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"MOLECULAR BEAM EPITAXIAL GROWTH OF $Hg_{1-x}Cd_xTe$"

to

The United States Air Force
Office of Scientific Research

by

K. L. Moazed
Professor of Materials Engineering
North Carolina State University
Raleigh, North Carolina 27650

INTRODUCTION

The II-Vi compound $Hg_{1-x}Cd_xTe$ is a material of considerable interest as a semiconductor for optical devices, particularly in the infrared spectral regions. Solid solutions appear to exist for all values of x, and for values of x between about 0.2 and 1.0 the material behaves as a semiconductor with a variable bandgap between about 0.05eV(x=0.2) and 1.53eV(x=1) at room temperature [1]. Thus, in principle, the fundamental absorption edge of the semiconductor varies with composition through the wavelength range of 0.8μm to beyond 20μm, and specifically covers both atmospheric "windows" of high infrared transmission at 3-5μm and 8-14μm. While there are other semiconductors which can address optical device needs at either of these windows, there are no other materials which can cover this entire range. The III-V compound $InAs_{1-x}Sb_x$ does cover the range of 4μm - 12μm at 300K and 3.5μm - 9μm at 4.2K [1], and the lead salts $Pb_{1-x}Sn_xTe$ and $Pb_{1-x}Sn_xSe$ also cover similar ranges, depending on the temperature.

The ternary $Hg_{1-x}Cd_xTe$ system may be considered as a pseudo-binary isomorphous system consisting of the components HgTe and CdTe as shown in Figure 1. Although there is some degree of uncertainty regarding the position of the liquidus line and that of the solidus line [1], there is general agreement regarding the phase relationships that exist in this system [2]. The lattice structure of the binary compounds (HgTe and CdTe) as well as alloys in the ternary system $Hg_{1-x}Cd_xTe$ consists of two interpenetrating face centered cubic sublattice corresponding to a zincblende structure; where one sublattice accommodates the cations (Hg or Cd in the binary compound or Hg and Cd in a random solid solution in the ternary compounds) while the other sublattice accommodates the anions (Te). Thus, $Hg_{1-x}Cd_xTe$ is crystallographical compatible with both the III-V semiconductors, such as GaAs, and the elemental semiconductors, such as Si. This fact allows the consideration of these materials in a variety of ways with $Hg_{1-x}Cd_xTe$ materials and devices, for example as substrates or multiple-level detector arrays.

The lattice parameter of alloys in the HgTe-CdTe pseudo-binary isomorphous system varies less than 1% across the ternary and corresponds to a slight negative deviation from Vagard's rule with values of 6.46Å for HgTe and 6.48Å for CdTe [3,4]. The density of the ternary alloys varies linearly from a value of approximately 8 gm/cm^3 for HgTe to a value of approximately 5.8 gm/cm^3 for CdTe. Although a great deal of information about the physical properties for alloys within this ternary is not available, a number of these parameters have been determined for $Hg_{0.8}\,Cd_{0.2}Te$ by interpolation from the experimental values obtained for the binaries as shown in Table 1. Among other parameters that have been examined for this system at various alloy composition are the conduction band edge effective mass, the temperature dependence of the energy gap and the optical absorption edge.

Much of the early work on $Hg_{1-x}Cd_xTe$ is attributed to three main groups [5]. These groups are W. D. Lawson and co-workers at the Royal Radar Establishment in England, M. Rodat and co-workers at the Laboratories de Magnetisme et de Physique de Solide, CNRS, Bellevue, France and P. W. Kruse and Associates of the Honeywell Corporate Research Center in Minnesota. Further research has been carried out by research teams at Lincoln Laboratory, MIT, at the Institute of Physics, Warsaw, Poland and at several locations in the Soviet Union and Eastern Europe.

The growth of $Hg_{1-x}Cd_xTe$ structures having a variable composition was achieved by the interdiffusion of CdTe and HgTe by a number of investigators [6-10]. Evidently this was the initial method for the production of ternary alloys. The interdiffusion process produces a range of alloy compositions which is not always desirable. The growth of homogeneous alloys of fixed composition which are free from impurities has posed a problem of some considerable difficulty.

The usual method of producing $Hg_{1-x}Cd_xTe$ crystals for detectors is by liquid-solid transformation from a melt. There are a number of variations in the method employed for solidification from the melt; these include mixing of the elements in a sealed evacuated container, usually quartz, then heating to produce a melt and then cooling slowly to prevent consitutional supercooling in advance of the liquid-solid interface which would lead to formation of solid crystals within the liquid in advance of the liquid-solid interface [3,11,12]. The growth methods include the Bridgman as well as a number of variations [13,14]. Another method used is to melt and solidify a mixture of the binary compounds HgTe and CdTe to produce the ternary of desired composition [4]. There are a number of problems associated with solidification from a melt when the solidification process occurs incongruently, as is the case for $Hg_{1-x}Cd_xTe$ alloys. One of these problems is the rather large degree of segregation of the alloying elements that occur both on a macroscopic as well as on a microscopic level, commonly referred to as macrosegregation and microsegregation, respectively. These problems are accentuated as the value of x (atom % Cd) increases from 0, reaches a maximum for $x \simeq .5$ and decreases as $x \to 1$. This means that the segregation problem is less serious for detectors in the 8-14μm range than for the 3-5μm range when produced by liquid-solid transformation techniques. Another source of compositional inhomogeneity occurs in this system because the slope of the solidus line is much smaller than the slope of the liquidus line in the mercury rich end of the phase diagram ($0<x<1/2$), as shown in Figure 1. Therefore, a small variation in the temperature of the melt will produce a large variation in the composition of the solid crystallizing from the melt. In any case the segregation effect can be alleviated by a homogenization anneal slightly below the liquation temperature, following the solidification process.

Another problem associated with solidification from the melt in this system is the possibility of gravity segregation due to the large variation in the density of the components in the melt. A slush rocking method has been developed to reduce gravity segregation. Another method for reducing segregation is to quench the alloy rapidly from a temperature slightly above the liquidus temperature. The quenching process leads to a more uniform composition of the solid; however, because of a large degree of supercooling, a fine grained polycrystalline product is produced. A high temperature anneal is used to allow for grain growth through either primary or secondary recrystallization.

Many of the problems inherent in the liquid-solid transformation may be circumvented by the utilization of vapor phase deposition. It should be noted that each component in the Hg-Cd-Te ternary has a sufficiently high evaporation rate and consequently a carrier gas method for increasing the rate of transport is not necessary in this system. For example, at approximate temperatures of 450^0 for Te, 400^oC for Cd and 125^oC for Hg, a vapor pressure of approximately 10^{-1} torr may be achieved for each component by free evaporation. Although the epitaxial growth of $Hg_{1-x}Cd_xTe$ may be accomplished by the vaporization of the three components with or without the aid of carrier gases, the majority of the research to date has used some variation of evaporation of binary or ternary compounds.

A serious problem with evaporation techniques where binary compounds are used as the source material in the evaporation process occurs when the rate of evaporation of the various components in the binary or ternary compounds is measureably different, as is the case for the HgCdTe system [15, 16]. Because the vapor pressure of Hg is considerably greater than that of Cd and of Te, in the evaporation process where $Hg_{1-x}Cd_xTe$ is used as the evaporation source material, the vapor flux will initially contain a higher concentration of Hg than the stoichiometric concentration present in the compound, but will eventually reduce to a concentration that is less than the stoichiometric concentration. This means that the concentration of the deposited film will vary as a function of deposition time. Nevertheless, much of the work on growth of $Hg_{1-x}Cd_xTe$ to date has used a variation of this method [17-26]. Some of the variations included the use of supplementary vapor sources of binary compounds and/or elements [27, 28]. Other variations include the use of sputtering techniques [29, 30] and of flash evaporation techniques [27, 31].

At the present time, there is considerable interest and effort toward applying state-of-the-art compound semiconductor synthesis techniques, such as are used to grow III-V semiconductors, to the $Hg_{1-x}Cd_xTe$ system. These techniques include liquid-phase epitaxy (LPE) and vapor phase epitaxy (VPE) growth systems [32] which are used to synthesize GaAs and other similar materials. These techniques have been prominent in the development of thin, uniform heteroepitaxial layers required for a electrooptical devices in optical communication systems [33], and the realization of high quality growth techniques are a major reason for the emerging impact of III-V semiconductors in a variety of electronic device applications.

One presently-developing growth technique which appears to offer much promise for unique material synthesis and new device concepts is molecular beam epitaxy (MBE). In this technique thermal beams of atoms or molecules impinge upon a heated substrate under ultrahigh vacuum conditions. This process differs from thermal evaporation since the beam intensities are separately controlled, which allows sticking-coefficient differences to be taken into account [34]. The MBE technique allows precise layer thickness control over large areas, and appears to be especially suited for the fabrication of a variety of optical devices [33].

The ternary II-VI compound $Hg_{1-x}Cd_xTe$ has not been synthesized using MBE growtn techniques. Potentially, the molecular beam epitaxial growth technique (MBE) offers many advantages over either the liquid phase epitaxial growth technique (LPE), or the vapor phase epitaxial growth technique (VPE) in the production of multicomponent compound semiconductors, such as $Hg_{1-x}Cd_xTe$. For example, whereas in the LPE, and to a lesser degree in the VPE, the composition of the compound is limited to quasi-equilibrium conditions, in the MBE much larger deviations from equilibrium composition may be achieved. This feature is of special importance for doping of compound semiconductors. Another feature of MBE that marks its potential superiority over LPE and VPE is the ability to produce extremely thin films with much closer control on the thickness of the films. Theoretically the thickness may be controlled to a monolayer.

Perhaps the most important feature of MBE is the possibility to control the composition of the film at a monolayer level. This composition control may be used to obtain a homogeneous film or a heterogeneous film with any compositional gradient that the design of the device dictates. In theory, it is possible to obtain with MBE complex doping profiles, graded junctions or abrupt junctions, in extremely thin layers. Although the potential superiority of MBE over LPE and VPE has not been demonstrated in actual devices produced to date, it cannot be denied that the stage of development of MBE lags the others by a decade as a minimum; consequently, much more experimental work is required before a final judgement about the relative merits of each technique for a given application can knowledgeably be made.

In the author's judgement, the MBE techniques that are currently in wide use have three major deficiencies that detrimentally affect the quality of films produced. The deficiencies are, the relatively low level of vacuum maintained in the system during deposition (which affects background contamination), the configuration of the various sources with respect to the substrate, and large variations in the source temperature. The purpose of the research effort was to design and construct a basic MBE system for the growth of $Hg_{1-x}Cd_xTe$ which would overcome these deficiencies, and to evaluate the feasibility of the MBE technique for the growth of this important ternary compound for infrared device applications. The system is a glass system designed to incorporate a manifold which will ensure homogeneous composition over the surface of the substrate.

DESIGN OF THE MOLECULAR BEAM EPITAXIAL GROWTH SYSTEM

In order to eliminate the deficiencies cited above, a system was devised that substantially deviates from those in use today as shown in Figure 2. This system permits the attainment of better vacuum through the use of subsidiary pumping in the region of the evaporation cells. Subsidiary pumping is accomplished with the use of mercury diffusion pumps. The apparatus consists of a mercury diffusion pump with a capacity of 80 liters/sec. in the region of each of the three evaporation cells. A fourth mercury diffusion pump with the same capacity is used in the substrate section of the system. Therefore, during the deposition there will be a dynamic pumping capacity of over 300 liters/sec. Ideally high speed pumping can be achieved more effectively with other pumping systems such as molecular turbine pumps and closed cycle L He cryopumps. However, because one of the components in the compound being investigated is mercury, and furthermore, because mercury has high reactivity with many of the alloys commonly used in the manufacture of pumps, it was deemed advisable to use mercury diffusion pumps as the main pumping regime.

Associated with each mercury pump is a liquid nitrogen trap to effectively eliminate mercury back-streaming into the system. Incorporated in the vacuum system is a pladium diffuser to allow for the introduction of pure hydrogen gas into the system. The system is purged with hydrogen gas prior to growth to react with and remove reducible impurities within the vacuum system. Hydrogen gas is also used to purge the source cells during their warm up period prior to growth to remove impurities that may be present. Therefore, it is anticipated that residual pressures of 10^{-9} torr or better will be attained during film growth. A separate vacuum system is used to pump the substrate section of the system as shown in Figure 2. The evacuation of this section is accomplished with the utilization of a mercury diffusion pump. It is anticipated that the level of vacuum in this system during film growth will be one or two orders of magnitude better than in other MBE systems and hence the amount of film contamination from residual gaseous impurities will be substantially reduced. The reduction in the impurity level of the films should produce electronic properties that would more nearly conform to those theoretically predicted. The lack of high speed pumping, especially in the evaporation cells section of most MBE systems that are currently in use results in contamination of the films which is a deficiency that was mentioned earlier.

A valve is used to isolate the two parts of the vacuum system prior to the initiation of film growth. This isolation allows independent evacuation and outgassing of the source cells without the danger of contaminating the substrate. The substrate section will remain isolated until the sources have been individually outgassed and raised to the evaporation temperature and stabilized at that temperature before the valve is opened to initiate the growth on the substrate. Of course, the substrate is also outgassed and its temperature adjusted and stabilized prior to the opening of the gate valve and the initiation of growth. At the termination of the growth the gate valve is closed and the two sections of the system are once again isolated from one another.

Perhaps the most important feature of the system that is being proposed here for the growth of $Hg_{1-x}Cd_xTe$ is the addition of a manifold shown in Figure 2. All of the MBE systems, known to the author, use substrate-sources configuration that approximates a cone. Therefore, the normal to the substrate is not colinear with the axis of the cell. This means that the normal to the cell orifice is at an angle to the normal of the substrate. Therefore, the flux of vapor arriving at the substrate is not uniform over the substrate surface. This nonuniformity increases as the angle of the cone increases, that is as the source-substrate distance is reduced. In fact, the nonuniformity in the flux is a function of the cosine of the conical angle. The nonuniformity of flux at the substrate for two cells that are diametrically opposite one another is, of course, opposite in intensity. This means that where the flux of vapor from cell A arriving at the substrate is high, the flux of vapor from cell B (diametrically opposite cell A) is low and vice versa. This results in a totally heterogeneous concentration of fluxes from the two cells at every point over the substrate surface. This lack of homogeneity of the various components over the substrate surface is a serious deficiency that the proposed design will eliminate through the utilization of the manifold.

The manifold serves two main purposes. First, each cell has an opening into the manifold only and therefore there is no direct line between each cell and the substrate except through the manifold. The vapor flux from each cell impinges on the surface of the manifold and therefore the manifold in effect becomes a composite cell for all of the evaporation cells. That is, the manifold acts as a "mixing cell" for all of the components. The exit part of the manifold is situated such that its axis is colinear with the normal to the substrate and hence the flux of vapor of each component will be homogeneous over the substrate surface. Through the adjustment of the temperature of each cell it is possible to produce any composition of the compound having a homogeneous distribution over the surface of the substrate. The second purpose that the manifold serves is to equalize the temperature of the various species in the manifold. This is accomplished by maintaining the manifold at a constant temperature. In this way all species arriving at the substrate surface will have a uniform temperature, which is the manifold temperature, rather than their cell temperature which is different from the other cell temperatures. The film growth under these conditions would be a pseudo-isothermal condition for all species, whereas in the usual MBE systems, each component will be at a different temperature dictated by their individual cell temperatures.

Although the manifold serves as a "mixing cell" for all the components, under normal operation of the system, the impingement flux for the various components is extremely small to allow for compound formation in the gaseous phase. Furthermore, even if the collision frequency was high, the rate of compound formation homogeneously in the gas phase would be negligible for the components proposed in this study.

It may be postulated that alloy formation may occur on the surface of the manifold heterogeneously. This possibility is real under certain operating conditions. For example, if the sticking coefficient of a component on the surface of the manifold was greater than zero, at the temperature that the manifold was being held, then the adatom concentration can be determined under steady state condition by noting that the flux of vapor atoms that supply the surface must equal the flux desorbing from the surface. That is,

$$J_{ads.} = J_{des};$$

where $J_{ads.}$ is the adsorption flux and can be expressed by,

$$J_{ads.} = \alpha P(2\pi mkT)^{-1/2},$$

and where; α = sticking coefficient, P = vapor pressure of the component, m = mass of the component in the gaseous phase, k = Boltzmann's constant, T = absolute temperature. Likewise, the flux desorbing from the surface $J_{des.}$ can be expressed by,

$$J_{des.} = n\nu\exp(-\Delta G^{*}des./kT)$$

where; n = number of adatoms per unit area of the surface, ν = adatom vibration frequency, ΔG^{*}_{des} = Gibbs activation free energy for desorption. By rearranging terms one obtains,

$$n = \alpha P(2\pi mkT)^{-1/2}[\exp(-G^{*}_{des}/kT)]^{-1}.$$

It is obvious that the adatom concentration n increases as $\alpha \to 1$, as P increases and for large values of $\Delta G_{des.}$. Conversely as $\alpha \to 0$, $n \to 0$. The sticking coefficient α is a function of temperature and for temperatures approaching 100^{o}C α vanishes to zero for metals such as Hg and Cd on glass substrates. The sticking coefficient for Te is in general less than for Hg and Cd at the same temperature. Consequently at temperatures greater than 100^{o}C the adatom concentration for each of the components on the manifold surface would be negligibly small and therefore the possibility of compound formation on the surface of the manifold can be neglected.

Although for the components and the system proposed in this study the possibility of compound formation within the manifold under the anticipated operating condition can be neglected, for components, systems, and operating conditions where the condensation coefficient is not equal to zero an alternate design of the manifold has been devised to eliminate the possibility of compound formation. If, in the course of the testing program in the proposed study, evidence is found to indicate compound formation in the manifold does occur under the conditions that would be optimal for film growth, then the alternate design of the manifold which has been devised to offset that very problem will be used.

In some cases it may be desirable to produce films of variable composition as a function of time, i.e. graded films. These films would have a homogeneous composition laterally over the surface while their composition would vary in a controlled manner in the normal direction. In the proposed system the growth of such a film would be easily accomplished through the control of the various source temperatures to produce the desired variation in concentration as a function of distance in the normal direction to the substrate surface.

CONSTRUCTION OF THE MOLECULAR BEAM EPITAXIAL SYSTEM

The MBE system was constructed according to the design scheme outlined above with some modifications in order to simplify the glass blowing. The construction of the system was delayed to a considerable extent because of budgetary restrictions, procurement policy, delayed shipments and shipment of faulty parts.

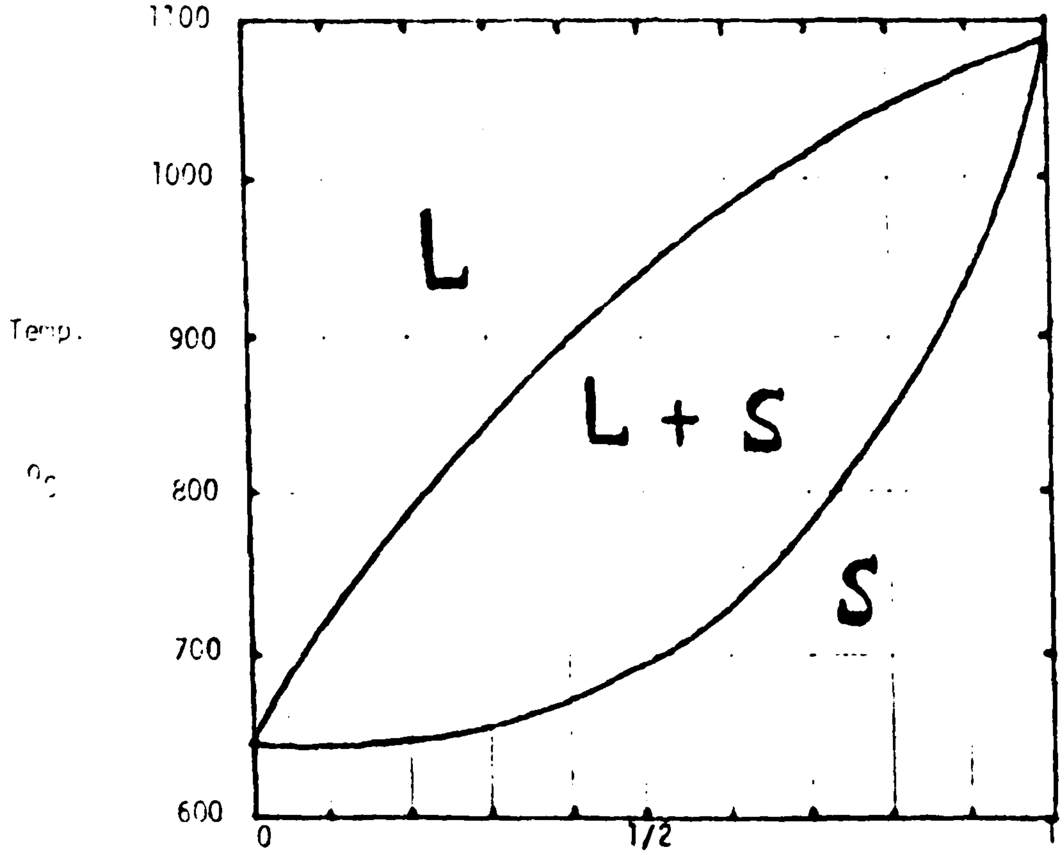
The growth system was initially constructed and operated using high vacuum valves while we awaited delivery of ultra-high vacuum valves in order to test the various components of the system. At this time it was discovered that the mercury diffusion pumps would not operate efficiently with tap water as a source of coolant for the condensation toweres of the pumps. Consequently, a closed loop water chilling unit had to be purchased and installed at NCSU's expense. This modification resulted in a six-months delay. Following the installation of the water chiller several additional experimental runs were made. These runs demonstrated a need for a modification of the substrate section of the system, which has been completed. At the present time we are awaiting replacement parts for several ultra-high vacuum valves that were shipped with defects which were the cause of vacuum leaks in the valves as shown in Figure 3.

CONCLUSIONS

A unique Molecular Beam Epitaxial growth system specifically designed for the growth of $Hg_{1-x}Cd_xTe$ has been constructed and will be operational in the near future.

The system cannot be operated because of termination of funding by the AFOSR.

It should be noted that the basic principles of film growth using "molecular beams" were discovered approximately a century ago. The application of these principles for the development of a reliable system for the growth of III-V compound semiconductors has consumed nearly three decades. When we (with the enthusiastic encouragement and support of the research personnel of the Electronics Branch of the Avionics Laboratory at WPAFB) proposed to undertake the design, the construction, and the operation of a unique system for the growth of $Hg_{1-x}Cd_xTe$ films, it was with the understanding that AFOSR would support this project for a minimum of three years. We were led to believe that although the AFOSR grant was for one year, continued support for at least two additional years would be provided to demonstrate the feasibility of the proposed MBE system, otherwise we would not have undertaken this task. To the author it appears as sheer folly for AFOSR to have: 1) spent over $65,000 of the taxpayers' money as well as our time and effort and then to abandon the project prematurely, or 2) expected that a project of this magnitude and complexity could have been completed with one year of funding which they provided.



Mole Fraction CdTe

Figure 1. Phase Diagram of the HgTe-CdTe Psuedobinary System.

TABLE I

Selected Physical Properties of $Hg_{1-x}Cd_xTe$

Parameter		Experimental Values		Interpolated
		HgTe	CdTe	$Hg_{0.8}Cd_{0.2}Te$
Static dielectric const.		20	10	18
Optical dielectric const.		14	7	12.6
Longitudinal optical phonon energy at zone center (eV)		0.0160	0.0212	0.0170
Transverse optical phonon energy at zone center (eV)		0.0143	0.0179	0.0150
Thermal conductivity at 77°K ($Wcm^{-1}°K^{-1}$)		0.15	0.4	0.2
Linear thermal expansion coefficient at 25°C ($X10^{-6}°K^{-1}$)		4	5.5	4.3
Elastic constant at 25°C ($X10^{11}dyn/cm^2$)	C_{11}	5.4	5.35	5.39
	C_{12}	3.8	3.681	3.78
	C_{44}	2.05	1.994	2.04

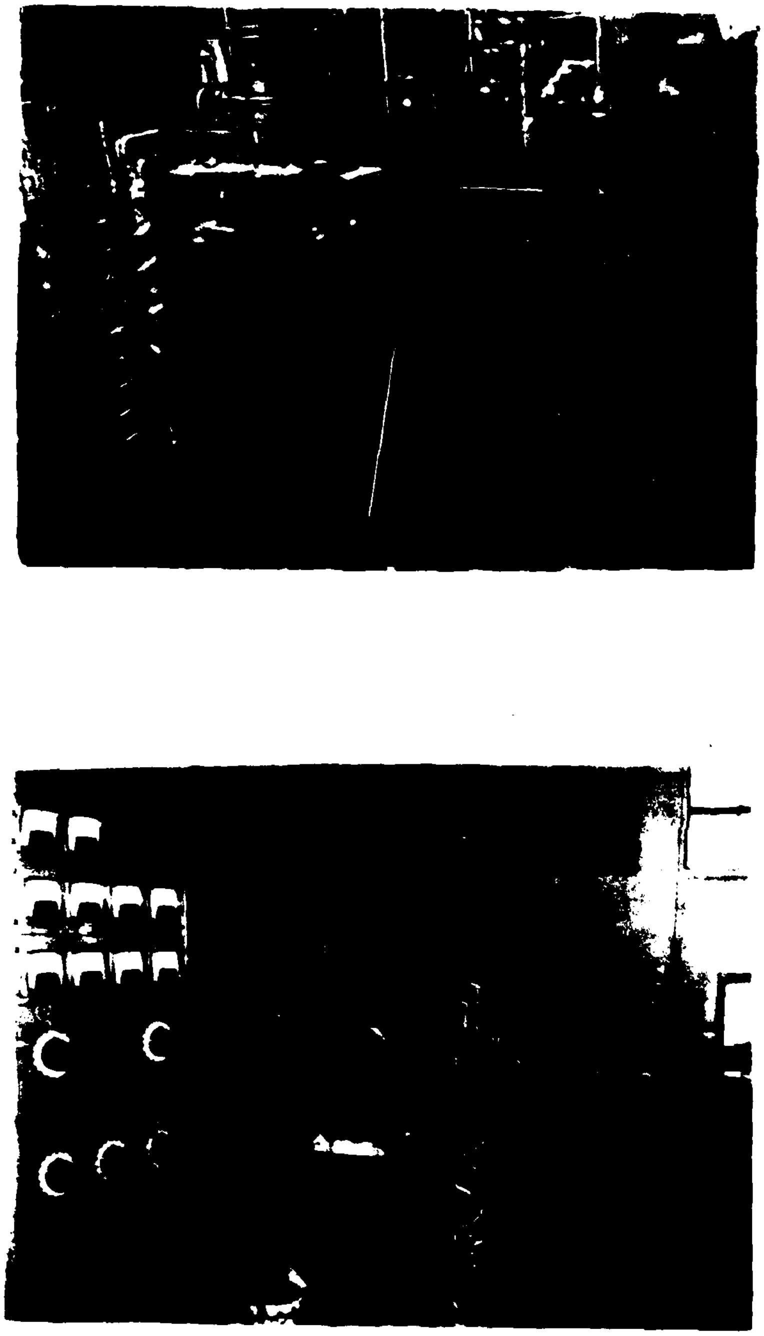


Figure 2. Two views of the Molecular Beam Epitaxial Growth System.

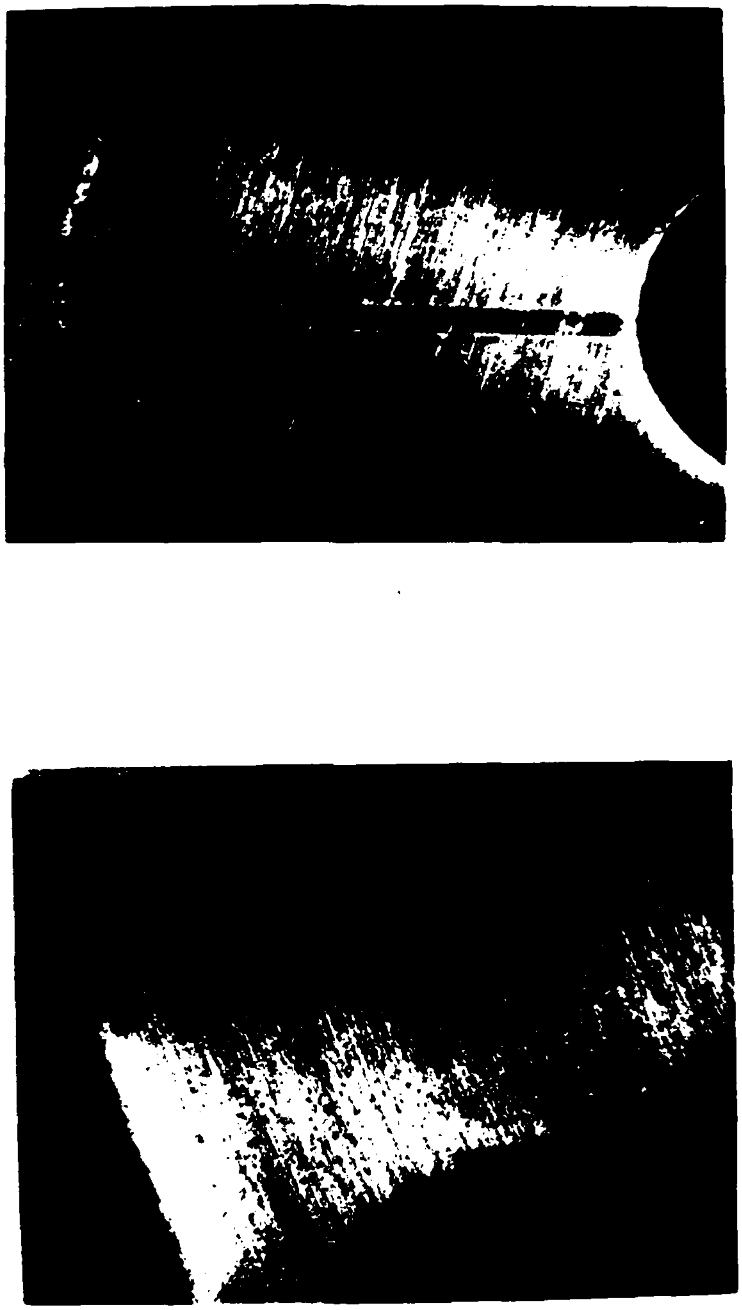


Figure 3. Photographs of defects in the sealing section of an ultra-high vacuum valve.

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