Final Report

ELECTRICAL COMPENSATION IN InP PRODUCED BY BACKGROUND IMPURITIES AND STRUCTURAL DEFECTS

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ABSTRACT

A steady-state liquid phase epitaxial growth system is being used to study the factors that influence the nucleation, growth and purity of InP. Humidity (>30% relative) has been found to be a major contaminant that leads to erratic growth morphologies and poor electrical properties. With the growth system and substrate preparations in a low humidity (<20% relative) room the reproducibility of epitaxial growths has been improved. The discoloration of the bone white pyrolytic boron nitride growth cell has helped to identify several sources of contamination that oxidize the In-melt and lead to premature nucleation. The nucleation and growth of epitaxial InP now appears to be limited by phosphorus transport instabilities in the P-saturated In-melt. A thermochemical analysis of phosphorus equilibria with In and InP may provide an insight into liquid-solid stability conditions. The results are in excellent agreement with the stability of the In-P liquidus curve and InP substrates under PH_3-H_2 mixtures. The analysis appears to indicate that the partial pressure of P_4 controls the nucleation of InP and the stability of the In-P liquidus curve. At temperatures below 700 °C the partial pressure of P_2 is greater than that for P_4 and appears to stabilize the InP substrate degradation.

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ELECTRICAL COMPENSATION IN InP PRODUCED BY BACKGROUND IMPURITIES AND STRUCTURAL DEFECTS

I. Introduction

Impurities and structural defects introduced into InP during growth produce compensation, scattering and traps that lower the electron mobility. The impurities arise from contaminants and chemical transport reactions between the growth system components. Structural defects arise from lattice mismatch with impurities and faults introduced during growth. Therefore, the purpose of this research is to minimize impurities and defects in the epitaxial growth of InP.

A steady-state liquid phase epitaxial (LPE) growth system has been developed to study the factors that influence nucleation and growth and the conditions necessary to control impurities. The steady-state technique is being used primarily because low temperature ($<500\ ^{o}C$) growths are possible which should minimize chemical reactivity and substrate degradation. In addition, the growth rate is controlled by a temperature gradient with the substrate held at a constant temperature which should allow detailed studies of nucleation, growth and impurity segregation under a variety of growth conditions to be studied.

The primary result during this final report period was the discovery that humidity drastically affects the growth system, growth materials and solvents which may be a major reason for nonreproducible epitaxial growths. The use of pyrolytic boron nitride (PBN) growth cells, also, has helped to isolate this problem and has lead to better procedures to identify and minimize contamination. In addition, thermodynamic analyses of phosphorus equilibria with In and InP may provide insight into vapor-liquid and/or solid and liquid-solid interface stability.

II. Growth System

The steady-state (LPE) growth system and technique was described in the August 1979 Final Report (Contract #N00019-78-C-o406). Breifly, the growth system consists of a semi-transparent furnace with an external heater that is controlled to maintain the substrate (or source) at a constant temperature, and an internal heater and heat transfer tube to induce a positive temperature gradient from the substrate to the source.

During this final report period, there was no basic change in the growth system except that the graphite growth cell was replaced with a pyrolytic boron nitride (PBN) cell, Fig. 1. However, at the end of this period a low humidity (<20% relative) room was constructed to house the growth system and for substrate preparations, Fig. 2.

The PBN growth cell is similar to the graphite cells, but the base was elongated so that a substrate cover could be used to minimize substrate degradation and to even the temperature over the length of the cell. The cover shields the substrate from the direct flow of H_2 and may locally restrain the loss of phosphorus.

A gas filter for N_2 and O_2, used in bakeout procedures to clean both the graphite and PBN growth cells, was also added to reduce their oil and water content. The H_2O content could be lowered to ∿0.1 ppm.

III. Preparations

Essential to high purity and reproducible growths of epitaxial layers are reliable procedures to prepare the growth system and the materials involved in growth. Separate from purified gases and materials are their handling and the preparation of the growth system components to avoid external contamination. A dominant factor of contamination is now believed to be related to exposure to humid conditions in the laboratory that vary from day to day. The final effort preceding the end of this final report period was to construct a low humidity room to house the growth systems and for final growth preparations. The reproducibility of growths have now been significantly improved so that growth related phenomena (transport, gradients, segregation, etc.) that affect nucleation and growth can be studied. The following discussion gives the preparation procedures that appear to lead to consistent reproducible growths.

A. System Preparation:

The fused quartz components of the growth system are thoroughly cleaned by degreasing and rinsing with solvents (MOS or electronic grade) and acids, and then baked out. The procedure steps are:

1. All surfaces are scrubbed with a brush and then with ultrasonic agitation (USA) in a detergent.
2. The surfaces are rinsed with deionized (DI) water to remove the detergents untill all surfaces remain uniformily wetted (the first step is repeated if DI water continues to bead or surface areas do not wet).
3. The surfaces are then rinsed with USA in
    a. acetone to remove all traces of water and repeated three times,
    b. trichloroethylene--two times,
    c. acetone to remove all traces of trichloroethylene --three times,
    d. methanol--three times,
    e. isopropyl alcohol--three times,
    and f. DI water.

4. The components are then soaked in aqua-regia (3 parts HCl and 1 part HNO_3) for a minimum of 20 minutes, to dissolve heavy metals, and then rinsed thorougly with DI water.
5. Step 3 a-e is repeated and then the components are dried in a clean room under low humidity conditions.

The components are then assembled and the external heater is attached to bake the fused quartz. First, however, the system is vacuum purged and He-leak checked through to the H_2-purifier. Then purified-H_2, at 1 l/min, is used to purge the system for 12 hours to further dry the system. The fused quartz is then baked at 100 °C above the expected maximum growth temperature. This temperature permits the quartz surfaces to equilibrate and to minimize excessive OH removal from the quartz. The effluent H_2 is monitored for H_2O content during the bakeout to determine when the quartz has equilibrated. The H_2O content is observed to drop below 0.01 ppm after baking for 6 to 8 hours. The thermocouples (TC) are not placed in their wells during the initial bakeout to avoid oxidizing and/or etching their inconel sheaths with trace acids and solvents. The external heater is controlled by a TC placed externally between the heater windings. After the bakeout the fused quartz system is only opened with flowing purified-H_2 to keep the interior continuously purged and dry.

B. Growth Cell Preparation:

The graphite and PBN growth cells are carefully cleaned to remove oxides and impurities. First the cells are rubbed with dry cotton swabs to remove loose materials.. The graphite cells are then loaded into the quartz furnace and cleaned <u>in situ</u> by drying for 12 hours under purified-H_2 and then baked at the anticipated growth temperature. The graphite is then quickly fired for 5 minutes at 700 °C under dried O_2 to burn off its surface and other contaminants. The cell is then baked under dried N_2 for 4 to 6 hours at the same temperature to purge the cell and system of O_2. Finally the gell is baked at the growth temperature in purified-H_2 for 12 hours.

The PBN cell cleaning procedure had to be developed to overcome several problems that only appeared after the cell had been baked. Dark stains were observed that were traced to indium oxides and handling with plastic tweezers. To remove the stains the following procedure was developed. First the cell is chemically cleaned in HCl (to remove In oxides) and then HNO_3 is added to form aqua regia. The cell is then thoroughly rinsed in DI water and dried in the clean room for 12 hours. The cell is then loaded into the growth system and dried under purified-H_2 for 12 hours. The PBN is then baked under dried O_2 at 800 °C for 15 minutes to burn off its surface and oxidize any matals present. In general, the cell will turn bone white, however, oxidized In is difficult to detect at this stage. The cell is further baked under dried N_2 and then purified H_2 for 24 hours each. If grey stains appear after firing in H_2 (probably reduced In_2O) the cell is removed and the acid and bakeout procedures are repeated. After 2 to 3 repeats of this cleaning procedure the PBN remains bone white after a 24 hour bakeout in purified-H_2. It was found that PBN must only be handled or touched with fused quartz implements--not metals. wood, paper or plastics. The latter materials leave trace deposits that stain the cell and will cause the In and P to oxidize. These stains cannot be reduced with extended bakeouts in purified-H_2 and have only been removed with the above cleaning procedure. In addition, PBN is a layered structure that can readily intercalate impurities and moisture. Therefore, the cell must be maintained under dry N_2 or H_2 with minimal exposure to the atmosphere and other materials not used in the growth system. With these precautions the PBN cell remains bone white after many months of use and does not appear to affect the oxidation of In and P (intially the dark stains around the melt well and under the In-melt caused premature nucleation of InP and the melt would stick to the base of the cell).

C. In-Melt Preparation:

Indium is very susceptable to oxidation due to handling in the atmosphere. Whenever the In is picked up, dropped or cut by hard objects small oxide particles (In_2O, InO and In_2O_3) were found floating on the In melt after baking in purified-H_2 . Below 300 °C In_2O_3 was found and above 300 °C In_2O formed. These particles could not be removed by extended (7 days) bakeouts above 300 °C (In_2O_3 reduces to In_2O). However, the reddish In_2O_3 particles diminished after extended bakeouts at 300 °C. Apparently, pressure from handling In in air oxidizes its surface. In order to handle In the following procedure was developed to load it into the melt well of the growth cell.

First the In was purchased from Johnson Matthey (A1A grade) as ingots cast in PBN crucibles to avoid oxide contamination (ingots are conventionally cast in fused quartz to which it wets but In does not wet PBN). However, this did not eliminate the problems discussed above. Presently the In as received is etched in HCl to remove oxides and then rinsed in methanol and isopropyl alcohol and dried. The In is then melted in a PBN crucible under dry N_2 and decanted through a hole at one end of the crucible into the melt well. The hole rim retains most of the remaining oxides as a scum in the crucible. The In in the melt well now remains bright and no oxide particles are observed on its surface after extended bakeouts in purified-H_2. In addition, no dark stains are observed to form on the walls of the PBN melt well or lid.

The melt is baked in the growth system for 24 hours at the growth temperature without the lid and InP source. During this bakeout the well is slid back and forth several times over the length of the cell base to assure that impurities are not present that will build up oxides during successive growths. The melt is not heated above the growth temperature in order to avoid contamination from higher temperature chemical transport reactions between the growth system components. Care is also taken not to slide the In when frozen, as this may induce oxidation in air.

D. Chem-Mechanical Polishing:

The InP substrates are chem-mechanically polished using a 0.1% Br in methanol solution with a "Pellon" pad mounted on a tilted rotating table. The substrates are cemented with beeswax to a rounded triangularly shaped stainless steel block that rotates against the rim of the rotating table. The triangular block gives a radial motion on the table that combined with its rotation and the rotation of the table gives the three basic strokes to generate a random motion of the substrate against the polishing pad. The tilt of the table and rotational speed is adjusted to balance the friction against the pad and the rotating rim for uniform strokes. Very flat, orange peel free polished surfaces are obtained for approximately 6 square inches of substrate area in about 8 hours. The substrates are polished on both sides so the each can be used for epitaxial growths. In addition, another rounded triangularly shaped block with 3 holes, each with smaller blocks similarly shaped, can be used to repolish used substrates for further growths.

E. Source and Substrate Preparation:

The InP source (large grain, high purity InP from NRL) and the substrates are cut to size and cleaned by the following procedure:

1. Steps 1 through 3e (Sec. III-A) are initially used with the InP resting of the bottom of a polyethylene beaker. The following steps are carried out in the low humidity room with solvents that have only been opened under low humidity.
2. The InP is dried, weighed, mounted in a fused quartz tweezer (the InP remains in this tweezed until finally loaded into the growth cell). and then placed in a fused quartz beaker.
3. Steps 3a-d (Sec. III-A) are repeated.
4. Methanol is then added to the beaker with ∿5% by volume HCl to chemically polish and remove oxides from the substrate surface for 1 minute under USA. The solution is then partially poured off <u>but</u> with care to not expose the InP to air. Then this step is repeated three times.
5. The methanol solution is partially poured off and displaced with more methanol <u>but</u> again not exposing the InP to air--three times.

6. Step 5 is repeated with isopropyl alcohol with the same precautions.
7. The beaker, with isopropyl alcohol is carefully heated to boiling.
8. The growth cell is removed from the growth system during step 7 for loading.
9. The quartz tweezer with InP is then carefully removed by maintaining the substrate surface perpendicular to the isopropyl surface. This allows the liquid surface tension to effectively wipe the substrate dry.
10. The source or substrate is finally loaded into the growth cell and then as quickly as possible into the growth system.

During the loading procedure the growth system is continuously purged with purified-H_2 to minimize moisture contamination of the inner fused quartz walls. Vacuum purging of the growth system after loading generally increases the amount of time required to lower the effluent H_2O content by several hours. Continuous H_2 purging lowers the H_2O content from 100 to 0.01 ppm in approximately 15 minutes. Before the furnace is turned on, the system is purged for at least 1 hour with the purified-H_2 flowing at 1 l/min.

F. Gas and Temperature Monitoring:

The effluent purified-H_2 is continuously monitored for flow rate and for H_2O content with a Panametrics hygrometer. The hygrometer signal is internally biased so that H_2O levels < 0.01 ppm can be recorded before and during growth. Providing the system has been purged for at least 1 hour there is no detectable increase in H_2O when the system is brought up to temperature and when the fused quartz push rod is used to slide the melt well. The hygrometer is also used to monitor the dried N_2 and O_2 gas before and during bakeout procedures. In general, there are large increases (>100 ppm) when the system and growth cells are initially baked. For graphite, with the H_2O content above the detectable limit a decrease is observed when the cell is locally heated. This indicates that the H_2O in the system reacts with the carbon and probably forms CO in the vicinity of the cell. This behavior is not observed with PBN cells.

The temperature and temperature gradient are continuously recorded during growth runs. The temperature can be controlled to better than 0.25 °C when the melt is slid over the substrate with temperature gradients up to 100 °C/cm. The temperature gradient and temperature uniformity can be adjusted by positioning the incoming N_2 flow under the cell in the heat transfer tubes and the internal heaters. With no temperature gradient, the P-saturated In melt can be placed over the InP substrate without producing meltback or growth. Thus temperature and gradient related nucleation and growth phenomena can be extensively studied under controlled conditions.

IV. Growth Procedures and Results

Note: The low humidity room was not completed until the end of this final report period. Therefore, the impact of low humidity conditions has not been fully evaluated on improved growth results.

After low humidity conditions were attempted during loading procedures by using a glove box the time-temperature and - temperature gradient sequences could be used to create a variety of very reproducible nucleation and growth effects. (the effect of humidity on solvents had not been realized until >60% relative humidity conditions brought about very erratic results). The conditions necessary for good growths have not yet been determined, i.e., hillocks and irregular surface terraces are still dominant but excellent terrace and miniscus line free morphologies are obtained on occasion, Fig. 3. The reason for the few good results have not been determined even though all of the growth conditions were reproducibly repeated.

At present the best results are obtained for the first growths from a new melt and source without an initial bakeout together in a bone white PBN growth cell. Further, growths from the same melt continue to degrade and finally only hillocks are obtained. Thus there appears to be a degradation effect in the melt that affects transport and/or nucleation. These conclusions are based on over 100 growths during this report period.

There are several modes or combinations of applying the temperature gradient and sequences of sliding the melt over the substrate to initiate nucleation and growth. Some of the following procedures have been tried:

1. Temperature controlled underneath the substrate with
    a. the temperature raised and held for saturation of the melt and then
        i. the melt is slid over the substrate and the temperature gradient is applied

        or ii. the temperature gradient is developed and then the melt is slid over the substrate.

b. the temperature gradient is developed during the heat-uo and stabilized during melt saturation and then the melt is slid over the substrate.

2. Temperature controlled over the source and then the preceding options are applied.

The latter mode has not been tried. In general these options have not been fully tried for different growth temperatures and temperature gradients. Growth temperatures at 475 °C appear to yield as good results as at 600 °C. High temperature gradients (>30 °C/cm) do produce more hillocks but are necessary for growths at the lower temperatures. These results, however, do not reflect what may be achieved under controlled low humidity conditions of all stages of growth preparation.

The electrical properties of the better layers appear to indicate that bakeouts of the melt and source together lower the mobility and raise the carrier densities. At room temperature mobilities as high as 4000 $cm^2/V \cdot sec$ for carrier densities in the $10^{16}/cm^3$ range have been achieved. However, the 77 °K mobilities do not significantly increase above this value. Therefore, impurity scattering remains high which means that compensation must also be high.

The use of the PBN compared to graphite for the growth cell appears to lower the InP substrate degradation. This may be related to the result that carbon reacts with H_2O in the growth system to create CO which might oxidize or reduce the InP surface to free phosphorus. In addition, the bone white PBN allows oxides and InP particles to be visibly seen which can't be confused with graphite particles. Hence, its superior inertness but discoloration to contamination amy help to improve the electrical properties of InP epitaxial layers.

V. Phosphorus Equilibria with In and InP

The degradation of InP substrates is primarily due to the loss of phosphorus at temperatures above 400 °C. To stabilize InP above this temperature phosphine (PH_3) is generally added to purified-H_2 to form partial pressures of P_2 and P_4. In order to study the equilibrium of P with In and InP the above results and those related to the equilibrium of PH_3-H_2 mixtures with the In-P liquidus curve will be analysed. This will be done by comparing results from thermochemical data of Barin and Knacke, and the JANAF tables with the experimental data on the liquidus curve by Panish and Arthur, and on InP decomposition by Drowart and Goldfinger and Clawson et al.

A computer program was developed to determine the equilibrium decomposition of PH_3 in H_2 to form P_2 by the reaction

$$2PH_3(g) = P_2(g) + 3H_2(g),$$

and similarly for P_4, at 1 atm from the thermochemical data. Initial mole fractions of PH_3, $(X_{PH_3})_i$, at temperatures from 700 to 1300 K were assumed to calculate the equilibrium partial pressures of P_2 and P_4. These results are then compared with the experimental data on the liquidus curve for the In-P phase diagram and the stabilization of InP with PH_3 over the same temperature range.

The results of this analysis are summarized in Fig. 4. The JANAF thermochemical data are in good overall agreement with both the experimental data on the stability of the In-P liquidus curve and of InP with initial molar fractions of PH_3 in H_2. The Barin and Knacke data depart from the experimental results by an order of magnitude for temperatures below 700 °C. It is interesting to note that there is very close correspondence between the stability of InP and the In-P liquidus for temperatures below 700 °C. The dissociation of PH_3 into P_2 and P_4 is also shown for $p_{P_2} = p_{P_4}$. This curve indicates that there is a strong correlation between this equilibrium and the stability of phosphorus in liquid In and solid InP with $(X_{PH_3})_i$.

When the stability results are replotted against the p_{P_2} and p_{P_4}, Figs. 5 and 6, there appears to be a marked tendency for p_{P_2} to stabilize InP and for p_{P_4} to stabilize the liquidus curve. This conclusion is based on the InP curve being approximately parallel to and below the $p_{P_2} = p_{P_4}$ line where $p_{P_2} > p_{P_4}$, Fig. 5, and the In-P liquidus data being approximately linear to $\ln(p_4)$ vs. 1/T, Fig. 6. In addition, the liquidus curve data nearly extrapolate linearly to the InP melting temperature (1070 ^{o}C) for the equilibrium pressure with phosphorus (25 atm).

Panish and Arthur based their experimental data evaluation of the liquidus curve on nearly complete dissociation of PH_3 over this temperature range. This is in agreement with the JANAF thermochemical data, but disagrees with the Barin and Knacke data for temperatures below 700 ^{o}C. In addition, Ban's experimental data indicate that there is only partial dissociation of PH_3 in this low temperature range. Ban's experimental results were obtained by time of flight mass spectrometry. This technique suffers from the transfer of gas species through a capillary tube before being analysed by a mass spectrometer. The effect of capillary walls and cooler temperatures suggest that P_2 and P_4 in H_2 transform back to PH_3, thus yielding low PH_3 decomposition results.

When the experimental results are compared to $\ln(p_{P_2} + p_{P_4})$, the stability of InP under $(X_{PH_3})_i$ becomes linearly related to 1/T and the stability of the In-P liquidus curve smoothly departs to higher partial pressures of phosphorus above 700 ^{o}C.

Several possible conclusions can be made from this analysis:

1. The correspondence between the stability of InP with the In-P liquidus curve probably occurs because the decomposition of InP produces P-saturated In droplets, i.e., an In-P liquid is present. At low temperatures P_2 dominates and competition develops between In and InP for the phosphorus.

For the equilibrium of In and P (without InP) the p_{P_4} is the primary stabilizing influence. Thus there is a stronger tendency for departure at higher temperatures when $p_{P_4} > p_{P_2}$.

2. The correlation of p_{P_4} with the In-P liquidus curve extending to the melting temperature of InP (where InP becomes and In-P liquid) may relate to P_4 acting as a critical nucleating configuration to establish InP. This would be in agreement with experimental and theoretical studies of stable atom clusters for III-V compounds by Mattes. From these studies the group V element forms a tetrahedral P_4 configuration at the center of the cluster. Thus the stabiltiy of the liquidus curve with p_{P_4} may correspond to the critical In-P conditions necessary to form stable clusters of InP.

3. Based on these conclusions and the results of Clawson et al for growth temperatures below 700 °C when slightly higher $(X_{PH_3})_i$, than for the stability of InP, are used there should not be an significant increase in the P-saturation of the In melt. This may be related to $p_{P_4} < p_{P_2}$. For $p_{P_4} > p_{P_2}$ at temperatures above 700 °C higher $(X_{PH_3})_i$, than for the liquidus stability, should have a marked effect on the P-saturation of the In melt.

These conclusions, based on thermochemical equilibrium of stable phases and cluster stability, appear to be consistent and may yield an insight into vapor-liquid and/or solid interface equilibria. Further, the coincidence of the In-P liquidus with the InP stability curves below 700 °C may be significant to liquid-solid interface equilibria, i.e., LPE growths are usually performed below 700 °C. In addition, for steady state LPE growth the solute may be in the form of P_4 which would markedly affect the diffusion process of phosphorus from the source to the substrate. Under certain conditions (unknown at present) this could lead to premature nucleation and growth in solution which would yield erratic growth morphologies on the substrate.

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FIGURE CAPTIONS

Figure 1. III-V growth systems located in a low humidity room. The preparation areas are in the laminar flow hoods (right background) and the loading area is between the systems.

Figure 2. The pyrolytic boron nitride (PBN) growth cell with a 1x2 cm InP substrate.

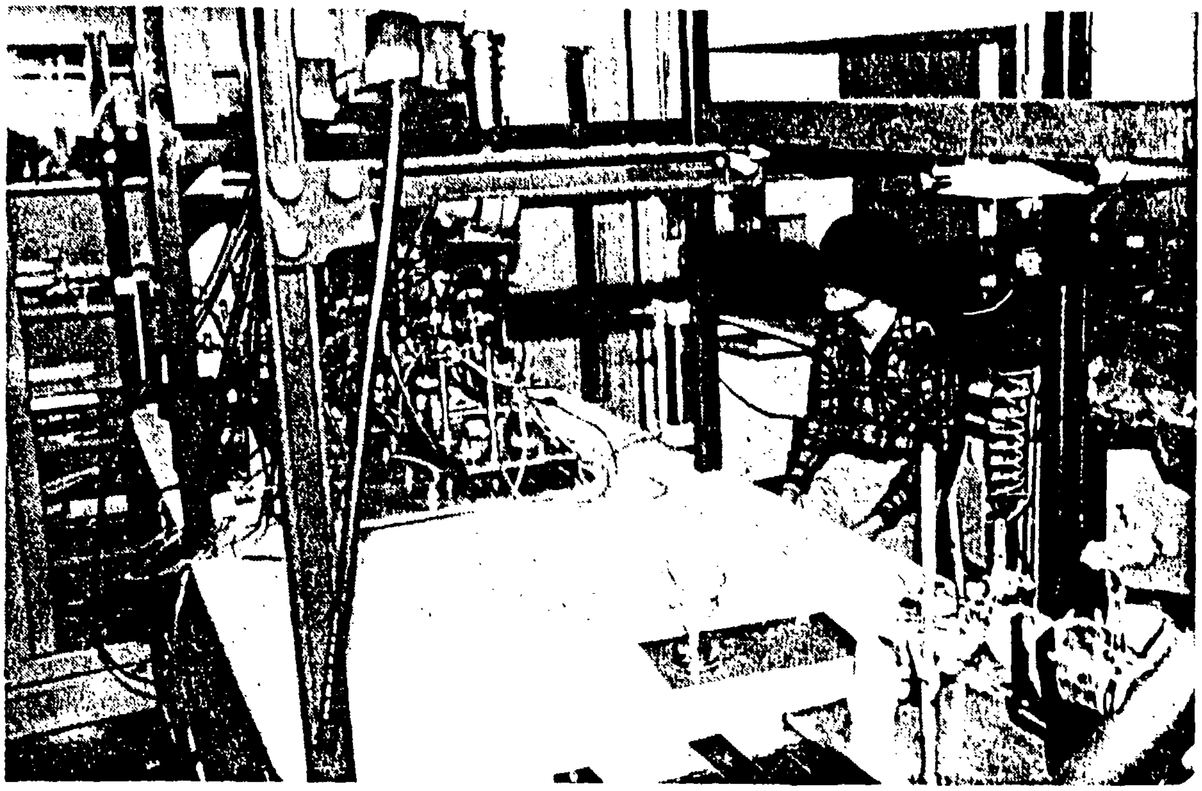
Figure 3. Sequential InP epitaxial layers grown in the PBN growth cell. Note the deterioration in growth morphologies from a new P-saturated In-melt, (a), and the subsequent InP growths from the same melt, (b) and (c). Each substrate has an initial (upper) and final (lower) growth. Each final growth degrades due to nucleation and growth in the melt that eventually dominates over subsequent growths on the substrate. The circular miniscus lines are due to incomplete In-melt removal at the end of each of the three growths.

Figure 4. Comparison between the thermochemical data from the JANAF Tables with the Barin and Knacke data for the experimental stabilization of the In-P liquidus and InP with initial PH_3 in H_2 mole fractions.

Figure 5. The P_2 partial pressure in equilibrium with the In-P liquidus and InP for different initial PH_3 in H_2 mole fractions.

Figure 6. The P_4 partial pressure in equilibrium with the In-P liquidus and InP for different initial PH_3 in H_2 mole fractions. Note that the In-P liquidus appears to be dependent on the partial pressure of P_4 up to the InP melting point.

Figure 7. Comparison of the experimental results with the total partial pressure of P_2 and P_4 for the different initial PH_3 in H_2 mole fractions.



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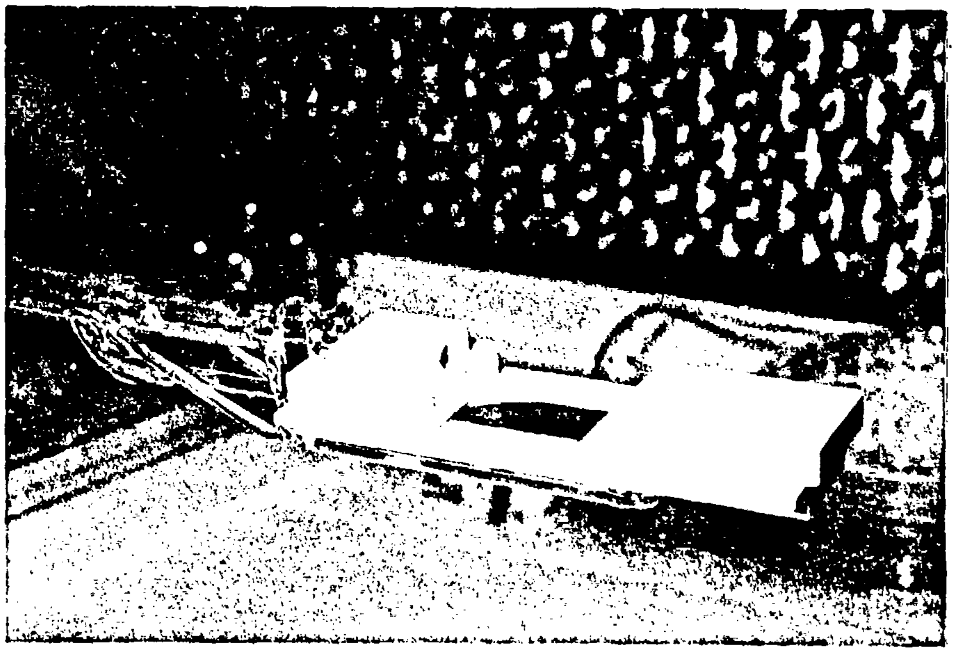

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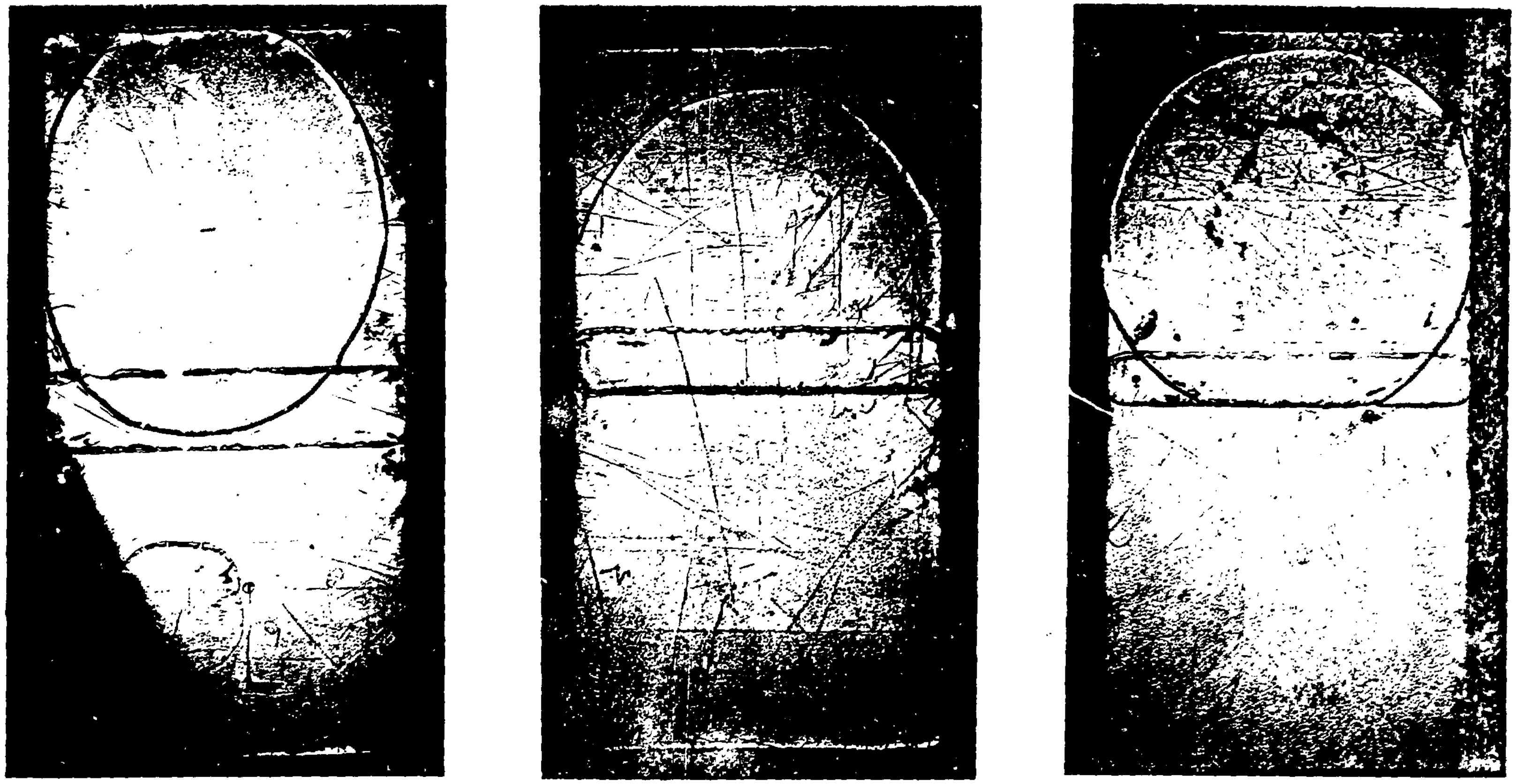

(a) (b) (c)

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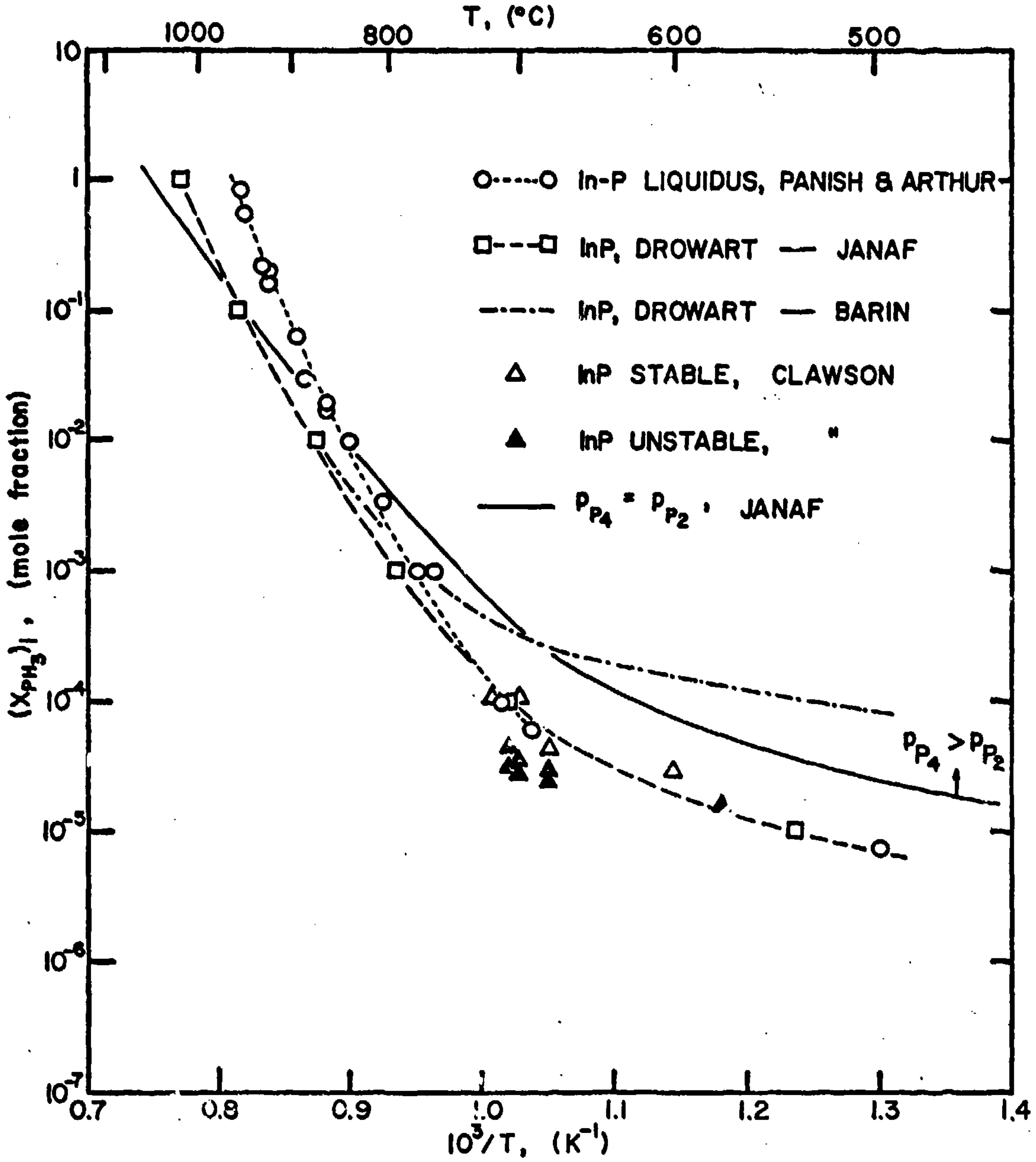

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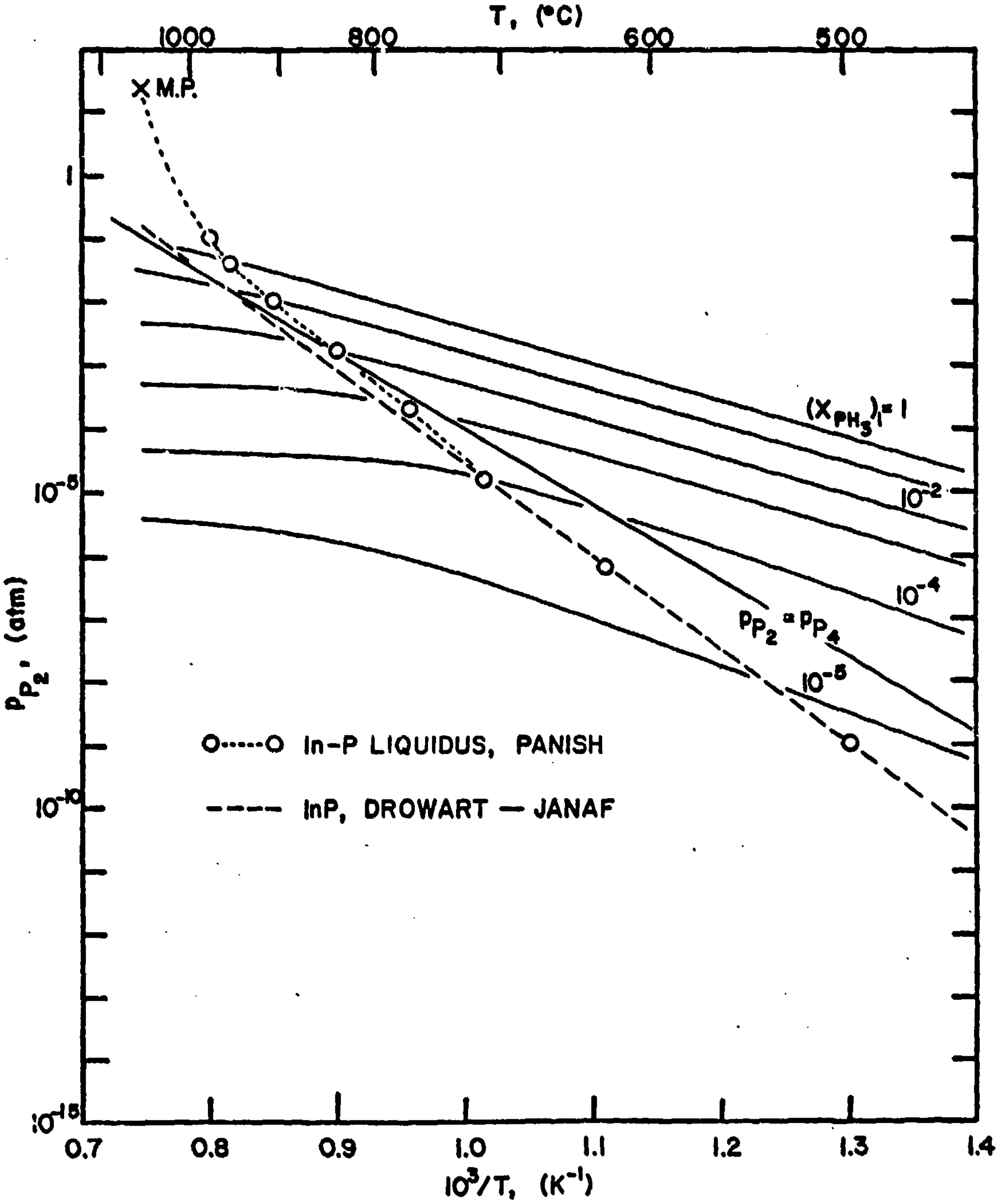

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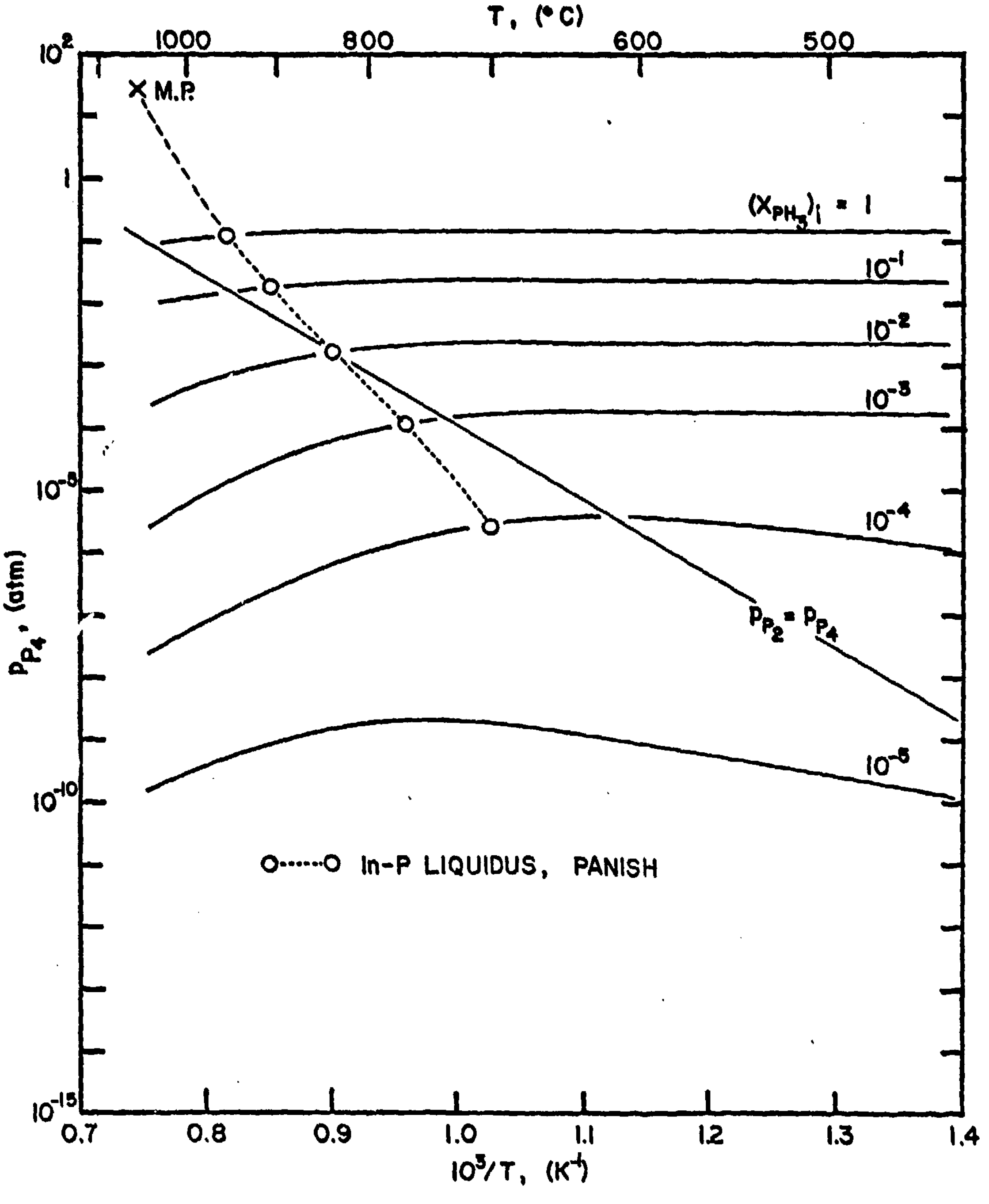
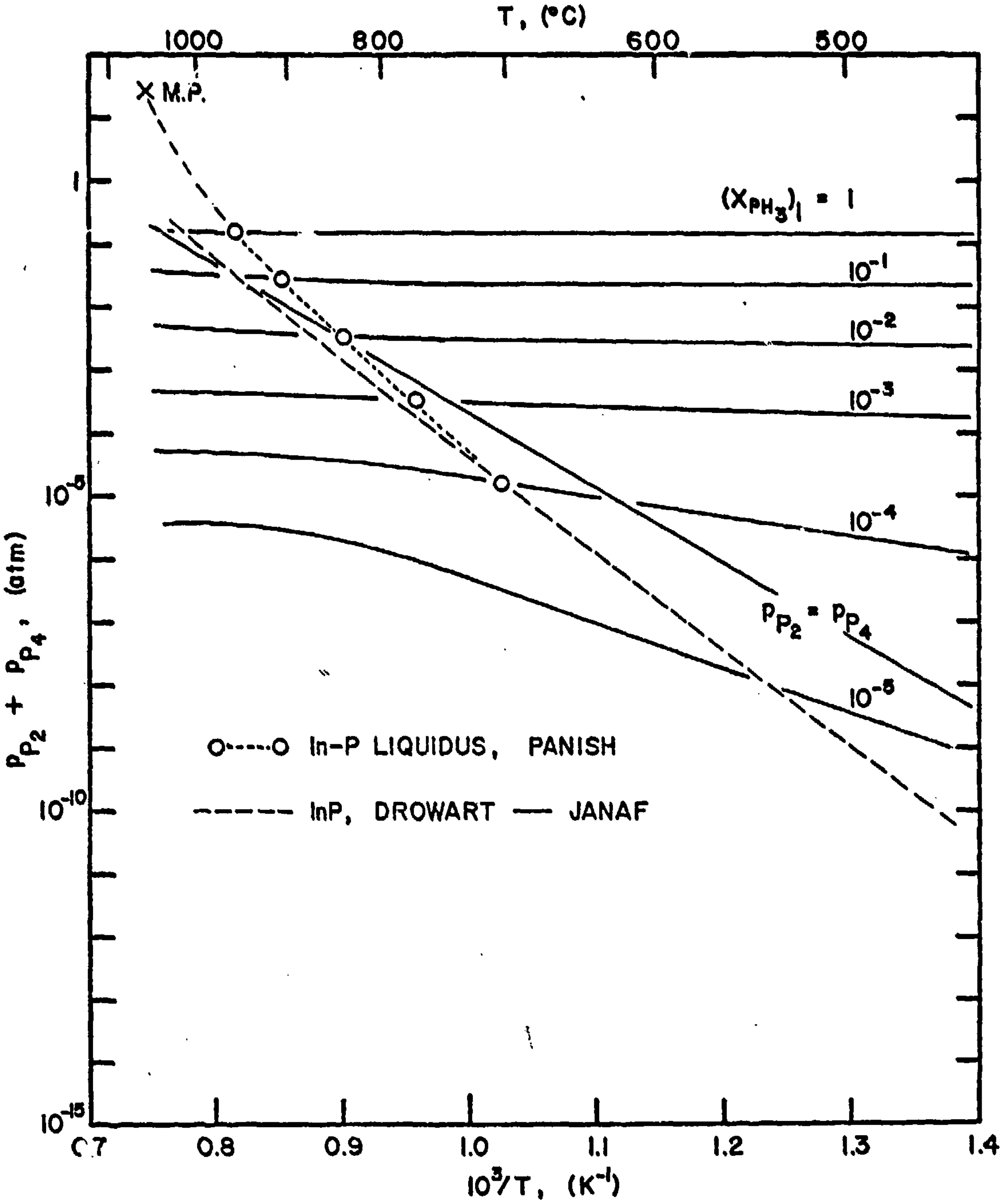


Figure 6. The P_4 partial pressure in equilibrium with the In-P liquidus and InP for different initial PH_3 in H_2 mole fractions. Note that the In-P liquidus appears to be dependent on the partial pressure of P_4 up to the InP melting point.



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