CHAPTER IV

RESULTS AND DISCUSSION

We have constructed a liquid phase epitaxy system to implement growth of α-Sn from Sn-saturated Hg solution on InSb substrate. Associated with the growth system a hydrogen generation/purification system was also constructed, so that the epitaxial growth process can be carried out in non-oxidizing atmosphere. A temperature control system was also constructed to provide the necessary growth condition, i.e. saturation, supercooling, and temperature-time rate.

Studies of the growth results were carried out with optical microscope for morphological features, with scanning electron microscopic techniques - imaging at higher magnification, chemical microprobing to identify the elements, and chemical mapping, with x-ray back scattering technique to identify the crystal structures.

We have identified the existence of α-Sn, that it can be epitaxially grown from liquid phase epitaxy as we have aimed to, and that it does undergo phase change from α to β phase at $\simeq 60^{\circ}C$ similar to the α- Sn grown in thin film via other techniques, such as molecular beam epitaxy.

However we succeeded in obtaining the α -Sn only in islet form, with an indication (not firmly verified) that we might also have

observed a thin film growth also, from our liquid phase epitaxy technique. We encountered several technical difficulties which prevented us from achieving all of our objectives. These difficulties are fundamental, inherent in α-Sn formation, i.e. the low temperature at which experiments must be carried out. Ordinarily epitaxy techniques are done for materials at much higher temperatures (≃ 800 °C for GaAs, for example), whether it is liquid or vapor-phase epitaxy. We understand from thermodynamics that the activity is strongly temperature dependent. If the activation energy is of the order of, say, 0.5 eV, then the reaction rate may be $\sim \exp(-0.5/281\ k)$ for an 8 °C process where k is the Boltzmann constant, and $\sim \exp(-0.5/1073\ k)$ for the 800 °C process. This means that we have to wait 4 x 10^6 times as long for a process at 8 °C as compared to that at 800 °C. The reaction time, or the necessary waiting to establish the equilibrium state for α-Sn epitaxy, therefore, is impractically long (of the order of days).

Such long waiting time makes it impossible to charaterize the melt status to an adequate accuracies for epitaxy control; the process is not now reproducible. More importantly, long waiting time at a temperature lower than the ambient causes the substrate surface to be re-contaminated, after careful preparation, by (most likely) the finite presence of oxygen. The presence of an oxide layer is confirmed. thus epitaxy is possible through pin holes in the oxide, and islet epitaxy obtains.

It appears that if the liquid phase epitaxy growth of α-Sn in the form of continuous layer on InSb substrate is to succeed a means must be found to etch the substrate uniformly (polishing etch)

immediatly prior to dipping into the Hg-Sn melt. Such etch perhaps should be a liquid layer above the higher density Hg, maintained already at the melt temperature. The existence of such an etch, which does not react with Hg and Sn, has not been pursued and is left for a future investigator.

In conclusion we have succeeded in setting up the necessary liquid phase epitaxy equipment, carried out LPE experiment to obtain α-Sn on InSb substrate, verified the epitaxy nature of the growth, and confirmed the high temperature stability of α-Sn up to 60 °C. We also have identified the nature of the experimental difficulties, and made a recommendation for possible improvement.