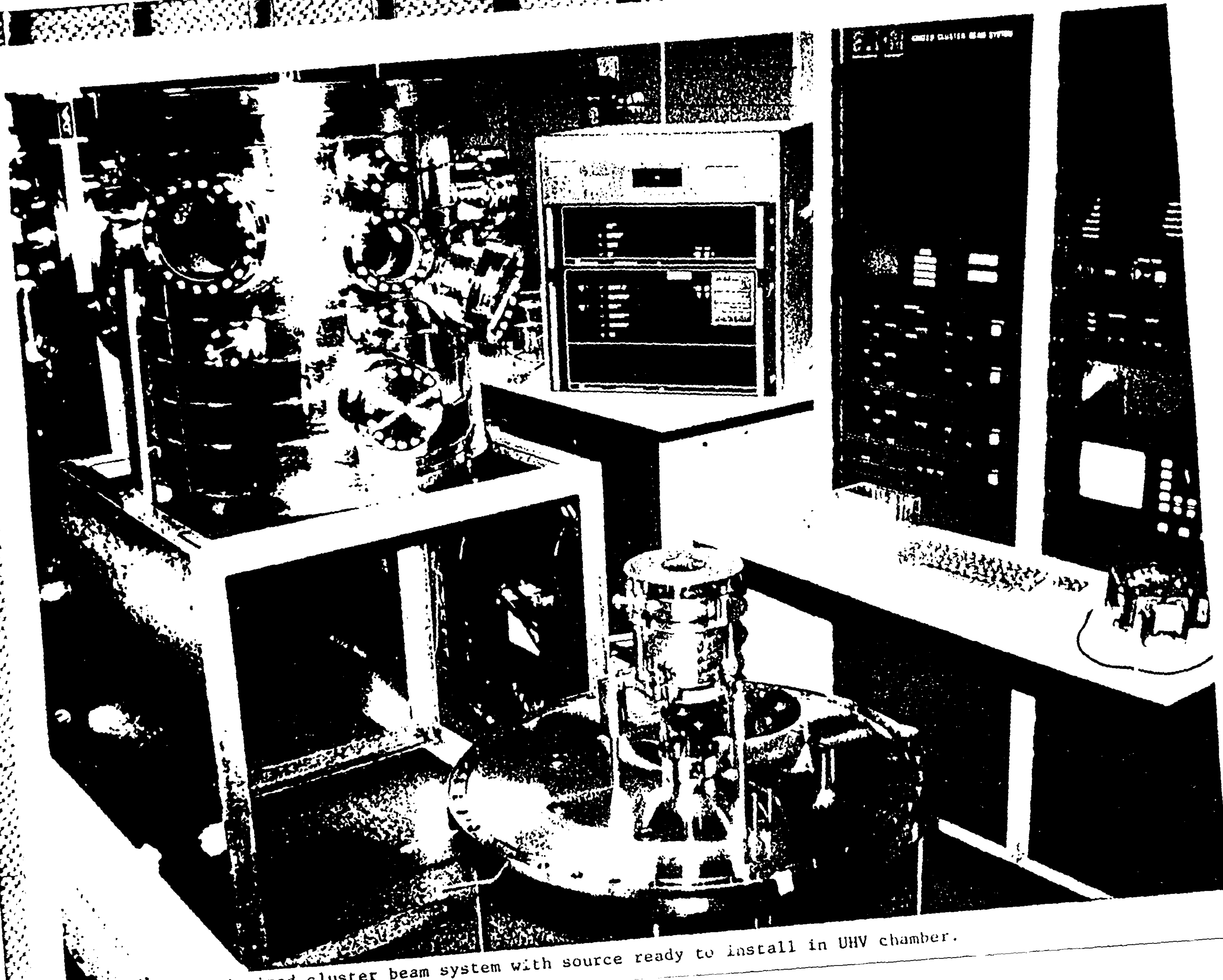

Figure 5. Ionized cluster beam system with source ready to install in UHV chamber.

Figure 6. Ionized cluster beam system with Ti sublimation ... being attached.

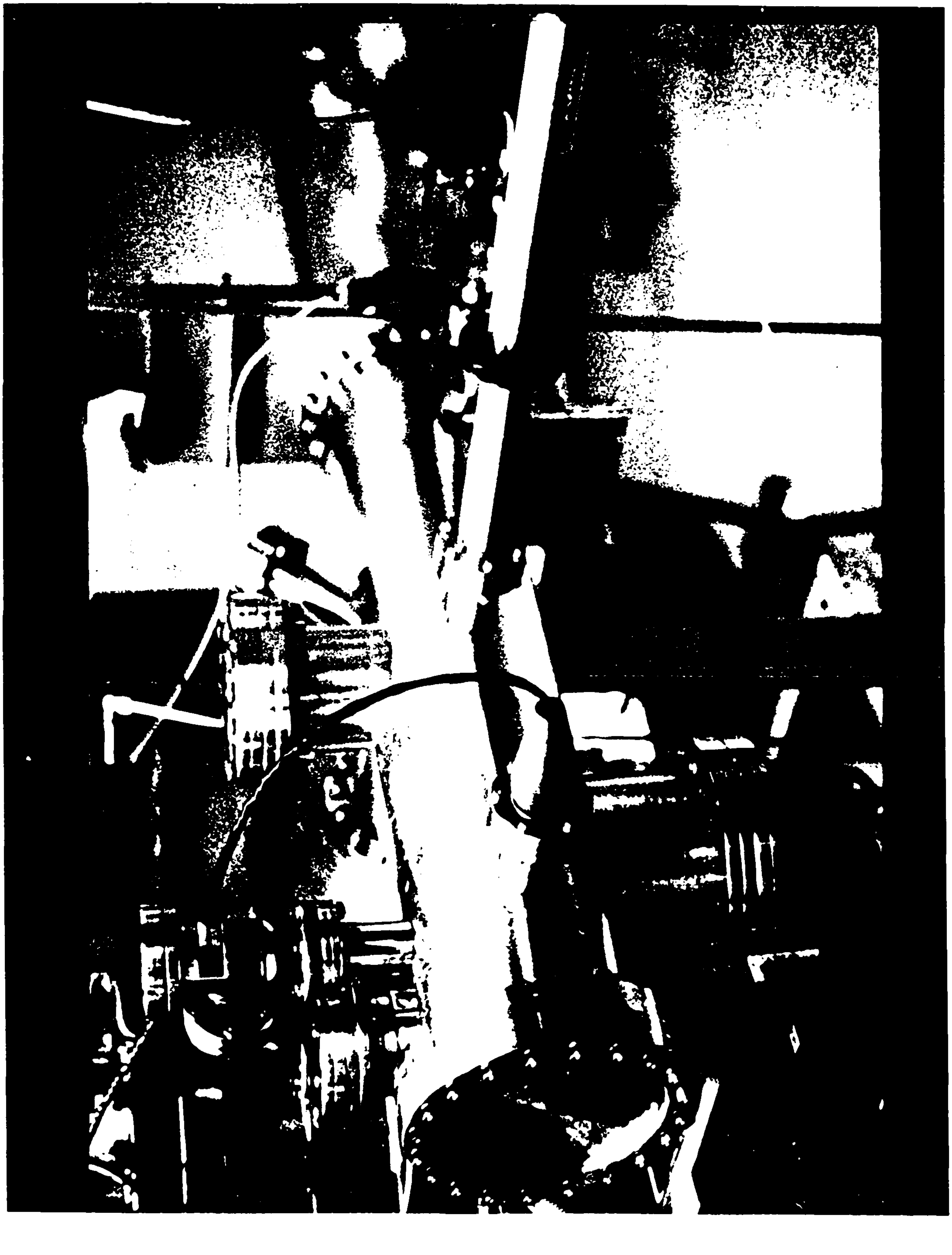

Figure 7. Section of wafer [illegible] attached to the [illegible] chamber

After initial bakeout and testing of the ICB system, a series of silicon depositions were attempted on silicon substrates. The upper limit on the source temperature that could be obtained was 1700°C. Vacuum at this temperature was maintained at $\sim 2 \times 10^{-8}$ torr. Silicon was deposited on the substrates but the crucible temperature was below the 2000-2200°C required for cluster formation. The electron bombardment crucible heater was found to be very prone to burnout and to heat the crucible in a very nonuniform manner. In addition, there was severe warpage of the radiation shields. Negotiations for a replacement source were started with Eaton Corporation.

In an effort to get work started with the ICB, experiments were redirected to the higher vapor pressure metals, aluminum and zinc. Several metallic films were deposited although the crucible heater repeatedly shorted out to the radiation shields. Several depositions of zinc were done as a function of cluster acceleration voltage. Improved adhesion of the zinc films to the silicon substrates was clearly demonstrated as the voltage was increased.

Because of our interest in piezoelectrics, we attempted to deposit AlN films by the addition of nitrogen gas to the chamber to a pressure of $\sim 5 \times 10^{-5}$ torr. We succeeded in incorporating nitrogen into the films but were unable to get stoichiometric compositions.

An alternate approach was tried for ZnO films. A tube was inserted into the chamber such that oxygen could be introduced directly into the ionization region of the source. This technique allowed the deposition of stoichiometric ZnO films but the presence of oxygen in the chamber further shortened the life of the filaments.

In order to gain a better understanding of the source design, the source was modelled on the computer and equal potential contour lines were plotted. Figure 8 shows the model with equal potential lines at 1000V intervals. To further clarify the system, the potential lines were also plotted every 100 volts above the source as shown in Figure 9. As can be seen from these figures, the fields in the system tend to spread the

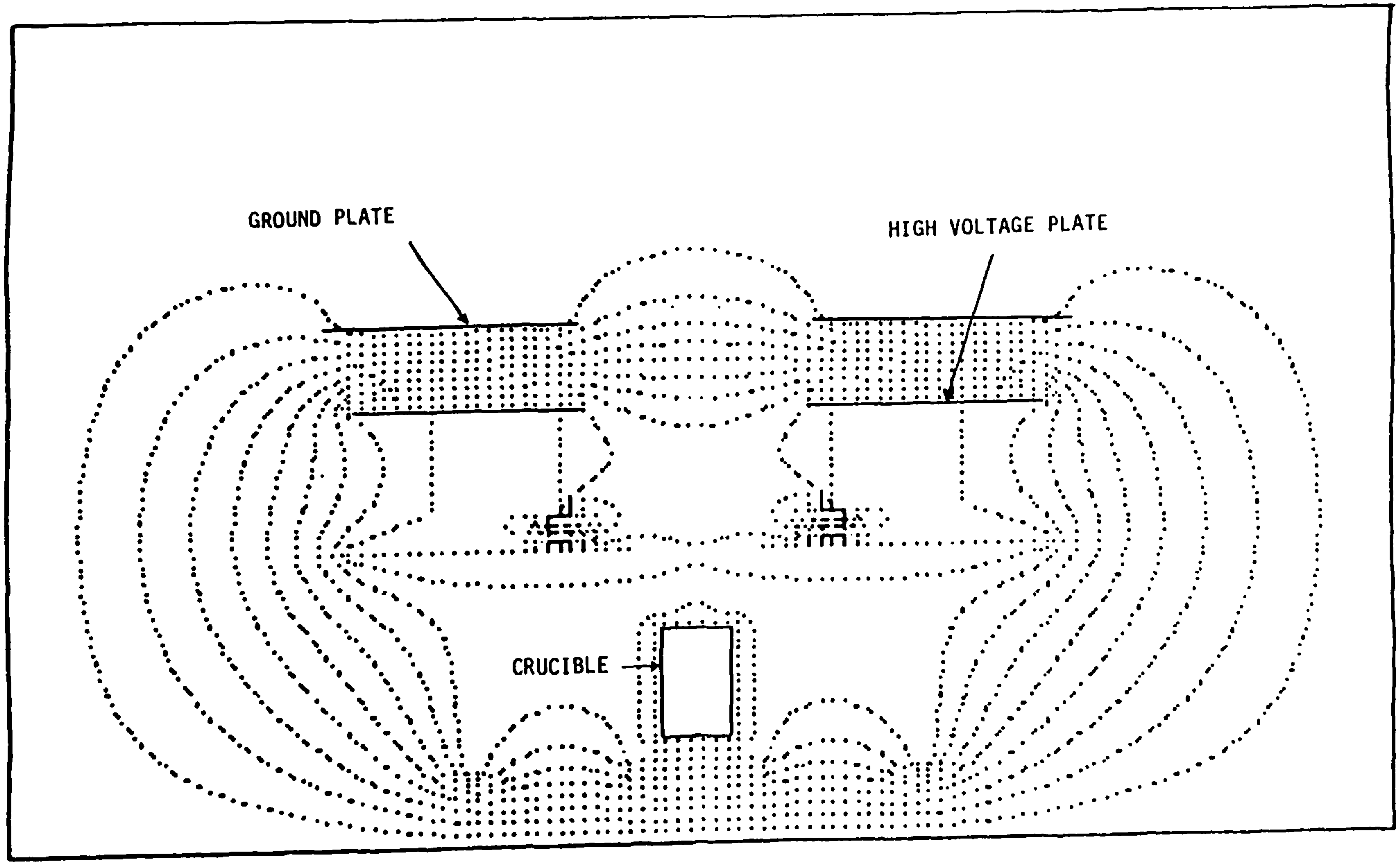

Figure 8. Equal potential lines every 1000 volts around the ICB source. Data is from a computer model of the Eaton source with 10kV accelerating voltage.

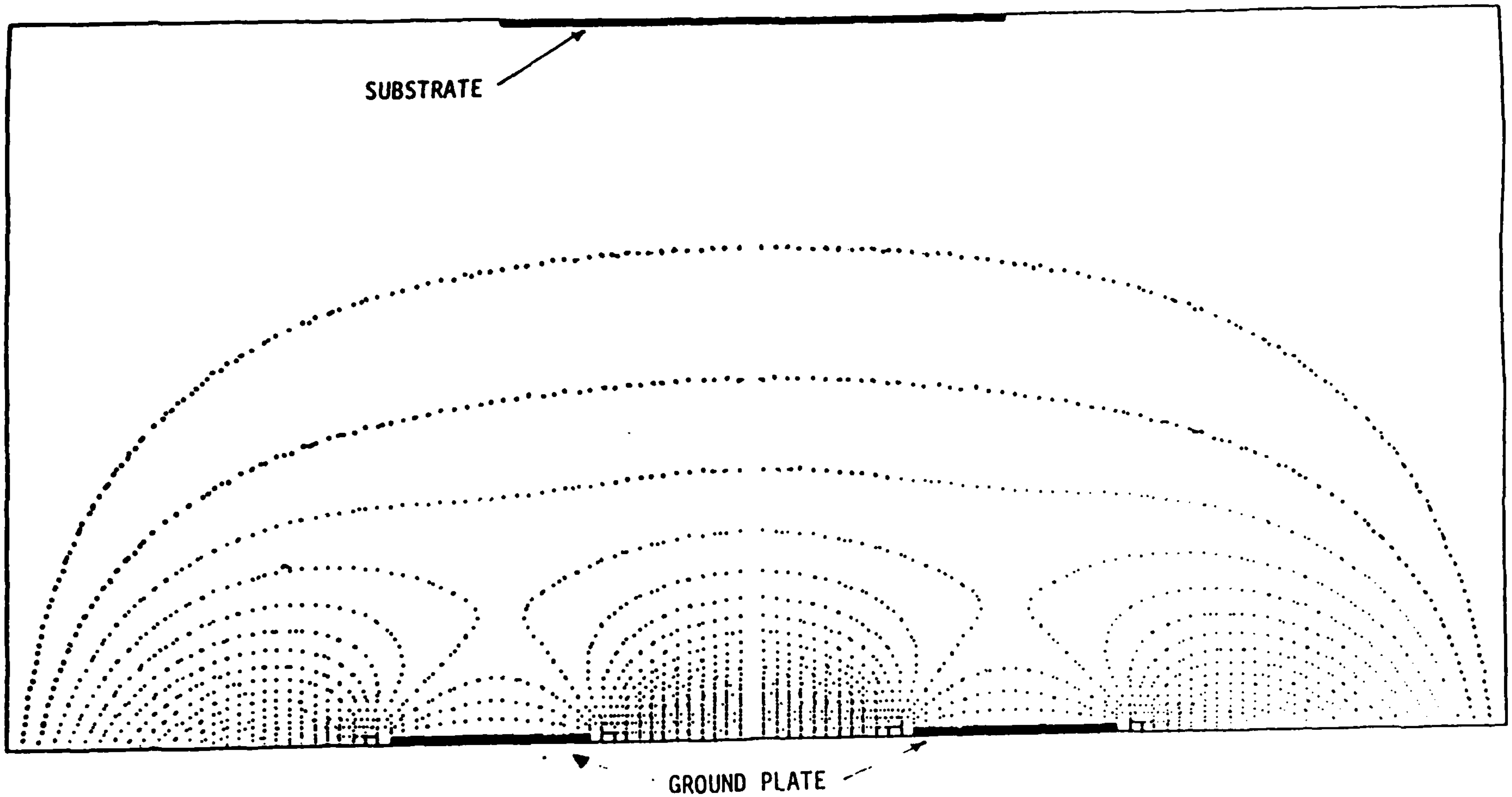

Figure 9. Equal potential lines every 100 volts from the ground plate of the source to the substrate.

charged beam which is consistent with excessive amounts of material deposited elsewhere in the system.

After considerable negotiation with Eaton Corporation, we were provided with a new style source which uses a graphite picket heater and a water cooled shroud. This new source is currently being installed.

The high vacuum sputtering system in the TFF (Figure 10) has been used to deposit Al and AlN films. A base pressure of 2×10^{-9} torr can be obtained in this system before backfilling with the sputter gas. AlN films with the desired columnar structure and which exhibit piezoelectric properties has been obtained.

The electron energy loss spectrometer shown in Figure 11 is currently being used to investigate amorphous silicon surface phonons with a 6meV resolution. This chamber with a base pressure of 5×10^{-11} torr is also used for LEED and UPS studies.

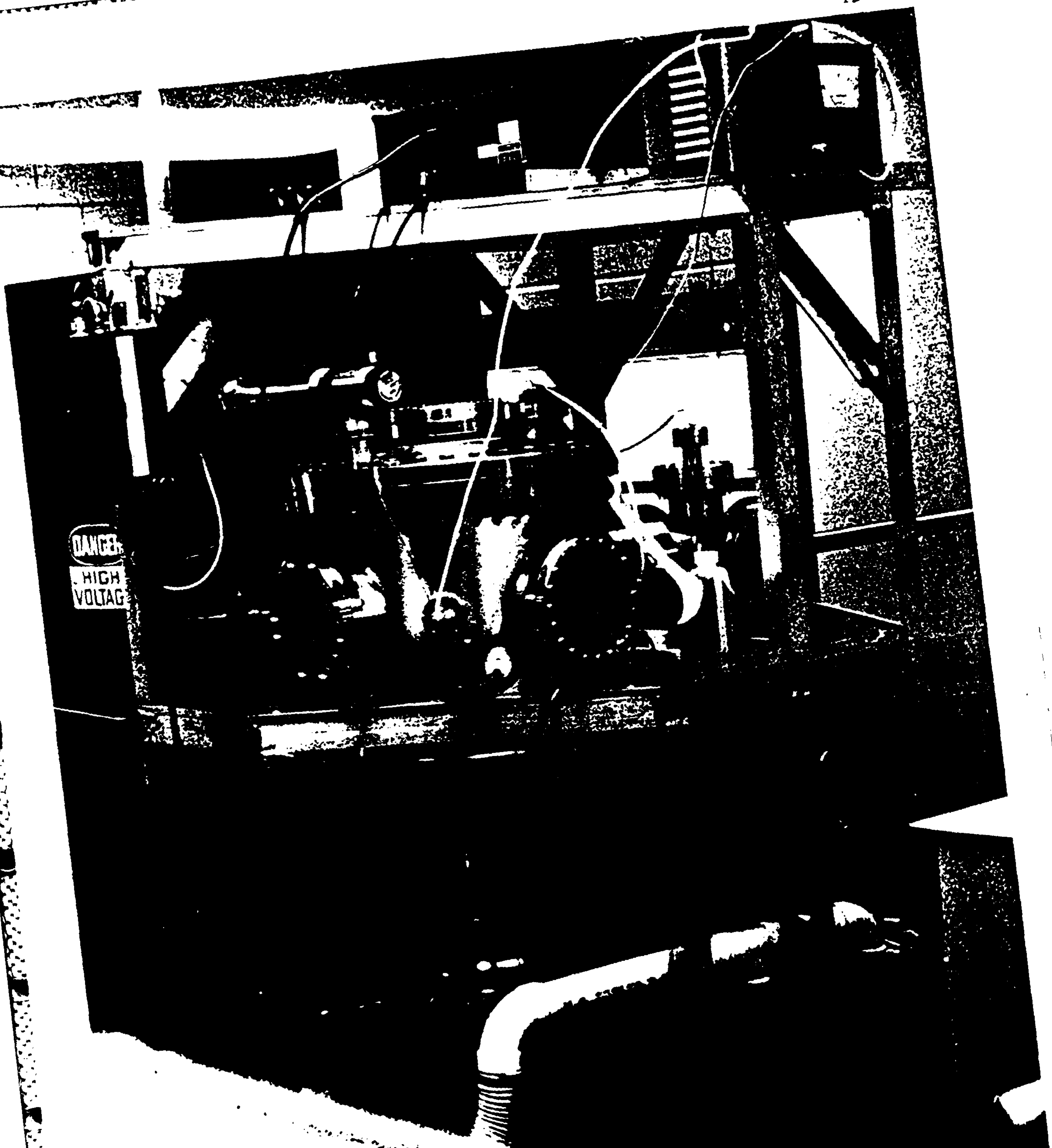

Figure 10. Sputter deposition system in thin film facility.

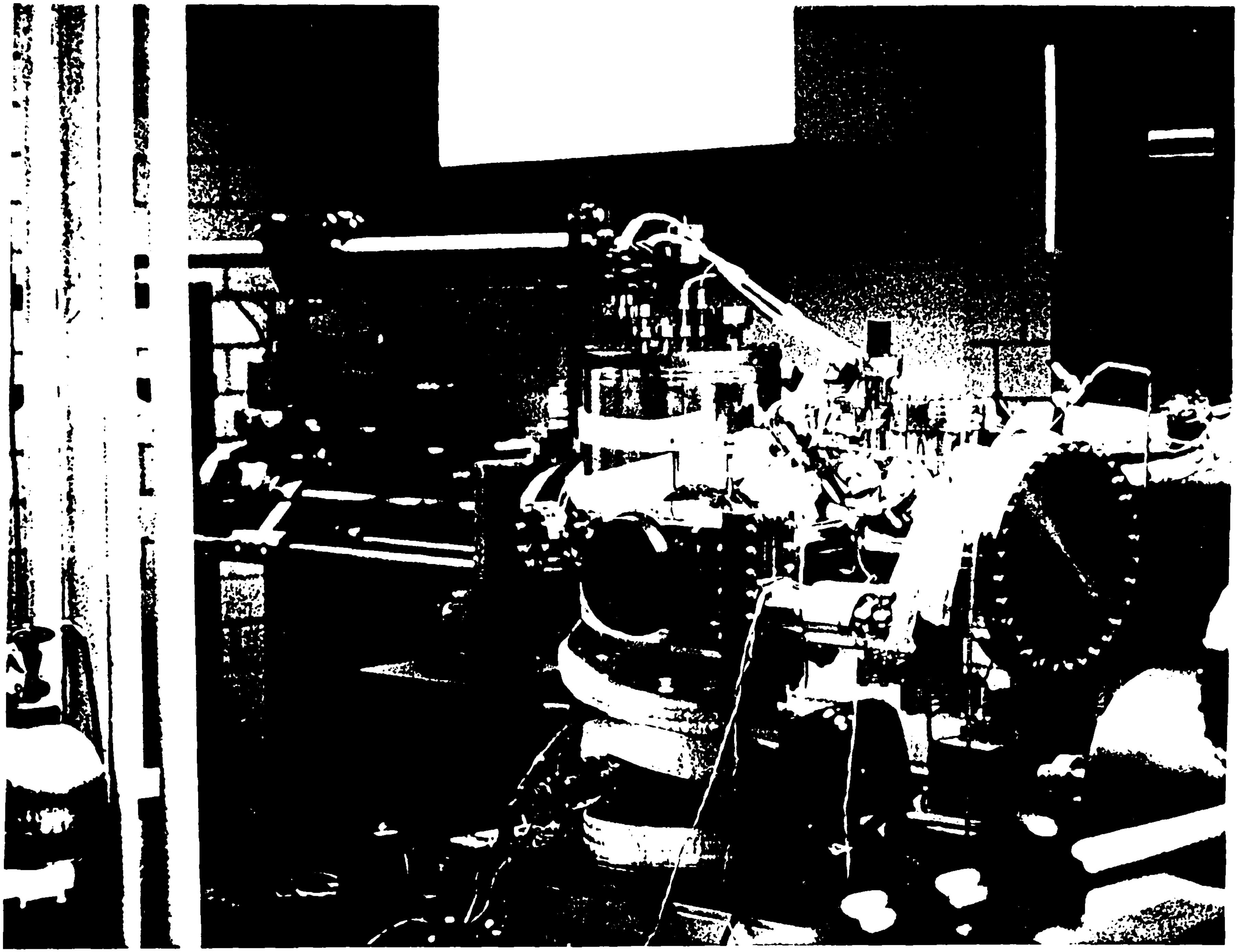

Figure 11. High resolution electron energy loss spectrometer.

3.0 PHASE II - ICB

The 1984 DoD University Research Instrumentation Program Grant to ISU was for Phase II of construction of a major thin film research facility. The grant was to provide for a three source ultrahigh vacuum (UHV) ionized cluster beam (ICB) deposition system to be connected to the UHV transfer system constructed in Phase I. Cost sharing on the grant by ISU provided for a scanning Auger system.

The overall plan for the facility is shown in Figure 1. The sputtering system was constructed previously as part of a research program on deposition of AlN thin films supported by AFOSR. It is currently being used to deposit high purity oriented AlN films on silicon substrates. The electron energy loss spectrometer (EELS) with LEED and UPS was constructed with partial support from DoE and ISU funds. The EELS is being used to study surface phonons on single crystal and amorphous silicon. These measurements are supported by theoretical first-principle calculations of silicon surface phonons by a local theorist Dr. Kai-Ming Ho.

The single source ICB is being used for epitaxial film studies. A number of oriented films of aluminum have been grown on silicon substrates. The films show no oxygen or carbon impurities but have some interaction with the silicon at the interface. Films of epitaxial (100) germanium are now being grown on (100) silicon at 400°C with sharp interfaces. No carbon crucible contamination is found.

The three source ICB system constructed with this grant has a locally designed vacuum chamber with a base pressure of $< 5 \times 10^{-10}$ Torr. In addition to three ports for ICB sources, the base plate also has ports for conventional style MBE effusion cells to increase the flexibility of the system for compound deposition. The chamber has ports for RHEED and ellipsometry. Three second generation sources and power supplies were purchased from Eaton Corporation. As modified for our systems, these sources have been repeatedly heated to ~1700°C with little difficulty. This is in contrast to the initial source for the single source system which had to be rebuilt after each run.

The three sources, with all support leads, are built on individual 8 inch OD flanges for rapid removal for servicing. Each also has a port for crucible introduction. These sources are water cooled to reduce heating of the chamber and undesirable outgasing.

The three power supplies were damaged in shipment and have required extensive repair before use. The three supplies and associated controls are totally independent so that each can be operated separately or from a single control.

A scanning Auger based on the Perkin Elmer 545 system has been constructed. It incorporates a Kratos rastorable ion gun for depth profiling. Lateral resolution for imaging and Auger mapping is about 1 μm. A depth profile of an AlN film is shown in Figure 2.

The Auger chamber makes use of differential pumping of the ion gun and a load-lock to maintain a low pressure at all times in the analysis chamber. A base pressure of $< 2 \times 10^{-10}$ Torr. is routine.

As the lowest possible base pressures are required in all the systems, efforts have been concentrated on getting each system operational with its own load-lock. This allows independent operation during initial test periods when the vacuum systems must be repeatedly cycled to atmospheric pressure. Each system is integrated with the transfer line once it is fully operational.

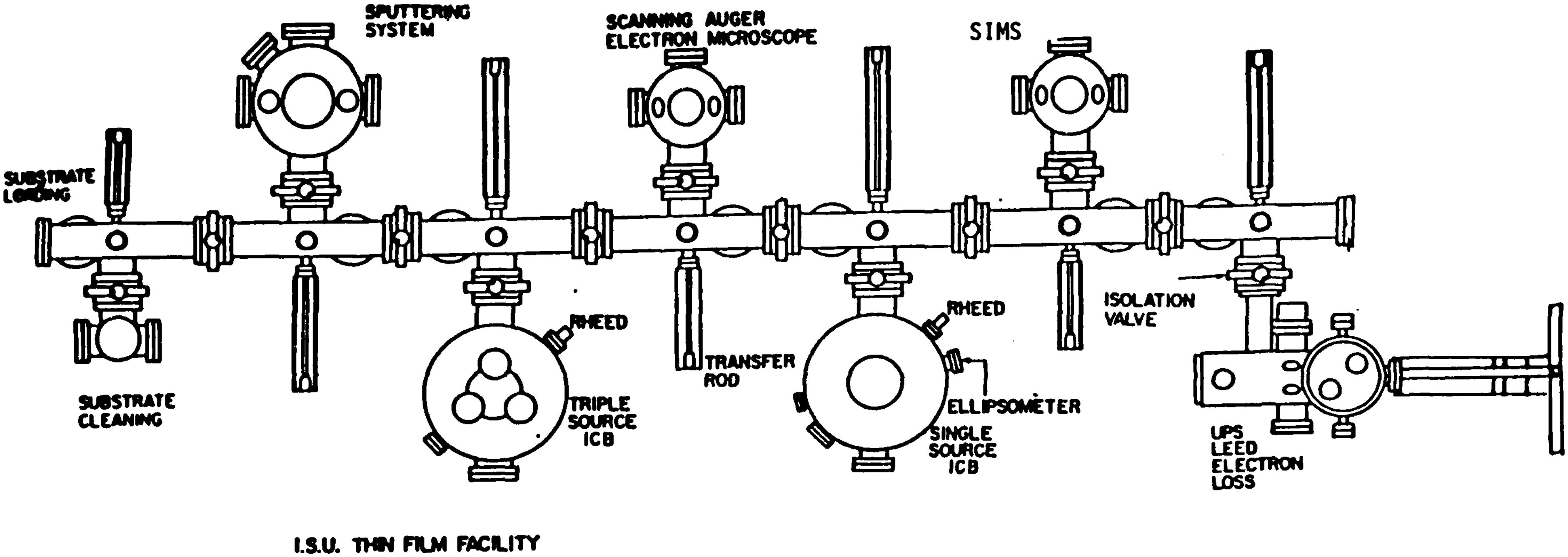

Figure 1. ISU Thin Film Facility. This grant provided for the purchase of the three source ICB system and the scanning Auger system.

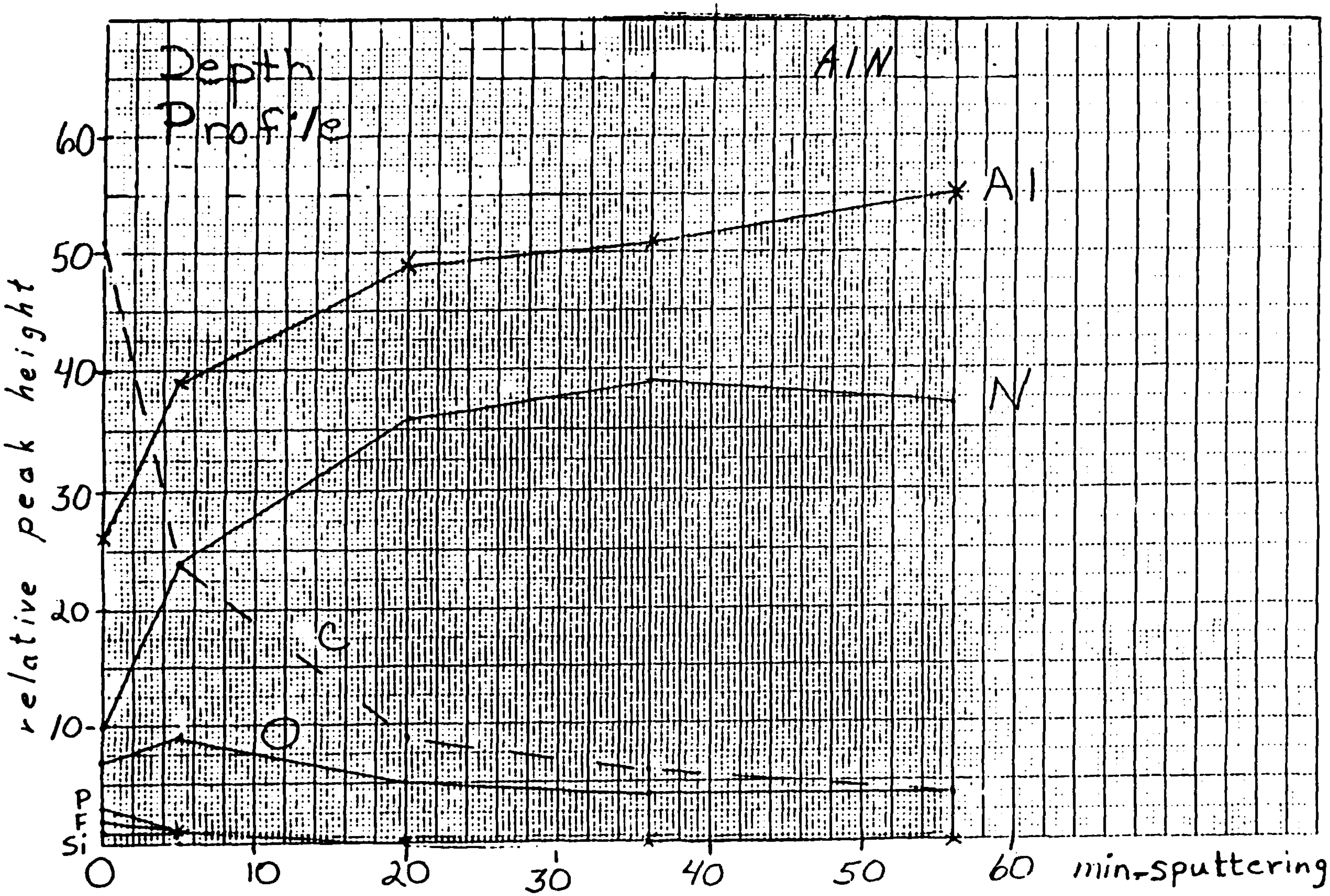

Figure 2. Auger depth profile of AlN sample.

4.0 SPUTTER DEPOSITION OF $LiNbO_3$ THIN FILMS

The sputter deposition of thin films of $LiNbO_3$ was initiated during this reporting period and was completed in the spring of 1986. The research work is reported in the MS Physics degree thesis of J. Martin entitled, "The deposition and characterization of lithium niobate thin films." The following is a summary of that work.

Lithium niobate films were sputter deposited and analyzed to determine their suitability for transducer or resonator material. A set of sputtering parameters was dete-mined as being the most relevant variables in the r.f. sputter deposition of $LiNbO_3$ thin films. The parameters were then optimized with respect to each other to obtain the best quality (in regard to crystallinity) thin films. The optimized parameters were:

An applied radio-frequency power of 300 watts.

A target-substrate separation of 8 cm.

A sputtering pressure of 3 mtorr.

An oxygen content of 50% in the sputtering gas.

A substrate temperature of 550°C - 600°C.

A sapphire substrate.

With these optimized parameters in effect, the resulting films were deposited with a deposition rate of approximately 1300 Å/hour. The DC bias during the deposition was around 350 volts. Infrared scans of the films revealed a definite $LiNbO_3$ crystalline curve with distinctive peaks (Figure 23). X-ray diffraction patterns demonstrated that the films were highly oriented polycrystalline $LiNbO_3$ (Figure 19). Auger electron spectroscopy on these films indicated an almost identical stoichiometry as bulk single crystal $LiNbO_3$, with little contamination (Figures 24 and 25). The relative permittivity of these films was determined to be 15 - 16. No piezoelectric effect was observed using the composite resonator structure.

Several possible explanations were supplied concerning this lack of piezoelectric response. While $LiNbO_3$ deposited on p+ membranes appears unsuitable for use as a composite resonator, very possibly due to the p+ membrane, sputtered $LiNbO_3$ could be used for other piezoelectric devices which require thinner films and do not demand a substrate with a poor lattice match or p+ doping. SAW devices on sapphire would be an example.

Further work could be done on what was begun here. A better sputtering system (more automated) could be used so that slower deposition rates, and thus possibly better films could be produced. An applied DC bias could be added to the sputtering system so as to more likey pole the sputtered films. Another possibility would be to try an entirely different method of sputtering lithium niobate, such as completely reactive sputtering.

5.0 ELLIPSOMETRY

High speed ellipsometry is viewed as a potentially key diagnostic tool in the real-time monitoring of thin film deposition processes. It is particularly attractive for ICB or other ion-assisted techniques where high electric or magnetic fields may make RHEED or LEED measurements impractical.

Details of the work on ellipsometry are given in the MS Physics thesis of M.L. Fleshner entitled, "Construction of two very-high-speed photometric ellipsometers" (144 pages). The following is a summary of that work.

Two very-high-speed photometric ellipsometers were constructed for this work. The ellipsometers are to be used to monitor and observe the growth of thin films by various deposition techniques. Examples of such techniques are ionized cluster beam (ICB), molecular beam epitaxy (MBE), sputtering, evaporation etc.... Both ellipsometers are photoacoustic modulated ellipsometers, PMEs. The first is a dynamic photometric ellipsometer capable of obtaining a set of ellipsometric angles, (ψ, Δ) in 20 μs. The second is a static photometric ellipsometer, capable of obtaining a set of ellipsometric angles in 1 μs. The additional information necessary to obtain this increased speed was brought about by splitting the light beam in the analyzer arm and simultaneously processing the two resulting signals. The ellipsometer is called a dual analyzer ellipsometer (DAE), since it has two analyzer arms. The concept of creating two analyzer arms is original, and can be implemented in any dynamic photometric ellipsometer, e.g., the rotating polarizer/analyzer ellipsometer.

The two very-high-speed ellipsometers were constructed almost completely from scratch. Using a Jones matrix formalism, generalized expressions for the intensity of light were calculated especially for this work. Based on these expressions, unique alignment procedures were developed for both ellipsometers. An original electronic feedback mechanism was implemented. Most of the optical mounts were constructed from a single aluminum sheet. Finally, all software was written exclusively for this project. This includes: a complexmath module, procedures to calculate a discrete Fourier transform, the value of any Bessel funtion with any argument, procedures, which control the digitizer and reduce the raw data to (ψ,Δ), and a program, which interprets (ψ,Δ) to provide the film thickness, the index of refraction, and the reflectivity of the unknown optical sample.

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