THE PREPARATION OF THIN FILMS OF InSb ON CRYSTALLINE SUBSTRATES

by

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INTRODUCTION

Studies of the properties of thin films of indium antimonide have been performed using polycrystalline films. (3),(6). The interpretation of experimental data from such polycrystalline films is complicated by effects which are due to structural nonuniformity. It would therefore be of value to be able to study monocrystalline films. The ultimate goal of the work described in this paper was the production of monocrystalline films of InSb suitable for further studies of the properties of thin films.

A promising and widely used method for preparing monocrystalline films is epitaxial growth. This method is based on the fact that films deposited from the vapor phase onto suitable crystalline substrates often are composed of large oriented crystallites.

The phenomenon of one crystal grown upon another with some definite and unique orientation relationship between their crystal axes was first observed in the form of natural growths among minerals. The term epitaxy denotes this phenomenon. Many naturally occurring cases of oriented intergrowth have been observed and comprehensive lists of the known examples have been compiled. (7).

Observation of these intergrowths led to attempts to obtain oriented intergrowths in laboratory experiments. The parallel growth of sodium nitrate from solution on the surface of a calcite crystal by Frankenheim in 1836, appears to have been the first successful attempt. The first systematic experiments were performed by Barker who studied the growth from solution of crystals with related structures, such as the alkali halides, upon each other. He found that some alkali halides oriented upon each other while others did not, and concluded that oriented growth is more likely to

occur if the molecular volumes of the two alkali halides are nearly equal.(2).

After the discovery and development of x-ray diffraction and the consequent tremendous increase in knowledge of crystal structure, Royer repeated and extended the work done by Barker. On the basis of his results he developed a theory of epitaxy. This theory centered on the necessity of a parallelism of the lattices accompanied by a small misfit value for the lattices. (7).

Electron diffraction was discovered about the time Royer was doing his work. The application of electron diffraction to the study of epitaxy greatly increased the possibilities for study, making it possible to study much thinner growths and deposits other than those grown from solution. With the application of electron diffraction to work on various types of deposits, it was found that epitaxy occurs in such varied deposits as chemically grown layers, electrodeposited metal films, and metal layers condensed from the vapor phase. Van der Merwe compiled a comprehensive summary of known cases of epitaxy and Pashley has compiled a detailed survey of those cases which have been studied using electron diffraction techniques.(7),(12).

The investigation of epitaxy of metals upon non-metals has been carried out almost exclusively by electron diffraction. Lassen's discovery of silver in parallel orientation when deposited from the vapor phase on a rocksalt cleavage face was the first known occurrence of this type.(7).

Lassen and Bruck reported the growth of good, single crystal, thin films of silver by deposition on heated rocksalt substrates. They reported the films grew in parallel orientation, which corresponds to a misfit value of -27%. Royer challenged this observation on the basis that other possible orientations would result in much smaller misfits and would therefore be the ones expected to occur. Lassen and Bruck, while agreeing one might expect

other orientations on the basis of misfit, confirmed that it was nevertheless the parallel orientation which occurred. (9).

Bruck and Rudiger, in their independent studies of metals deposited on rocksalt, fluor, and calcite, found that orientation of the films was markedly dependent on the substrate temperature during deposition. When the deposition was made onto substrates held at temperatures above a certain critical temperature (the epitaxial temperature) well oriented films were obtained. For depositions made on substrates at temperatures below the epitaxial temperature there was not complete orientation. The epitaxial temperature was found to be different for different metals.(2),(7).

Shirari carried out many experiments with metals on non-metals and found that in some cases where the substrates were preheated to several hundred degrees Centigrade and then cooled to some lower temperature for the deposition there was an improvement in the degree of orientation achieved. (7).

Oriented growths of metals upon mica, calcite, fluorspar, and mineral sulphides have been studied extensively. These are characterized, in general, by the occurrence of mixtures of orientations which are temperature dependent. The misfits found in these films are often very high.(7).

More recently, Kehoe examined the growth process of silver films by continuously monitoring the diffraction patterns from films being deposited in the diffraction camera. He found that good orientation occurred at temperatures appreciably below previously reported epitaxial temperatures. Kehoe, however, used much slower rates of deposition than had been used previously. (5).

Sloope and Tiller, working primarily with silver on rocksalt, were the first to perform systematic experiments to establish some of the important

factors for the production of good single crystal thin films by epitaxial growth. They found the most perfect films, structurally, were produced on substrates that were given a preliminary heat treatment to reduce the amount of absorbed gas and other foreign matter on the surface. Short periods of preheating were found to be as effective as extended periods. It was also noted that if the substrates were preheated to temperatures where thermal etching took place the resultant films were porous. In studying the relation between the substrate temperature and deposition rate they found that there is actually no epitaxial temperature, but that the required substrate temperature varies with the rate of deposition.(10).

Kalinkin, Aleskovskii, Sergeeva, and Straknow have obtained monocrystalline films of CdSe on surfaces of NaCl, KCl, and KBr. On the basis of their studies they concluded that the orientation of the films did not depend to any significant degree on the surface relief of the substrate and that in obtaining monocrystalline films the most important parameters were the substrate temperature during deposition and the rate of deposition. They also observed that a preliminary heating of the crystal substrate increased the ability of the substrate to cause orientation of the film.(4).

Extensive studies have been made of the occurrence of and conditions for epitaxy, but there still does not exist a good theory of epitaxy to provide guideposts for work with previously untried materials.

There have been many theoretical considerations of epitaxy but the majority of them were based on the concept of a good geometrical fit between the lattices of the substrate and the overgrowth and completely failed to explain the many large misfits and varied orientations that have been observed. It now seems to be commonly agreed that a greater knowledge of the process of nucleation and of surface forces must be obtained before any comprehensive theory of epitaxy can be evolved.

EQUIPMENT AND PROCEDURE

Plate I shows the vacuum chamber arrangement used throughout this investigation. All evaporations were made inside a 3 inch glass tee. The tee was sealed to a brass flange on the throat of the pumping system by means of a soft rubber gasket. The pumping system used to evacuate the chamber consisted of a chain of two oil diffusion pumps and a mechanical forepump and was capable of an ultimate vacuum of 10^{-6}mm Hg. The main pumping system could be sealed off just ahead of the chamber and the chamber itself roughed down by a second mechanical pump after it had been opened to the atmosphere. Thus the pumps were never turned off or exposed directly to the air. The gas pressure in the chamber was measured with a Consolidated Vacuum Corporation, Philips Gauge type PHG - 09.

Bulk InSb was evaporated from a molybdenum boat which was mounted on copper high current leads. These leads passed out of the chamber through a brass end-piece on the side arm of the tee. The power for the boat was supplied by a 3 kva, 4 to 1, stepdown transformer regulated by means of a 100 amp Transtat Voltage Regulator in the primary.

The brass end-pieces on the top and side arm were sealed to the glass by means of Teflon gaskets, the pressure being applied by bolts passing between opposing aluminum flanges. The top piece supported the substrate holder and had the heater and thermocouple leads passing through it. The substrate holder held both the substrate material and a glass optical flat approximately 11cm above the bottom of the boat. The substrate was sandwiched between the substrate heater in contact with the rear surface and a brass mask over the face. The optical flat also was covered by a brass mask having a rectangular slit so that the film deposited on it had two,

long, parallel edges. The thickness measurements were made on this film by an optical method employing interference by reflection of sodium light with a wavelength of 5890A. The fringe shifts were measured with a telescope equipped with a micrometer head.

The optical flat was not backed with a heater and therefore remained at a different temperature than was being used for the substrate during the deposition of the film. The actual thickness of the films deposited on the crystalline substrates may have been somewhat different from those measured on the optical flat due to re-evaporation from the heated substrate. The relative rate of deposition for the various films was of more interest than the actual thickness of the film in this study. This was calculated from the thickness measurement and length of time to evaporate the charge. The deposition rate, as measured, represents only an average rate since the actual deposition rate varied somewhat throughout the deposition due to factors such as the reduction of the size of the charge in evaporating and temperature changes of the crucible.

The substrate heater was a ceramic slab on one side of which chromel wire was threaded through thin parallel cuts and cemented into place with Eccoceram-CS ceramic bonding and sealing compound. The other side was lapped flat to provide thermal contact with the substrates. Power was supplied to the heater from a transformer regulated by a variac.

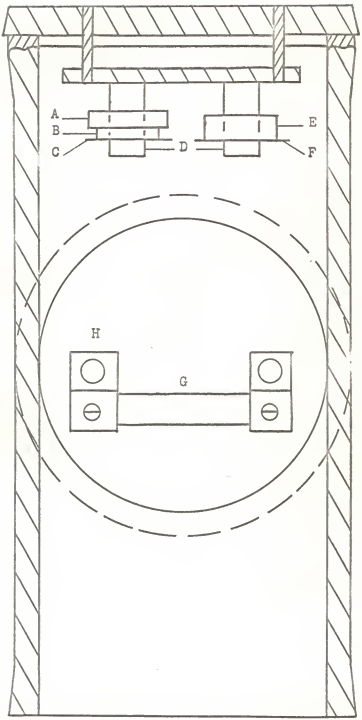
The substrates were freshly cleaved from bulk samples of CaF_2, KBr, LiF, and NaCl. The only exception to this was a thermally etched (111) surface of NaCl which had been exposed to the air for an extended length of time. CaF_2 was used because it cleaves easily along the (111) plane. The (111) plane was thought to be the most likely to produce epitaxy since InSb has the zinc blende structure and if it grew on a (111) face in parallel orientation

EXPLANATION OF PLATE I

Cutaway view of vacuum chamber arrangement.

A. Substrate heater
B. Substrate
C. Brass mask
D. Substrate holder
E. Optical flat
F. Brass mask
G. Molybdenum boat
H. Current leads

PLATE I



the crystal lattice would grow as alternate parallel layers of In and Sb. KBr was tried because it has a lattice constant nearly the same as that of InSb. NaCl was tried because most of the good single crystal thin films of various metals have been grown on rocksalt substrates. LiF was used because it was available in large pieces with good crystal structure.

After the substrate was cleaved, a copper-constantan thermocouple was attached to its surface with Eccobond 58C solder and the substrate was heated approximately 1 hour to dry the solder. The substrate was then immediately mounted in the vacuum system. Prior to evaporation the substrates were heated in vacuo to a temperature somewhat higher (50°C to 200°C) than the intended substrate temperature during deposition, and were held at that temperature about 2 hours or more. The substrate was then cooled in a few minutes to the desired substrate temperature and evaporation was begun. The pressure in the chamber never rose about 10^{-4} mm Hg during deposition of the films. The substrates were allowed to cool slowly to room temperature immediately upon completion of the deposition. The substrate temperature was determined from the thermocouple emf measured with a Rubicon potentiometer model No. 2700.

Small pieces of substrate with the overgrowth of InSb were carefully cleaved from the rest of the substrate for study by reflection electron diffraction. In one situation it became necessary to examine the film by transmission diffraction. In several cases pieces of the substrate not covered by the InSb were also cleaved off for examination by electron diffraction. The electron diffraction patterns were obtained in a RCA model EMU-2D electron microscope equipped with a reflection diffraction attachment.

After examination by electron diffraction, Pt-Pd shadowed carbon surface replicas were made of the film surfaces. The shadowing was done by evapor-

ating the Pt-Pd wire from approximately 15° with respect to the film surface. The carbon was then deposited at normal incidence. The replicas were removed in dilute nitric acid and mounted on grids for examination in the electron microscope.

RESULTS AND CONCLUSIONS

Fifteen usable InSb films were deposited on (111) cleavage faces of CaF_2. These were deposited with the substrates at various temperatures ranging from 25°C to 300°C and at various rates of deposition ranging from 200A/min to 1000A/min. When examined by electron diffraction all of these films gave good InSb diffraction patterns, but there were significant variations in the types of patterns and in the appearance of pure In and pure Sb lines in the patterns. The pertinent data for the films are compiled in Table 1.

Slower deposition rates were found to be more favorable for the formation of InSb films with larger crystallites. Films A-11 and A-3 were both deposited on substrates held at 200°C, but the deposition rate for film A-3 was double that of A-11. The diffraction pattern for film A-11 (Plate II, Fig. 1) shows spots and grainy rings whereas the diffraction pattern for film A-3 (Plate II, Fig. 2) shows only continuous rings. Films A-14 and A-15 were also deposited on substrates held at the same temperature (300°C), but the deposition rate for film A-15 was double that of film A-14. Here, as before, the diffraction pattern from the more slowly deposited film, film A-14 (Plate III, Fig. 1), contained well-defined spots while the pattern from the more rapidly deposited film, film A-15 (Plate III, Fig. 2), contained only continuous rings. The spotted patterns from the films deposited with the slower deposition rates show the crystallites in these films are somewhat larger than those in the corresponding films formed at faster deposition rates and having only continuous rings in their patterns.

There was a definite darkening of segments of the rings in the diffraction pattern from film A-4 (Plate IV, Fig. 1) deposited at a substrate

Table 1. Data for InSb films.

Film	Sub. Temp. °C	Dep. Rate A/min	Diffraction Pattern (d-spacings for lines present)
A-4	25	1000	InSb: 3.74,2.29,1.95,1.49,1.32; In: 1.68; Sb: 3.10,1.77 Unknown: 2.87
A-8	100	400	InSb: 3.74,2.29,1.95,1.62,1.49,1.145; In: 2.71; Sb: 3.10 Weak spots
A-9	125	300	InSb: 2.29,1.95,1.62,1.49,1.32; Sb: 3.10; Unknown: 3.46 Spots
A-7	150	200	InSb: 3.74,2.29,1.95,1.62,1.49,1.32,1.25,1.145,1.09,0.935, 0.907 Weak spots
A-5	150	300	InSb: 3.74,2.29,1.95,1.62,1.49,1.32,1.25,1.145,0.907; Sb: 3.10 Weak spots
A-6	150	800	InSb: 2.29,1.95,1.49,1.32,1.145; In: 2.72,1.68,1.47
A-1	150	800	InSb: 3.74,2.29,1.95,1.49,1.32; In: 2.72,1.68; Sb: 3.10,1.76 Weak spots
D-1	150	200	InSb: 2.29,1.95,1.145 Very faint pattern
E-1	150	700	InSb: 2.29,1.95,1.49,1.32,1.25,1.145
A-10	175	400	InSb: 2.29,1.95,1.62,1.49,1.32,1.25,1.145; In: 2.72
A-11	200	500	InSb: 2.29,1.95,1.62,1.49,1.32,1.25,1.145,1.02; In:272 Sb: 3.54 Spots
A-3	200	1000	InSb: 2.29,1.95,1.62,1.49,1.32,1.145,1.09,0.907,0.810
D-2	200	400	InSb: 2.29,1.95,1.49,1.32,1.145; In: 2.72,1.40; Sb: 1.76, 1.55,1.36
E-2	200	300	InSb: 2.29,1.95,1.49,1.32,1.25
A-16	225	300	InSb: 3.74,2.29,1.95 Transmission pattern
A-13	250	300	InSb: 3.74,2.29,1.95,1.62,1.49,1.32,1.25,1.145,1.09,1.02, 0.935,0.907,0.866
A-12	250	400	InSb: 1.95,1.62,1.49,1.32,1.25,1.10,0.935,0.907
A-14	300	200	InSb: 1.95,1.62,1.32,1.25,1.145,1.09,0.935,0.866; Sb: 310 Spots
A-15	300	400	InSb: 2.29,1.95,1. 2,1.49,1.32,1.25,1.145,1.09,1.024, 0.935,0.907,0.866
C-2	?	400	InSb: 2.29,1.95,1.145; In: 2.72,1.68; Sb: 1.55

* A denotes CaF_2 substrate; C, LiF substrate; D, KBr substrate; E, NaCl substrate

EXPLANATION OF PLATE II

Fig. 1. Electron diffraction pattern from film A-11.

Fig. 2. Electron diffraction pattern from film A-3.

PLATE II

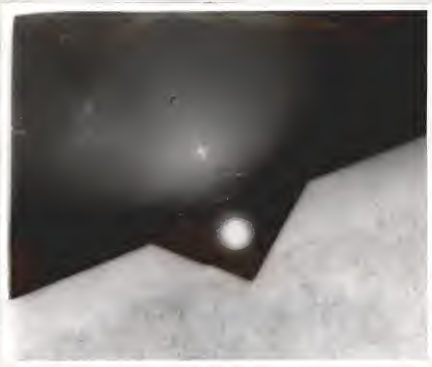


Fig. 1.

Fig. 2.

EXPLANATION OF PLATE III

Fig. 1. Electron diffraction pattern from film A-14.

Fig. 2. Electron diffraction pattern from film A-15.

PLATE III

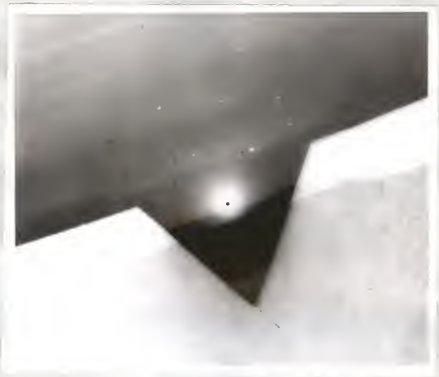


Fig. 1.

Fig. 2.

EXPLANATION OF PLATE IV

Fig. 1. Electron diffraction pattern from film A-4.

Fig. 2. Electron diffraction pattern from film A-14.

PLATE IV

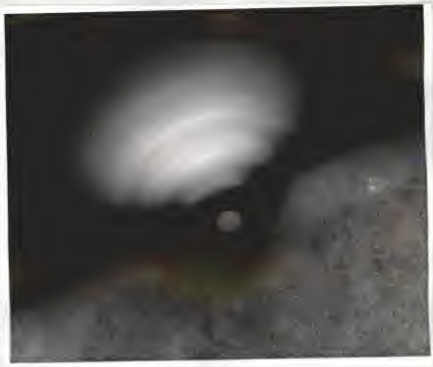


Fig. 1.

Fig. 2.

temperature of 25°C. This was interpreted as due to a slight tendency towards orientation of the crystallites composing the film. For materials producing monocrystalline films, a higher degree of orientation of the crystallites is found in films deposited at higher substrate temperatures. The only evidence for orientation of the crystallites in InSb films deposited at substrate temperatures above 25°C was from film A-14, deposited at 300°C. The major spot pattern of multiple spots in the pattern from one setting of film A-14 (Plate III, Fig. 1) suggests that several crystallites were in nearly the same orientation. This orientation was not characteristic of this entire film, however, as diffraction patterns from other areas (Plate IV, Fig. 2) did not show evidence of crystallite orientation.

In general, the best polycrystalline InSb films produced on CaF_2 substrates were produced on substrates held between 200°C and 300°C during deposition. This is evidenced by the good InSb diffraction patterns obtained from these films with notably fewer pure In and pure Sb lines occurring in the patterns. A very excellent InSb diffraction pattern was obtained from film A-13 (Plate V, Fig. 1) deposited with a substrate temperature of 250°C.

The peculiar background associated with the pattern from film A-13 was also found in the diffraction patterns of film A-8 (Plate V, Fig. 2). The background changed in orientation with different settings of the film with respect to the electron beam but remained of the same general nature for all orientations of the samples. No reference to this phenomenon has been found in the literature and nothing was observed which would possibly explain its existence in the patterns from these films.

All films deposited on CaF_2 substrates at temperatures above 200°C were extremely loose on the substrates when removed from the vacuum system. In some instances the whole film or a portion of it fell off the substrate

EXPLANATION OF PLATE V

Fig. 1. Electron diffraction pattern from film A-13.

Fig. 2. Electron diffraction pattern from film A-8.

PLATE V

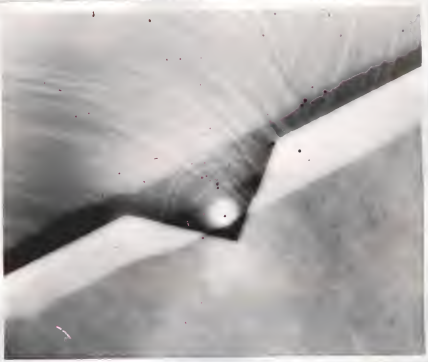


Fig. 1.

Fig. 2.

during subsequent handling. This looseness was attributed to the difference in the coefficients of expansion for the two materials.

In several cases good electron diffraction patterns were obtained from the substrate material of the samples before serious charging effects set in. These patterns, obtained from the substrate material of films A-7 (Plate VI, Fig. 1 and Fig. 2), A-14 (Plate VII, Fig. 1), A-16 (Plate VII, Fig. 2), and A-17 (Plate VIII, Fig. 1), show the substrate material had the very good crystal structure desired for epitaxy. This is evidenced by the good spot patterns and Kichuchi lines obtained from these samples. The symmetrical elongation of the spots in the patterns is characteristic of a lack of penetration of the electron beam into the surface with a corresponding relaxation of the Bragg condition.

For a given set of conditions, CaF_2 seemed to foster better films of InSb than the other substrate materials used. Diffraction patterns were obtained from InSb films deposited on (100) faces of KBr and NaCl at 150°C, on a (100) face of KBr and a (111) face of NaCl at 200°C, and from one film on a (100) face of LiF at unknown substrate temperature. Some of these patterns were very faint. No evidence of orientation or crystallite growth was found in these films. A much greater separation of components, giving rise to pure In and pure Sb lines in the diffraction patterns, was found to exist in the films on KBr and LiF than in comparable films on CaF_2. Good InSb patterns were obtained from the films on NaCl.

The surface micrographs (see Plate VIII, Fig. 2, and Plate IX, Fig. 1 and Fig. 2, for representative examples) were prepared with the idea that some information as to crystallite size, shape, or orientation might be obtained but such was not the case. A correlation between the appearance of the film surface and its diffraction pattern was sought, but there was no

EXPLANATION OF PLATE VI

Fig. 1. Electron diffraction pattern from the substrate of film A-7.

Fig. 2. Electron diffraction pattern from the substrate of film A-7.

PLATE VI

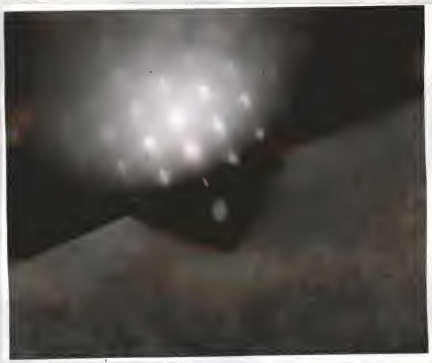


Fig. 1.

Fig. 2.

EXPLANATION OF PLATE VII

Fig. 1. Electron diffraction pattern from the substrate of film A-14.

Fig. 2. Electron diffraction pattern from the substrate of film A-16.

PLATE VII

Fig. 1.

Fig. 2.

EXPLANATION OF PLATE VIII

Fig. 1. Electron diffraction pattern from the substrate of film A-17.

Fig. 2. Electron miscroscope micrograph of a surface replica of film A-9.

PLATE VIII

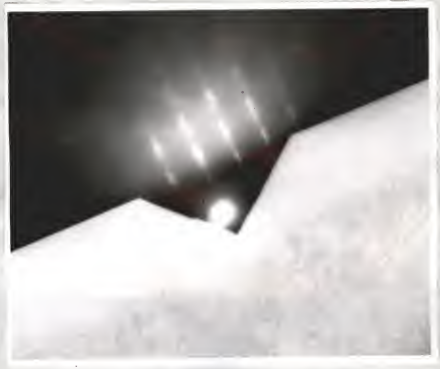


Fig. 1.

Fig. 2.

EXPLANATION OF PLATE IX

Fig. 1. Electron microscope micrograph of a surface replica of film A-4.

Fig. 2. Electron microscope micrograph of a surface replica of film A-2.

PLATE IX

Fig. 1.

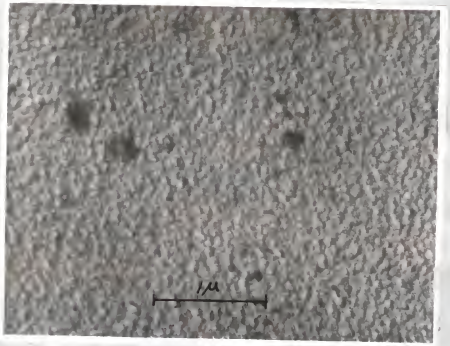


Fig. 2.

recognizable difference between the surface features of films giving diffraction spots on their patterns and those of films giving only rings. The micrographs did show that the films produced were, with one possible exception, continuous. Much of the value of the surface micrographs was lost because no way was found to remove the carbon replicas from the CaF_2 without serious damage to the replica and thus it could not be determined which structures observed on the film surfaces were due to substrate surface features and which were inherent with the films.

DISCUSSION

It was shown that slower deposition rates favor the growth of InSb films with larger crystallites. The slowest rate of deposition used in this investigation was 200A/min. Slower rates were not used because bulk InSb undergoes component separation upon evaporation and the more volatile Sb initially evaporates at a much faster rate than the In. Thus, the first layers on the substrate are almost pure Sb and the last layers almost pure In with varying combinations of the two components in between. The slower the evaporation rate the more pronounced this separation tends to become. This separation requires that some sort of diffusion mechanism must be operative after the components reach the substrate in order to produce good InSb films. The rebuilding of the InSb lattice by diffusion of the components is probably not very conducive to formation of extensive crystallites in the films. Thus it would be of great interest to attempt epitaxial growth of InSb by use of the three temperature technique whereby the components are evaporated from separate crucibles onto a substrate held at some given temperature. With this method, deposition rates in the order of a few Angstroms per minute could be worked with and good InSb layers would be developed at all stages of growth rather than by some process after deposition is completed. Thus, films with rather large oriented crystallites might be obtained.

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KANSAS STATE UNIVERSITY
Manhattan, Kansas

1962

ABSTRACT

In studies of the properties of thin films of InSb it has been found that the interpretation of the experimental data has been complicated by effects due to the polycrystalline nature of the films studied. The purpose of this work was to develop methods of preparing monocrystalline films of InSb suitable for further studies of the properties of thin films. The epitaxial growth of films by deposition from the vapor phase on crystalline substrates has proved highly successful for the production of monocrystalline films of many materials and thus was the method employed for this study.

The crystalline substrates used were primarily (111) cleavage faces of CaF_2 but several films were also produced on (100) cleavage faces of NaCl, KBr and LiF and on one (111) face of NaCl. These substrates were given a preliminary heat treatment in vacuum and were allowed to cool to the desired substrate temperature for the deposition of the film. The substrate temperatures ranged from 25°C to 300°C and the deposition rate ranged from 200A/min to 1000A/min for deposition of the various films. Small pieces of the substrate with the InSb film were cleaved from the rest of the substrate for examination of the films by reflection electron diffraction. After this examination a Pt-Pd shadowed carbon surface replica was made of the film for examination in the electron microscope.

Evidence was found that, other conditions being similar, slower rates of deposition favor growth of larger crystallites. A slight orientation of the crystallites was found in the film produced on CaF_2 at 25°C. The only evidence for orientation of crystallites in films produced on substrates at higher temperatures came from one area of a film deposited on a substrate held at 300°C. Study of the diffraction patterns from all of the films

showed that, in general, the best polycrystalline InSb films were obtained from deposition on CaF_2 substrates in the temperature range of 200°C to 300°C. The micrographs of the carbon surface replicas showed that the films produced were, with one possible exception, continuous. There was a great variation in the surface characteristics of the films but no correlation was found between the surface features and the type of diffraction patterns obtained from the films.