end which acts as the nucleation center for the crystal growth process. The solid–liquid phase boundary thereafter travels down the length of the sample. This is essentially the Bridgman method of growing single crystals. The rate of growth is controlled by the motor driven powerstat. The temperature gradient was determined by making several dummy runs with thermocouples, sealed in boron-nitride tubes, placed at various positions along the length of the sample. The gradient was found to be fairly linear between 7 and 20 mm from the thick end of the furnace. It ranged from 20°–30°C/cm for furnace temperatures between 300° and 400°C.

Before inserting the cell into the pressure vessel, the vessel was lubricated with dry molybdenum disulfide power and the cell wrapped in a 0.002 in. layer of lead foil to decrease friction between the cell and vessel walls.

The sample was first pressurized to 25 Kbars for about 12 h. It was then heated until the thermocouple, which was at the cooler end of the furnace, read 400°C. The sample was kept at this temperature for one hour in order to make certain that it was entirely in the liquid phase. The pressure was then set at 26 Kbars. The starting pressure was very important since starting with too high a pressure would cause the sample, upon cooling, to enter a region where both the orthorhombic and β-tin phases are in equilibrium. The resulting sample would then be composed of a mixture of the two phases. On the other hand, if the starting pressure was too low, the sample would revert back to the zinc-blend phase upon cooling. This would occur because there is a pressure drop of 2–3 Kbars due to the thermal contraction of the oil and press materials. This could have been overcome by increasing the pressure while the crystal was being cooled. However, it was feared that this would further strain the crystal. The press controls were never touched once the crystal growth process was started. The crystal was grown by using the motor driven powerstat to slowly reduce the power being supplied to the furnace. The most successful growth rate was estimated to be about 5 cm/h.

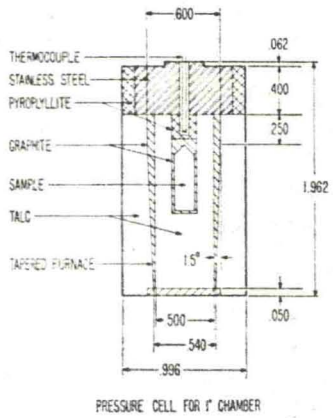

FIG. 2. Pressure cell for 1 in. chamber.

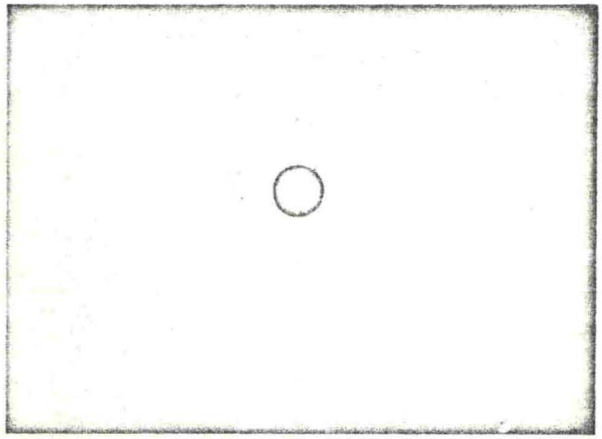

FIG. 3. Laue photo of InSb single crystal.

Once the crystal was grown, the temperature was further reduced to about 150°C at which point the driving motor was turned off and the crystal allowed to anneal at this temperature for two–three days. The temperature was then reduced to room temperature and liquid nitrogen introduced into the aluminum trough surrounding the pressure chamber. The crystal could only be cooled to about −125°C due to the large thermal path that the steel backup plates provided. The pressure was then reduced to one atmosphere over a period of four or five hours. The crystal was then removed from the press and stored in liquid nitrogen.

X-ray studies were made of these crystals using a Polaroid Laue camera with a Picker x-ray diffraction unit. Since the crystals could not be etched, the x-ray photos were not of the highest quality. However, it was easy to identify the major symmetry axes and confirm that the crystal had the β-tin structure. A typical x-ray Laue pattern is shown in Fig. 3. In order to verify that the crystals were indeed single, x-ray Laue shots were taken at 2 mm intervals as the crystal was translated horizontally from one end to the other without changing its orintation. The resulting Laue patterns were identical in every respect. Thus, it was concluded that the samples were indeed single crystals.