

# Growth and recovery of $\alpha$ -Sn on InSb(001) using He scattering and LEED

B.F. Mason and B.R. Williams

*Institute for Microstructural Sciences, National Research Council of Canada, Ottawa, Ontario, Canada K1A 0R6*

Received 13 July 1990; accepted for publication 27 August 1990

The deposition and growth of pseudomorphic  $\alpha$ -Sn has been studied on InSb(001) using He scattering and LEED. The InSb gave a  $(1 \times 4)$  reconstruction which changed to a  $(1 \times 1)$  structure for a monolayer of  $\alpha$ -Sn. The growth process was followed using the intensity of the  $(00)$  He peak as a function of coverage (as in RHEED) and the shapes of these curves were used to interpret the events. Recovery curves (for different coverages and surface temperatures) show times similar to those observed by RHEED and the times indicate that growth and recovery are concurrent phenomena. Recovery curves for very low coverages  $\sim 0.5\%$ , are thought to observe the process of local reconstruction towards the  $\alpha$ -Sn structure. Finally, the effects of ion bombardment on InSb as well as the deposition of  $\alpha$ -Sn are briefly presented.

## 1. Introduction

There has been worldwide intense interest in the properties of semiconductor surfaces as well as in the deposition and growth of several types of material layers on such surfaces, e.g., semiconductors, insulators and metals. Several reasons account for such diverse interest. They include, amongst others, the different phenomena in growth behaviour at low coverages, the influence of small amounts of metal on the electronic properties of semiconductors and interest in the adsorption of gases for capping or catalysis purposes. In this study experimental findings are presented for the deposition of a metal, Sn, on InSb(001) single crystal which forms a good lattice match to InSb in the tetrahedral  $\alpha$ -Sn metastable phase. The knowledge of the stability of this phase is of interest at lower coverages than has been studied to date. The study uses the technique of He scattering and LEED to yield structural, and growth properties of the chosen system, at low coverages. Interest in any system starts with the structure or particular reconstruction of the substrate upon which growth is to occur. In the case of InSb(001) many reconstructions have been

noted [1] depending on either the method of preparation or the overpressure of In or Sb and much effort is directed to the understanding [1] of these different structures. In this particular instance we follow the early procedure of Farrow [2] and others [2c,2d] which involved ion bombardment and anneal cycles. Disagreement in the reconstruction of the InSb substrate and the absorbed layers of  $\alpha$ -Sn has been observed between workers using RHEED techniques. RHEED yields either a  $(2 \times 4)$  or a  $c(2 \times 8)$  structure but two other studies using LEED [2d] have shown a basic  $(1 \times 4)$  structure with streaks in the  $\times 2$  position so that disagreement also occurs depending on the techniques used. Farrow was able to grow films of  $\alpha$ -Sn in a metastable state up to thicknesses of  $0.5 \mu\text{m}$  and which remained stable to about  $60^\circ\text{C}$  above the bulk  $\alpha \rightarrow \beta$ -Sn transition of  $\sim 13.2^\circ\text{C}$ . The subject of the growth of metastable surface phases has been more recently reviewed [3a]. In the present case we have concentrated on the early stages of growth ( $\sim 1\%$  monolayer up to 5 monolayers) using He elastic scattering and LEED as opposed to the more traditional RHEED technique. The former techniques act as a complement to RHEED and can be expected to yield ad-

ditional information on the surface structures and the concurrent growth processes. He atoms have been used recently [4] to study metal deposition on both similar and dissimilar metal substrates where oscillations in the (00) peak were observed as in many instances of deposition carried out using RHEED patterns. It is thought that He scattering might not be as plagued by multiscattering effects as is RHEED. In the present case however, we have applied He scattering, probably for the first time, to the deposition of a lattice-matched metal on a III-V semiconductor with concurrent reconstruction, i.e., the flux was low enough that deposition and reconstruction of the new selvedge were occurring simultaneously.

By interrupting growth and observing the slow recovery of RHEED patterns much insight can be obtained about the growth process itself [5]. In fact, it is through that such experiments could well yield more useful information than morphological observations [5,6]. Similar recovery experiments have been attempted in this study using very low coverages ( $\sim 1\%$ ) up to monolayer values.

The choice of Sn deposition was prompted by several factors: (1) like CdTe, InSb is very closely lattice-matched to  $\alpha$ -Sn; (2) the metastable  $\alpha$ -Sn was considered to have potential for interesting thickness dependent band-gap devices [3b]; (3) the study of the stability of the  $\alpha$ -Sn at low coverages is of interest and finally [4] the work can be carried out at room temperature to  $\sim 200^\circ\text{C}$  – a range where the elastic (00) peak for He is still adequately larger than background inelastic effects.

The arrangement of the paper is as follows: After a description of the experimental arrangement the results will be presented and discussed. Diffraction patterns for He scattering along with LEED will be discussed so that the particular reconstruction is established. (The authors have already presented such studies at low temperatures – from the oxide layer to the cleaned surface [7].) Then the variations of the He (00) peak intensity with deposition will be presented in an attempt to understand the mechanisms of growth at different stages of coverage. Recovery times for various temperatures and coverages will then be discussed and compared with RHEED

results and finally the effect of ion bombardment on reconstruction and deposition will be shown.

## 2. Experimental

The beam generating system is similar to that used in our previous low temperature atom scattering experiments [8]. Changes were made to the UHV chamber to accommodate high temperature treatments of semiconductor specimens. The detector was changed to a modified (Extrel) quadrupole mass spectrometer. The line of sight of the fixed detector to the sample was at  $90^\circ\text{C}$  to the incident beam and the crystal was rotated to change scattering conditions. The beam was chopped by a wheel with four slits at 300 Hz and was located 50 cm from the sample. The sample-detector distance was 109 cm. Time of flight data was obtained using a multichannel scaling board (EG and G Ortec) installed in a computer (Zenith). The UHV chamber was pumped with a turbopump (Varian V1800) and the detector chamber was pumped by a smaller similar pump (Varian V200). An intermediate chamber (installed between the UHV and detector chambers) served to reduce the background for a better signal to noise ratio.

The LEED apparatus was a back reflection instrument (Princeton Inst.) and was operational at a distance of 10 cm from the He beam plane along the axis of the manipulator. An Auger analytical spectrometer (V.G.) was located at a similar distance.

The Sn source (99.999%, CERAC INC.) was made of a Mo container and could be heated to the temperature range of interest for evaporation ( $800$ – $900^\circ\text{C}$ ) by electron bombardment. The source was situated at about 30 cm from the sample while the flux was measured with a quartz microbalance. Source temperatures were arranged to yield monolayers in the range of 20–200 min for accurate small doses. No contamination of the Sn layers was observed, within the sensitivity of the AES.

Prior to loading, in the UHV chamber the InSb the sample was dipped in a CP-4A etchant [7] for

2 min to remove any polishing damage. The surface was cleaned in situ by ion bombardment and anneal cycles (500 V Ar ions for 1 h at  $\leq 1 \mu\text{A}/\text{cm}^2$ ; anneal, 40 min at  $\sim 300^\circ\text{C}$ ). Sn layers were likewise removed after deposition. During the whole set of experiments the diffraction pattern for a given incident energy did not change appreciably indicating that ion bombardment damage was not a major variable or that islands of In which are known to form on the surface did not become a parameter on the scale of the diffraction technique, e.g., 100–200 Å.

### 3. Results and discussion

Before proceeding with a discussion of the results it is useful to describe briefly the surface interactions that influence atom scattering and render it a very surface sensitive tool and a useful addition to the study of deposition on semiconductors. The He atom is about 3 Å in diameter (a large probe) but scatters exclusively from the surface by the repulsive potential developed as the atoms electrons overlap the surface electronic structure. The attractive portion of this van der Waals interaction does not play a major role in deflecting the He atom except in the vicinity of a step or a polarized molecule or atom on a terrace. If, during reconstruction the electronic environment of the surface is changed locally, then such a situation could also change the scattering cross section and influence peak intensity. Because of its physical dimension the probe atom is comparable to steps on the surface, kinks in steps and vacancies on terraces so that intuitively it is seen that the interplay and development of such features, during growth and recovery for instance, will influence scattering considerably. Semiconductors, as a group of materials, have basically relatively open structures so that, not only is the spacing between rows of atoms comparable to the He atom (and its wavelength  $\sim 1 \text{ \AA}$ ) but the depth of the corrugations are relatively large. This leads to a scattering pattern with many diffraction peaks [9] (relative to, e.g., close packed smooth metals).

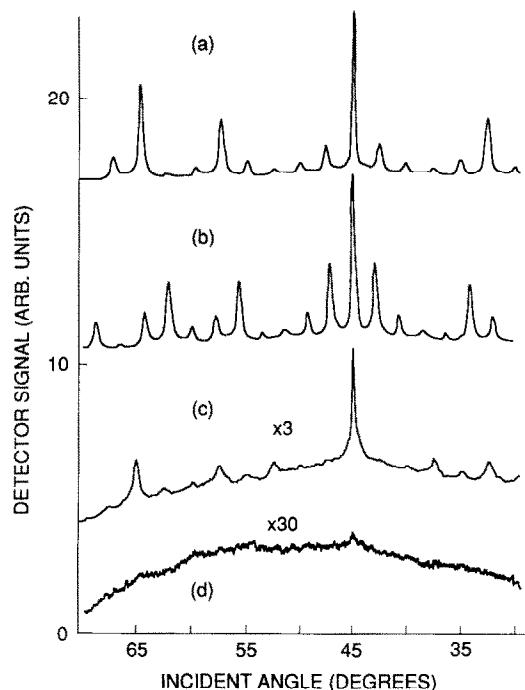


Fig. 1. (a) and (b) show the diffraction pattern for InSb(001) after ion bombardment and annealing in the (110) direction for beam energies ( $E_0$ ) of 16 and 20 meV respectively. Many peaks are detected at this surface temperature (RT) such that rainbow effects are visible. The reconstruction in this direction is  $\times 4$  the bulk lattice. (c) and (d) show the changes in the diffraction patterns for  $E_0 = 16$  meV when a coverage of 0.4 and 1, respectively, of  $\alpha$ -Sn has been deposited. No diffraction peaks are visible for a coverage of unity.

Figs. 1a and 1b show the diffraction pattern from InSb(001) in its reconstructed form after ion bombardment and anneal cycles to remove the oxide layer. As mentioned earlier, many reconstructions have been observed [1] and the structure reported here is different from the  $(2 \times 4)$  structure observed by Farrow et al. [2a] during experiments on  $\alpha$ -Sn layers and using RHEED to analyze the results. Höchst [2c], using similar techniques (RHEED, etc.) observed a  $c(2 \times 8)$  reconstruction. However, more recently and employing LEED techniques, Jones et al. [2d] have observed a different reconstruction to the above. This group observed a structure which was basically  $(1 \times 4)$  with streaks in the  $\times 2$  positions. Figs. 1a and 1b indicate, as did our study at low temperatures, a  $\times 4$  reconstruction in the  $\langle 110 \rangle$  both for an energy

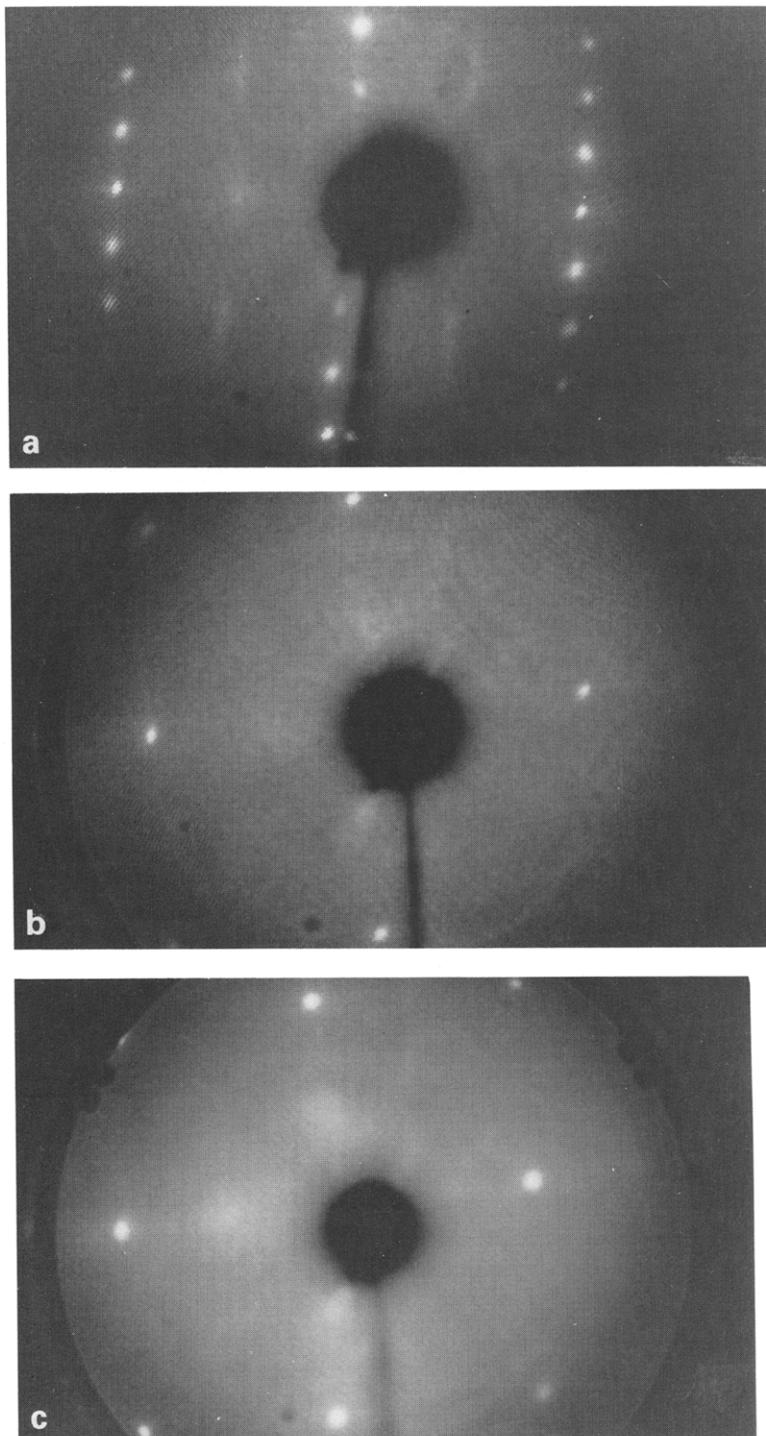


Fig. 2. LEED patterns (a) for clean InSb giving a  $(1 \times 4)$  reconstruction,  $V = 48$  V (b), for a monolayer of  $\alpha$ -Sn showing a  $(1 \times 1)$  reconstruction,  $V = 43$  V and (c) for 5 layers of  $\alpha$ -Sn,  $V = 45$  V. The voltage range for well defined  $(1 \times 1)$  spots was much wider in this latter case.

of 16 meV (a) and 20 meV (b). As it was not possible to rotate the sample azimuthally in situ, LEED was used to yield the general structure in the out of plane directions. Such a LEED pattern is shown in fig. 2a where it is clear that the predominant structure is a  $(1 \times 4)$  reconstruction. For a very narrow energy range extra very broad streaks were obtained in the  $\times 2$  positions similar to Jones et al. but they were also very weak and suggest that the electrons in this instance are interacting with an arrangement of longer range order such as steps. Jones et al. interpret the differences in the following way. They associate the streaks with those of RHEED which would give the same result (i.e., streaks) in the  $\times 2$  position at glancing incidence. Nevertheless, they describe the structure as either a  $(4 \times 1)$  or a  $(4 \times 2)$  with one-dimensional disorder. The ion bombardment and anneal conditions for all these studies are not very different so that it is not likely that nuclei of In on the surface could cause the differences. Perhaps these results emphasize the difficulty in assigning a definite reconstruction without taking the intensities of many diffraction peaks over a wide range of incident energies and comparing the results theoretically with a model. Returning to figs. 1a and 1b it can be seen that the He patterns are remarkably symmetrical about the  $(00)$  position for both energies and the grouping of intensities around the  $(00)$  and other diffraction peaks suggest the commonly observed rainbow features which in principle can yield structural information for the surface provided the intensities can be measured over a wide range of incident conditions. Changes in the relative heights of the diffraction peaks provide a sensitive measure of any changes to the corrugation of the surface such as occurs on adsorption.

Figs. 1c and 1d show the diffraction pattern for a coverage ( $\theta$ ) of  $\alpha$ -Sn of 0.4 and 1 respectively. It is seen that by  $\theta = 0.4$  the signals have fallen considerably but the  $(1 \times 4)$  structure is still visible. This feature indicates that the surface is becoming covered with a layer of single thickness as opposed to three-dimensional nuclei. LEED, being slightly less surface sensitive, gives at  $\theta = 0.4$  also a  $(1 \times 4)$  structure but with a greater emphasis on the  $(1 \times 1)$  reconstruction that finally emerges at

full monolayer and even more prominently (the spots are apparent over a greater range of incident electron energies) at about 5 monolayers (figs 2b and c). This result is again in disagreement with Farrow and Höchst who observed a  $(2 \times 2)$  and  $(2 \times 1)$  reconstruction respectively for  $\alpha$ -Sn layers up to a thickness of  $0.4 \mu\text{m}$  although very broad features can be seen in the  $\times 2$  positions for 5 layers. Interestingly, Jones et al. also find a conversion of the  $(1 \times 4)$  to a  $(1 \times 1)$  structure on the adsorption of both  $\text{I}_2$  and  $\text{Cl}_2$  as in the present case of  $\alpha$ -Sn and H atoms (see below). Unfortunately, atom scattering was not able to shed light on this matter because the diffraction patterns continuously decreased in intensity as  $\theta$  increased to yield only a broad scattering pattern, fig. 1d, which did not change further in the multi-layer region. This is an unusual result for atom scattering except for the adsorption of some gases at low temperature [10]. The answer most likely arises as a result of the large Debye-Waller effect of a heavy relatively loosely bound atom at room temperature as energy analysis of the layers, fig. 3

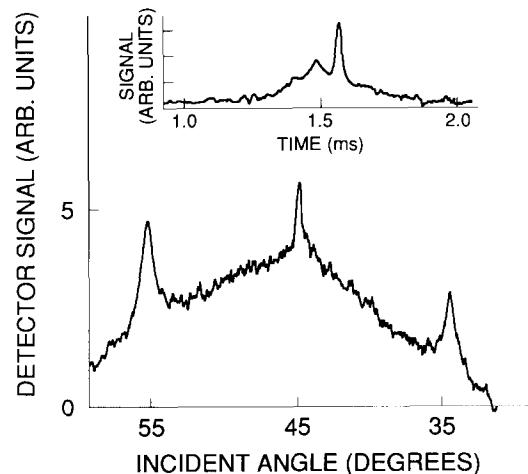


Fig. 3. He scan for H atoms (generated from a tungsten filament) adsorbed on InSb to give a monolayer reconstructed to a  $(1 \times 1)$  structure. This shows that the basic substrate is sufficiently flat to yield diffraction peaks for an adsorbed monolayer. The inset is a time of flight scan for a monolayer of  $\alpha$ -Sn. It shows that, although some elastic incoherent scattering is present (the sharp peak) the scattering yields no well defined single phonon processes. The result shows mainly multiphonon events where the surface has predominantly given energy to the He atom.

(inset), yielded only multiphonon scattering along with some elastic incoherent component. It is difficult to accept that the  $\alpha$ -Sn monolayer becomes completely rough as the LEED pattern shows well defined and well ordered spots. In contrast hydrogen atoms also yielded an ordered reconstructed  $(1 \times 1)$  monolayer as seen by LEED but, in this instance, He scattering gave small but well defined (00) and first order diffraction peaks – see fig. 3. It is possible that alloying of the Sn can occur by cation exchange as this effect is well known in II–VI compounds. This has not been mentioned as a problem in other studies of  $\alpha$ -Sn–InSb or  $\alpha$ -Sn–CdTe. The only reference to a film not having a sharp interface of  $\alpha$ -Sn is that by Farrow where a small fraction of  $\beta$ -Sn nuclei form at defects at the interface (for thicker films).

As referred to earlier the Sn atoms when adsorbed on InSb (and CdTe), they are constrained by the substrate into pseudomorphic growth due to the close lattice matching which minimizes the development of elastic strain. Farrow discovered that thick films grew quite perfectly and were stable against the transition to  $\beta$ -Sn up to 70 °C. It was our objective to discover the stability of the monolayer films to higher temperatures. Heating to 200 °C did not influence the properties of the film as evidenced by LEED and He scattering and deposition of the film up to temperatures of 160 °C yielded the same LEED  $(1 \times 1)$  reconstruction from  $(1 \times 4)$ . Such stability of the first layer to high temperatures has been observed also for Sn on Si(100). Furthermore, the He scattering was still broad and diffuse on cooling the films to room temperature suggesting that the scattering behaviour is an intrinsic property of the  $\alpha$ -Sn layer rather than a rough surface. (LEED would also yield diffuseness if a rough surface was causing the effect.) On adsorption of Sn atoms several processes are occurring concurrently. First, the atoms are mobile enough at room temperature to form two-dimensional nuclei and reach existing steps on the surface to produce an ordered layer. The mobility could possibly be described as being ballistic but most probably the atoms have to surmount a surface barrier, especially at the lower temperatures used – thus favoured sites for reconstruction probably exist. Further, the chemical

forces cause a reconstruction of the surface within times (for the given incident flux) comparable to the rate of increase of  $\theta$  (see below for discussion of recovery curves). Finally, the chemical bonding is stable to higher temperatures, once established. It is also clear from the LEED behaviour that the films grow layer by layer as opposed to developing three-dimensional nuclei after having grown a monolayer.

We have studied the low surface coverage regions in an attempt to shed some light on the growth process using low fluxes of Sn atoms and He scattering. The coverage was measured using a

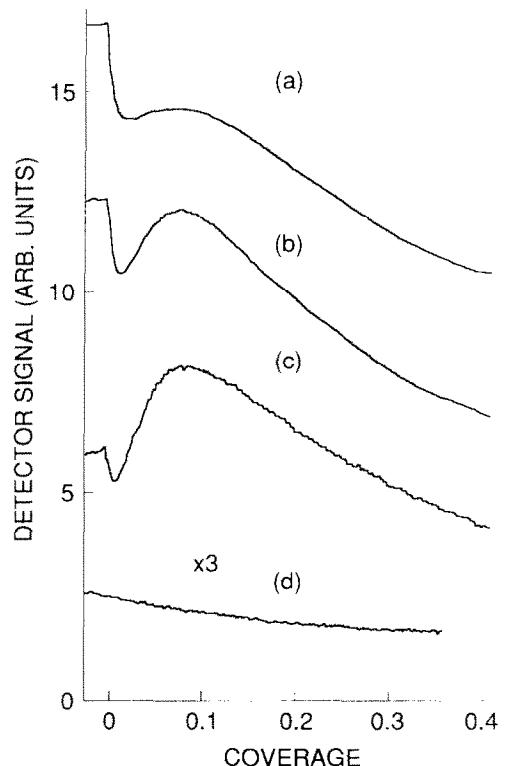


Fig. 4. Three curves that represent the fall of the (00) peak ( $E_0 = 16$  meV) for three different surface temperatures, (a) 36 °C, (b) 82 °C and (c) 150 °C respectively. Each result shows an initial rapid fall in the peak height at low coverages, then a broad maximum at a coverage of 8% as growth proceeds. The ratio of the initial (00) to the broad peak falls as the temperature increases suggesting an influence of increased lateral mobility. The curve gradually decrease after a coverage of 0.1 due to the changed nature of the surface yielding a large Debye-Waller factor. The part of curve (c) near monolayer coverage is shown in (d).

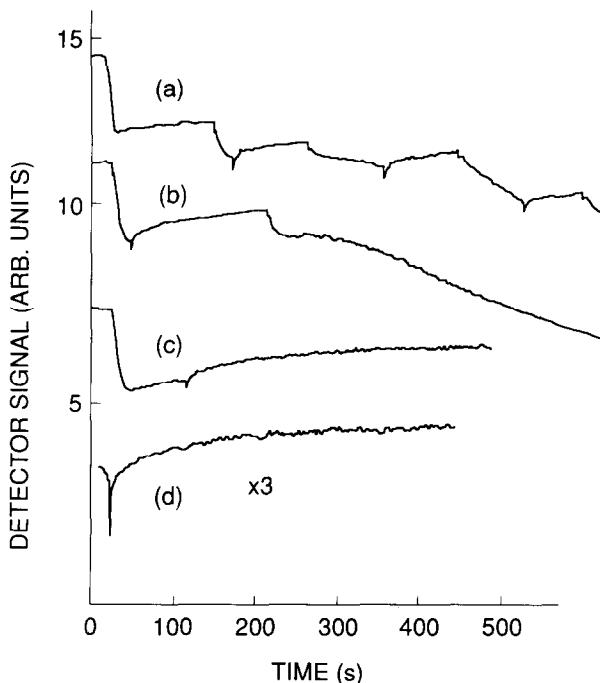


Fig. 5. Portions of curves seen in fig. 4 but where growth has been interrupted at different coverages to observe the recovery process. (a) Interruption and recovery sequentially for  $E_0 = 20$  meV at room temperature; (b) interruption and recovery at the minimum of curve 4a ( $E_0 = 20$  meV); (c) similar results at the maximum at a coverage of 8% ( $E_0 = 20$  meV,  $T_s = 56^\circ\text{C}$ ); (d) the case for  $E_0 = 16$  meV and  $T_s = 73^\circ\text{C}$ . Results (b), (c) and (d) suggest, like RHEED, that two regions of recovery exists but dependence on coverage is not marked.

quartz microbalance as well as the disappearance of He diffraction peaks. In fig. 4 the source was adjusted to yield a monolayer in 20–23 min so that in 1 min only 5% of the surface was covered. In 1 min at least 50% of the final reconstruction is complete as can be seen from the recovery curves in fig. 5. Fig. 4 shows the variation of the (00) peak height as a function of the exposure to Sn atoms (sticking coefficient assumed to be unity) for different surface temperatures. Such curves are similar to those found in RHEED work to follow MBE growth where oscillations are observed as a function of step edge concentration (roughness) as islands grow and aggregate to form monolayers sequentially. Such oscillations have been observed using He scattering [4] where the authors claim, from Monte Carlo simulations and experiment,

that the results can be treated kinematically and not be confused by multiple scattering effects as seems to be the case for RHEED. However, the current situation is not exactly equivalent as pointed out above. It is seen that in all the curves of fig. 4 an initial sharp drop occurs at very low coverage – less than 1% in all cases. The sharpness of the fall for the amount of Sn adsorbed indicates a very large cross section for deflecting He atoms in this dilute region of adsorption. Such a large cross section could not arise from the physical size of the Sn atoms blocking (or shielding) the He probe – a fact known from adsorption studies. The result is similar to that observed for CO–Cu (110) where similar sharp decreases were observed [12]. Such results have been attributed to isolated atoms or molecules (as opposed to small nuclei) having been polarized so that both the repulsive and attractive parts of the He atoms interaction with the surface region have to be taken into account. Such situations can lead to very large scattering cross sections. Mobile isolated atoms can also lead to a similar observation.

Therefore it is postulated here that in this dilute, low  $\theta$ , region that the Sn atoms are not only laterally mobile but are active chemically to a degree that a new electronic environment is being created with the result that charge transfer can occur. This might occur at preferred sites on a terrace or after migration to a step or kink.

The next region of interest in the curve is a broad maximum, initiating from the dilute phase and reaching about  $\theta = 0.2$  depending on the surface temperature. The maximum, relative to the initial (00) peak increases in height as temperature increases. In this denser surface region the atoms can begin to interact to form two-dimensional nuclei, a process that reduces the total cross section compared to isolated randomly located atoms. This is a view incorporated in theories that attempt to interpret RHEED oscillations as well as atom scattering oscillations. In this denser region atoms are also on average closer to existing steps on the surface, into which they can be incorporated and proceed with reconstruction. Although the maximum is always at  $\theta \sim 7\text{--}8\%$ , it increases in height with temperature suggesting that the mobility of the atoms does play some role in the

growth. The only observational change that can be observed in the continuously falling diffraction peaks is a brief very small increase in the  $\times 2$  peak suggesting some degree of ordering at this stage in the reconstructing layer. This feature was observed at the maximum in the curve. The final phase in the development of the monolayer is the continued deposition of Sn atoms to yield a surface which is reconstructing to  $(1 \times 1)$   $\alpha$ -Sn and as discussed above is a poor scatterer of He atoms and the (00) falls as in many adsorption systems studied. The whole of the above is equivalent in time to one oscillation in RHEED experiments and the regrettable part of this system is the decline in the signal at a monolayer. This feature makes other systems worthy of study – those that do not yield a metastable phase and soft modes of vibration. A final conclusion can be made – being that the  $\alpha$ -Sn is deposited as a single layer without the formation of Volmer–Weber type nuclei.

Fig. 5 demonstrates the nature of the recovery process mentioned above and is the subject of active theoretical interest currently [4–6]. The technique involves intercepting the Sn beam after particular values of  $\theta$  have been reached and observing the subsequent change in the (00) peak. The small vertical changes in the signal at these points result from the He beam being briefly interrupted at the same time.

Fig. 5b shows a recovery curve just before the minimum of a curve like that in fig. 4a, except the beam energy was  $\sim 20$  meV in this case as opposed to 16 meV. The shape would seem to indicate that, as has been found by RHEED [14,15], an initial faster recovery region exists followed by a much longer reordering phase. Fig. 5c shows a similar recovery curve for the same energy but at  $\sim 50^\circ\text{C}$ . The result is somewhat similar although the interruption was made at  $\theta = 8\%$ , i.e., the maximum in the curve and as a result in a region having a slightly different surface environment. In either case within 30–60 s half of the recovery is complete, i.e., times not dissimilar to those found for RHEED. Fig. 5d shows a recovery curve taken at higher coverage,  $\theta = 0.4$  for an energy of 16 meV where there might be an indication that the initial recovery is slightly slower. Finally, referring back, Fig. 5a shows a composite of recovery curves

taken in sequence starting at very low coverage. Reviewing this data it is felt that differences in the initial recovery do not seem to vary significantly with coverage in this instance. However, as opposed to RHEED it must be borne in mind that scattering cross sections for He scattering including shadowing effects and changing Debye–Waller effects would have to be incorporated into any interpretation and that direct comparison is not necessarily proper.

In an attempt to separate out the influence (presence) of many atoms at high coverages the temperature of the source was lowered to yield a flux ten times less than used up to the present. By

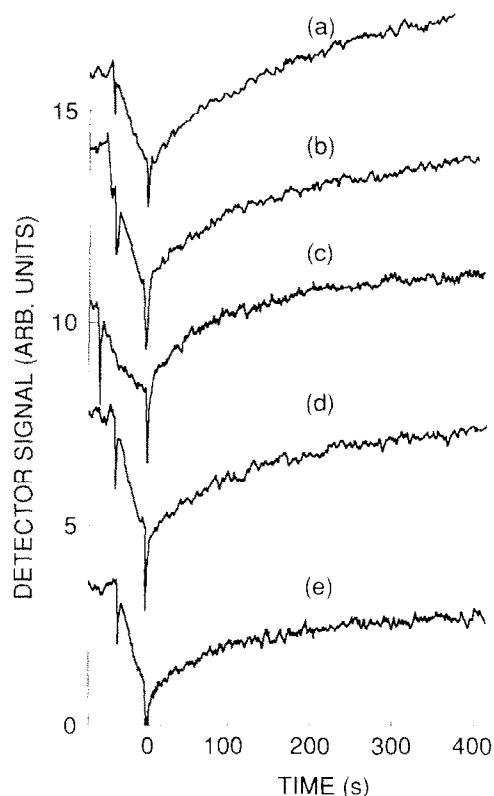


Fig. 6. The results of recovery in the dilute region of adsorption of  $\alpha$ -Sn. The Sn flux was reduced so that doses of  $\sim 0.5\%$  of a monolayer could easily be deposited. The (00) signal was suppressed and detection at lower level accomplished. The results are for surface temperatures ranging from  $160^\circ\text{C}$  down to near room temperature. (a)  $160^\circ\text{C}$ , (b)  $118^\circ\text{C}$ , (c)  $75^\circ\text{C}$ , (d)  $43^\circ\text{C}$ , and (e)  $34^\circ\text{C}$  ( $E_0 = 16$  meV in all cases). It is postulated that the recovery process represents local restructuring at this dilute level of coverage.

suppressing the (00) signal and looking into the background it was possible to produce small doses equivalent to  $\theta < 0.5\%$  and then follow the recovery process in the dilute region where isolated atoms, which probably induce charge transfer, are observed as indicated by the rapid initial fall. The results are shown in fig. 6 as a function of surface temperature ( $160^\circ\text{C}$  to RT). It would be expected that the mobility of the Sn atom might be reflected in the results, however unlike the dramatic time changes in RHEED results as a function of mobility we were not able to observe sufficiently variable results with temperatures to be significant, the recovery process being very uniform in this range of  $\theta$ . The current thought on the two stages of recovery [5] is that initially dendritic effects, i.e., kinks and small protrusions are annealed out rapidly, after which longer time effects such as filling in depressions and the overall decreasing of the perimeters of islands account for intensity changes. The results here, where the coverage would only be in the dilute region would not fit that scenario as it is unlikely that individual Sn atoms would have the chance to form small nuclei before encountering a preferred site for initiating reconstruction or join an existing step. Consequently, it is possible that what is being observed primarily in this dilute region is the actual change from an adsorbed atom (inducing a large cross section) slowly being incorporated into the top terrace at preferred sites as one process and the absorption into existing steps as the other possibility. The distance to a preferred site can only be of the order of a lattice separation or two. However, the distance to a step site can be many times more, rendering the former process as being more likely as the initial process. The slow migration of a "localized defect" to lower its energy by moving to a step can then account for the longer times of recovery. If this is so it is much related to the lattice matching and the subtle extra chemical driving force of the lattice to constrain the Sn into the  $\alpha$ -Sn cubic form rather than the  $\beta$  tetragonal form. It is also of interest to note that even for the very lowest flux depositions of Fig. 6, on continued deposition of  $\alpha$ -Sn a maximum was still observed in the (00) peak height. This means that even with a factor of ten more time to move about

on the surface and reconstruct relative to the rate of impingement the same phenomena were observed. At the other extreme of higher fluxes (such as used by Farrow and others to make thick films, it is a wonder that the lattice of the newly created

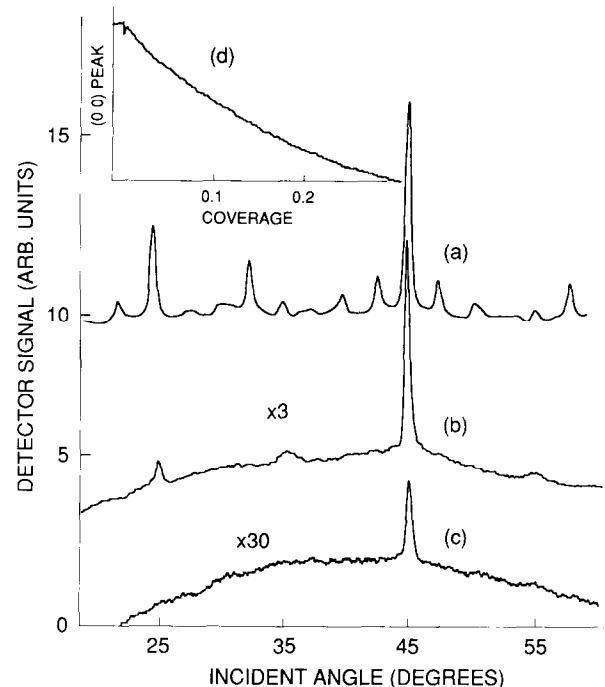


Fig. 7. The influence of ion bombardment on InSb and the deposition process. (a) Diffraction scan similar to fig. 1a ( $E_0 = 16$  meV). The influence of extended bombardment and anneal cycles is to make two relatively small changes: (1) the (00) peak gains in height relative to the diffraction peaks and (2) some diffraction peaks broaden or distort. The symmetry about the (00) peak is still maintained. (b) The effect of ion bombardment without a subsequent anneal cycle. The (00) has fallen by only a factor of 3 but the diffraction peaks have all been removed except for two broad first order peaks (giving a  $1 \times 1$  reconstruction) and a sharper second order peak. LEED confirmed the  $(1 \times 1)$  ion bombardment induced rough surface. Deposition of Sn on this surface is shown in curve (d) where it is clearly seen that a rough surface changes the nature of the growth process. A recovery mechanism was still visible at monolayer coverage (not shown). (c) He scan for the Sn monolayer. It is similar to the  $\alpha$ -Sn layer (fig. 1d) except a better defined (00) peak is visible.

film becomes sufficiently relaxed to grow defect-free films.

Finally, in this study, it was decided to measure the effects of ion bombardment on the deposition process. Others have failed to produce  $\alpha$ -Sn on either contaminated or ion-bombarded surfaces. The effect of prolonged ion bombardment and anneal cycles on the InSb substrate is shown in fig. 7a. (We could not measure the total number of ions needed to reach this condition.) The effect is not very striking which lends confidence to the results above. If the pattern is compared to fig. 1a, two effects are apparent: (1) the (00) peak has gained height relative to the diffraction peaks, and (2) some orders of diffraction peaks (in the minority) have broadened or distorted on one side. Broadening of peaks is a general trend when loss of longer range order of a particular nature is developed. Fig. 7b is very interesting and shows the effect of the standard ion bombardment cycle but without anneal. It shows a definite diffraction pattern with a sharp (00) peak, broad first order ( $1 \times 1$ ) peaks and a sharp second order peak. In other words although the pattern is weaker (the (00) has fallen by a factor of  $\sim 3$ ) the ion bombardment has induced at least a partial restructuring to the ( $1 \times 1$ ) configuration. This fact is confirmed by LEED which like the He background scattering, shows a bright background due to spurious scattering. Compared to the ion bombardment of metals [16] the result is entirely different. For metals the (00) peaks fall exponentially with  $\theta$  and broaden considerably, e.g., by a factor of 3 with the base of the peak broadening much more due to elastic incoherent scattering.

In fig. 7d however, the effect of the induced damage becomes apparent when Sn was deposited. The shape is entirely concave to the abscissa and the features of (a) initial rapid fall and (b) a maximum at  $\theta = 8\%$  were not present. Nevertheless, a recovery curve was still observed after the deposition of a monolayer with the relaxation time being of the same order as the previous films. The other interesting feature in this process is found in fig. 7c. Although the He scattering from the Sn layer is still broad, cf. fig. 1d, a sharp (00) can be observed. LEED still gave a very diffuse ( $1 \times 1$ ) structure for the Sn monolayer. The con-

clusion is that it is basically easy to form  $\alpha$ -Sn layers on InSb.

#### 4. Summary

A study has been made of InSb(001) using He scattering and LEED. Pseudomorphic films of  $\alpha$ -Sn have been deposited as a function of temperature. The process of adsorption has been followed from the very dilute phase up to 5 monolayers and attempts made to understand the phenomena. Recovery curves were observed for all coverages up to a monolayer but the results did now show a marked dependence on coverage. Very small doses of Sn ( $\theta < 0.5\%$ ) were deposited at different temperatures and the recovery in the dilute stage monitored. It is thought that the process of reconstruction is being followed for the small number of non-interacting atoms. The effects of ion bombardment on InSb has been observed to reconstruct the surface to a ( $1 \times 1$ ) structure but it has the effect of modifying the adsorption process.

#### References

- [1] A.G. de Oliveira, S.D. Parker, R. Droopad and B.A. Joyce, *Surf. Sci.* 227 (1990) 150.
- [2] (a) R.C.F. Farrow, D.S. Robertson, G.M. Williams, A.G. Cullis, G.R. Jones, I.M. Young and P.N. Dennis, *J. Cryst. Growth* 54 (1981) 507;  
(b) R.F.C. Farrow, *J. Vac. Sci. Technol. B* 1 (1983) 222;  
(c) I. Hernández-Calderón and H. Höchst, *Phys. Rev. B* 27 (1983) 4961;  
(d) Robert G. Jones, Nagindar K. Sing and C.F. McConville, *Surf. Sci.* 208 (1989) L34;  
D.P. Woodruff and K. Horn, *Vacuum* 33 (1983) 633.
- [3] (a) D.A. King and D.P. Woodruff, *The Chemical Physics of Solid Surfaces and Heterogeneous Catalysis*, Vol. 5 (Elsevier, Amsterdam, 1988) p. 399;
- [4] (b) S. Takatani, Y.W. Chung, *Phys. Rev. B* 31 (1985) 2290.
- [5] J. Ferron, J.M. Gallego, A. Cebollada, J.J. Miguel and S. Ferrer, *Surf. Sci.* 211/212 (1989) 797.
- [6] D.D. Vvedensky and S. Clarke, *Surf. Sci.* 225 (1990) 373.
- [7] C.T. Foxon, *J. Cryst. Growth* 95 (1989) 11.
- [8] B.F. Mason and B.R. Williams, *Z. Phys. D* 10 (1988) 369.
- [9] B.F. Mason and B.R. Williams, *Surf. Sci.* 111 (1981) 609.
- [10] M.J. Cardillo, *Faraday Disc. Chem. Soc.* 80 (1985) 1.
- [11] B.F. Mason and B.R. Williams, *Surf. Sci.* 139 (1984) 173.

- [11] W.A. Jesser and J.W. Matthews, *Philos. Mag.* 15 (1967) 1097.
- [12] B.F. Mason, R. Caudano and B.R. Williams, *Phys. Rev. Lett.* 47 (1981) 1141.
- [13] H. Johnsson, J.H. Weare and A.C. Levi, *Surf. Sci.* 148 (1984) 126.
- [14] J.H. Neave, B.A. Joyce, P.J. Dobson and N. Norton, *Appl. Phys A* 31 (1983) 1.
- [15] B.F. Lewis, F.J. Grunthaner, A. Madhukar, T.C. Lee and R. Fernandez, *J. Vac. Sci. Technol. B* 3 (1985) 1317.
- [16] B.F. Mason and B.R. Williams, *Surf. Sci.* 180 (1987) L134.