

AD-A151 014 INVESTIGATION OF THE MBE GROWTH OF INSB AND NOVEL
QUANTUM WELL STRUCTURES. (U) WESTINGHOUSE RESEARCH AND
DEVELOPMENT CENTER PITTSBURGH PA R F FARROW ET AL.
UNCLASSIFIED 15 JAN 85 84-9F3-QUADS-R1 N00014-83-C-0617 F/G 20/2

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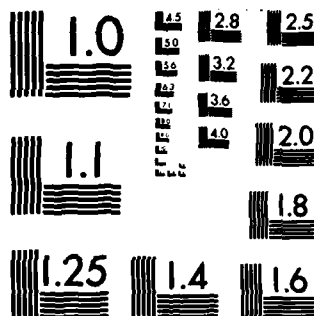
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R&D REPORT 84-9F3-QUADS-R1

INVESTIGATION OF THE MBE GROWTH OF InSb
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R. F. C. Farrow, A. J. Noreika, T. A. Temofonte,
W. J. Takei, F. A. Shirland

Final Report for the Period October 1983
to October 1984

ONR Contract N0014-83-C-0617

January 15, 1985

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1. INTRODUCTION

The discovery⁽¹⁾ of the existence of a high-mobility electron gas in GaAs, confined at the interface between GaAs and $\text{Al}_x\text{Ga}_{1-x}\text{As}$, has had profound consequences in the development⁽²⁾ of semiconductor physics and in the origin⁽³⁾ of a new generation of high-electron-mobility transistors (HEMT), also known as modulation-doped field effect transistors (MODFETS). This has led to a search for other semiconductor materials systems in which confined electron or hole gases are present. The work outlined in this report was stimulated by the recognition^(4,5) that the conduction band offset at an InSb-CdTe interface favored the existence of an electron gas confined in the InSb. Figure 1 illustrates the expected energy band diagram for this materials system for an n-InSb/n-CdTe interface. InSb has a greater electron affinity (4.59 eV)⁽⁶⁾ than CdTe (4.28 eV,⁽⁶⁾ 4.5 eV⁽⁶⁾) and a much smaller bandgap. Lattice-matched, abrupt interfaces between n-type InSb and n-type CdTe should therefore exhibit a conduction band-edge discontinuity (ΔE_c) of the same type as that for GaAs- $\text{Al}_x\text{Ga}_{1-x}\text{As}$. Estimated room temperature values of ΔE_c vary from 0.09 eV⁽⁶⁾ through 0.31 eV⁽⁶⁾ and 0.46 eV⁽⁷⁾ with the latter value being more reliable. As is the case of GaAs, very high-electron-mobility values are expected at 77K. In fact, the intrinsic carrier concentrations in InSb are negligible ($< 10^{15} \text{ cm}^{-3}$) at temperatures below 200K, and carrier mobilities at 77K are expected to approach the values measured⁽¹²⁾ for MBE-grown undoped ($n_D - n_A \sim 2 \times 10^{14} \text{ cm}^{-3}$) InSb, i.e., $\geq 300,000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. Even at room temperature, electron mobilities in bulk InSb are as high as $50,000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, for $n_D - n_A \leq 2 \times 10^{17} \text{ cm}^{-3}$, so that a very high-mobility two-dimensional electron gas should exist at room temperature. In fact at 300K it may not be necessary to dope the CdTe film to achieve such a gas since the two-dimensional well will be populated by intrinsic electrons.

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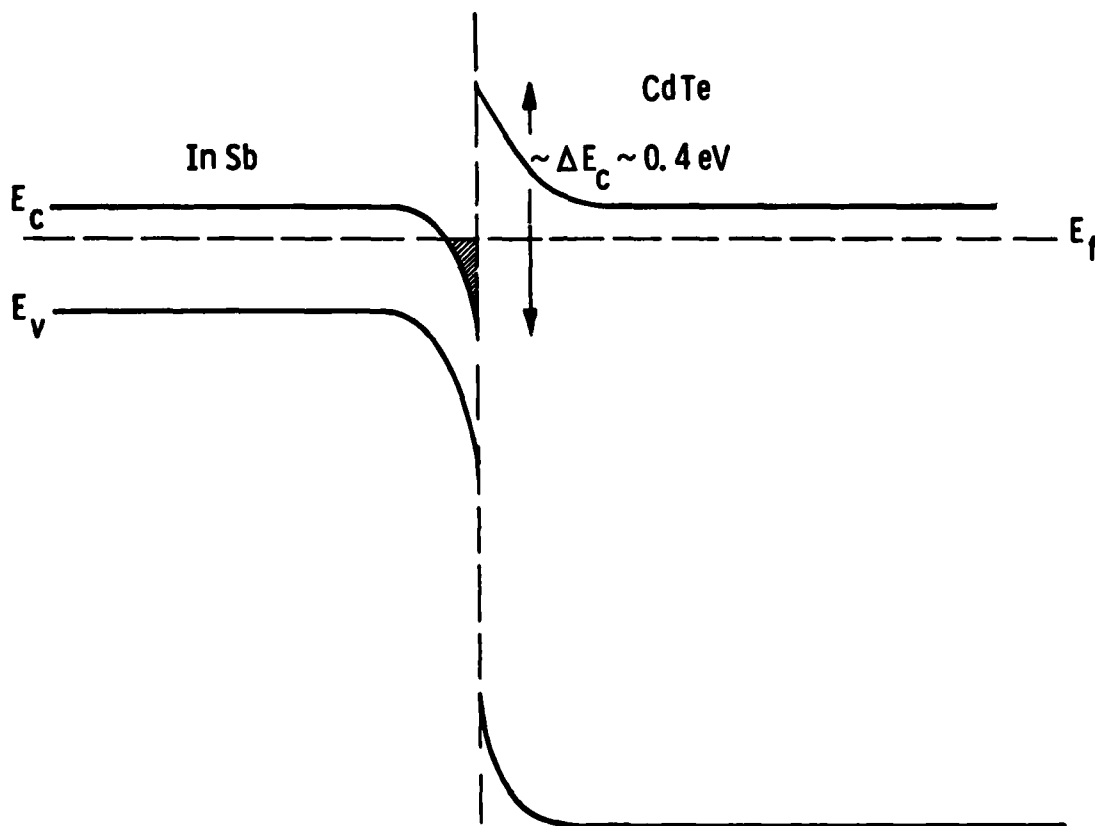


Figure 1. Energy band diagram showing expected discontinuities in conduction and valence band edges for abrupt n-InSb/n-CdTe heterojunction at 300K.

In order for the MBE growth of InSb/CdTe heterostructures to be optimized, full characterization of the structures by a range of diagnostic techniques was essential. Figure 2 illustrates the organization and relationship of the growth and characterization work reported here. MBE growth and structural characterization of CdTe/InSb heterostructures was carried out at the Westinghouse R&D Center. This work was also supported by ERADCOM (Ft. Monmouth, NJ) because of their interest in the unique transport properties of InSb films and in novel approaches for generating lateral superlattices in the CdTe/InSb materials system. Magneto-photoconductivity measurements of the structures were carried out at the Naval Research Laboratory by Dr. R. J. Wagner and at the State University of New York (SUNY), Buffalo, by Dr. B. D. McCombe. Photoluminescence studies were carried out during the first half of 1984 by Dr. J. Furneaux (NRL) and subsequently at the University of Pittsburgh by Dr. W. J. Choyke.

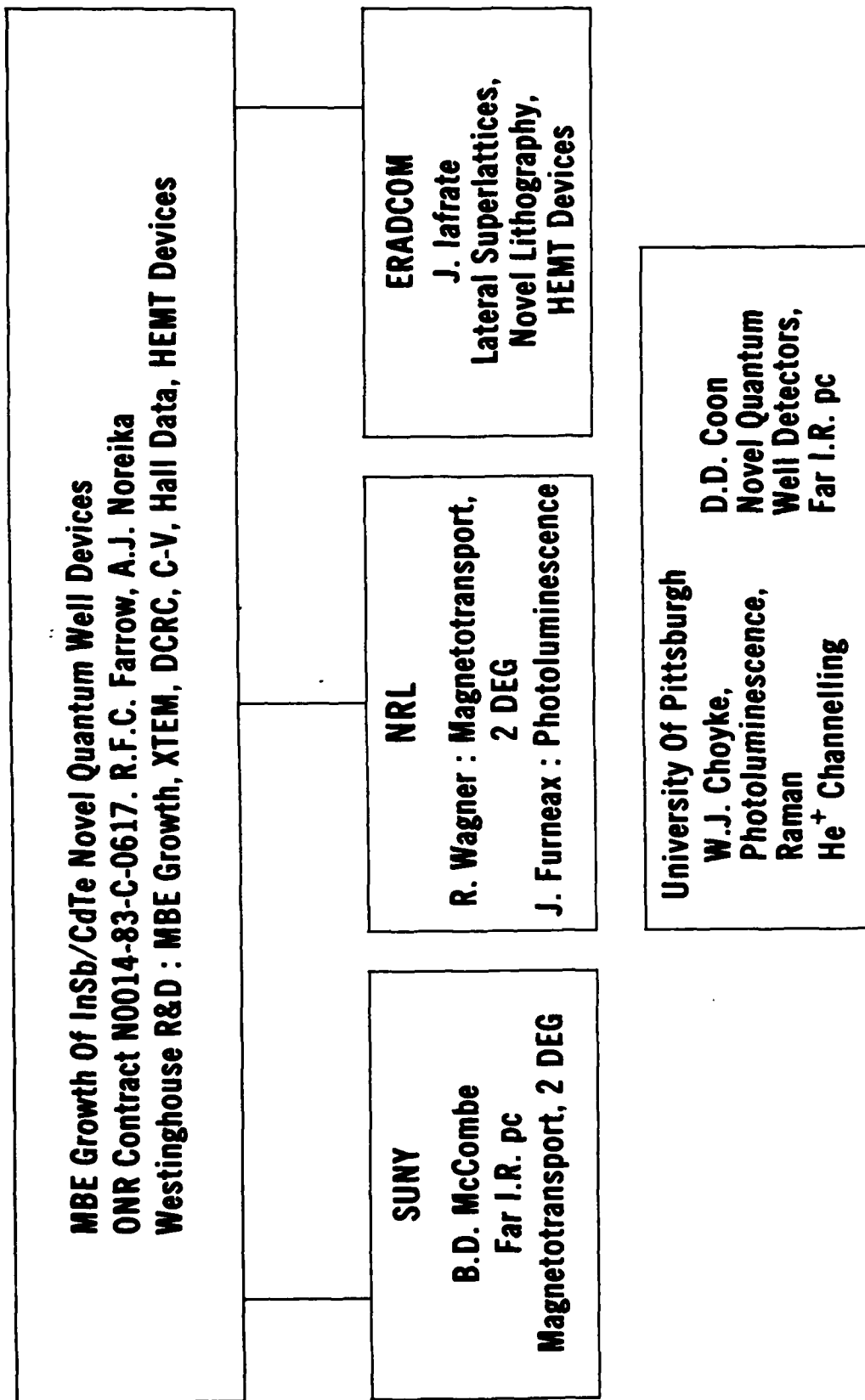


Figure 2. Diagram showing program organization and relation to growth and characterization.

2. OBJECTIVES

The prime objectives of this contract are twofold: (a) the preparation and characterization of InSb/CdTe interfaces to investigate the existence of a two-dimensional electron gas confined in the InSb by the interface potential; (b) the preparation and characterization of InSb films grown onto lattice-matched CdTe substrates.

3. PREPARATION AND CHARACTERIZATION OF InSb/CdTe INTERFACES

3.1 MBE-Growth of CdTe on InSb (001) Substrates

High-perfection, high-purity CdTe films were grown onto ion-bombarded and annealed InSb (001) substrates using the method⁽⁸⁾ developed by the author at RSRE, Malvern, U.K. CdTe films were also grown onto intermediate buffer layers of InSb grown by MBE. Both methods yielded high-perfection, lattice-matched InSb-CdTe interfaces. This was confirmed by extensive cross-section transmission electron microscopy (XTEM) studies⁽⁹⁾ and double-crystal X-ray rocking curve (DCRC) and topography analysis of the structures. Figures 3 and 4 illustrate the defect-free abrupt nature of the InSb-CdTe interfaces. The interface is delineated by a dark line in the bright-field micrographs. The width of this line is $\leq 25\text{\AA}$ (see Figure 4) in the most abrupt structures. This dimension is believed to be an upper limit to compositional interdiffusion and is consistent with SIMS depth profiles of the structures. A detailed description of microstructural studies of the MBE-grown CdTe films is given in Reference 9 (copy appended). Room temperature C-V measurements of high-perfection CdTe films revealed that the films were n-type with free carrier concentrations in the 10^{14} cm^{-3} to 10^{15} cm^{-3} range (see Figure 5). The C-V data were consistent with a flat profile of carrier concentration through the film.

The high structural perfection of CdTe films on InSb (001) substrates was confirmed⁽¹⁰⁾ by both X-ray topography and double-crystal X-ray rocking curve analysis. Figures 6 and 7 show double-crystal X-ray rocking curves for two representative films. These curves reveal several significant facts about the films. The width of the InSb peak is close to the theoretical value (~ 10 arc sec) for a perfect crystal. This is consistent with the very low ($\sim 200\text{ cm}^{-2}$) dislocation

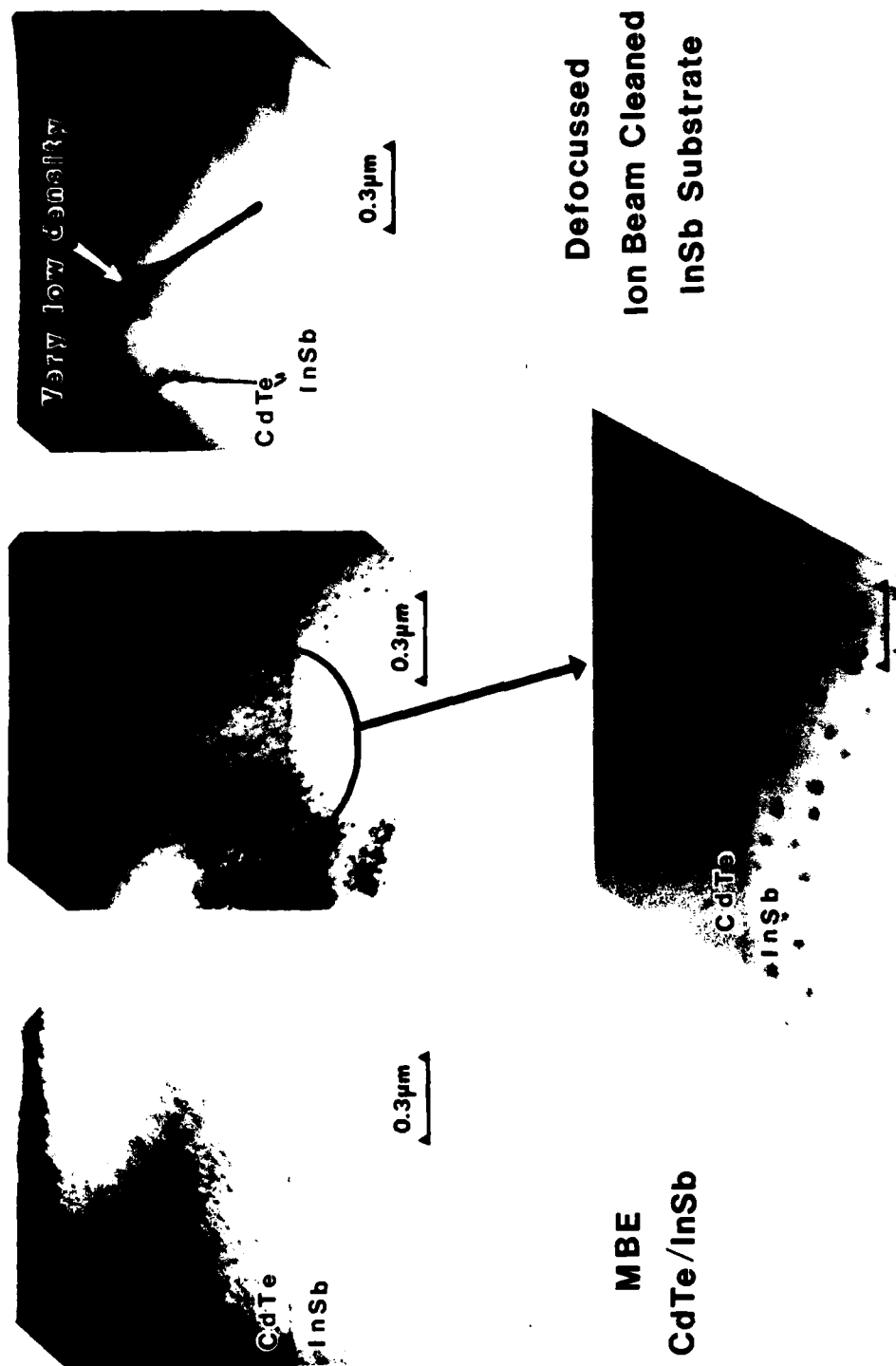


Figure 3. Bright-field, cross-sectional transmission electron micrographs for high-perfection CdTe film, 1 μ m thick, grown at 200°C on InSb (001) surface prepared by argon-ion bombardment and annealing. Few extended defects were present and virtually no misfit dislocations.

Top surface of CdTe

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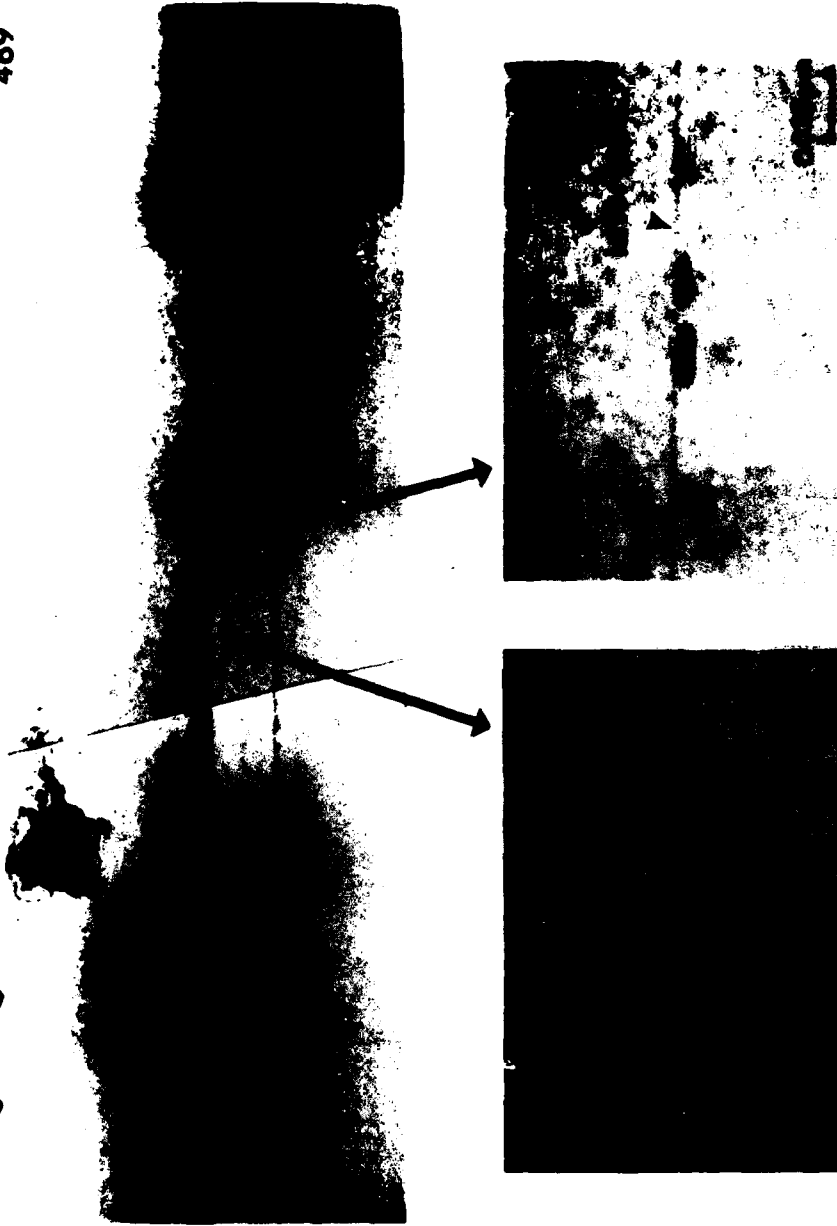


Figure 4. Bright-field, cross-sectional transmission electron micrographs for a good-quality CdTe film grown on a thin (0.2 μ m) InSb buffer layer. The CdTe/InSb interface is clean, whereas the InSb/InSb interface is defined by residual surface contamination and In-rich precipitates.

Curve 745920-A

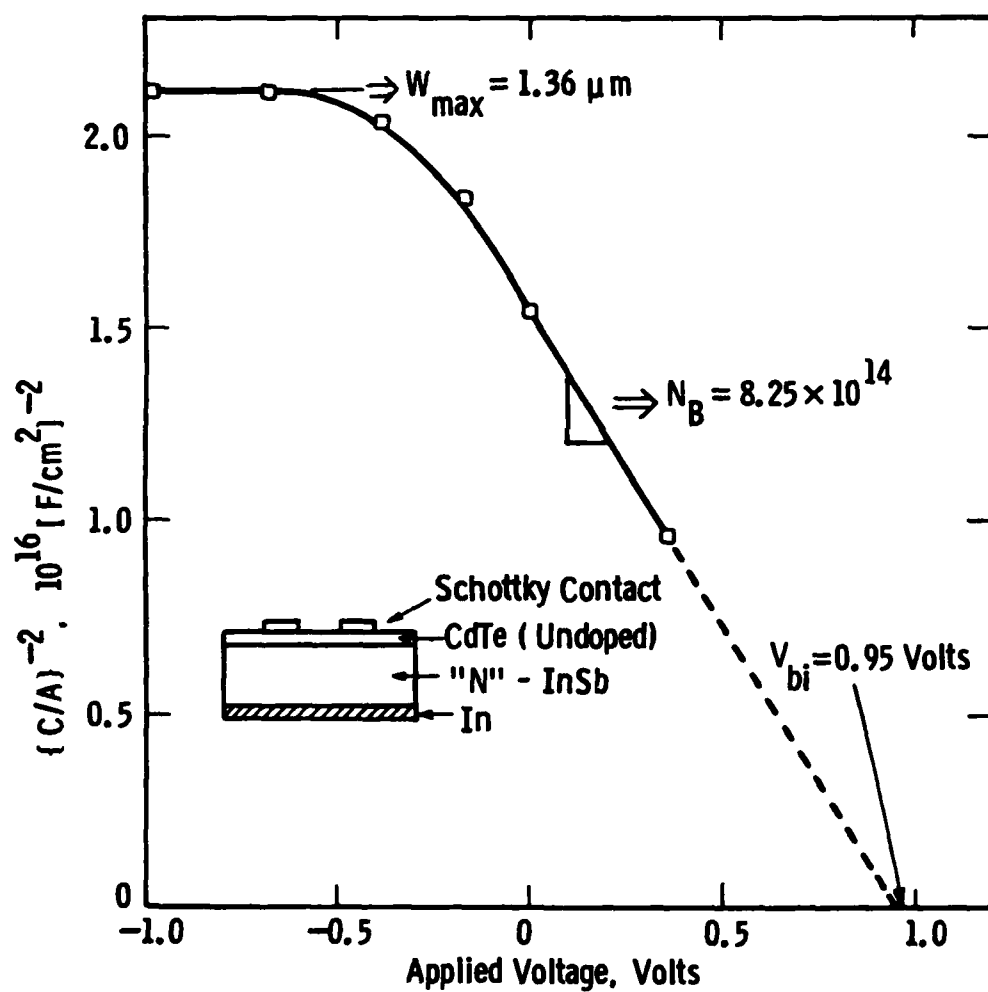


Figure 5. Capacitance-voltage plot for an undoped CdTe film grown on (001) InSb at 200°C.

Curve 747598-A

DCRC MBE CdTe/InSb (001) #207
1.4 μm $T_g = 186^\circ\text{C}$

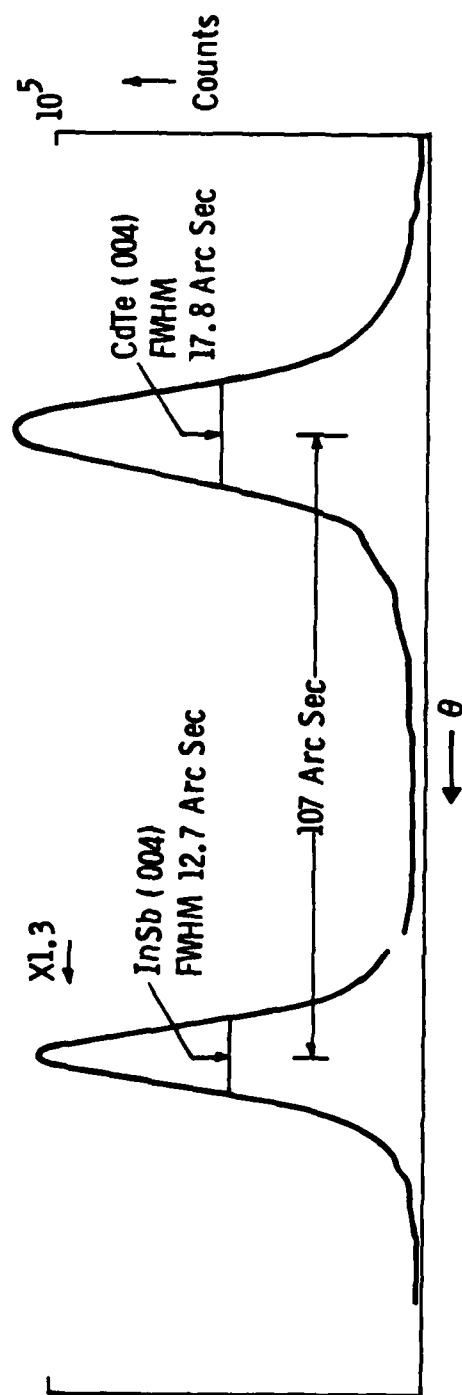


Figure 6. Double-crystal X-ray rocking curve of a 1.4 μm thick MBE-grown CdTe film on (001) InSb substrate. The single, symmetric CdTe peak is consistent with a film which has a dislocation density of $10^4 - 10^5 \text{ cm}^{-2}$, is free of low-angle grain boundaries, and is exactly lattice-matched to the InSb substrate in the growth plane. Lattice misfit between InSb and CdTe is accommodated by a uniform elastic strain in the film.

Curve 747596-A

DCRC MBE CdTe/InSb (001) #215

2.5 μm $T_g = 186^\circ\text{C}$

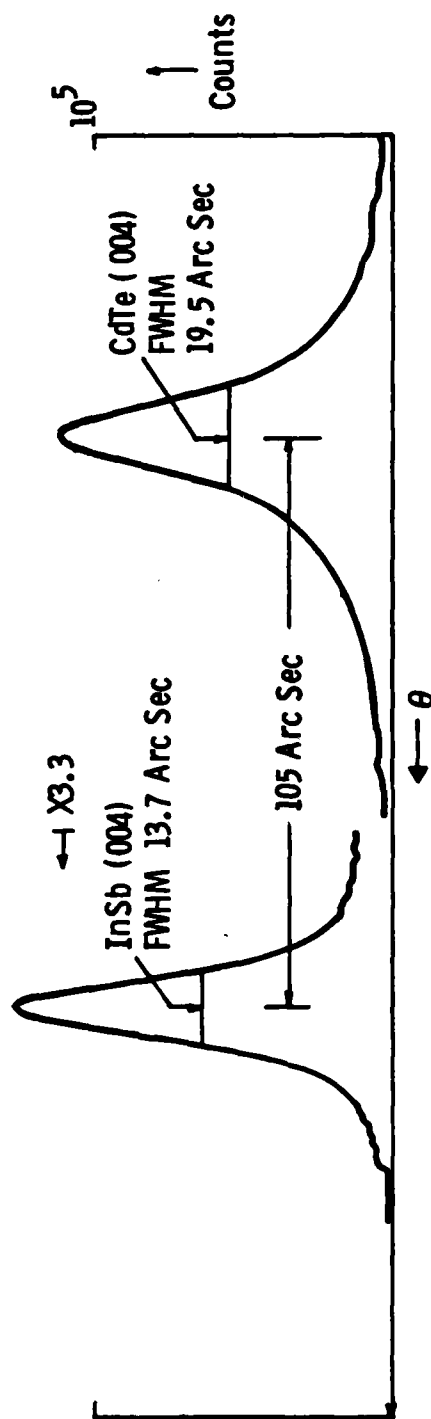


Figure 7. Double-crystal X-ray rocking curve of 2.5 μm thick MBE-grown CdTe film on (001) InSb substrate. The single symmetric CdTe peak is consistent with a film which has a dislocation density of $< 10^5 \text{ cm}^{-2}$, is free of low-angle grain boundaries, and is exactly lattice-matched to the InSb substrate in the growth plane. Lattice misfit between InSb and CdTe is accommodated by a uniform elastic strain in the film.

density of the InSb wafers and the considerable care taken in surface preparation prior to epitaxy. The CdTe peak width is significantly greater than the theoretical value (~ 11 arc sec) for a perfect film. This is consistent with a dislocation density in the 10^4 - 10^5 cm $^{-2}$ range. However, the single symmetric CdTe peak confirms the absence of low-angle grain boundaries in the film. The films are considerably more perfect than any bulk CdTe crystals yet produced and represent the state of the art in CdTe film perfection. This point is substantiated by the results of X-ray topography studies illustrated in Figure 8. The X-ray topograph of the CdTe film in (a) exhibits a near-uniform grey scale of density with isolated linear features. This is indicative of a highly perfect film, free of strain inhomogeneities and low-angle grain boundaries. The isolated linear features are due to surface scratch marks on the film surface. The topographs of CdTe wafers shown in (b) and (c) exhibit contrast variations indicative of long-range strain, low-angle grain boundaries, and lineage features. These defects are present through the entire thickness of the wafers since they remained after extensive (100-200 μ m) free-etching.

High-resolution cross-section transmission electron microscopy (HRTEM) studies⁽⁹⁾ of the InSb-CdTe interface confirmed that the interface was coherent as indicated⁽⁸⁾ by the double-crystal rocking curve analysis.

3.2 MBE Growth of InSb on CdTe Substrates

Extensive studies^(11,12) of InSb homoepitaxy and of heteroepitaxy on GaAs substrates have led to a complete mapping of the growth conditions necessary for high-structural and electrical-quality InSb films. These studies revealed: (a) near-bulk electron mobility values could be achieved⁽¹²⁾ in thick (> 30 μ m) homoepitaxial or heteroepitaxial films grown near 400°C; (b) the near-unity condensation coefficient of Sb $_4$ on InSb at substrate temperatures below 300°C⁽¹¹⁾ led to problems in sustaining single-phase films at this growth temperature or lower.

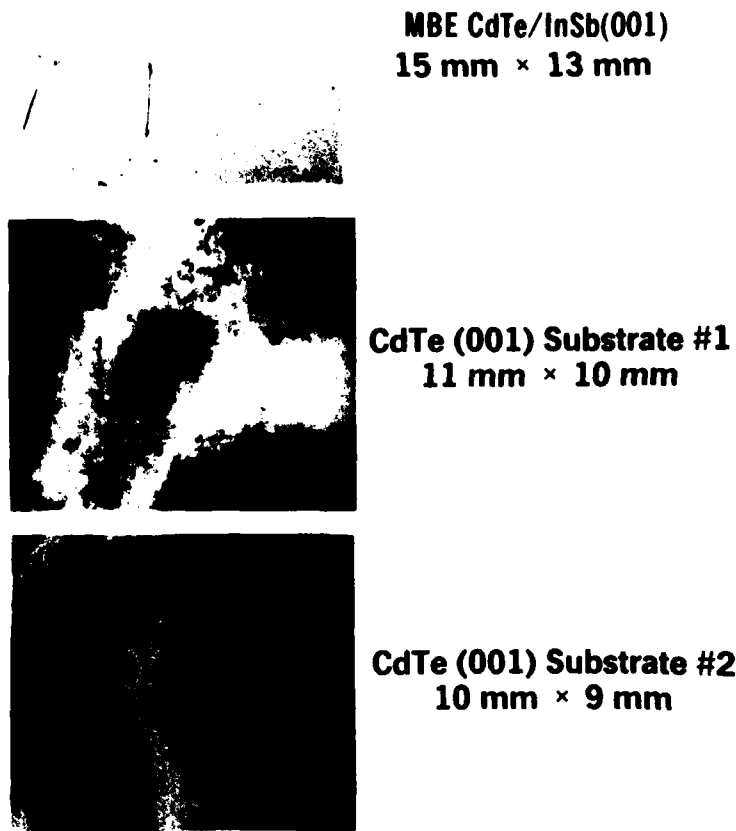


Figure 8. X-ray topographs of CdTe: (a) MBE-grown CdTe film, 1.2 μm thick on (001) InSb substrate; (b) (001) orientation wafer from Bridgman-grown bulk CdTe ingot (substrate #1); (c) (001) orientation wafer from Bridgman-grown bulk CdTe ingot (substrate #2).

Note: The near-uniform grey scale of the CdTe film in (a) is indicative of a highly perfect film, free of strain inhomogeneities and low-angle grain boundaries. The CdTe wafer topographs, on the other hand, are characteristic of mosaic crystals with low-angle subgrain boundary structure, long-range strain and lineage features. The presence of long-range strain is indicated by the fact that only part of the wafer operates in the Bragg diffraction at the setting used. The reflection topographs were recorded with $\text{CuK}\alpha$ radiation and the operating Bragg diffraction was (531). Note that CdTe substrates #1 and 2 are representative of the great variability in structural quality of commercially available substrates.

In view of the poor and irreproducible quality of available CdTe substrates, as discussed in Section 3.1, it is clear that MBE growth of InSb on CdTe substrates will inevitably result in highly defective films. In addition, the requirement of a growth temperature near 400°C for useful electrical-quality InSb films is likely to compound this problem by leading to an interdiffused interface. For any reasonable chance of detection of a two-dimensional electron gas in the InSb, at the interface, interdiffusion must be absent. This is because the conduction and valence band offsets will be reduced by compositional and doping gradations. In addition, carrier scattering by ionized impurities in the InSb will degrade carrier mobilities, especially at low temperatures. For these reasons, emphasis has been placed on MBE growth of CdTe on InSb substrates (rather than vice versa) in order to achieve the highest purity and most perfect interfaces. MBE growth of InSb films on CdTe substrates has, in fact, been reported.⁽¹³⁾ However, the structural quality of the films was clearly limited by the poor quality of substrates used. No electrical data for the films were reported. It is likely that MBE growth of InSb onto high-perfection CdTe films (grown onto InSb substrates) may be a more promising route to high-perfection InSb on CdTe interfaces, although the problem of interdiffusion at growth temperatures $\geq 300^\circ\text{C}$ remains.

4. PHOTOLUMINESCENCE OF CdTe FILMS ON InSb (001)

Extensive photoluminescence studies of MBE-grown CdTe films on InSb were undertaken at NRL during the first half of 1984 and subsequently at the University of Pittsburgh. These studies provide information, complementary to the structural studies, on film quality and in addition give information on free-carrier concentration and on impurity and defect-induced states in the film. Figures 9 and 10 show photoluminescence spectra, recorded at NRL, from CdTe films grown⁽¹⁴⁾ at temperatures in the range 220° to 160°C. Several points emerge from this photoluminescence study. Figure 9 reveals that the intensity of the broad deep-level peak centered at ~ 1.48 eV decreases in comparison with the near band-edge emission peak (~ 1.59 eV) as the growth temperature is reduced. It is believed⁽¹⁵⁾ that the broad, deep-level peak arises from point defect-impurity complexes of the type:

$(V_{Cd}^{2-} D^+)^-$, i.e., a doubly ionized Cd vacancy (V_{Cd}) combined with a donor impurity (D). The intensity of the peak has been observed⁽¹⁵⁾ to scale with the chemical concentration of In in In-doped, bulk CdTe crystals. In addition, the peak has been correlated⁽¹⁶⁾ with structural damage in the surface region of undoped bulk CdTe wafers. In the present case the systematic decrease in the intensity of this peak with decreasing growth temperature may indicate a decreasing concentration of Cd vacancies with decreasing growth temperature. This is qualitatively consistent with a CdTe film growth model in which growth occurs by reaction between Te_2 molecules and Cd adatoms in chemisorbed states. Entry to the chemisorbed states is via a physisorbed precursor state. Because of the greater volatility of Cd compared with Te_2 (suggested by the elemental vapor pressures) at high (> 200°C) substrate temperatures, desorption of Cd from its physisorbed state, before reaction with Te_2 , is increasingly likely as the substrate temperature is increased. Hence

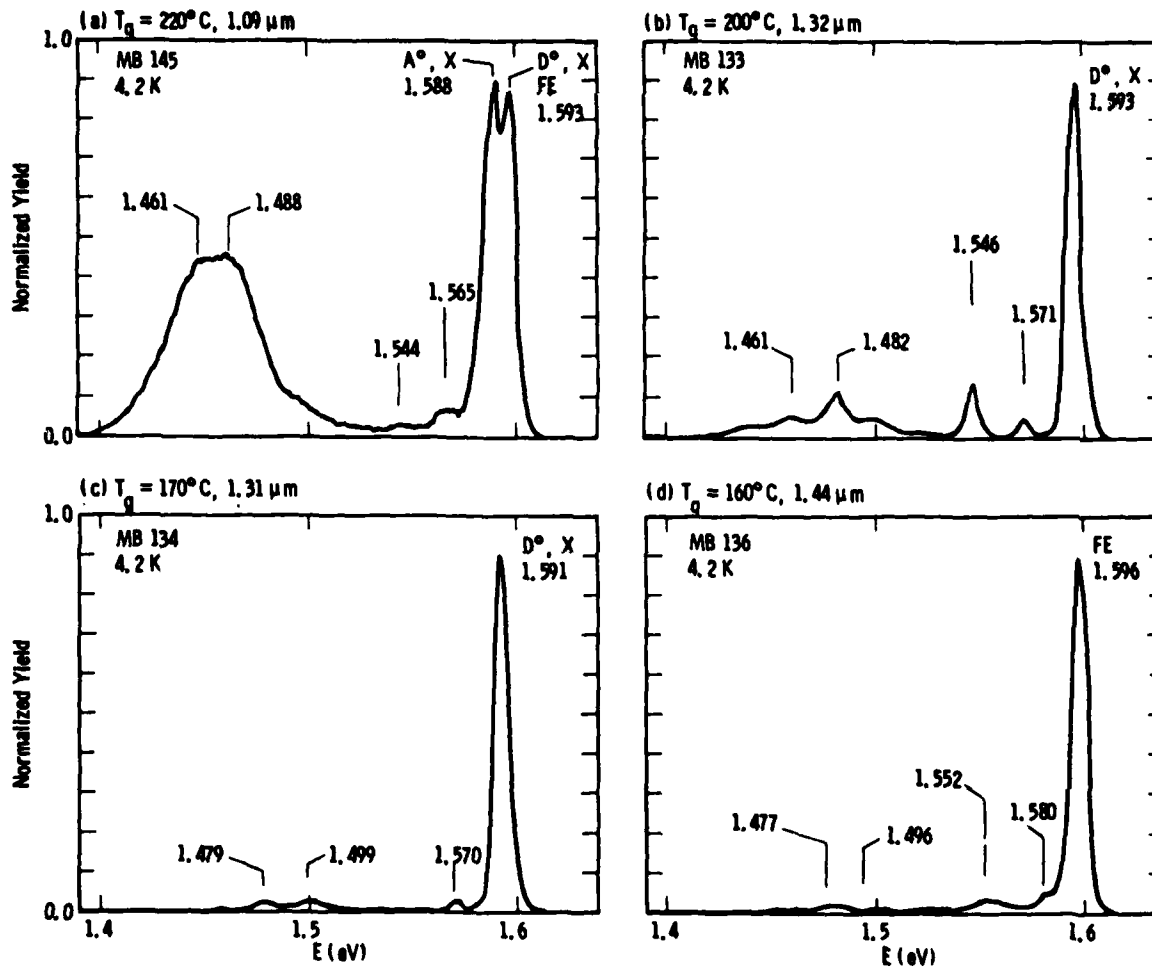


Figure 9. Photoluminescence spectra of MBE-grown CdTe films grown at temperatures from $T_g = 220^\circ\text{C}$ to 160°C . Spectra recorded at low-energy resolution to cover the range $1.4 - 1.6\ \text{eV}$. Note the decrease in the broad, deep-level peak centered at $\sim 1.4\ \text{eV}$ compared with the near band-edge emission as the growth temperature decreases.

CdTe/InSb <100>

Curve 746954-8

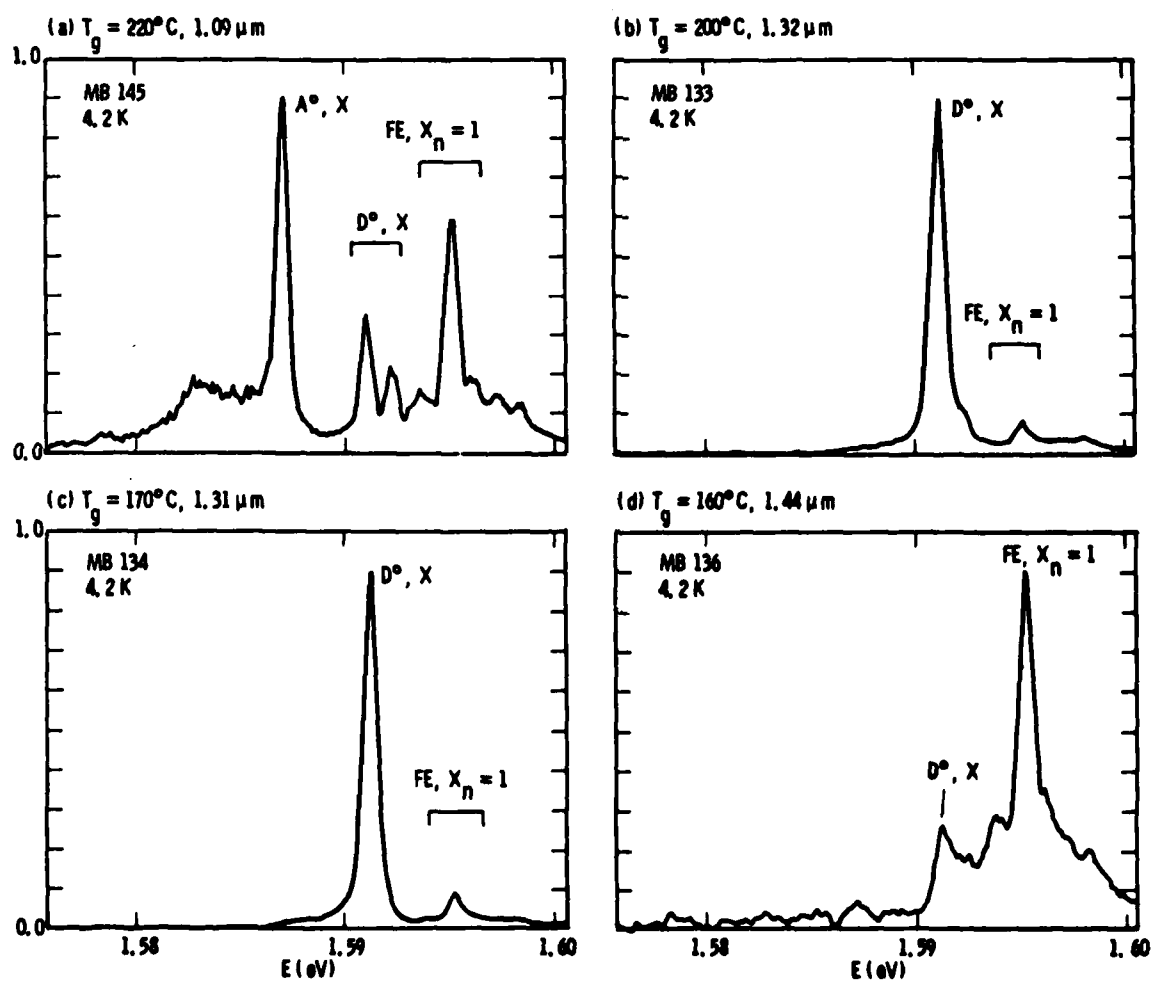


Figure 10. Photoluminescence spectra of same MBE-grown films as in Figure 9. Spectra recorded near band-edge region as higher resolution.

one expects an increasing (decreasing) concentration of Cd vacancies as the growth temperature is increased (decreased).

Another feature in the spectra is the sharp (FWHM ~ 1 meV) well-resolved free-exciton lines seen in the high-resolution spectra shown in Figure 10. This is indicative of a low ($< 10^{16}$ cm $^{-3}$) free-carrier concentration in the films, consistent with the C-V data.

Assignment of the impurity-related bound exciton lines ($\tilde{A}$, X) and ($\tilde{D}$, X) must await a detailed photoluminescence study. Such a study is now underway at the University of Pittsburgh. However, the main conclusion from the photoluminescence study is already clear: the CdTe films are of good structural and optical quality and of high purity with the lowest concentration of point defects at the lowest growth temperature.

5. MAGNETO-PHOTOCONDUCTIVITY STUDIES OF MBE-GROWN CdTe/InSb STRUCTURES

Far-infrared magneto-photoconductivity studies of MBE-grown CdTe films on InSb (001) were carried out at NRL (by Dr. R. J. Wagner) on samples prepared prior to October 1983. The technique and experimental method is described in Reference 17. These studies revealed no features in the photoconductivity spectra attributable either to carrier transport in the CdTe films or to an inversion layer in the p-type InSb substrate. In the period from October 1983 to July 1984, considerable improvements were made in the structural and optical quality of the CdTe films by optimizing⁽¹⁴⁾ InSb surface preparation, growth temperature, and conditioning of the CdTe effusion source. The high-perfection films described in Section 3.1 were prepared in this period. Several of these films were sent to Dr. B. D. McCombe (SUNY) for magneto-photoconductivity studies. Here, the results of a study on one of the samples, MBE 189, are presented. Additional studies on further samples are presently in progress. The growth conditions and InSb substrate parameters are summarized in Table 1.

Figure 11 shows the photoconductivity response for MBE 189 versus photon energy for several different magnetic fields (top trace to bottom trace: $B = 0$; 1.5T; 2.5T; 3.0T and 3.5T). All data were taken with a Fourier Transform Interferometric Spectrometer with the sample in a light-pipe system immersed in liquid helium and cooled by helium exchange gas. The photoconductive spectrum is rich in structure. The features which can be assigned with near certainty are identified by the light lines and labelling. The minimum which occurs at about 144 cm^{-1} and which is independent of magnetic field is due to TO phonon absorption in the CdTe epilayer. The line labelled "InSb substrate" is due to acceptor impurity transitions in the substrate that contribute to the photoconductivity due to contacts being made right through the CdTe film to the InSb substrate.

TABLE 1
GROWTH AND SUBSTRATE PARAMETERS FOR SAMPLE MBE 189

InSb Substrate: p-type, Cd-doped, (001) wafer plane

77K electrical data:

$$n_A - n_D = 5.10^{14} \text{ cm}^{-3}$$

$$\mu_H = 9000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$$

Supplier: Metal Specialties Inc.

Crystal Grower: MCP Ltd. Wembley, U.K. Ingot 1SC 119

CdTe Film Growth Parameters:

Etch pit density $< 300 \text{ cm}^{-2}$

CdTe Effusion Source Orifice

1 mm

CdTe Effusion Source Temperature

670°C

(Cd + 1/2 Te₂) Beam Pressure

at sample

$2.1 \cdot 10^{-7}$ Torr

Growth Temperature

185°C

Growth Rate

0.5 $\mu\text{m hr}^{-1}$

Film Thickness

1.0 μm

Film Grown on April 10, 1984

Double-Crystal X-ray Diffraction Linewidth: 21.5 arc sec for (004)

Bragg diffraction

CuK α radiation

The line labelled CdTe 1s + 3p appears to be a transition from the ground state (1s) to a higher excited state of the hydrogenic system of a donor impurity. The 1s + 3p transitions are clearly observed as labelled. Photoresponse from the 1s + 2p transitions is not observed because kT at the ambient temperature is not sufficient to ionize this excited state. The 2p + continuum energy at zero magnetic field is about 3.5 meV, 10 times kT at 4.2K, and this energy increases with magnetic field.

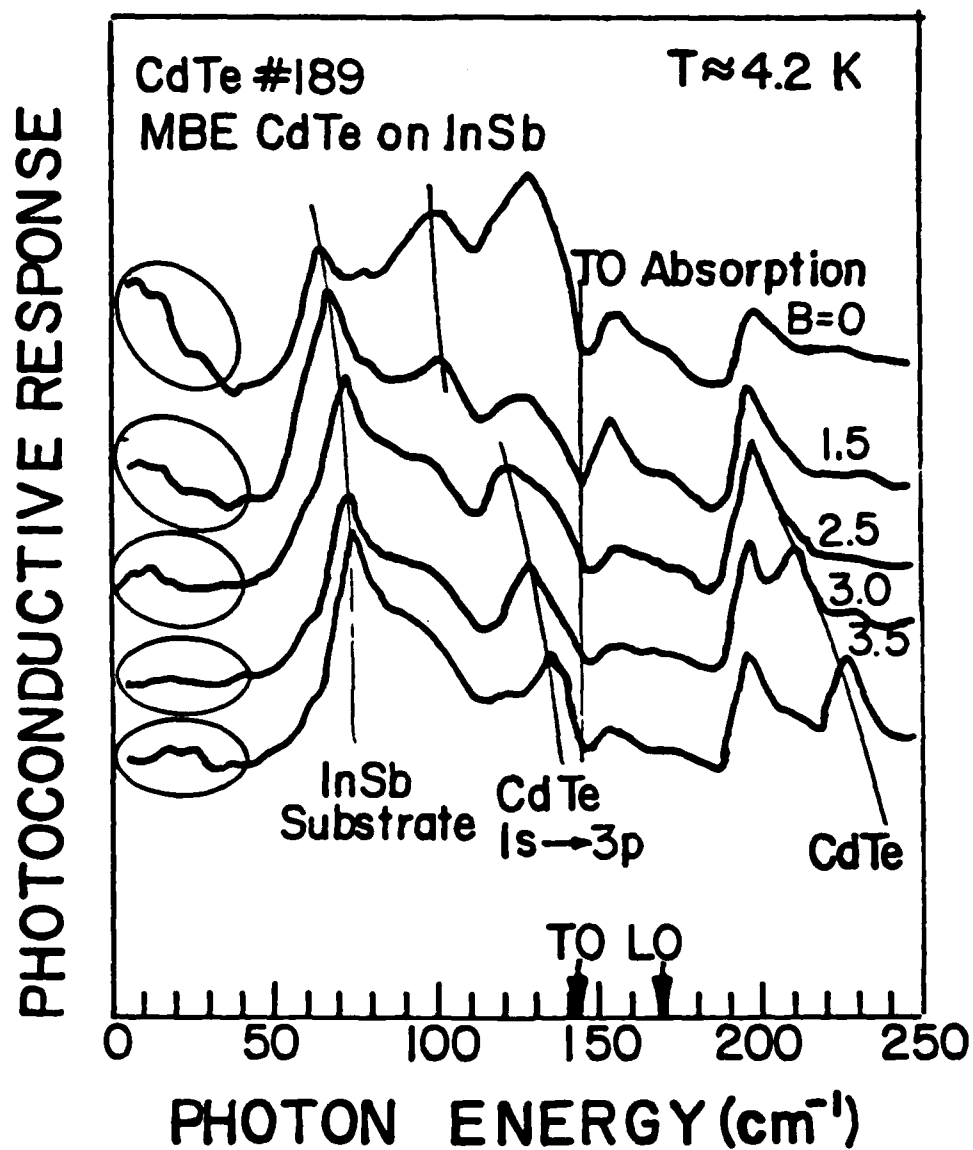


Figure 11. Photoconductive response of MBE-grown CdTe film on InSb (001) orientation substrate: sample MBE 189 (recorded by Dr. B. D. McCombe; see text for details).

The circled region at very low photon energies has the character of free-carrier photoresponse (intensity decreases with increasing photon energy). As the magnetic field is increased this photoresponse decreases; it is very weak at 3T. At higher magnetic fields it takes on the character of a resonant response. This may⁽¹⁸⁾ be due to free electrons in an inversion layer in the InSb at the InSb-CdTe interface resulting from charge transfer from the CdTe donors to the p-type InSb (as in modulation-doped GaAs-AlGaAs heterostructures). Further investigations are in progress at SUNY to investigate this possibility.

Figure 12 shows a compilation of the major features in the photoconductive signal that can be identified with some degree of certainty. The transition energy, at which the peaks are observed, is plotted as a function of magnetic field. For comparison purposes, data from donors in bulk CdTe⁽¹⁷⁾ (solid lines) and acceptors in bulk InSb⁽¹⁹⁾ (dashed lines) are also shown. The data points attributed to CdTe donor transitions are shown as solid circles, while the data points attributed to acceptor transitions in the InSb substrate are shown as solid triangles. By heating the samples to about 10-15K it should be possible to observe the $1s \rightarrow 2p$ transitions easily, and by studying them at high resolution it should be possible to determine the chemical nature of the donor impurities.

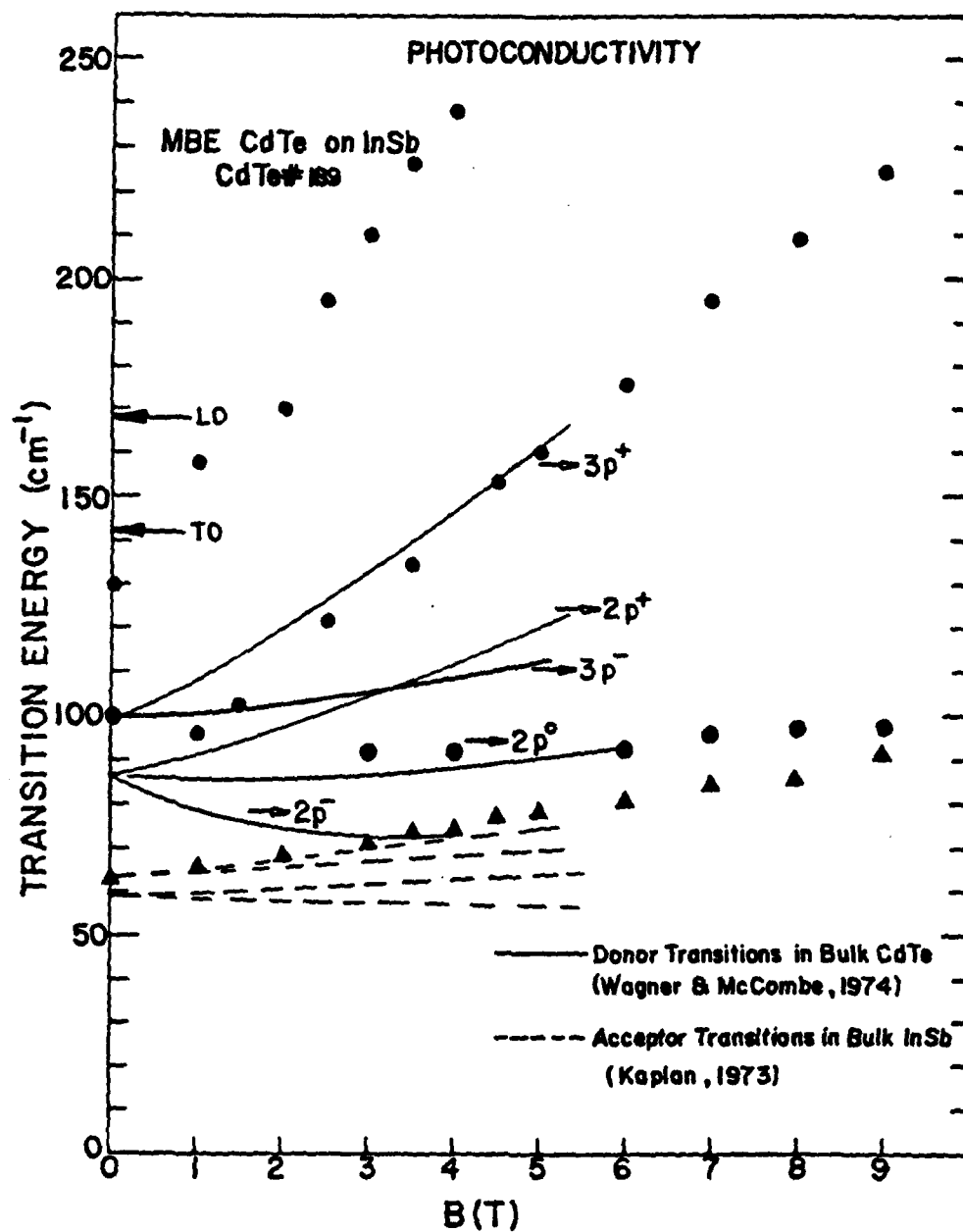


Figure 12. Transition energies as a function of magnetic field for sample MBE 189 (recorded by Dr. B. D. McCombe; see text for description).

6. CONCLUSIONS

The main objective of this contract has been achieved. MBE growth of CdTe films on InSb (001) orientation substrates has been optimized and has resulted in lattice-matched structures of the highest structural perfection yet achieved in this materials system. Magneto-photoconductivity data suggest the existence of an inversion layer in the InSb at the CdTe-InSb interface. Magneto-transport investigations of this inversion layer are in progress and will be supplemented by Hall mobility investigations to determine whether electrons in the inversion layer have enhanced mobilities compared with electrons in bulk InSb.

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Microstructural studies of CdTe and InSb films grown by molecular beam epitaxy

APPENDIX

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Epitaxial thin films of CdTe (1–5 μm) have been grown directly onto (001) InSb substrates or onto intermediate buffer layers of InSb (0.25–0.5 μm) by molecular beam epitaxy. Cross-sectional transmission electron microscopy and high-resolution transmission electron microscopy have been used to characterize the film and interfacial microstructures. Inferences about film quality were also compared with single-crystal x-ray rocking curve data and agreed well. Resulting microstructural features were correlated with various experimental growth parameters and substrate cleaning procedures. Results show that near-perfect CdTe films can be grown on InSb substrates, but film quality is critically dependent upon substrate cleaning. Other factors observed to influence defect formation in the films include growth rate, total growth time, or a change in growth rate during film growth. Extended defects which form include twins, line dislocations, or looplike defects. Lattice imaging has demonstrated the lattice matching across the InSb film/InSb substrate interface, despite the formation of In precipitates during the heat cleaning procedure.

PACS numbers: 68.55. + b, 68.60. + q, 68.48. + f

I. INTRODUCTION

The interest in producing large areas of high-quality, single-crystal CdTe is driven by the need for an adequate substrate for $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ growth for infrared detectors. This application requires CdTe crystals which are essentially free of extended defects, precipitates, and low-angle boundaries; however, twins occur readily in Bridgman, solvent evaporation, and Czochralski grown material.¹ Molecular Beam Epitaxy (MBE) growth of CdTe on a lattice matched substrate offers one possible alternative for producing high perfection single-crystal material. MBE techniques permit the growth of epitaxial CdTe (Ref. 2) and $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ (Ref. 3) films of device quality at temperatures well below those of other growth technologies. InSb is of particular interest as a CdTe film substrate for MBE growth because of the nearly perfect lattice match between the two zincblende structures ($|\Delta a|/a \leq 5 \times 10^{-4}$ at 25 °C).² Thus it is expected that MBE growth of essentially defect-free films of CdTe on InSb should be possible.

In this work we have employed cross-sectional transmission electron microscopy (XTEM) and high-resolution transmission electron microscopy (HRTEM) to characterize the microstructures of thin films of CdTe (1–5 μm) grown by MBE directly onto (001) InSb substrates or onto intermediate buffer layers of MBE grown InSb (0.25–0.5 μm). XTEM provides information about the perfection of the MBE films throughout their entire thickness as well as allowing direct observation of the CdTe/substrate interface. Film quality and interface structure observed by XTEM have been correlated with experimental growth parameters and substrate cleaning procedures. Furthermore, it has been shown that film quality determined by single-crystal x-ray diffraction data correlates very well with the quality determined by XTEM characterization. This is the first comprehensive microstructural study of MBE grown CdTe films, although Chew, *et al.*⁴ have presented some preliminary results of their work.

II. EXPERIMENTAL PROCEDURE

Pertinent experimental parameters for the films grown by MBE and characterized by XTEM are listed in Table I. Most of the CdTe films were grown directly onto ion beam cleaned InSb substrates, but in two experiments (designated as samples 469 and 470) intermediate InSb buffer layers were grown on the substrate by MBE prior to CdTe growth in a continuous process. These latter films were grown in a Varian MBE 360 whereas those grown directly onto the substrate were grown in a Westinghouse built apparatus of similar design. All growth temperatures were < 200 °C. For samples 469 and 470, the InSb substrate was cleaned by heat cleaning at ~ 400 °C in a beam of Sb_4 , rather than by ion beam cleaning, as is shown in Table I. Additional experimental details are described in Ref. 2.

To prepare cross-sectional specimens for TEM observations, the InSb substrate-film composite was first cut into 2×2 mm squares. Approximately 300 nm of SiO_2 was then put onto the CdTe surface by uv-enhanced low-temperature pyrolysis as a protective measure. Specimens were sandwiched together and mounted end on in a low viscosity embedding medium (Ladd LX-112 resin), cured, and ground and polished to produce wafers ~ 0.1 mm thick. After mounting on tungsten support rings, final thinning to obtain transparent regions was achieved by ion milling with Ar ions using the liquid nitrogen cooled stage of a Gatan dual ion miller. Initial milling conditions of 5 keV and 15° angle of incidence produced adequate thin regions throughout the InSb substrate/MBE layers but left small In-rich islands on the surface of the InSb. Later a final milling at 1.5–2 keV and 10° greatly minimized or even eliminated this surface In segregation. A Philips 400T electron microscope operating at 120 keV was used for all TEM evaluation. This instrument is equipped with a Kevex System 7000 x-ray energy dispersive spectrometer (EDS) for chemical analysis. In general, the TEM specimens were prepared such that the foil normals were [110].

TABLE I. Film parameters.

Sample no.	InSb film thickness (μm)	CdTe film thickness (μm)	CdTe growth time (h)	InSb cleaning	Anneal temp. ($^{\circ}\text{C}$)	Single-crystal x-ray rocking curve FWHM, arc min		
						InSb	CdTe	R_{sc} ^a
053	None	1.35	3	Focussed Ar ion beam, 500 V	200	3.6	12	3.3
058	None	1.05	4.83	Defocussed Ar ^b ion beam, 500 V	200	3.6	6.8	1.9
086	None	1.0	3.0	Defocussed Ar ion beam 500 V	200	3.6	4.4	1.2
094	None	2.34	7.5	Defocussed Ar ion beam, 500 V	200	3.6	7.6	2.1
099	None	3.77	3.0	Defocussed Ar ion beam, 500 V	200	3.6	6.0	1.7
108	None	1.4	3.5	Defocussed Ar ion beam, 1 KV	200	3.6	4.7	1.3
469	0.21 ^c	0.66 ^d	2.0	400 $^{\circ}\text{C}$ in Sb ₄	None	3.6	4.0	1.1
470	0.6 ^c	0.61 ^d	1.5	400 $^{\circ}\text{C}$ in Sb ₄	None	3.6	5.0	1.4

^a R_{sc} is the ratio of the FWHM of the (008) Bragg diffraction for the CdTe film compared with the InSb substrate and is an inverse measure of film perfection. Subsequent analysis of the best films (086, 469) by double crystal rocking curve analysis has revealed line widths of ≈ 25 arc sec for the (004) Bragg diffraction ($\text{Cu } K_{\alpha}$ radiation) from these films.

^b With a beam current of $\sim 0.1 \mu\text{A}/\text{cm}^2$ for typically 2 h.

^c InSb buffer layer grown at 380 $^{\circ}\text{C}$.

^d CdTe layer grown at $\sim 176^{\circ}\text{C}$.

Single-crystal rocking curve data were obtained using a Siemens Omega drive diffractometer on as-grown films prior to XTEM sample preparation. A single-crystal diffractometer has considerable instrumental broadening of the Bragg peak and double crystal diffractometry is necessary for an accurate quantitative assessment of film perfection when R_{sc} [the ratio of the FWHM of the (008) Bragg diffraction for the CdTe film compared with the InSb substrate, Table I] is close to unity (i.e., < 1.4).

III. EXPERIMENTAL RESULTS

A. Growth of CdTe directly onto InSb substrates

Figures 1(a)–1(c) show BF XTEM micrographs of three CdTe films of progressively increasing crystallographic quality. Film thicknesses range between 1–1.35 μm . Early experiments, of which sample 053 [Fig. 1(a)] is representative, used a scanned focussed, 500 V ion beam to clean the InSb substrate prior to growth. Bright field XTEM [Figs. 1(a) and 2] reveal a nonplanar, undulating interface between the InSb and CdTe and a very high twin density in the CdTe film. A high dislocation density (not clearly visible at these imaging conditions) is also present within the CdTe film. Twin reflectins were utilized to obtain dark field (DF) images of the two sets of twins visible at this [110] orientation. One set of twins is shown in Fig. 2. The twins are highly faulted and nucleation appears to have primarily occurred at the "high" points of the InSb substrate. Note that, although many of the twins and dislocations have grown throughout the CdTe film, no extended defects propagate into or are generated within the InSb substrate. The spotty texture on the InSb are In-rich surface islands caused by ion milling.

In comparison to film 053, film 058, shown in Fig. 1(b) under two-beam ($g = [311]$) dynamical conditions, has a much lower twin density and the twins are much narrower. For this experiment the substrate was cleaned using a defocussed 500 V ion beam. Twin nucleation has again occurred at the InSb-CdTe interface, often in pairs, and the dislocations are often observed associated with the twins. The small (< 70 nm) defects imaged under these diffracting conditions in the CdTe film are probably due to ion milling damage,⁴ although somewhat larger looplike defects have been observed in other films as reported below.

Further improvements in CdTe film quality are evident in specimen 086 [Fig. 1(c)]. Substrate cleaning was again performed with a defocussed 500 V ion beam. It is difficult in the better quality films such as 086 to determine extended defect concentrations from cross-sectional microscopy. However, a region in 086 extending $> 150 \mu\text{m}$ in length was examined

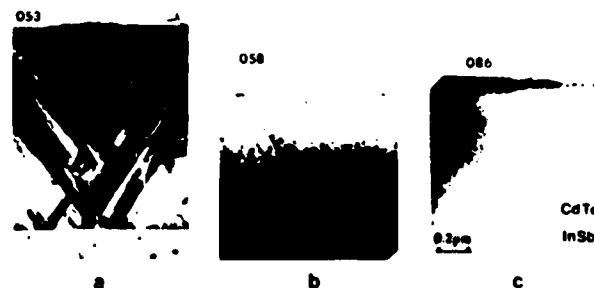


FIG. 1. BF XTEM micrographs of CdTe films on InSb substrates. Film quality improved with improved substrate cleaning from (a) a highly twinned and large dislocation density film to (c) a near perfect film with very few extended defects.

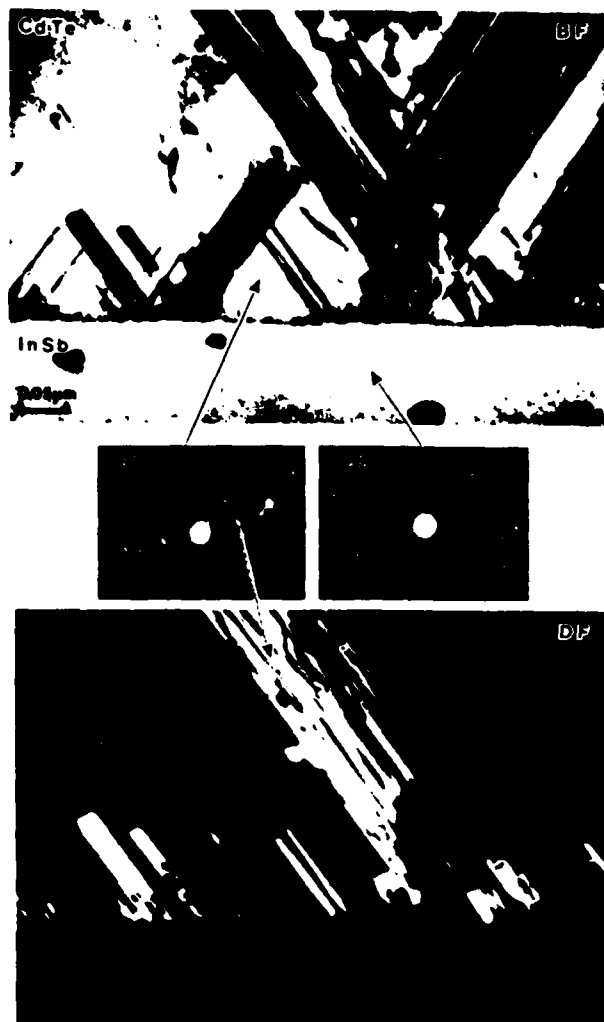


FIG. 2. XTEM micrographs of film 053 showing details of the CdTe/InSb interface structure and the twins.

in the TEM in which no defects were detected. Other areas contained an occasional dislocation, and no twins were observed in this film. In part, the clear delineation between the CdTe and InSb at the interface is thought to occur from the differential milling between the CdTe and InSb, although impurity decoration of the interface or interfacial strains have also been proffered as an explanation of the interfacial delineation.⁴

The conclusions concerning the CdTe film quality obtained from the electron microscopy observations correlate well with the single-crystal rocking curve data presented in Table I. The full width at half maximum (FWHM) of the (008) CdTe reflection decreases considerably from film 053 to 086 with 058 having an intermediate value. The correlation is more remarkable considering that the x-ray beam samples a larger area of film than generally observed by XTEM. TEM, however, has the further advantage in being able to delineate the specific microstructural feature responsible for film degradation.

Once it was established that high-quality films could be grown, subsequent experiments have concentrated on

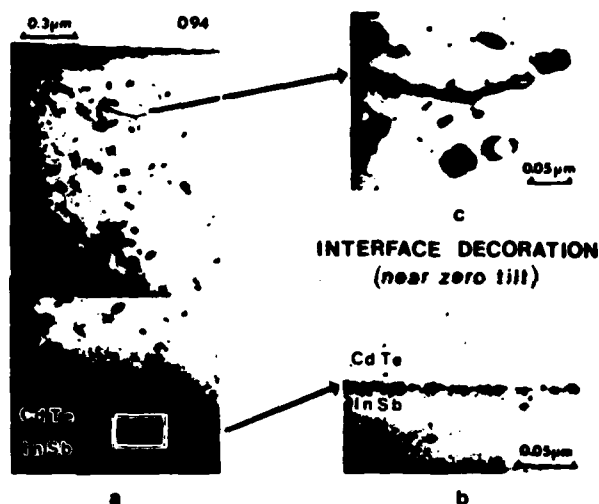


FIG. 3. BF XTEM micrographs of a relatively thick ($2.34 \mu\text{m}$) CdTe film showing (a) the defect distribution throughout the entire film thickness; (b) details of the interface structure; and (c) details of the bulk defects.

changing other variables such as growth time, growth rate, film thickness, and substrate cleaning procedures. Figures 3 and 4 show XTEM micrographs from, respectively, a thicker ($2.34 \mu\text{m}$) CdTe film (specimen 094) with a correspondingly larger growth time, and a film grown on an InSb substrate cleaned with a 1 kV ion beam instead of the more generally used 500 V beam (specimen 108). The thick film 094, contains no twins and very few dislocations, but it does have a high number density of looplike defects $<50 \text{ nm}$ in diameter. The density of these defects is low both near the InSb-CdTe interface and the CdTe surface, but for the remaining film thickness no gradation in number density is apparent. However, the size of these defects is a maximum in the center portion of the film. Further work is in progress to determine if they are dislocation loops or precipitates.

Specimen 108 (Fig. 4), grown on an InSb substrate cleaned with a 1 kV ion beam instead of the usual 500 V defocused ion beam, contains a relatively high dislocation density and very few twins. Most of these dislocations originate near interfacial precipitates, evident by the Moire fringe contrast. The small size of these precipitates and the relatively low number observed at the interface, especially in the cross-section configuration, makes their determination by either EDS or diffraction techniques difficult. However, they are probably indium rich, and their formation is due to the same ion beam induced instabilities which produce the indium-rich islands on the TEM specimens during thinning.

Specimen 094 (Fig. 3) also exhibits some sort of interfa-



FIG. 4. BF XTEM montage of a CdTe/InSb film interface showing interfacial precipitation (arrowed) and dislocation generation.

cial decoration. These defects, however, were not easily resolved precipitates as in specimen 108 and did not generate dislocations in the CdTe layer. This observation would suggest that they occurred after growth had proceeded. One explanation is that this decoration is a finer version of the looplike structure observed near the center of the CdTe film and would explain the denuded region in the CdTe near the interface. Note again that specimens 094 and 108 show intermediate values for the single-crystal rocking curves.

Chew *et al.*⁴ have observed that MBE growth of CdTe on InSb at lower temperatures than those utilized here results in highly faulted, columnar polycrystalline CdTe. In this study a very minimum amount of polycrystalline CdTe was observed in only one specimen (099) which was grown at 3–4 times the rate of the previous specimens. As shown in Fig. 5(a) these crystals were also highly faulted and columnar. These crystals did not originate at the CdTe-InSb interface but extended from a very complex defect as shown in Fig. 5(b). This defect, which did nucleate at the interface, is bounded at least on two opposing sides by a series of small microtwins. Figure 5(c) shows several examples of smaller defects of this nature originating at the interface. It is highly probable that the other two sides of these defects are composed of the remaining two sets of microtwins out of contrast in this orientation.

B. Growth of CdTe with an InSb buffer layer

Two examples of XTEM micrographs from CdTe films grown on MBE InSb buffer layers of different thicknesses are presented in Figs. 6 and 7. Both substrates were heat cleaned at $\sim 400^\circ\text{C}$ prior to InSb film growth at 380°C . Both specimens show precipitates at the InSb substrate-InSb

film interface; and, in this situation, the size and number density are sufficient to determine that the precipitates are In rich by EDS techniques. Electron diffraction suggests that the precipitates are metallic indium by showing a precipitate reflection corresponding to the c axis of tetragonal indium normal to the interface. This orientation would then allow (100) and (010) planes of tetragonal In ($a = b = 3.24 \text{ \AA}$) to be matched with the (200) and (020) planes of InSb ($a_0/2 = 3.24 \text{ \AA}$). In some locations a thin residual layer of surface contamination which defines the actual interface shows these precipitates to be contained within the original substrate. Despite the presence of these precipitates the InSb buffer layers are mostly free of extended defects, and are lattice matched across the InSb/InSb interface as shown by Fig. 8.

For both specimens the InSb buffer-CdTe interface always appeared to be sharp and smooth. No decoration or precipitation on this interface was observed, and consequently, no defects were found to be nucleated at this interface. The few dislocations observed in the CdTe layer above the interface of specimen 470 (Fig. 6) were correlated with an experimental change of growth rate of the CdTe after $\sim 0.25 \mu\text{m}$ of film growth. Problems, however did arise in specimen 470 when the residual surface contamination at the InSb/InSb interface was more extensive in localized regions. Figure 9 shows the comparison between the two interface morphologies observed in specimen 470. The foils are tilted to observe the interface more closely. In Fig. 9(a) the interface shows only precipitates with a clean interface between them, whereas, in Fig. 9(b) the interface between the precipitates is covered with a thin coating. A small denuded region is observed around each precipitate. In this latter situation twins are observed to nucleate at the InSb/InSb interface, as shown in Fig. 10. Some of the twins are completely contained within the buffer layer, but many continue to grow throughout the entire thickness of both InSb and CdTe films. Twin-dislocation interactions similar to those seen in 058 are observed. Notice that for specimen 469, in which no extended defects are observed, the x-ray rocking curve data are as good as for 086. Also this specimen received the second-

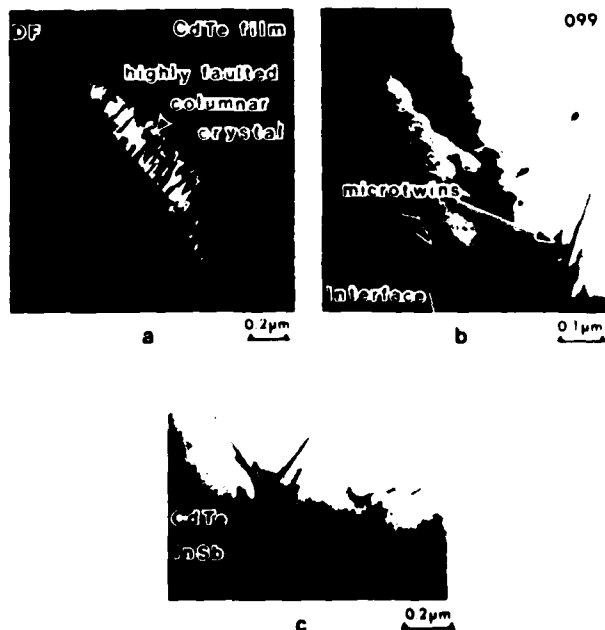


FIG. 5. XTEM micrographs of polycrystalline regions in a rapidly grown, thick ($3.77 \mu\text{m}$) CdTe film. (a) DF of faulted polycrystals; (b) complex defect at base of polycrystal near interface; and (c) other examples of complex defect nucleated at interface.

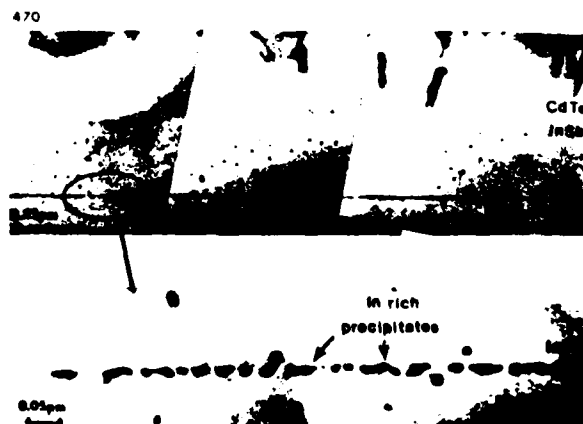


FIG. 6. BF XTEM montages of a CdTe film grown on an InSb buffer layer. Note the formation of In-rich precipitates in the heat cleaned InSb substrate surface. Dislocation generation in the CdTe film is due to a change in the growth rate.



FIG. 7. BF XTEM micrographs of a good quality CdTe film grown on a thin ($0.2 \mu\text{m}$) InSb buffer layer. The CdTe/InSb interface is clean whereas the InSb/InSb interface is defined by residual surface contamination and In-rich precipitates.

dary ion milling operation during preparation and shows no evidence of the In droplets on the InSb regions.

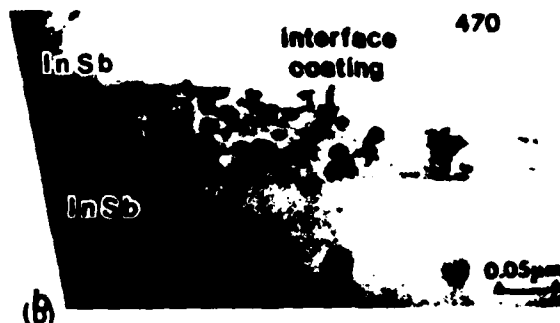
IV. DISCUSSION OF RESULTS

In this study cross-sectional electron microscopy has been employed to characterize films of CdTe grown by molecular beam epitaxy onto (001) InSb substrates. The film quality as determined by XTEM has been correlated with a number of experimental parameters such as growth rate, thickness, and InSb substrate surface preparation. A number of general comments can be made at this time concerning MBE growth of CdTe films.

One result of this work is that high-quality CdTe films, i.e., free of any extended defects, can be grown by MBE. This result in itself is significant since twin formation which occurs readily in bulk CdTe, as discussed by Vere *et al.*,¹ can be



(a)



(b)

FIG. 9. BF XTEM micrographs comparing two InSb/InSb interface morphologies in specimen 470. Interface is tilted away from vertical.

eliminated in thin films grown by MBE. Twins, however, can occur in the MBE grown CdTe films. Although the tendency for twinning in bulk CdTe may be related to the reported low stacking fault energy of $10.1 \pm 1.4 \text{ ergs/cm}^2$,⁵ twins in the MBE films depend upon the availability of suitable nucleation sites. In the case of severe twinning these sites are related to surface irregularities on the InSb substrate surface. Also, any twins in the InSb substrate which intersect the surface are replicated in the CdTe by the MBE growth process. In general, dislocations are associated with the twins—the dislocation density being proportional to the twin density. However, situations do exist when dislocations can exist alone. Two cases observed here are the nucleation of dislocations on precipitates introduced at the InSb/CdTe

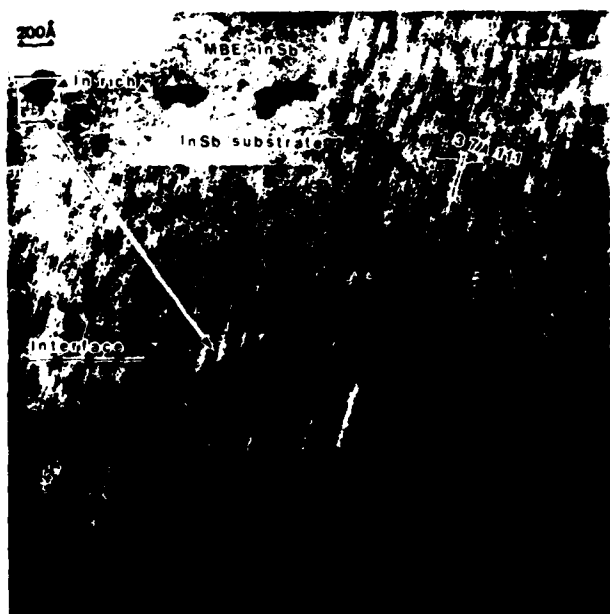


FIG. 8. High resolution TEM micrograph (lattice image) and XTEM micrograph (inset) of InSb film-InSb substrate interface with In-rich precipitates. Lattice matching occurs across the interface despite precipitate formation.



FIG. 10. Twin nucleation at the InSb/InSb interface in specimen 470 occurs only in those regions which have a surface coating between precipitates.

interface as a result of improper surface preparation and the generation of dislocations in the film incurred by a sudden change in the CdTe growth rate.

Substrate cleaning procedure and surface preparation are obviously of primary importance for growth of high-quality films. Similar conclusions have been reached by Chew *et al.*⁴ Nucleation of twins at irregular interfaces and generation of dislocations on precipitates at the interface are two consequences of improper surface cleaning previously mentioned. The columnar polycrystals observed in specimen 099 appear to be a result of the rapid growth rate employed. However, we note that these polycrystals nucleated on complex faults composed of twins which, in turn, were nucleated only at localized areas on the InSb/CdTe interface. It is believed, therefore, that adequate substrate surface preparation could eliminate these faults and thereby allow growth of high-quality films over a wider range of growth rates than achieved here. The importance of the interface in determining film quality becomes more important when CdTe films grown on ion beam cleaned InSb substrates are compared with those grown on MBE InSb buffer layers. Although high quality films were grown on ion beam cleaned InSb, adequate surface preparation depends critically on ion energies, beam geometry, and cleaning time. In comparison the InSb buffer layer/CdTe interfaces always appear to be sharp and uniform, and, consequently, no defects were observed in the CdTe which had nucleated at the interface. (The exception, of course, are the replicated microtwins which nucleated at the InSb/MBE InSb interface.)

To date, no other microstructural data from MBE CdTe and InSb films has been published, but our microscopy analysis and general conclusions are in basic agreement with the data presented by Chew *et al.*⁴ TEM analyses of the defect and precipitate structures observed in bulk CdTe have been performed⁶⁻⁸ and compared with those in ZnSe.⁹ Microtwins, extrinsic stacking faults,⁸ and interstitial, or Frank, loops⁷ have been observed by Ponce. Bean⁸ in CdTe material obtained from II-VI Inc., recorded both Te and In₂Te₃ precipitates, the former being associated with dense dislocation tangles. In₂Te₃ particles were identified from Moire fringe spacings. In Bean's study⁸ vacancy loops were only observed after ion implantation. Thus, the types of defects observed in the MBE films, when present, are consistent with those in bulk material.

Although, the precipitates associated with the heat cleaned InSb/InSb interface are either In or In-rich In-Sb compounds, those observed on the ion cleaned InSb/CdTe interface could be the In₂Te₃ type observed by Bean. Their analysis in the present case is complicated by the low number density along the interface. Similarly, the looplike defects present in specimen 094 are most likely interstitial dislocation loops—possibly containing segregated Te. Further characterization will address this question. We note, however, that this film provides the single example in which the film quality was not determined predominantly by the nature of the InSb/CdTe interface.

It should be noted that the authors had some initial concern about the introduction of extended defects in the MBE film due to stresses generated by sample preparation

for XTEM. It is now apparent that XTEM samples with very low extended defect densities can be prepared by the techniques employed, as evidenced by the microstructures of films 086 and 469. Microstructural data also correlates well with single-crystal x-ray rocking curve broadening. Furthermore, as discussed by Vere *et al.*,¹ twins are not generally introduced by post-growth mechanical deformation, that is, they are not strain induced. Instead, they nucleate and propagate during growth. Some plastic deformation (dislocation motion) can occur, however, at room temperature, but in the samples evaluated in this work, this was prevented by maintaining adequate sample thickness prior to the final ion milling operation. Thus, we conclude the XTEM does yield a true representation of the microstructures of these films, but several specimens should be examined to identify any inhomogeneities across the original wafer. This was achieved in this work by mounting several specimens and evaluating in a comparative fashion. The variation in specimen 470 elucidates the misconception which might have arisen, had only one small portion of the wafer been examined.

A brief comparison with the ZnSe/GaAs system is also worthy of note since this is the only other example of growth of a II-VI compound on a III-V substrate for which XTEM data has been reported.⁹ Ponce has examined the defect structures of highly doped ZnSe epitaxial layers grown by organometallic CVD on (100) GaAs substrates and observed Frank-type interstitial loops near the interface and in the bulk using HRTEM. In contrast, layers grown on (111) substrates exhibited a large twin density. Since this system has a much larger mismatch ($\Delta a/a = 2.5 \times 10^{-3}$ at R.T.)¹⁰ than the CdTe/InSb system, it is not clear how to interpret these comparative results at present. Furthermore, details of the substrate cleaning procedures, etc., are not known. It is of interest, however, that similar extended defects can be observed in the two systems.

V. CONCLUSIONS

(1) Near-perfect epitaxial CdTe films can be grown by MBE on InSb substrates with or without an InSb buffer layer, but film quality is critically dependent upon substrate cleaning.

(2) Effective substrate cleaning can be obtained at an ion energy of 500 V: higher energies can produce precipitation and subsequent dislocation nucleation during MBE growth. Residual irregularities on the InSb substrate surface leads to microtwin nucleation at the InSb/CdTe interface. Dislocations are generally associated with these microtwins.

(3) Other factors which induce defect formation in the MBE CdTe films include high growth rates, long growth times, or a change in growth rate during film growth. Extended defects formed include twins, line dislocations, or looplike defects. Defects in the InSb which intersect the surface, such as microtwins, are replicated in the CdTe during MBE film growth.

(4) Heat cleaned InSb substrates can produce In precipitates in the surface layer which does not, however, induce defect formation in the InSb buffer layer. Twin nucleation does occur at this interface if the indium is more uniformly

distributed (i.e., coated) over the substrate prior to film growth.

(5) Lattice matching across the InSb MBE film/InSb substrate interface has been demonstrated.

(6) The defects delineated in the MBE CdTe films are consistent with other analyses in bulk CdTe.

(7) The film quality observed by XTEM is in excellent agreement with the single-crystal x-ray rocking curve data.

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