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PRECIS FLUX CONTROL FOR LATTICED MATCHED SUPERLATTICE MATERIALS

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13. ABSTRACT (Maximum 200 words)

WE HAVE INVESTIGATED FLUX TRANSIENTS IN MOLECULAR BEAM EPITAXY (MBE) SOURCES. A DETAILED STUDY OF THE RELEVANT FACTORS HAS BEEN CARRIED OUT. FOR THE FIRST TIME Ga TRANSIENT WAS MEASURED IN THE PRESENCE OF AS IN A REALISTIC GROWTH ENVIRONMENT. GROWTH RATE VARIATION DUE TO TRANSIENTS CAN BE ACCURATELY MONITIRED BY RHEED ANALYSIS. WE DEMONSTRATED THAT TRANSIENTS COULD BE MINIMIZED OR EVEN ELIMINATED BY SOURCE TEMPERATURE RAMPING AND MASS SPECTROMETER FEEDBACK CONTROL.

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Precise Flux Control for Lattice Matched MBE Growth

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1.0 PROJECT OBJECTIVE

To demonstrate the feasibility of eliminating flux transients in molecular beam epitaxy (MBE). Specifically the technical objectives are as follows:

o Investigate transient mechanism and modify current MBE source assembly design and its temperature ramping procedure to test flux transient reduction

o Perform experiments to demonstrate elimination of transients

2.0 PROJECT SUMMARY

MBE has become a critical technique for fabricating a wide variety of semiconductor devices. Source flux transient during growth however is a major cause of composition and layer thickness variation and is especially problematic for structures of very thin layers. During the course of this project we have investigated several possible causes of the flux transient, designed remedies and demonstrated elimination of the problem. Flux transient is a result of thermal perturbation due to source shutter opening. To isolate factors that could reduce this perturbance we carried out detailed experiments with controlled source environment. Source geometry, shutter material and action, and various source cell temperature routines have been experimented. The results are very promising. We have clearly shown that the flux transients can be eliminated with proper control of the source operation.

We found in this investigation that the mass spectrometer (MS) sensitivity varies with As partial pressure in the system and therefore the Ga flux measured by the MS is distorted. We have developed a technique to measure flux transient by RHEED intensity oscillation. The flux variation as measured by RHEED intensity oscillation differs from that by the MS. Since the former measures the growth rate, the true effect of the transient is manifested by this measurement. Because the flux transient depends on so many dynamic process variables, we believe in future studies the process can be optimized by using Artificial Intellience (AI) and fuzzy logic type of control.

Summary of Phase I achievements are listed below:

Tested refined designs of the shutter and source, including heated PBN shutter and dual filament cell

Demonstrated minimized transient with temperature ramping

Investigate for the first time Ga transient in As pressure in realistic growth enviornment

Discovered MS sensitivity altered by As pressure

Developed technique to measure growth rate change due to the transient

Eliminated transient with a mass spectrometer feedback control of the sources

3.0 FLUX TRANSIENT EXPERIMENTS

3.1 Ga Flux Transient Measurement and Correction

In contrast to other transienet studies, this investigation included measurements done in real growth conditions. The experiments were carried out in a SVT-GaAs MBE system that is designed to provide uniform 3" wafer growth. Figure (1) shows a schematic of the MBE system. This system is equipped with a full complement of eight effusion sources and a substrate manipulator that is capable of heating the sample to 1,000 C. A combination of ion and cryopump was used to achieve a base pressure reaching about $1x10^{-10}$ Torr. A unique feature of the growth chamber is that a mass spectrometer is situated next to the substrate such that the meeasured flux signals represent realistic growth conditions. Reflection High Energy Electron Diffraction (RHEED) was used to verify the transient level. Realistic flux transient effects were found under typical growth conditions.

METALLIZATION STATION

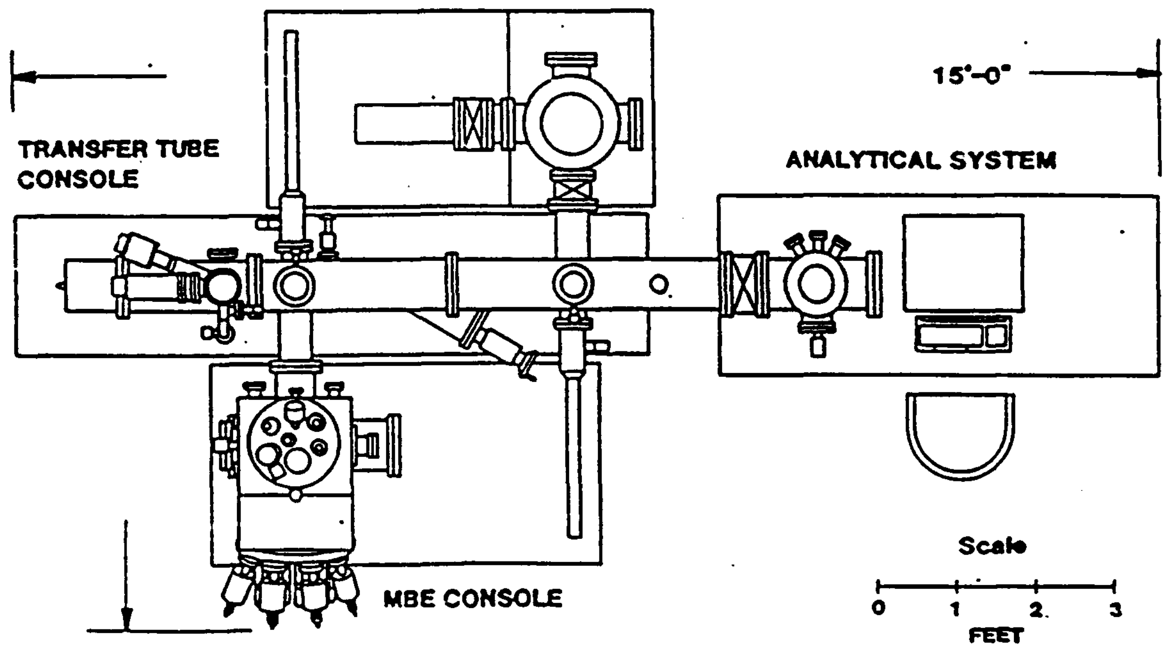


Figure 1. Laboratory MBE System at Superior Vacuum Technology (SVT)

Molecular beam flux is formed by evaporating material from an effusion cell. Since the vapor pressure of any material is an exponential function of the temperature, the magnitude of the molecular flux is affected strongly by the thermal environment in the cell. Opening and closing of the source shutter in front of the cell invariably perturb this thermal equilibrium, and it has long been noted that the shutter actuation cause flux transient in the molecular beam making exact control of the composition and thin layer thickness difficult. During Phase I we have carried out a systematic investigation to isolate factors that could reduce this perturbance. Since the partial vapor pressure of the evaporant, and hence the beam flux, depends exponentially on the material temperature, the flux can be approximately described by

$$F(t) = F(T_0) + \delta F(T_1 - T_0) \;\; e^{-t} \qquad (1)$$

where F(t) is the time (t) dependent flux, after the shutter is opened,
$F(T_0)$ is the equilibrium beam flux
T_0 is the operating temperature of the cell during growth,
T_1 is the initial cell temperature, and
δF is the flux difference due to temperature change.

We have looked into the effects of source geometry, shutter material and action, and various source cell temperature routines. In the following we will present the results and discuss solutions to eliminate the flux transient problem.

Figure (2) shows a typical mass spectrometer (MS) trace of the Ga flux signal that shows approximately 13% transient (the second term in equation 1) as the shutter is opened. Note there is also the more long term (on the order of several minutes) down drift even though the cell temperature is regulated by a thermal couple. This level of transient is actually less than many other commercial MBE systems would generate but it is a significant problem for precision control of the layer thickness and composition.

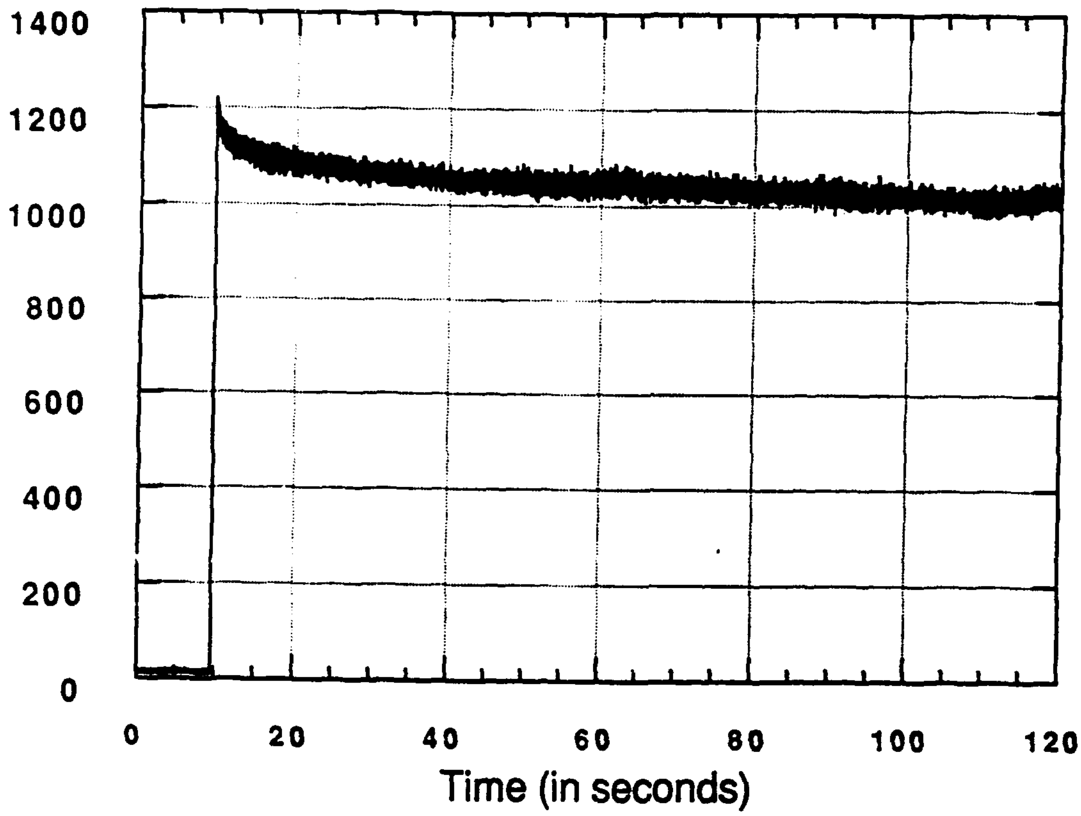


Figure 2. Flux Transient in a Gallium Cell

The most common strategy for reducing flux transient is by varying the cell temperature. By quickly ramping up the temperature to a few degrees above the set point as the shutter is being opened, one could even out the thermal perturbation and significantly reduce the transient. This is to compensate for the heat loss due to shutter opening as described by the second term in equation (1). The amount of temperature variance depends on the cell set temperature, crucible shape and cell-shutter orientation. By monitoring with a mass spectrometer (MS) we were able to optimize the ramping sequence to obtain a fairly small flux transient, as shown in Figure (3). The cell temperature was raised quickly before the the shutter opening.

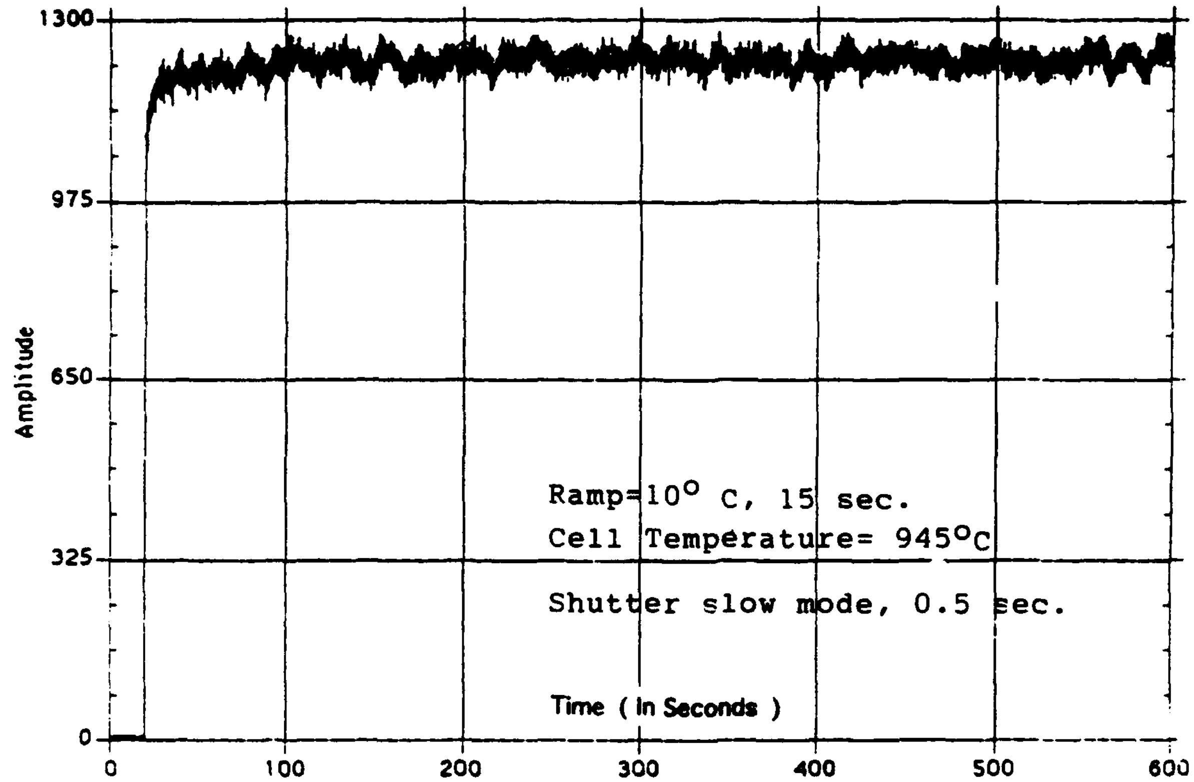


Figure 3. Flux Signal Transient Reduction Due to Temperature Ramping

Further improvement was realized with a multi-step ramping sequence. One example shown below used a three step ramping. The cell temperature was first raised 5 seconds and then lowered as the shutter is opened, before finally raised again to attain the equilibri um operating level. Figure (4) shows that the flux transient was totally eliminated. The reason of the improved result is due to compensation for the delayed thermal response of the cell.

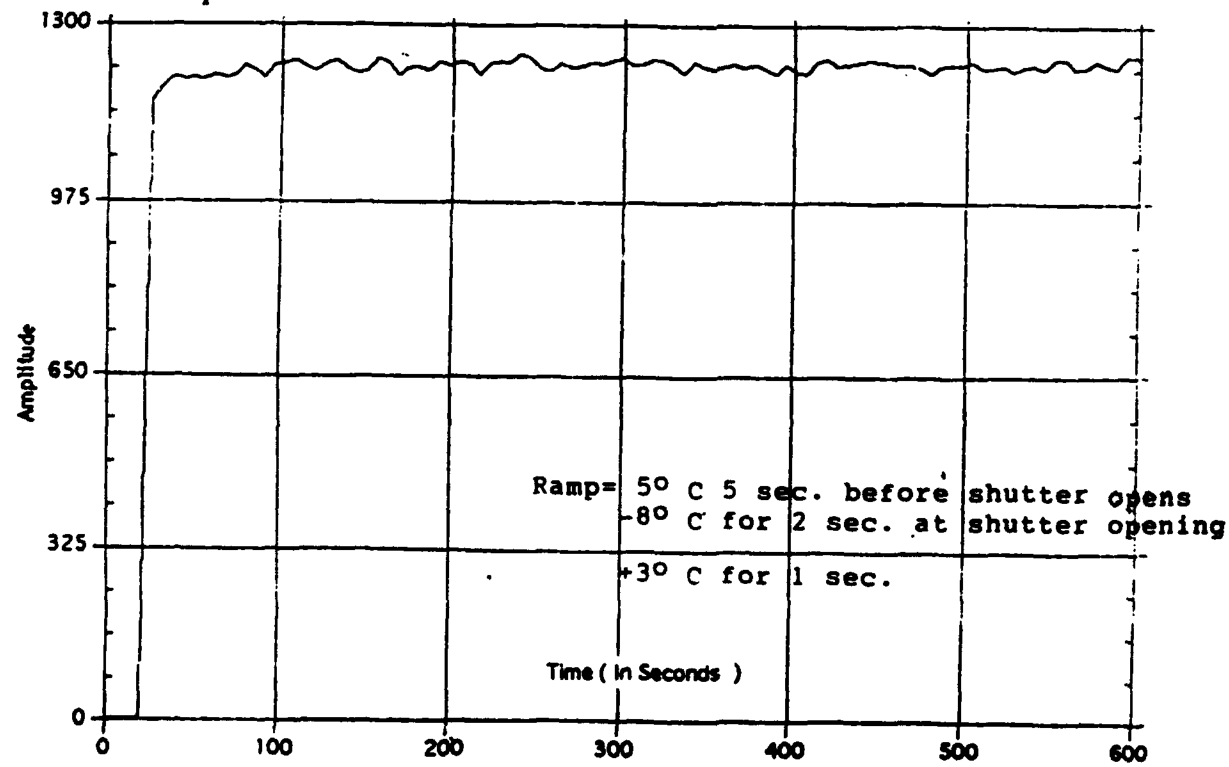


Figure (4). Stable Ga Flux by a Three-Step Cell Ramping Procedure.

Having established a significant transient reduction we undertook to investigate other relevant factors that cculd bring further improvement. We studied shutter action and alternate materials for shutter. A special cell/shutter flange with water cooling was built for this purpose as shown in Figure (5). Normally the shutter is made of Mo but it is changed to pyrolytic boron nitrite (PBN) in the photo. Both Mo and PBN have been used in the test. PBN transmits infrared radiation much better than Mo so it causes less thermal fluctuation during the shuttering action. To prevent condensation a shutter heater filament is wound to the PBN shutter. However our study did not show significant difference in the two cases.

Figure 5. Photo of a Cell/Shutter Assembly

We found that shutter speed is a factor in transient variation. Instead of the proposed double deck shutter we tried slower shutter speed to achieve the same objective of gradually varying the thermal environment. Figure (6) shows a trace of the transient with opening time of 1 second instead of normal 0.1 second. As might be expected the slow action shutter creates smaller transient. In particular the sharp rise is eliminated. Although in practice such slow time is not acceptable this measurement did verify the importance of the shutter action and could lead to more advanced shutter design.

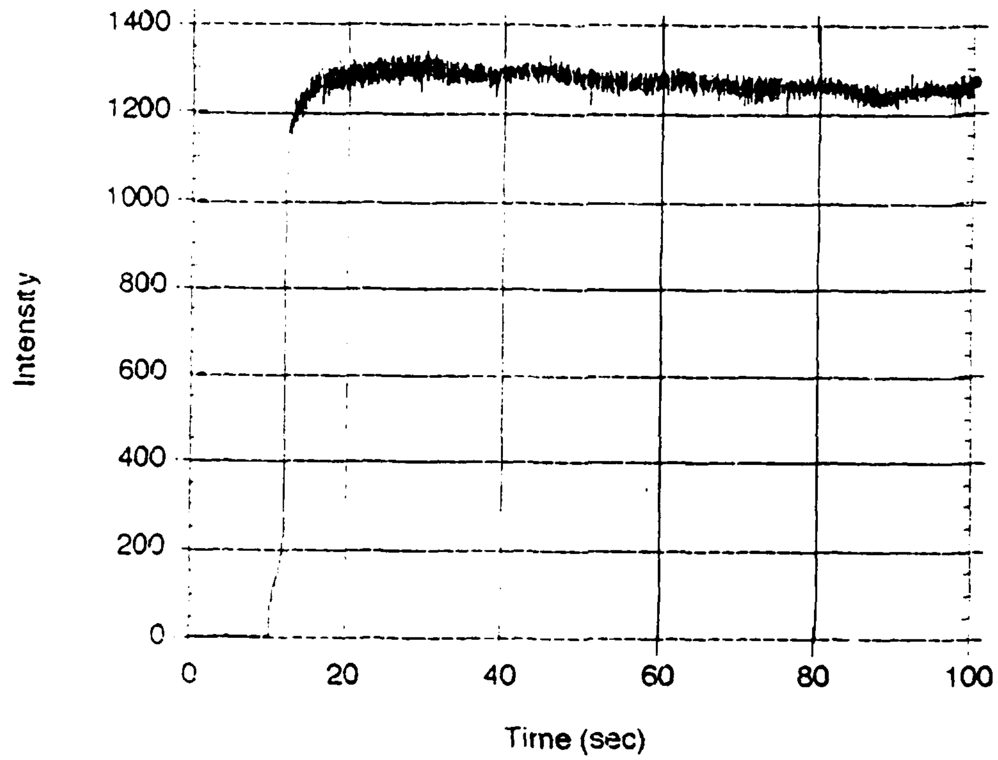
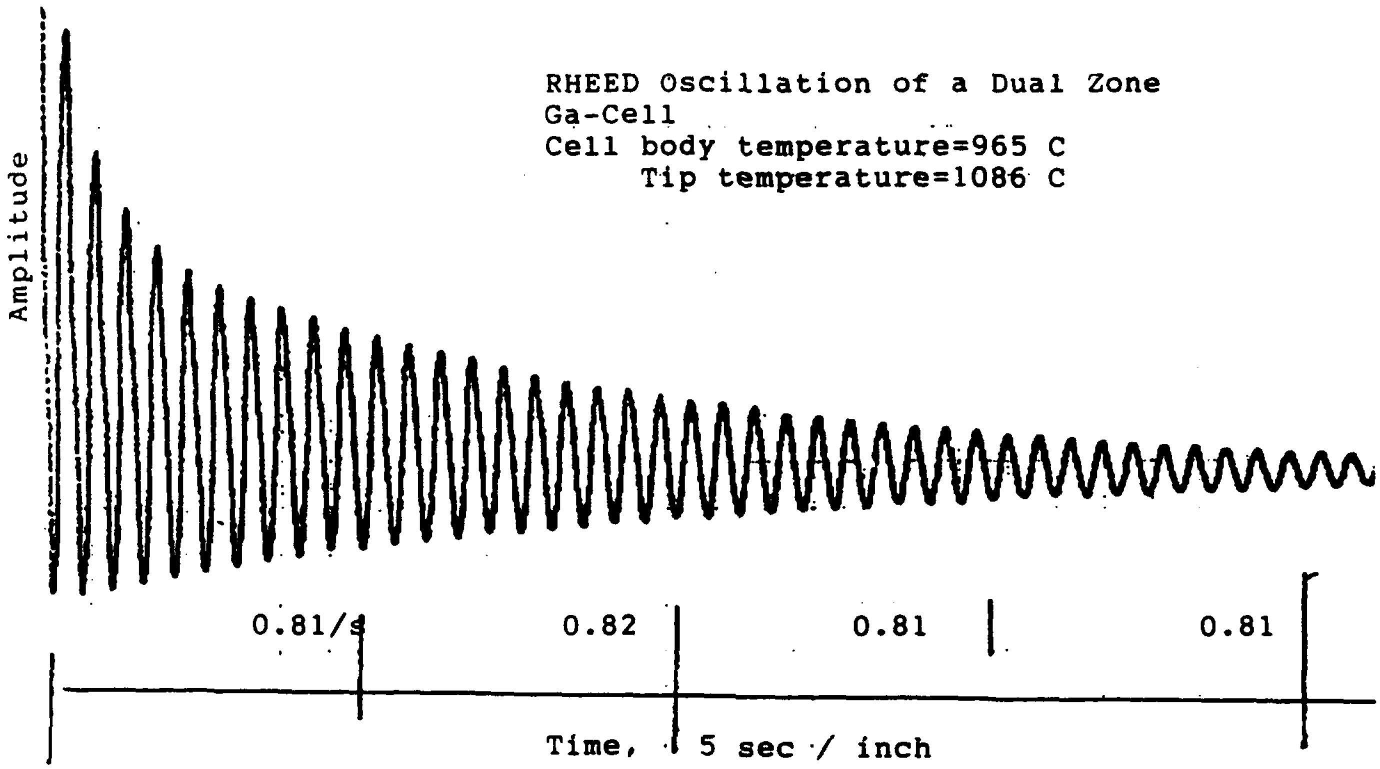


Figure 6. Transients of Slow Shutter Opening Speed.

We observed very stable flux oscillations using a two zone cell. Instead of the normal single heater filament winding there are two windings, one around the bottom half of the cell and the other around the upper part and the lip section of the cell. The purpose of the upper heater is to compensate the heat loss through the cell opening. Figure (7) shows the RHEED intensity oscillation of GaAs. It can be seen that the oscillation period is very constant even after many cycles. Since the GaAs growth rate is dependent on the Ga flux arrival rate, we are basically following the the Ga flux arrival. The result is indicative of lack of transient in Ga flux.



Fgiure 7. RHEED Oscillation of GaAs with a Two-Zone Cell.

The RHEED technique was exploited for more sophisticated application. In the Phase I proposal we planned to develop a real time surface lattice constant measuring technique using an electro-magnetic field to scan the primary RHEED electron beam. Since the lattice constant changes according to layer composition, the measurement would indicate any change in deposited layer composition due to flux variation. To illustrate the technique we present in Figure (8) an image screen of the RHEED analysis taken by a video camera. The upper right hand corner figure displays the RHEED diffraction spots as they appeared on the phosphor screen. The inset below shows the relative positions of the diffraction peaks and the distance between the diffracted peaks is inversely proportional to the lattice constant.

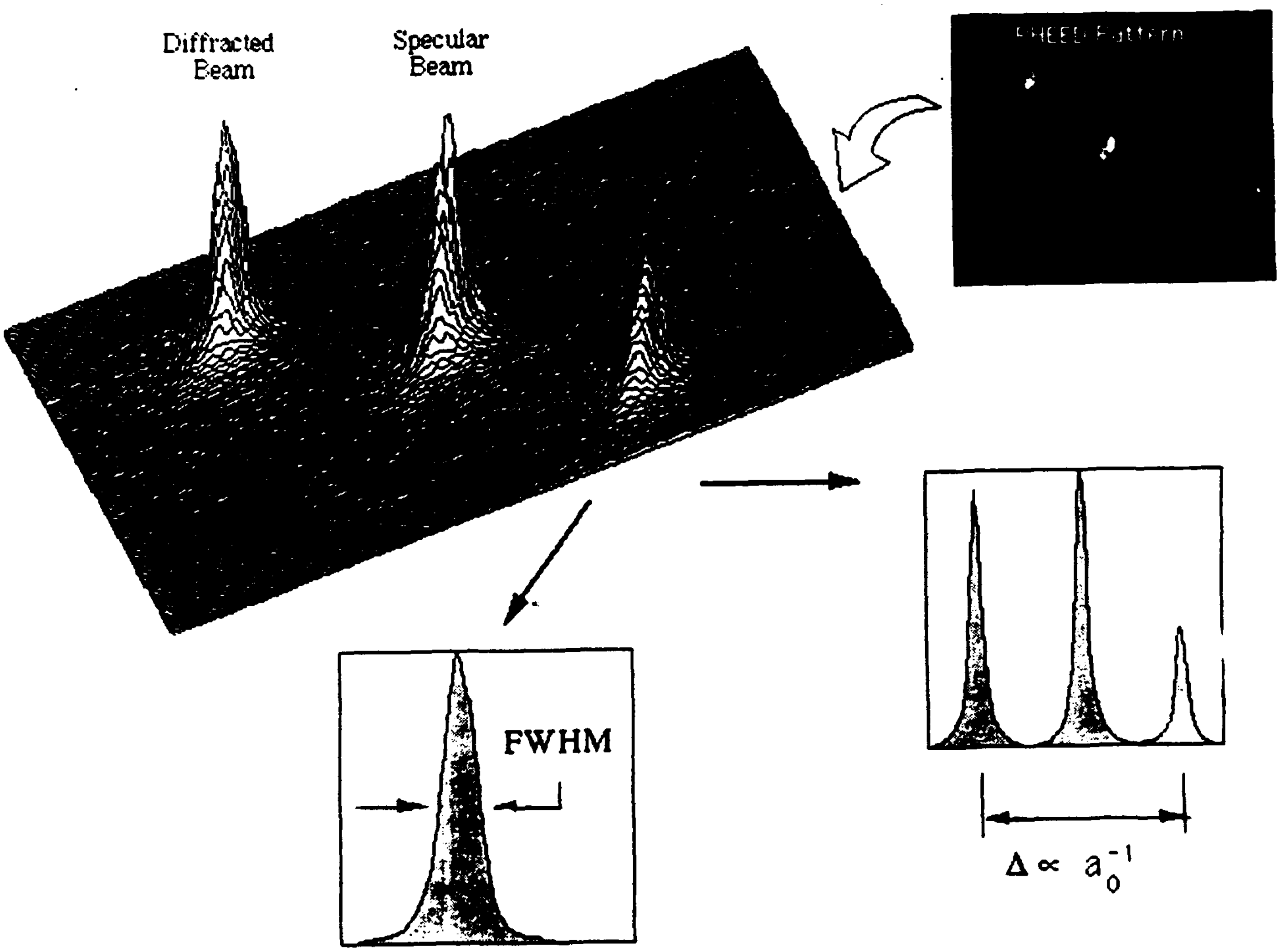


Figure 8. RHEED Image Analysis Display

The analysis is aided by electromagnetically sweeping the diffraction spots across a detector so that the distance between spots can be computed. The electromagnetic coil is wound to produce a peak magnetic field of 200 Gauss which is adequate to scan all the

first order spots. All the spots are swept across a photomultiplier whose signals are sent to an A/D converter and then to the computer for analysis. Figure (9) shows a schematic of the experimental set up. The lattice constant is calculated by measuring the electromagnetic field necessary to scan two diffraction spots. The intensity of the several diffraction spots in one scan is displayed in Figure (10). A computer program is written to compute the lattice constants automatically. A complete scan takes less than one second so the timing is compatible with typical MBE growth rate. By continuously monitoring the lattice constant one is able to detect any composition change due to flux variation. This technique can therefore provide feedback for flux control which will be pursued in future work.

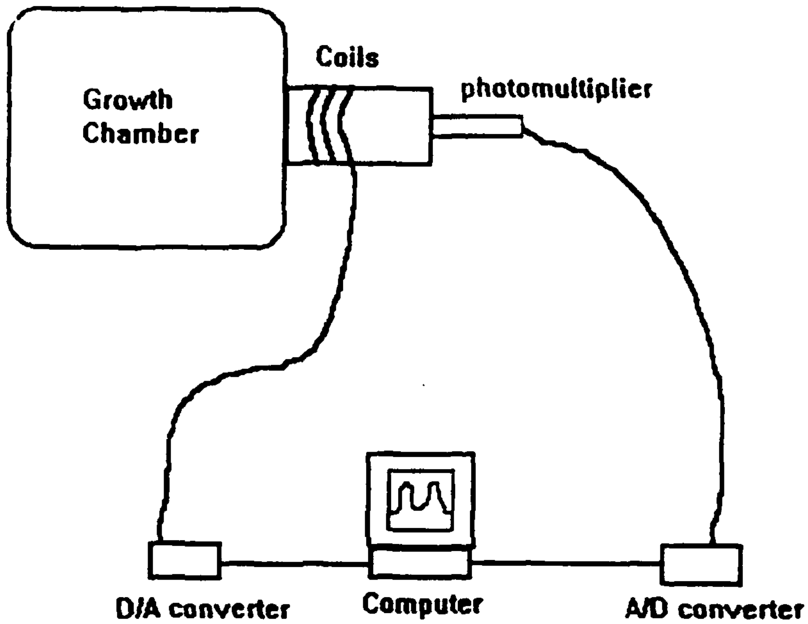


Figure 9. Distance of Movement of the Diffraction Spot as Function of Applied Voltage

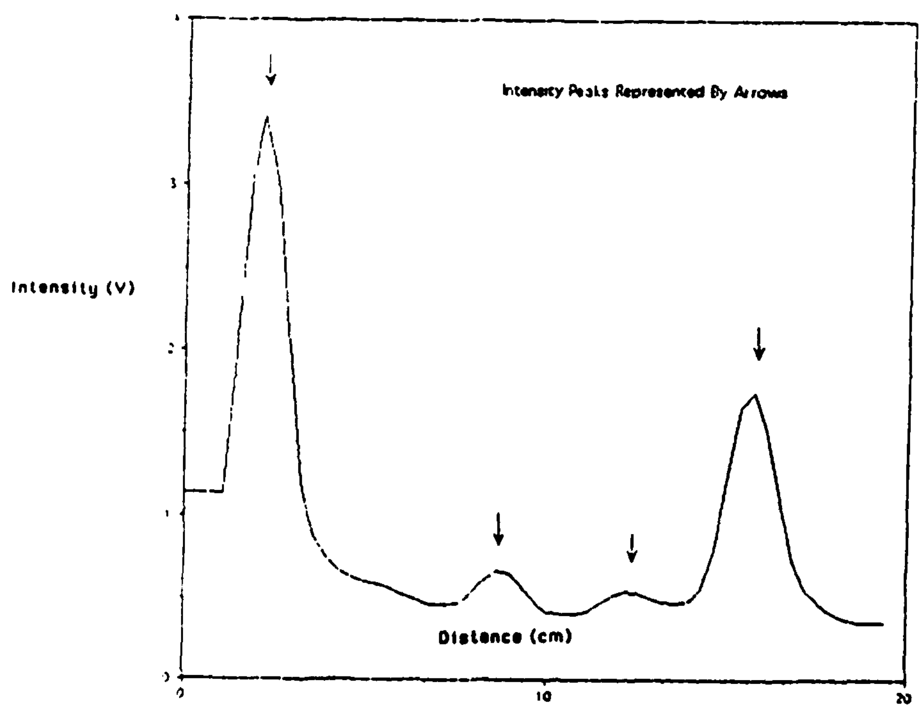


Figure 10. Diffraction Intensity as Measured by the Electromagnetic Coil Scan

3.2 Mass Spectrometer Feedback Flux Control

Since the mass spectrometer (MS) has proven to be a sensitive flux monitor, it leads naturally to experimenting with feedback from the MS for flux control. A special computer program was set up to compare the measured mass peak with a set value and then adjust the cell temperature accordingly. The experiment was very successful and clearly indicated that the feedback can control the flux very accurately and quickly to maintain stable flux. Figure (11) is a flux measurement that shows very stable flux level for the test interval. It demonstrated that using the MS feedback control can eliminate the flux transient.

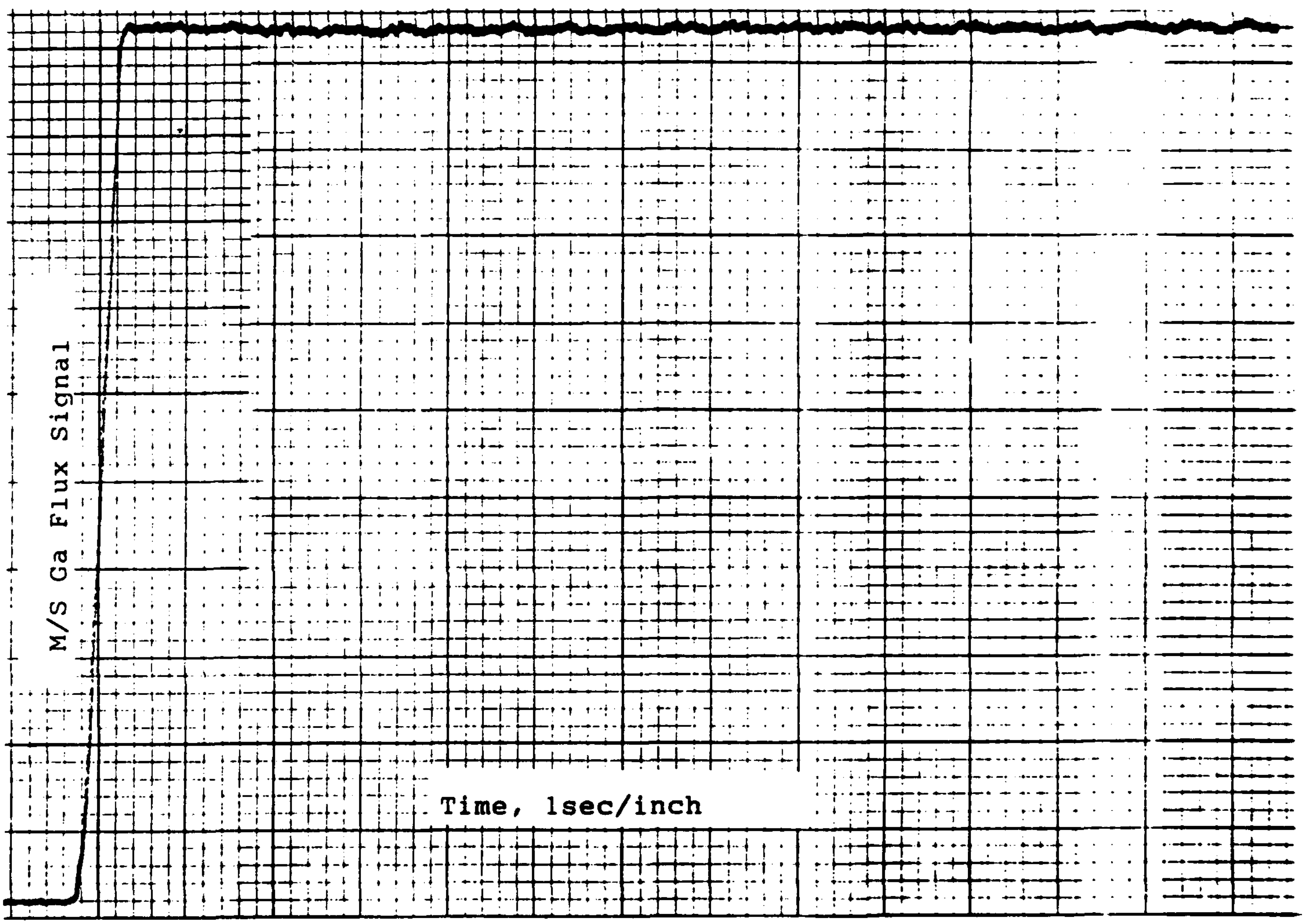


Figure 11 . Mass Spectrometer Controlled Ga Flux

Similar result was also obtained for the Al flux. We therefore believe that in future work it shall be possible to achieve real time control of the Al/Ga flux ratio, and thus the layer composition, very accurately using the MS in a multiplexed mode.

3.3 Flux Transients During MBE Growth

It is not straight forward, however, as we found out, to interprete the MS data as they are influenced by the presence of other species. The growth process takes place in a dynamic environment where a multiple of sources are simultaneously present. However, as far as we know there has been no investigation on flux transient during the growth. Therefore we undertook to study the transient behavior of the Ga source when As flux is also presen. It was immediately apparent that the As presence has altered the MS measurement. Figure (12) illustrates two traces of the Ga flux: trace (a) is a transient with the As off and trace (b) is with the As on. It can be clearly seen that the presence of As affected the transient behavior drastically. Note in particular the time interval immediately after shutter opening where the transients appeared very different. Although there is a possibility that this may be due to flux collision effect we believe that the real reason is changes of the ionizer characteristics due to

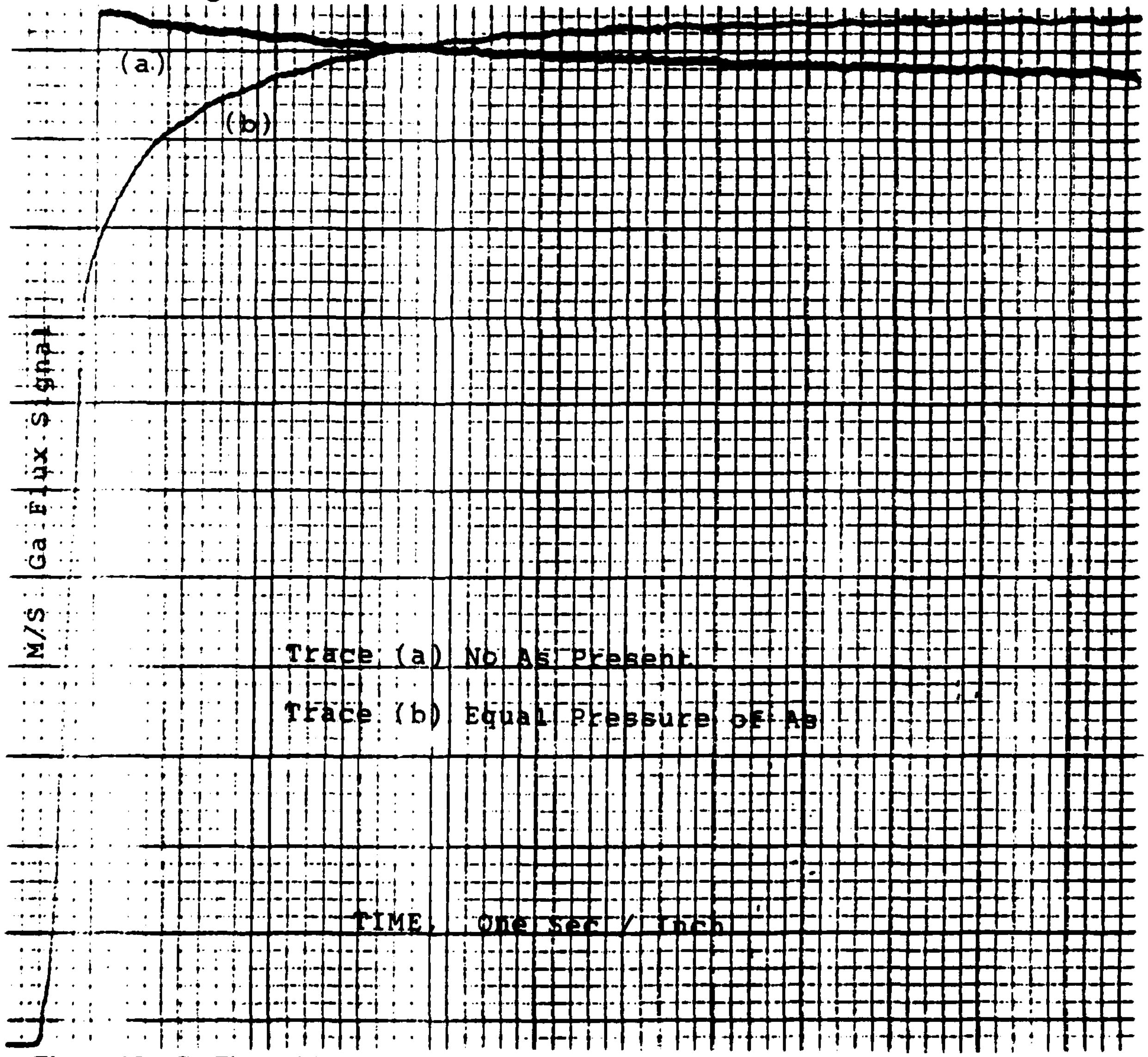


Figure 12. Ga Flux with As Off (a) and with an Equal Amount of As On (b)

rising As partial pressure. We were able to perform an experiment in that the Ga flux was maintained at a constant level while the As partial pressure was varied by adjusting the opening of an As valved cracker almost instantaneously. Figure (13) shows the Ga signals as a function of the As pressure which exhibited a very nonlinear behavior. Note that the As pressure covers the important growth operating range.

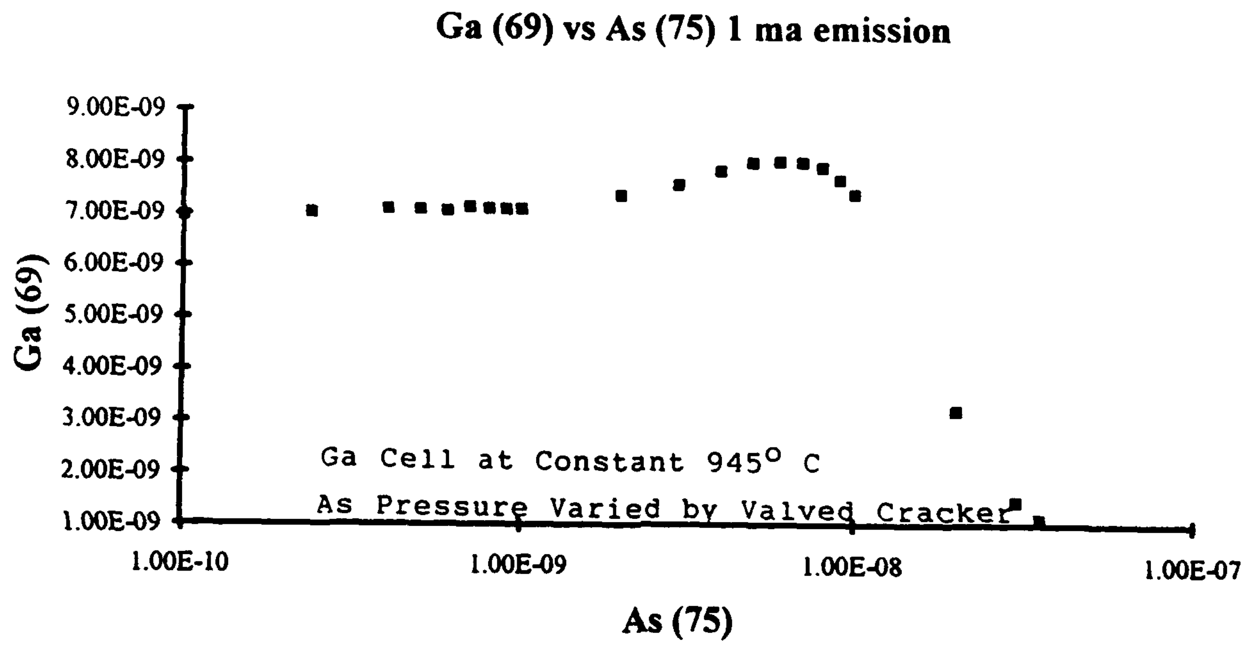
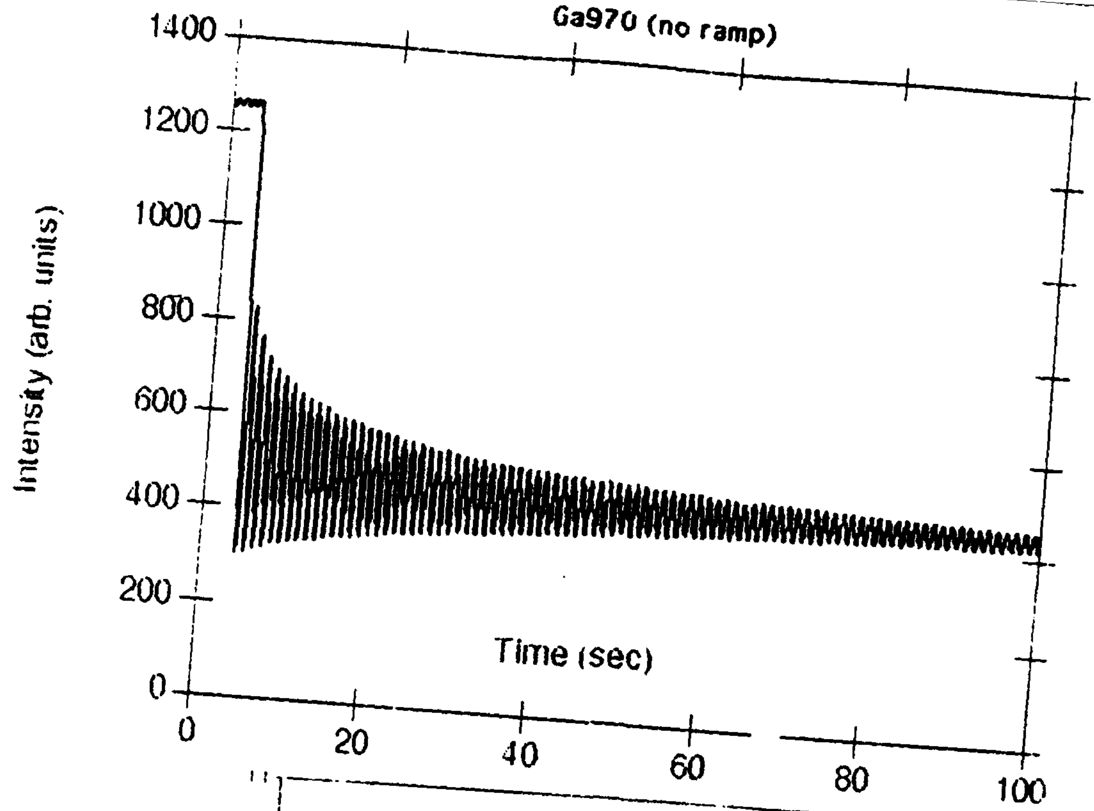
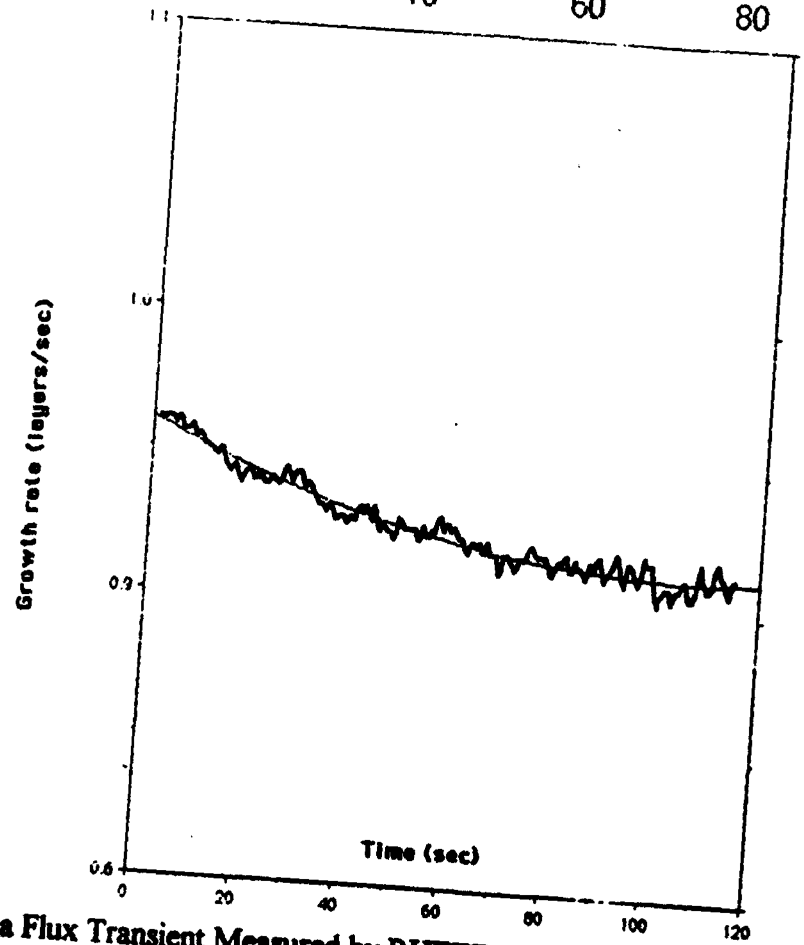


Figure 13. Mass Spectrum Signal of a Constant Ga Flux as Function of As Pressure.

To confirm our suspicion that the MS measurement may vary at different As levels we explore using RHEED to monitor the transient since it represents what really occurrs at the growing surface. Normally we use a Fourier Transform routine to compute the growth rate from RHEED intensity oscillations. However, because the computation averages over several periods it is not very sensitive to small variation from one period to the next. We therefore developed a special computer routine to calculate the rate from individual periods. Since the growth rate is linear with the Ga flux arrival it should vary exactly as the Ga transient. In Figure (14) we present the RHEED data (a) as well as the transient (b) measured in this manner. We can say in confidence that this represents the real transient and the transient is less than that obtained by MS of the Ga flux alone. We conclude that transient results reported in the literature is probably 40% too high.



(a)



(b)

Figure 14. Ga Flux Transient Measured by RHEED Oscillation. (a) RHEED Oscillation, (b) Growth Transient Computed from (a)

4.0 SUMMARY CONCLUSION AND RECOMMENDATION

Superior Vacuum Technology has carried out thorough characterization of source flux transient in III-V MBE. Substantial efforts have been spent on testing different experimental parameters. We have investigated several relevant factors affecting flux transients. It is determined that the flux transient can be much reduced by suitable heat ramping sequence; it should be especially effective with a dual filament cell. The shutter action affects the thermal environment and thus the flux transient but the shutter heater did not seem to make significant difference on its own. Real growth transient has been obtained for the first time using RHEED analysis. Most importantly real time feedback, either with a MS or some other technique, provides transient free flux.

The Phase I results provided an excellent foundation for future investigation in Phase II of the program. We will further optimize the performance of the sources and study the effects that could impact the MS behavior so that the MS may be used as a useful in-situ feedback sensor for MBE process control. AI control algorithm will be incoporated to optimize the control process. The MBE growth is a dynamic process in that many parameters are inter-related. Because there are often interaction between control loops a better control method would be to take into account these interactions. This is more difficult control application but would be well suited for a fuzzy logic controller because the interactions do not need to be characterized by a complicated mathematical model. It is possible to create a multiple input, multiple output fuzzy logic controller with relative ease. If this is coupled with AI it could automatically adjust the control parameters to obtain the best results.