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STUDIES OF SURFACE PHASE TRANSFORMATIONS BY LOW ENERGY ELECTRON DIFFRACTION

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ABSTRACT

The structure of low index surfaces of semiconductor and metal single crystals are often characterized by unit cells which are different from the bulk unit cell. Some of these ordered surface structures are stable up to the melting point of the solid, others undergo transitions in well-defined temperature ranges to form different ordered surface structures. The mechanisms of these surface phase transformations and theoretical models which predict many of their properties are discussed.

The mean square displacement of surface atoms in metal surfaces is markedly larger than the mean square displacement of bulk atoms. There are theories of melting which would predict the onset of disorder at the surface below the bulk

melting point. Low energy electron diffraction studies of surface melting of lead, bismuth and tin surfaces, however, indicate that the crystal faces remain ordered up to the melting point. These results and their implications concerning the understanding of the kinetics of melting are discussed.

Low energy electron diffraction studies of vaporizing metal surfaces indicate that the crystal faces remain ordered on an atomic scale during sublimation.

1. INTRODUCTION

When detailed studies of the electrical properties of the bulk solid had been initiated in the nineteen thirties, the atomic structure of the solids had already been established. X-ray diffraction studies have revealed the crystal structures of a large number of monatomic and diatomic solids and therefore the developing electronic band theory has already been utilizing the crystalographic data available at this time. In early studies of the electrical properties of surfaces during the nineteen fifties, the structure of the solid surface has been an unknown parameter. Until the advent of broad application of low energy electron diffraction, (LEED), the atomic structure at the surface has not been investigated experimentally. In the absence of such investigations, it has long been assumed that the surface structure is the same as one would expect by extrapolating the bulk unit cell to the surface.

In the last few years, there has been an exciting and revolutionary change taking place in our understanding of the structure and many other physical-chemical properties of surfaces. The number of experimental techniques which are available, to study well-defined surfaces in a definitive manner, has multiplied. First of all, we can study one face of a single crystal cleaved or cleaned by ion bombardment in a ultra high vacuum. Ultra high vacuum can be maintained during experimental times which are sufficiently long so that reproducible results as to the properties of clean surfaces could be obtained.[1] In addition to low energy electron diffraction, which is today the most important experimental tool in investigations of the structure of surfaces, we can use the inelastically scattered portion of the electrons to carry out electron spectroscopy (Auger spectroscopy) studies to reveal the chemical composition of the surface.[2] We can use ellipsometry to study the thermodynamic properties and the build-up of absorbed atoms on the surface.[3] Finally atomic beam scattering studies give us information about the energy transfer between the incident gas molecules and the surface and can be used to study the lattice vibration spectrum of surface atoms.

In this paper we shall be discussing the structure and phase transformations which take place at clean single crystal surfaces, in the absence of adsorbed gases, in ultra high vacuum. First, we shall discuss the order-order transformations which manifest themselves in changes of the surface structure as studied by low energy electron diffraction. Then, our melting

studies will be discussed where we have been investigating the possibility of surface pre-melting of metals such as lead, bismuth and tin. And finally, we shall describe our results of the investigation of the surface structure during sublimation of metal single crystal surfaces.

2. ORDER-ORDER PHASE TRANSFORMATIONS OF SEMICONDUCTOR SURFACES

Creation of a surface lowers the symmetry about the atoms at the interface, reduces their number of nearest neighbors, and places them in an anisotropic environment with respect to that for bulk atoms. These marked changes can give rise to electronic effects at the interface such as electronic surface states and space charge[4] or lattice vibration modes [which are often called surface phonons] which are restricted to propagate along the surface.[5] The creation of a surface not only changes the electronic properties and the atomic motion but also the atomic structure at the interface. There is no reason to believe that the surface atoms remain in the same position that they would occupy in the bulk or to maintain the same structure they would have in the bulk environment. Low energy electron diffraction studies reveal that all semiconductor surfaces which have been studied so far have surface structures which are different from that expected from their bulk unit cell. Table I summarizes the surface structures which have been found on semiconductor low index crystal faces which have been investigated.[6,7,8] These changes in surface structure are detected by changes in the diffraction pattern which are obtained as a function of

temperature. Figure 1 gives as example of such a change. The Si(111) - (1 x 1), surface structure is stable at room temperature up to about 600°C where it converts to (7 x 7) surface structure. The appearance of new diffraction features indicate the appearance of a new surface periodicity which is integral multiple of the periodicity which is characteristic of the bulk unit cell. Several similar ordered surface structures which appear as a function of temperature have been found on other semiconductor single crystal surfaces which have been investigated such as silicon, germanium, gallium arsenide, and cadmium sulfide.

It is somewhat difficult, due to the lack of adequate, detailed experimental data, to propose a mechanism which accounts for the appearance of these surface structures. There are certain features shared by these structures which allow us to classify them in some manner. First of all, we find that on all semiconductor surfaces, the surface structures have a well defined temperature range of stability. Below and above these temperature ranges, the surface structure may not be stable but it is converted to another ordered surface structures. We also find that on surfaces which show 6-fold rotational (hexagonal) symmetry at least one of the surface structures gives rise to a (2 x 2) diffraction pattern, which indicates a surface unit mesh which is twice as large as that of the bulk unit cell. This is clearly seen from Table I. It has also been found that these surface structures and their temperature ranges of stability is very sensitive to the presence of impurities which may be present on the surface. Impurities can change the temperature

range of stability of the surface structures, can eliminate them, or can catalyze the formation of other surface structures. In spite of the sensitivity of the surface structures to impurities however, there is little doubt that these surface structures are the property of the clean semiconductor surface. There have been several studies carried out to ascertain that these surface structures are not caused by impurities but they are properties of the clean semiconductor substrate. For example, Haneman et al.[9] has cleaved a (111) face of silicon at 700°C in ultra high vacuum and the cleaved surface immediately showed a surface structure. Jona[10] has depositied amorphous silicon at room temperature on the silicon (111) surface. Upon heating, the disordered silicon atoms had rearranged on the surface and assumed a (7 x 7) surface structure. The physical model which is presently accepted to explain the appearance of surface structures assumes that the surface is buckled by periodic displacement of surface atoms out of plane perpendicular to the surface to increase the overlap among localized electronic orbitals.[11] Thus the surface is not flat, atoms are periodically displaced normal to the surface plane by a few per cent of the interatomic distance to give rise to a new surface periodicity which is detected by the appearance of new diffraction features in low energy electron diffraction experiments.

3. THE ORDER-ORDER PHASE TRANSFORMATIONS ON METAL SURFACES

Surface structures with unit mesh which are integral multiples of that of the bulk unit cell are discovered on several but not all metal single crystal surfaces.[11] For example, Figure 2 shows the low energy electron diffraction pattern which can be obtained from the (100) face of platinum. It is seen that in addition to the diffraction spots which one would obtain if the surface would have the same structure as that characteristic of the bulk unit cell, there are other diffraction features. The appearance of four extra spots, that are the one-fifth order diffraction beams, indicate the appearance of a new (5 x 1) surface periodicity.[13] The (5 x 1) diffraction pattern indicates the presence of ordered domains on the surface in which there is a 5x periodicity in the X direction and the same periodicity as that in the bulk unit cell in the y direction and domains which are perpendicular, that is, have 5x periodicity in the y direction and the same periodicity as the bulk unit cell in the X direction. Such a diffraction pattern can be generated if we assume that the surface atoms in the (100) face, which have a square unit mesh (Figure 3) are distorted into a close packed hexagonal structure as shown in Fig. 3. The (5 x 1) diffraction pattern is generated by the coincidence of every sixth atom in the hexagonal over-layer with every fifth atom in the (100) substrate. Similar (5 x 1) type surface structure is observed on the (100) face of gold single crystals.[14] No surface structures or rearrangements have been found on the (111) faces of platinum or gold. One also finds in the case of metal surface structures

that, just like for surface structures on semiconductor surfaces, impurities of certain types stabilize these surface structures while others eliminate it. Sodium and other alkali metals for example on the platinum or gold surfaces seem to stabilize the (5 x 1) surface structure. On the other hand, carbon and adsorbed gases such as carbon monoxide and hydrocarbons, seem to eliminate the (5 x 1) surface structure and stabilize the (1 x 1) structure which is characteristic of the bulk unit cell.[13] Unlike semiconductor surfaces however, the surface structures which appear on metal surfaces seem to be stable in the whole temperature range of metal stability --from room temperature up to the melting point. Surface structures have been found, in addition to platinum and gold, on antimony,[15] bismuth[15] and palladium[16] surfaces as well. The observed surface structures which have been reported so far are listed in Table II. If appears that again, a slight periodic distortion of atoms out of the surface plane normal to the surface gives rise to the new periodicities which are observed by low energy electron diffraction. The extreme sensitivity of these surface structures to the adsorption of minute carbon monoxide seem to indicate that the new surface layer which appeared to form by periodic distortion is easily transformed by changing the surface environment. The cause of the formation of surface structures on metal surfaces, however, should be different from that which causes them to appear on semiconductor surfaces. It is likely that the observed surface structures which are different from those which are expected from the bulk unit cell are the result of surface phase transformations. The

explanation as to the properties of the surface structures could follow the same lines which have been applied to explain the formation of certain bulk metal crystal structures. The Hume-Rothery rules[17] which have been modified by Engel and Brewer[18] indicate that the number of unpaired s or p valence electrons per atom, which are available for bonding, determine the bulk crystal structure. For example, metals with 1-1.5 unpaired valence electron per atom crystalize in the body centered cubic structure. Examples of that are the alkali metals. Metals with 1.7-2.1 unpaired valence electrons per atom crystalize in the close packed hexagonal structure. Atoms with 2.5-3 unpaired electrons per atom are found in the face centered cubic crystal structure while atoms with four unpaired valence electrons per atom implies the diamond structure. In gold and platinum surfaces therefore, if as postulated, the number of bonding electrons per atom in less than in the bulk, then a transition from a fcc to a hcp structure at the surface would be possible. The stabilizing effect of alkali metals on the hexagonal (5 x 1) structure and the poisoning effect of carbon seems to indicate the correctness of this model.[18] Surface structures other then those corresponding to the bulk unit cell do not appear on nickel, copper and aluminum surfaces. Also, low index surfaces of body centered cubic metals which have been studied so for (W, Nb, Mo) do not appear to undergo surface phase transformations in the absence of impurities at the interface. More studies on a wider range of clean metals and crystal faces should be performed so that the physical parameters which govern surface phase transformations could be uncovered.

This semi-empirical model which has been successfully applied to explain the structure of bulk metals points to intriguing possibilities in forming surface phases by suitable and judicious addition of impurities to certain crystal faces.

4. THE MELTING OF BISMUTH LEAD, AND TIN SINGLE CRYSTAL SURFACES

A. The Mean Displacement of Surface Atoms in Metal Surfaces

Surface Debye-Waller measurements using the diffracted low energy electron beams have revealed that the mean square displacement of surface atoms in metal surfaces is much larger than that in the bulk.[19] In these measurements the intensity of a given diffraction spot is monitored as a function of temperature at a given beam voltage. The intensities attenuated due to the vibration of surface atoms which scatter a given fraction of the diffracted electrons out of the diffraction spot into the background. It is found experimentally and it is also predicted by theory that the intensity of a given diffraction spot falls exponentially as a function of temperature, with increasing temperature. The intensity of scattered electrons in the specular, (00) beam (neglecting multiple scattering events) is given by

$$I = |F_{hkl}|^2 \exp[-(16\pi^2 \cos^2\phi/\lambda^2)\langle u_\perp^2\rangle], \qquad (1)$$

where the exponential term is the Debye-Waller factor, λ is the electron wavelength, ϕ is the angle of incidence with respect to the surface normal, and $|F_{hkl}|^2$ is the scattered intensity by a rigid lattice. Using the Debye model of lattice vibrations

in the high-temperature limit the mean-square displacement is given by[20]

$$\langle u^2 \rangle = (3N\hbar^2/Mk)\,[T/(\theta^\infty)^2], \qquad (2)$$

where θ^∞ is the Debye temperature at the high-temperature limit, M and T are the atomic weight and the temperature of the solid, respectively, N is Avogadro's number, and k and h are the Boltzmann and Planck constants. Combining Eqs. (1) and (2) we have

$$I_{hkl} = |\,F_{hkl}\,|^2 \exp\left\{-(12N\hbar^2/Mk)(\cos\phi/\lambda)^2[T/(\theta^\infty)^2]\right\} \qquad (3)$$

The logarithm of the intensity plotted as a function of temperature T gives a straight line. From the slope the root-mean-square displacement in the direction perpendicular to the surface plane, $\langle u_\perp \rangle$, can be calculated. Figure 4 shows a typical intensity-temperature plot which is obtained for the (100) face of palladium single crystals.[21] The measured values of the root-mean-square displacements are strongly dependent on the electron-beam energy. It is apparent that with increasing electron energy a larger fraction of electrons scatter from atomic planes which lie below the surface plane. Thus, at higher electron energies the experimentally determined mean displacement approaches the bulk mean displacement value. The mean displacement which is characteristic of the surface atoms can be obtained from data taken using a very low-energy electron beam ($\leq$ 15 eV). At such low energies the largest fraction of the impinging electrons back-scatter from the surface atoms without penetrating deeper

into the lattice. The effective root-mean-square displacement, $\langle u_\perp^{eff} \rangle$ calculated from the $\log I_{oo}$-vs-T curves using Eq. (1) is plotted as a function of beam voltage for the different lead and bismuth surfaces (Figures 5 and 6). It is apparent that the surface root-mean-square displacement is much greater than that of the bulk value (dotted line) for both orientations. The root-mean-square displacements which were obtained at the lowest electron energies were taken as values characteristic of the mean vibrational amplitude of surface atoms. These along with the corresponding bulk mean displacements, the calculated surface, and bulk Debye temperature are listed in Table III. For comparison, we have included the values obtained for other metal surfaces as well.

B. Surface Melting Experiments

It has been proposed by several investigators[22,23] that melting is actually related to the mean square displacement of the solid atoms. Melting in these theories is considered to be a process in which the atoms, when they reach a critical mean square displacement, which is a certain fraction of the interatomic distance, are no longer able to maintain their equilibrium position and the lattice collapses. Using such a model a good correlation between the Debye temperature and the melting temperature has been obtained for large numbers of simple monatomic solids. This model, would predict however, that the surface should disorder or melt at a temperature which is lower

than that of the bulk melting point since the mean square displacement of surface atoms is markedly larger than their value in the bulk at the melting point. For this reason we have set out to study the structure of surfaces as the melting temperature is approached, at the melting point, and above the melting point as well. In these studies we have used lead, bismuth and tin single crystals because these crystals have low vapor pressure at the melting point, an important condition in low energy electron diffraction studies where ultra high vacuum is necessary. The experimental equipment used in these studies is schematically shown in Fig. 7. The diffraction chamber was rotated by 90° to enable us to support the molten surface and the diffraction pattern was viewed through a mirror as shown in the figure. The temperature in these studies could be controlled within half a degree.

We could melt a solid at the top first and then monitor the melting interface as it proceeds through the crystal or we could melt the crystal so that the melting interface moves along the surface and we could watch the diffraction pattern simultaneously. We have found that the surface diffraction features which are characteristic of ordered surface structures have disappeared <u>at the bulk melting point</u> within the temperature uncertainty of the experiment. The intensity due to scattered electrons from the liquid surfaces was fairly uniform but showed small fluctuations as a function of electron beam energy. These intensity changes were mapped out and were related to the atomic

scattering factor and the radial distribution function which is characteristic of surface atoms in the melt.[21] Under no conditions have we found indications of surface melting at temperatures below the bulk melting temperature. These studies were performed for three different crystal faces of lead, two different crystal faces of bismuth; thus we had ample opportunity to detect surface melting if it occurred along one crystal face even though it may not have occurred along another one. Our results indicate that the melting theory proposed by Lindeman[23] and others[22,24] certainly does not apply to the melting of face centered cubic metal single crystals. However, our studies and studies by Turnbull[25] and others[26] clearly indicate that melting has to be nucleated and nucleation takes place at the surface. Turnbull has found that solid SiO_2 and P_2O_5 crystals can be superheated by as much as 150 to 300°C while the molten interface was slowly moving due to the high viscosity of the melt. Also, it was found that in gallium,[27] superheating of the order of 1° could be obtained. In ice[28] similar superheating temperatures have been found. Up to 3° of superheating could be maintained in the bulk while the surface was kept below the melting temperature. It appears that as long as the surface is maintained at a temperature below that of the bulk melting point heating concentrated in the bulk does not cause instantaneous melting as soon as the melting temperature is obtained. Such a result can only be interpreted as indicating nucleation of melting as a necessary condition for the melting process. Since in most melting experiments there is adequate surface area and

thus nucleation may easily occur, superheating is not a common experimental occurence. The nature of the surface nucleation centers cannot be revealed by low energy electron diffraction experiments. These experiments are only sensitive to surface order. That is, we detect domains of ordered atoms, their structure and other properties, but a fraction of disordered atoms which may also be present at the surface simultaneously will not show up in low energy electron diffraction experiments. It is apparent however that the surface plays a very important role in determining the kinetics of melting, even though melting will occur only if the temperature is high enough so that the molten face becomes thermodynamically stable.

5. THE STRUCTURE OF VAPORIZING SURFACES

In the previous melting studies we have investigated the surface structure of materials which have low vapor pressure at the melting point. In these studies the surfaces of lead, bismuth and tin proved to be stable and ordered up to their respective melting points. In our vaporization experiments we have investigated the structure of solids which have large vapor pressure below their melting point. These solids will sublime at an appreciable rate before melting occurs. We have monitored the diffraction pattern due to the surface structure of silver and nickel at temperatures from 575 to 915°C respectively, where the vaporization rates are appreciable and the surface atoms are continually removed. The low energy electron diffraction studies revealed that the vaporizing surface is ordered on an atomic

scale. The diffraction spots were sharp and small indicating the presence of ordered domains of size as large as 500Å in diameter or larger. Scanning electron microscope pictures, which have been made on the same vaporizing crystal faces of silver and nickel, have revealed a rough surface on a 10,000Å scale. These are shown in Fig. 8. It appears that the surface may become extremely rough under conditions of vacuum vaporization on a scale of 10,000Å. On an atomic scale however, the surface remains ordered as deduced from the spot size and the intensity of the diffraction spots. Since the evaporation rate of single crystal metal surfaces in the vacuum is near or equal to that of the maximum rate which may be calculated from equilibrium considerations,[29] it has been proposed that all atoms have equal probabilities to vaporize. This can only happen if the surface takes up a liquid like character, since in a solid surface there are many atomic positions in which the surface atoms have different binding energies due to differences in the number of their nearest neighbors. Our low energy electron diffraction studies have revealed that the surface is far from being liquid-like during vaporization, it is ordered and heterogeneous. It appears that the number of surface atoms of lowest binding energy which can vaporize from the surface is increasing by roughening of the vaporizing surface until their concentration attain that of the concentration of the total number of surface atoms in a homogeneous flat surface. For this reason we find that the evaporation rate is at the maximum under conditions of vacuum evaporation from monatomic single crystal surfaces.

ACKNOWLEDGE

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TABLE I

Material	Surface Structures
Si	(100) - (4 x 4), (111) - (7 x 7)
Ge	(100) - (4 x 4), (111) - (8 x 8), (110) - (2 x 2)
GaAs	(111) - (2 x 2)
GaSb	(111) - (2 x 2)
InSb	(100) - (2 x 2), (111) - (2 x 2)
CdS	(0001) - (2 x 2)

TABLE II

Surface Structures	Temperature range of established stability
Pt(100) - (5 x 1)	25° - 1300°C
Au(100) - (5 x 1)	25° - 500°C
Au(110) - (2 x 1)	---
Pd(100) - (2 x 2)	250° - 550°C
Pd(100) - C(2 x 2)	550° - 850°C
Bi(11$\bar{2}$0) - (2 x 10)	25° - melting point
Sb(11$\bar{2}$0 - (6 x 3)	25° - 250°C

TABLE III

	Pb (111)	Pb (110)	Bi (0001)	Bi (01$\bar{1}$2)	Pd (100)	Pd (111)	Pt (100)	Pt (110)	Pt (111)	Ag (111)	Ag (110)	Ag (100)
$\langle u_\perp \rangle$ (surface) (Å)	0.298	(.395)	0.302		0.144		0.135			0.129		
$\langle u \rangle$ bulk (Å)	0.160		0.10		0.074		0.064			0.089		
θ_D surface (°K)	49	(37)	48		140±10		110±10			152	(104)	
θ_D bulk (°K)	90.3		118		273.4		234			225		

TABLE CAPTIONS

Table I. Surface Structures found on clean semiconductor surfaces.

Table II. Surface structures found on clean metal surfaces.

Table III. The Surface and bulk root-mean-square displacements and Debye temperatures of several metals.

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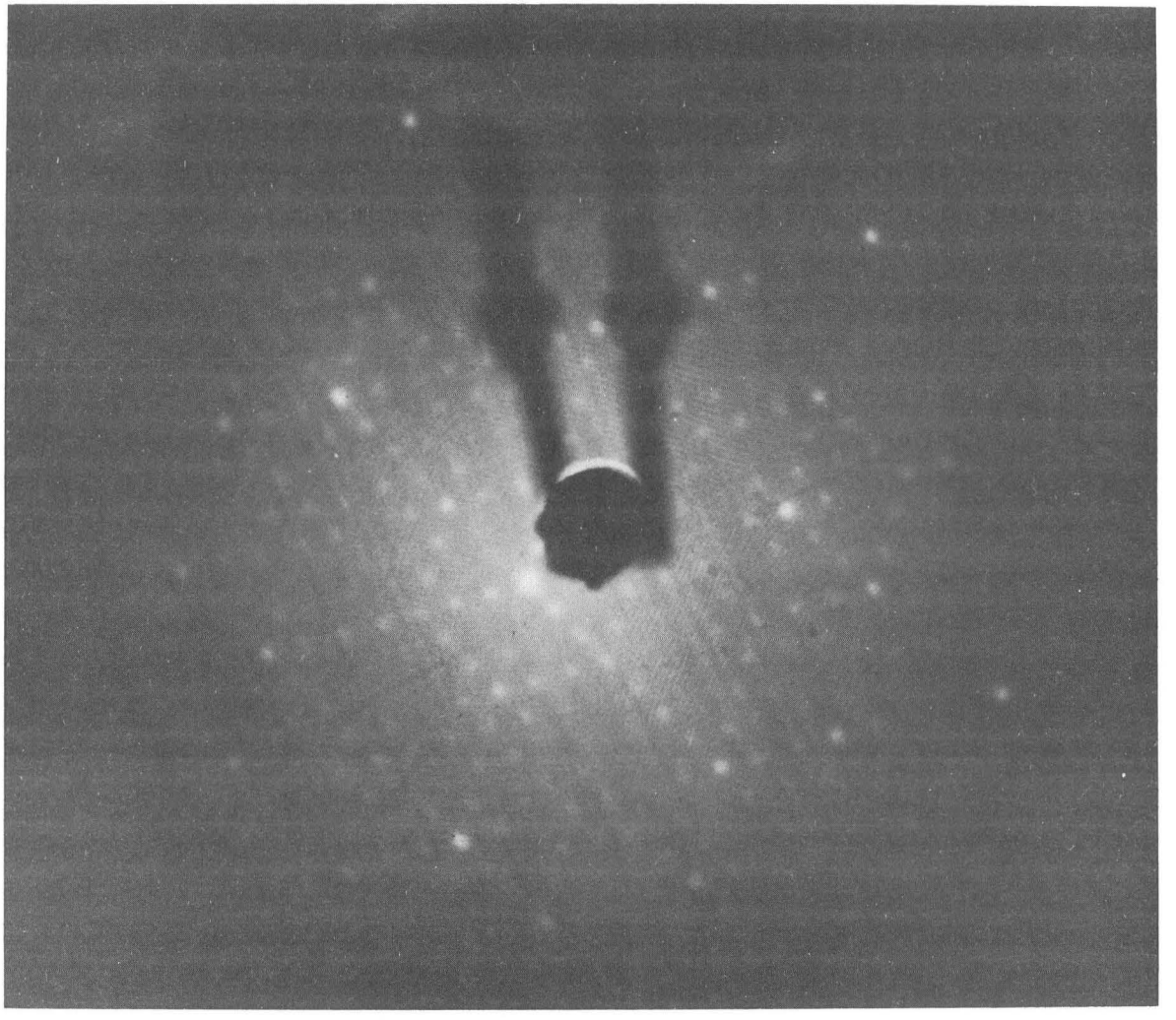
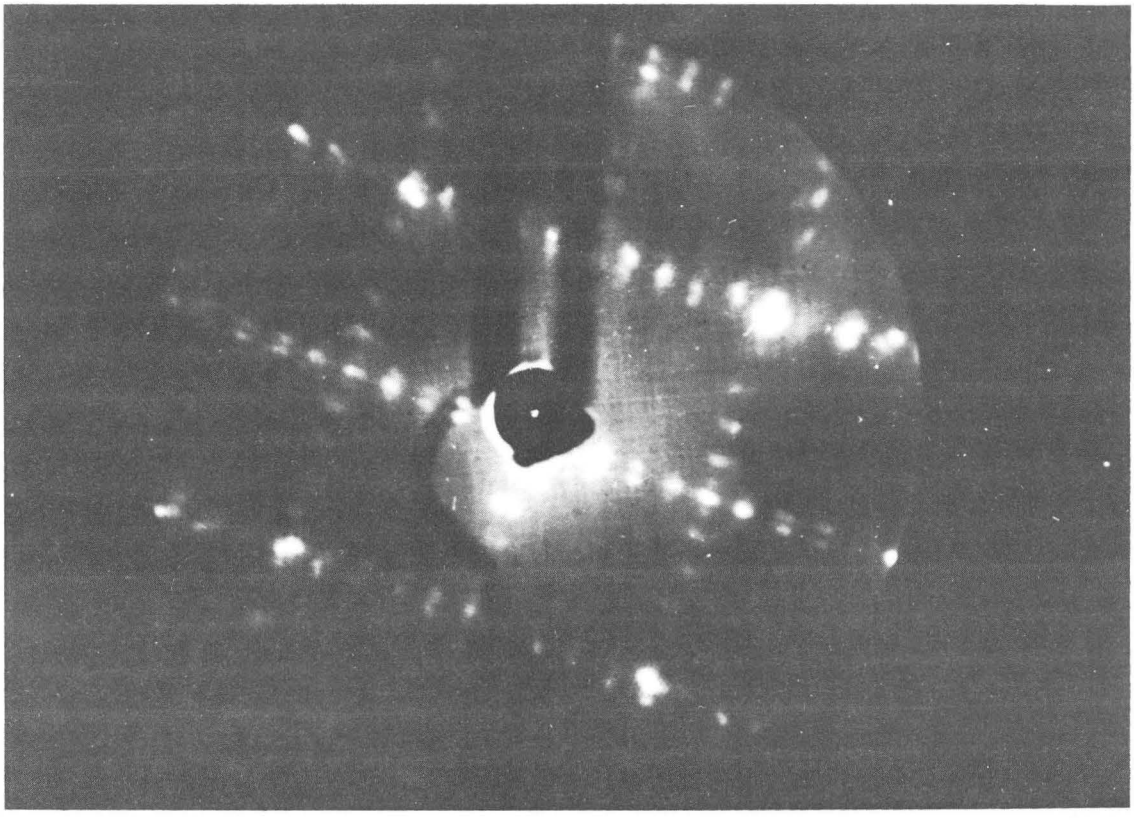


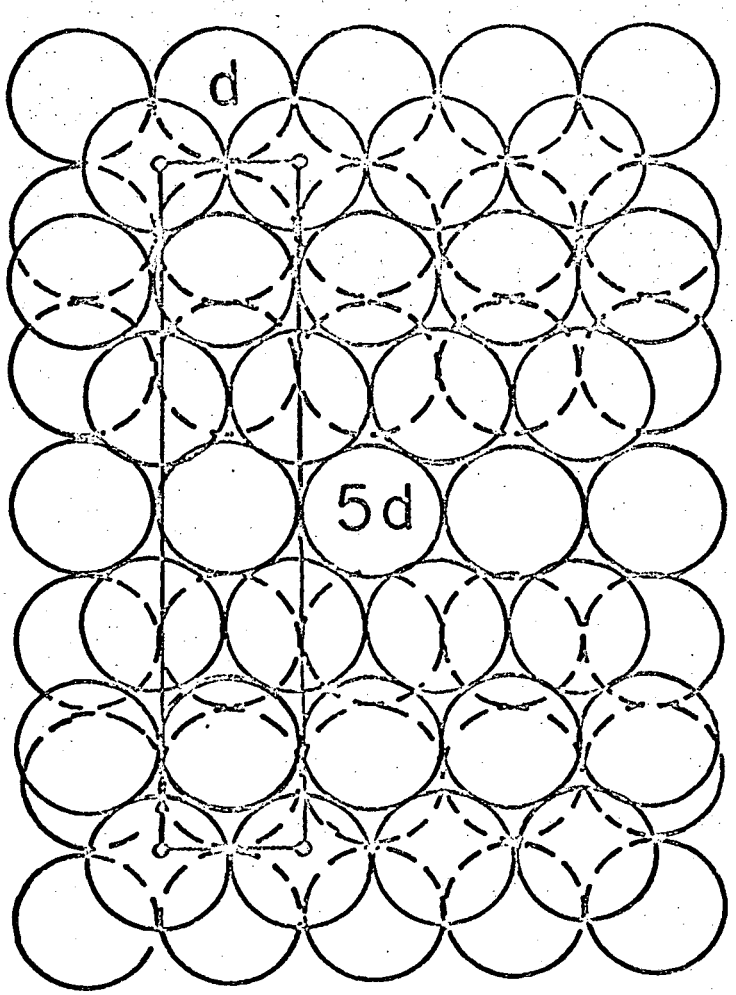
XBB 6910-6721

Figure 1a, b. Diffraction patterns of the a) Si (111) - (1 x 1) and b) Si (111) - (7 x 7) surface structures.



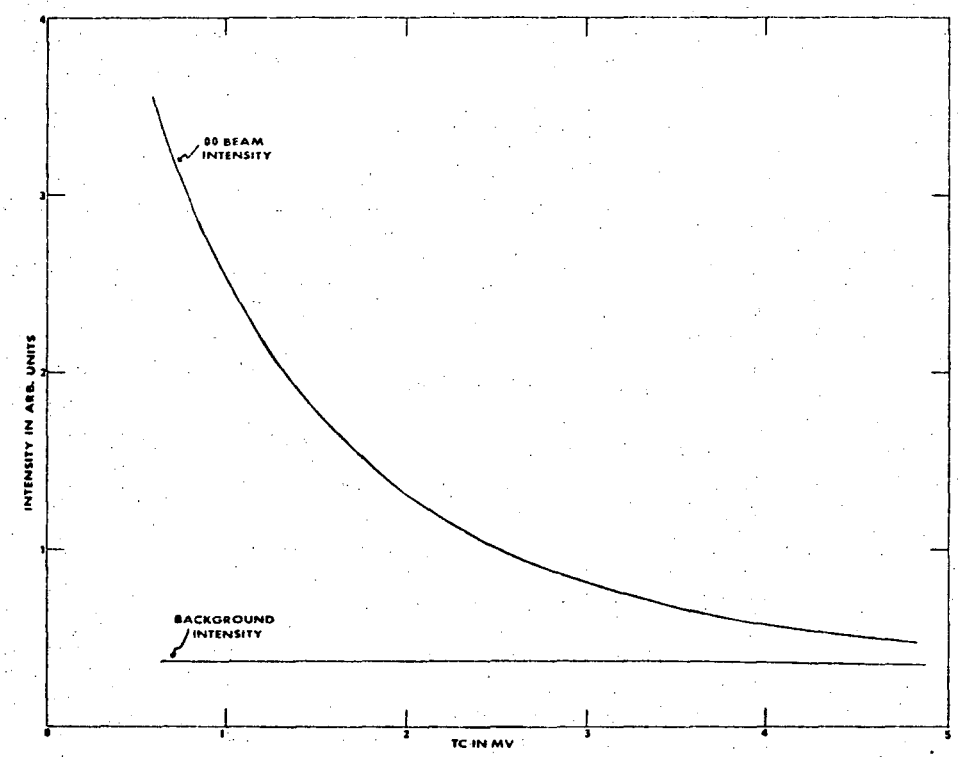
XBB 693-1973

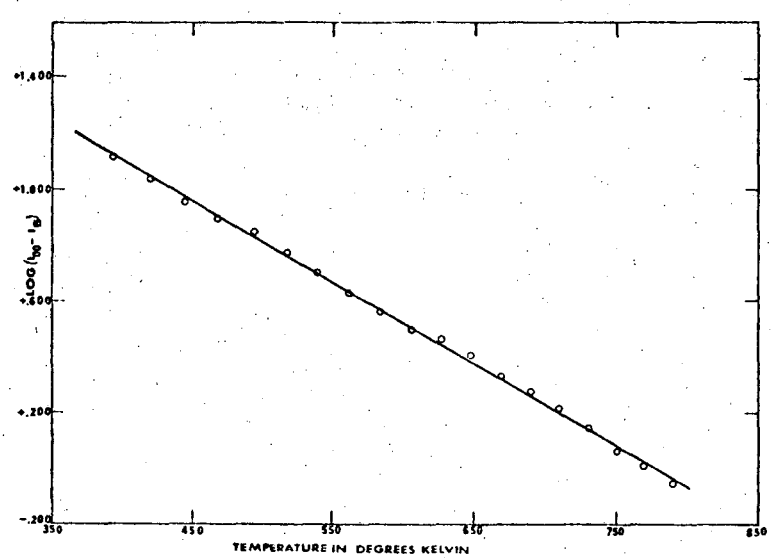
Figure 2. Diffraction pattern of the Pt (100) - (5 x 1) surface structure.



Reconstructed layer

Figure 3 - Schematic representation of the (100) surface of platinum with a hexagonal overlayer to simulate the hexagonal distortion which gives rise to the (5 xl) diffraction pattern.





XBL 696-642

Figure 4a. (Top) Temperature dependence of the intensity of the (00) reflection and background scattering from Pd(100) surface at 315 eV.

Figure 4b. (Bottom) Derived log $(I_{00}-I_B)$ vs T from Fig. V-1

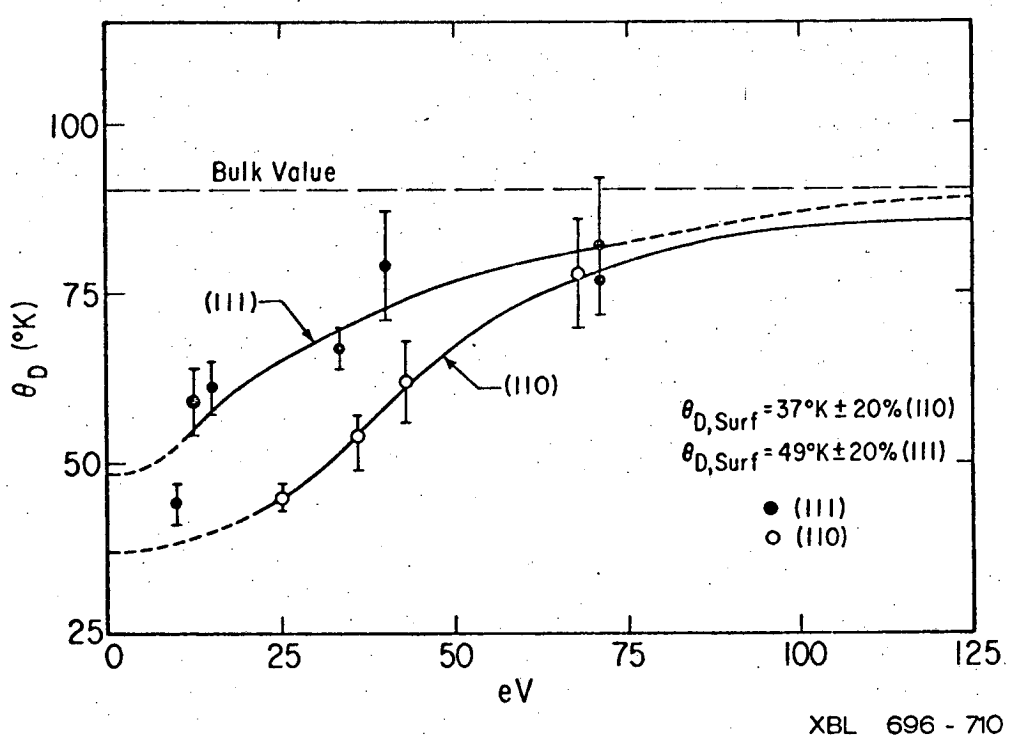


Figure 5 - $\theta_{D,EFF}$ versus eV for lead surfaces.

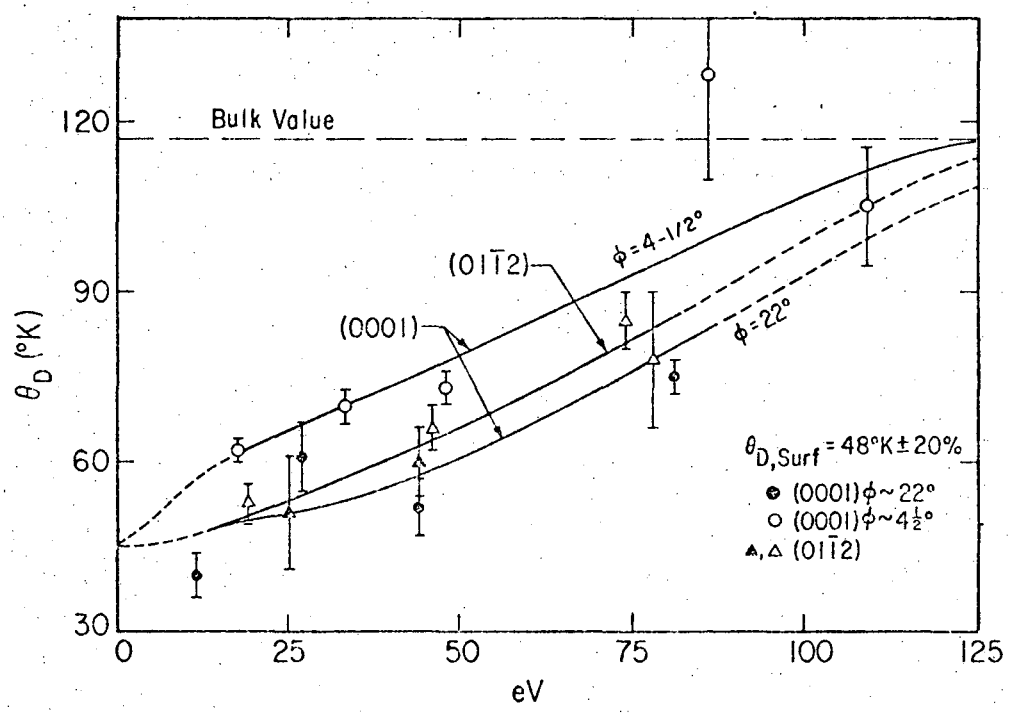


Figure 6 - $\theta_{D,EFF}$ versus eV for bismuth surfaces.

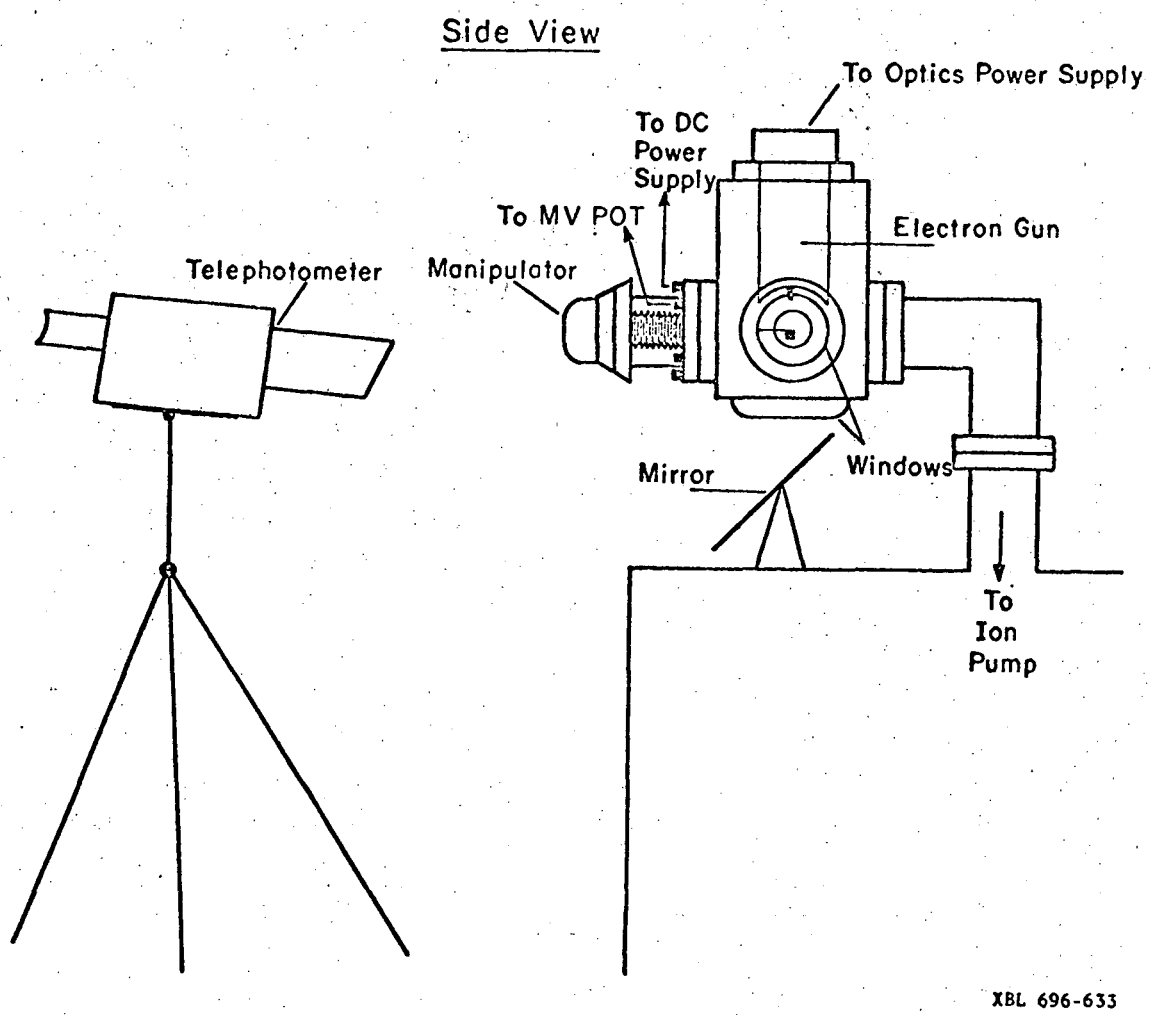


Figure 7 - Experimental arrangement for the melting experiments.

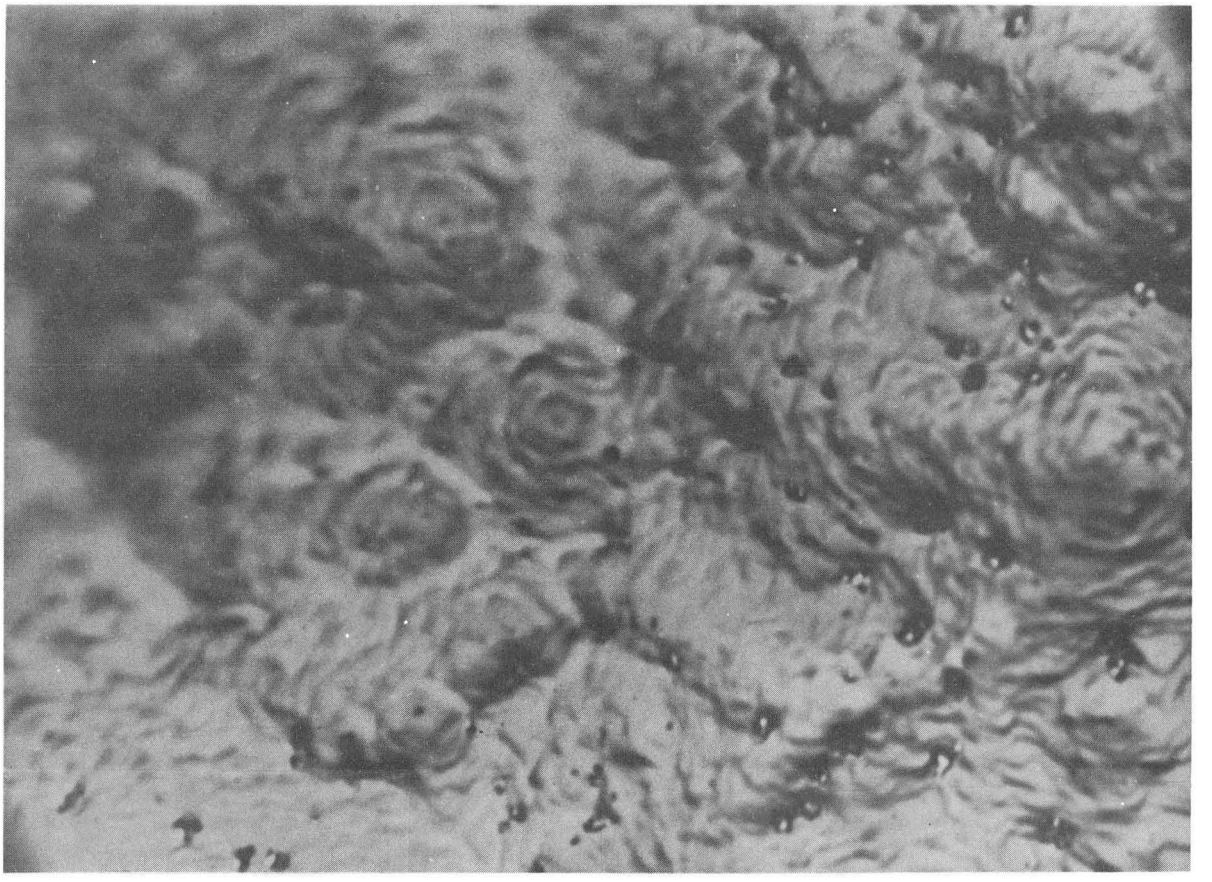


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Figure 8. Scanning electron microscope pictures of the vaporizing silver surface.

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