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1.06-Micron Image Intensifier Tube Development

(October-December 1972)

JANUARY 1973

Sponsored by  
Advanced Research Projects Agency  
ARPA Order No. 1988

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⑥ 1.06-MICRON IMAGE INTENSIFIER TUBE DEVELOPMENT.

⑨ QUARTERLY TECHNICAL REPORT NO. 3  
(Oct--Dec- 72)

⑩ Prepared by J. Edgumbe

Approved by R. L. Bell

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⑬

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## SUMMARY

The objective of this program is to incorporate "broadband" 18-mm diameter III-V photocathodes with high 1.06-micron sensitivity into an electrostatic inverter image tube. The cathode is to be sensitive in the spectral range from  $\sim 0.7$  to  $\sim 1.1$  microns. In parallel with the research effort necessary to develop the photocathode, problems associated with incorporating the cathode into a tube are to be solved using the recently-developed "narrowband" InGaAsP-on-InP photocathode. Particular attention is to be given to such problems as shelf life, operating life, attaining high sensitivity and low background in an operational device, and improving the overall cosmetic appearance of the output image (cathode uniformity and blemishes).

During the quarter, the majority of the effort was devoted to the slumpage and cosmetic problems. The characteristics of the slumpage problem are reviewed. Although the mechanism responsible for the slump has not been determined, it is shown that the fabrication procedures have been developed to the degree that good shelf life is possible.

The new LPE growth facility, solely for growing InGaAsP cathodes for tubes, has been completed and reoptimization of the doping has nearly been accomplished. Several pre- and post-growth procedures for improving surface cosmetics have been tested with encouraging results. Implementation of these procedures will commence as soon as the cathode sensitivities are at an acceptable level.

Calculations have been made which indicate excellent broadband performance potential using InGaAsP sealed to glass. Experimental verification is expected in the next reporting period.

## FOREWORD

The work reported here was sponsored by the Advanced Research Projects Agency under ARPA Order No. 1988, and has been carried out in the Varian Corporate Research Solid State Laboratory under contract DAAK02-72-C-0396 with the U.S. Army Mobility Equipment Research and Development Center, Fort Belvoir, Virginia 22060. The supervising project scientist is Mr. Frederick F. Carlson, Night Vision Laboratory. The main program objective is the development of an image intensifier with high quantum efficiency at 1.06-micron wavelength, using negative affinity III-V compound photocathodes. Materials development and research on high efficiency cathodes has been partially supported by other programs. Contributions were made by G. A. Antypas, L. W. James, S. Reita, C. W. Wilkinson, C. B. Jones, and M. L. Simkins. Acknowledgment is made to the Varian LSE Division for invaluable assistance in provision of facilities for image tube fabrication and evaluation.



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## 1. INTRODUCTION

The objective of this program is to incorporate "broadband" 18-mm diameter III-V photocathodes with high 1.06-micron sensitivity into an electrostatic inverter image tube. The cathode is to be sensitive in the spectral range from  $\sim 0.7$  to  $\sim 1.1$  microns. In parallel with the research effort necessary to develop the photocathode, problems associated with incorporating the cathode into a tube are to be solved using the recently-developed "narrowband" InGaAsP-on-InP photocathode. Particular attention is to be given to such problems as shelf life, operating life, attaining high sensitivity and low background in an operational device, and improving the overall cosmetic appearance of the output image (cathode uniformity and blemishes).

## 2. INVESTIGATION AND DISCUSSION

### 2.1 PERFORMANCE IN SEALED-OFF DEVICES

Pertinent tube design, EPIC processing, and general III-V tube technology information can be found in the first quarterly report on this contract. A detailed discussion of equivalent background illumination, resolution, and gated performance is contained in the second quarterly report.

#### 2.1.1 Cathode Sensitivities

An opaque infrared sensitivity probability distribution for InAsP and InGaAsP photocathodes is presented in Fig. 1; these data were obtained prior to the completion of the new growth facility solely for the purpose of growing InGaAsP cathodes for image tubes. The first cathodes grown in the new facility had very low yields. After considerable testing the low sensitivities have been found to be due to over-doping of the epitaxial layer. The doping of the cathode is established by adding a small amount of zinc to the melt prior to each growth; the amount of Zn to be added having been rather crudely determined some time ago. A comparison of the sensitivities obtained, using the same procedure for adding Zn prior to each run, in the two facilities is shown in Fig. 2. On reducing the amount of Zn added to the melt prior to the first growth a rather critical optimum, Fig. 3, was observed. Prior to this effort the amount of Zn necessary to achieve optimum sensitivity on the first growth of a new melt was never formally established. At this time a complete procedure for adding Zn has not been determined, but should be in a week or two, and a better probability distribution than shown in Fig. 1 should result.

The reason for the gross change in the amount of Zn now required (about a factor of two less) is believed to be due to improving the purity of the hydrogen source, possibly the primary Zn loss mechanism being due to a contaminant in the hydrogen

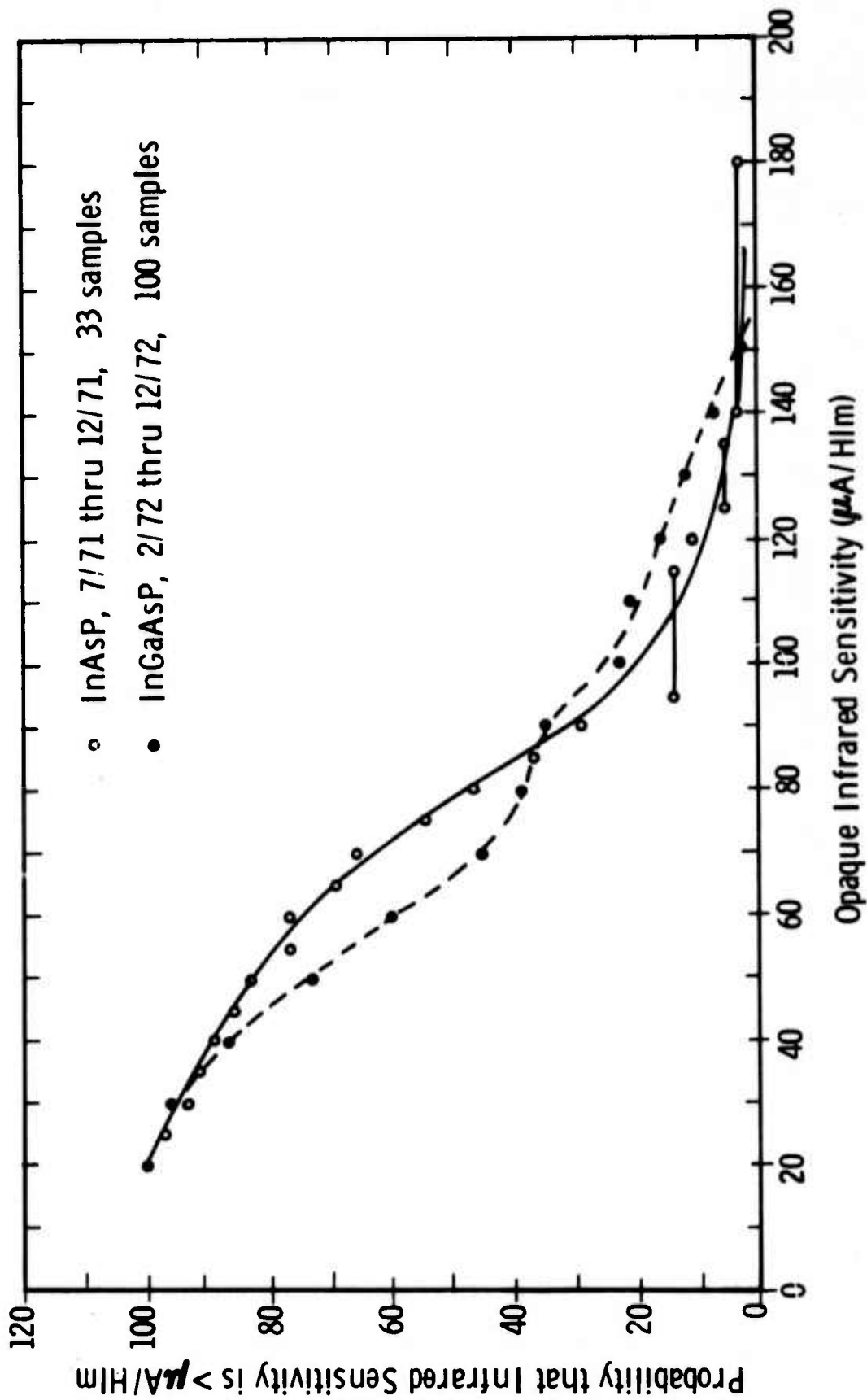


Figure 1. Probability distribution for the opaque infrared sensitivity obtained from InAsP and InGaAsP.

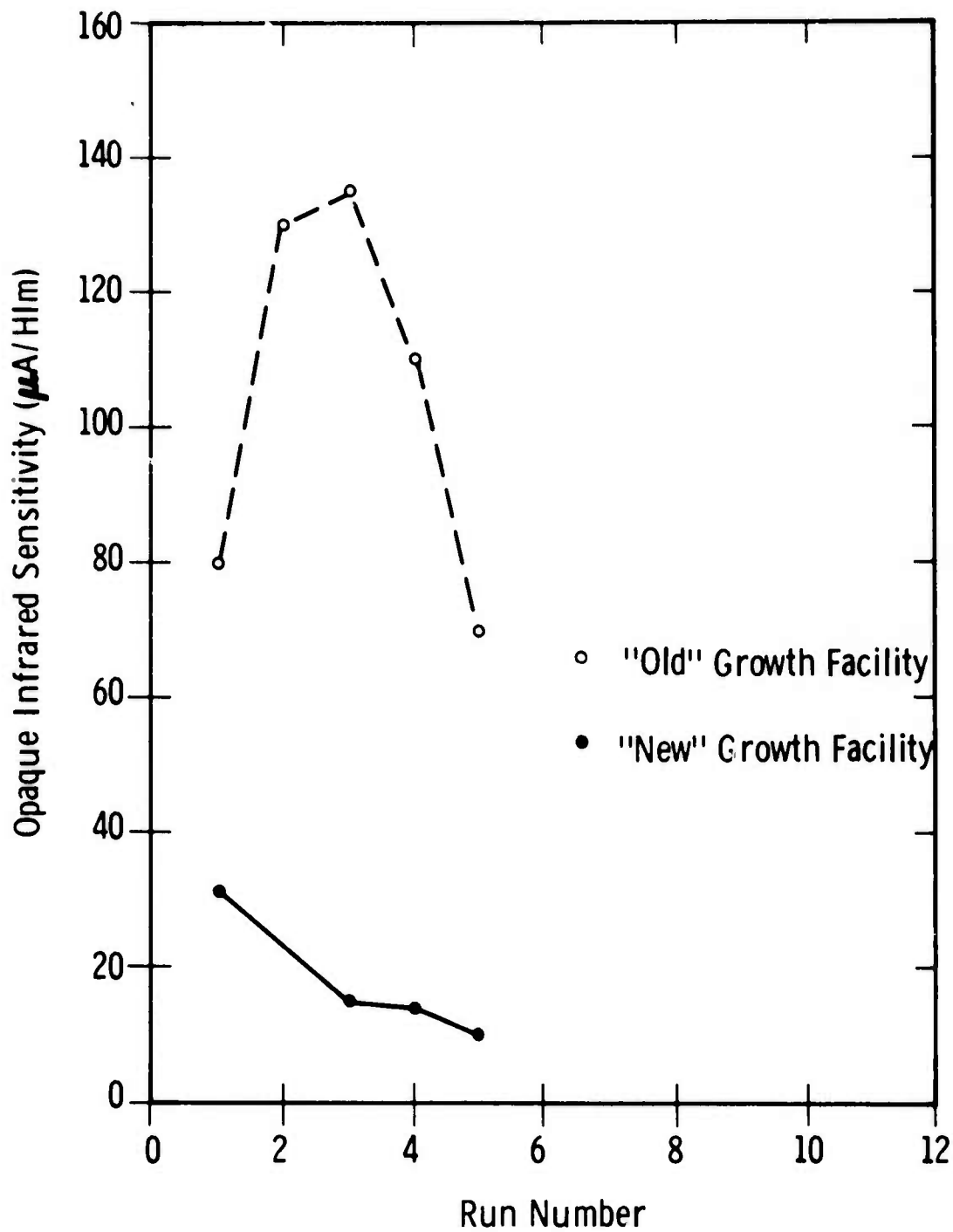


Figure 2. Infrared sensitivity vs run number for two growth facilities using the same melt composition and doping procedure.

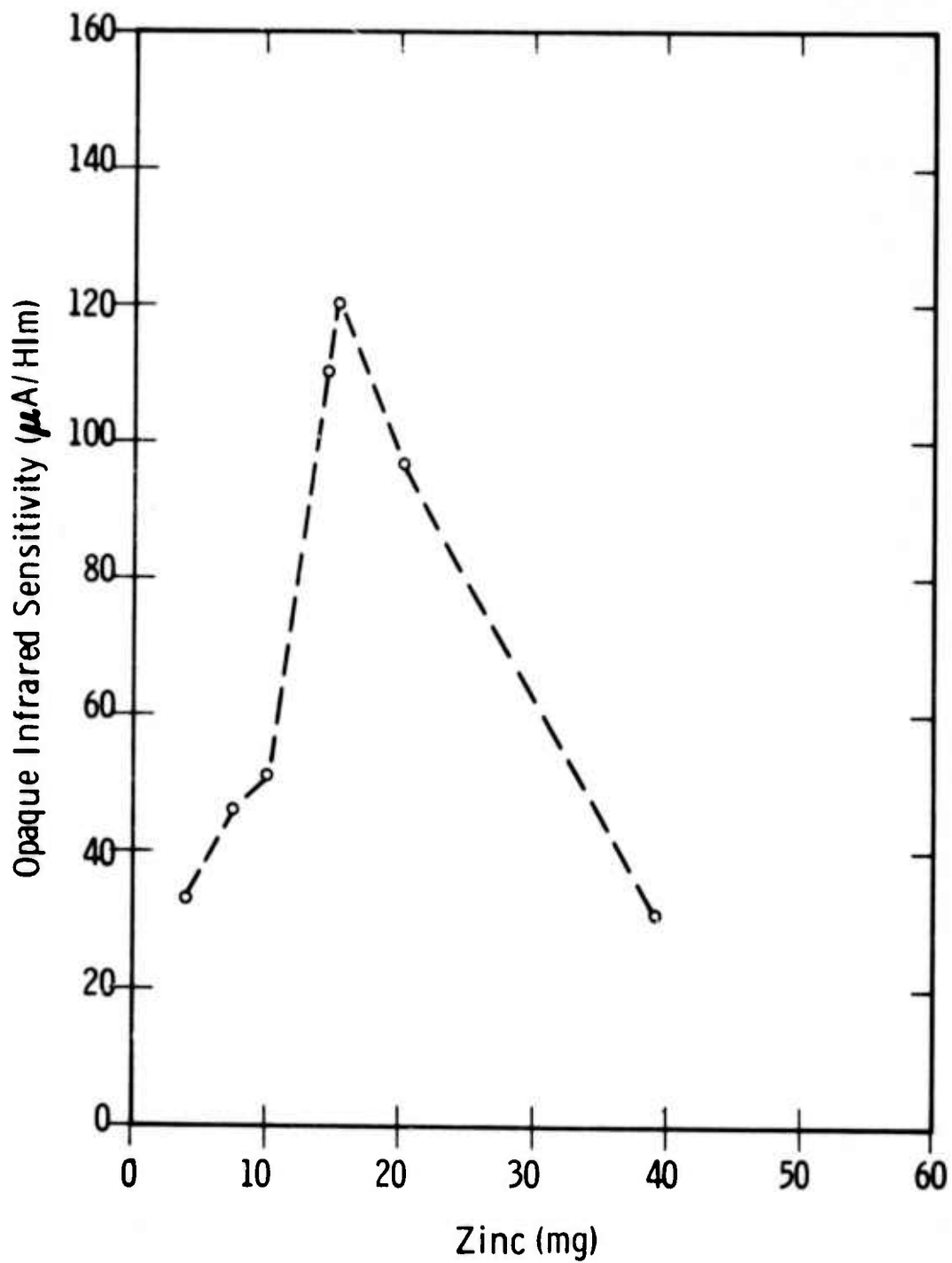


Figure 3. Opaque infrared sensitivity vs the amount of Zn added to the melt prior to the first growth.

(chemical transport as opposed to simple evaporation). During the coming quarter, the use of covered boats will be initiated to minimize the Zn loss, and improve the control of the doping.

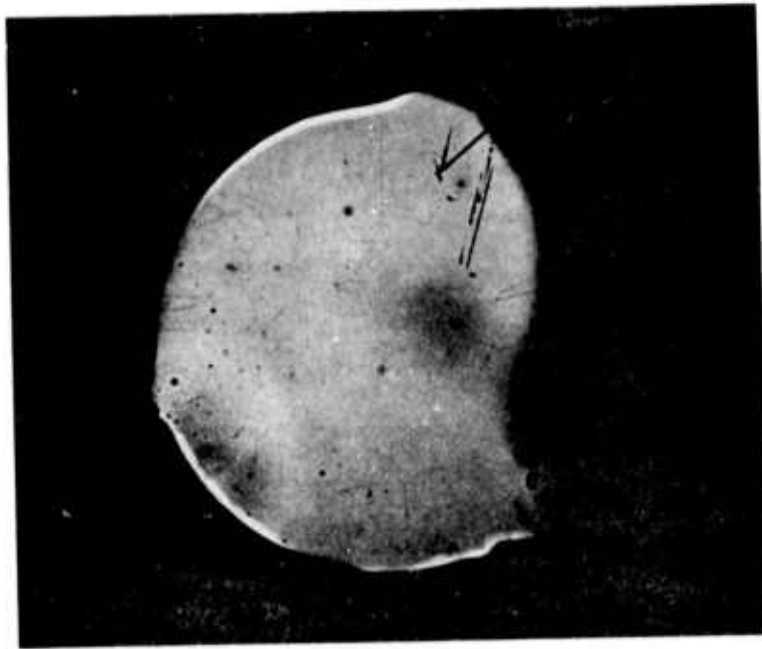
It now appears that a major factor in our inability to consistently obtain high sensitivity on each attempt has been due to variations in doping. Without some assurance that a cathode will have high sensitivity it is obviously very difficult to do meaningful experiments on post-growth procedures to improve surface cosmetics or variations in the activation to improve attainable sensitivities and performance in image tubes. Thus the time being devoted to this problem should simplify other experiments which are planned to improve the overall performance of the image tubes. Also, hopefully, some increase in the maximum attainable sensitivity should be obtained.

#### 2.1.2 Uniformity and Blemishes

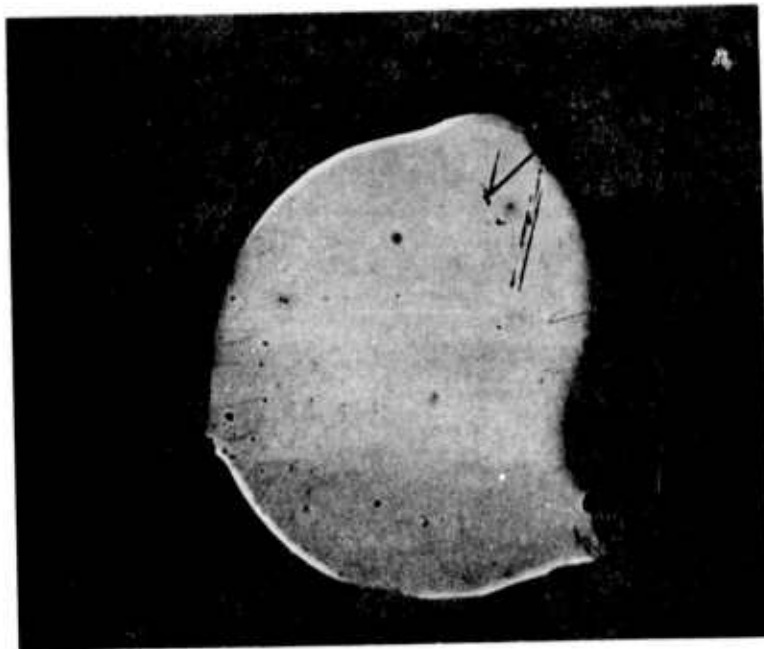
2.1.2.1 Nonuniformities Resulting from Poor Activation Geometry. As discussed in the previous quarterly report one non-uniformity associated with the activation remained to be solved, namely, the "black shade," see Fig. 4. This has been accomplished during this reporting period, by modifying the way in which oxygen is introduced into the preprocessor during activation--see Fig. 5. The activation geometry of the preprocessor now appears to be adequate to obtain uniform activation over the 18-mm cathodes required on this contract.

2.1.2.2 General Blemish Considerations. As a result of the time required to complete the new growth facility and reoptimize the doping, less effort than planned was devoted to the blemish problem. An appreciation of the cosmetic problem can be readily obtained by studying Figs. 4 and 5; the front surface photographs were obtained by reflecting 1.0-micron light off the gate electrode. Several types of blemishes are apparent in the photographs, as follows.



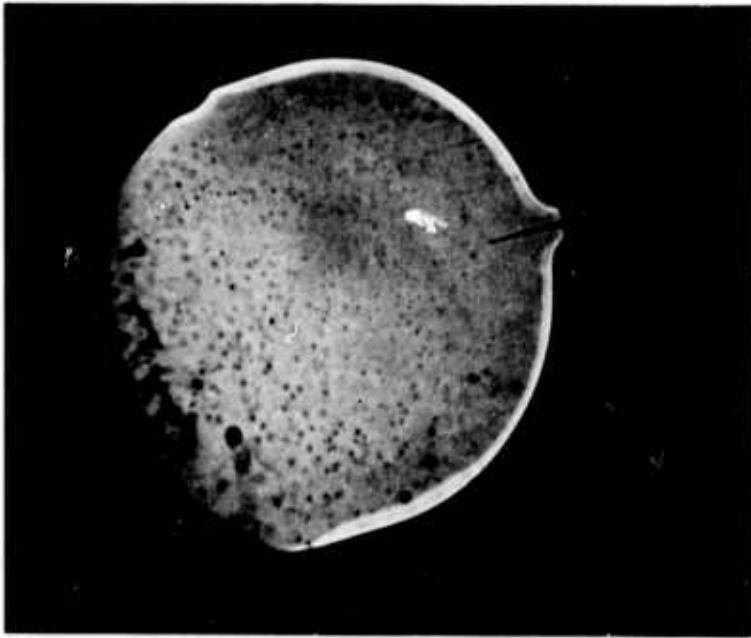


**(a) 1.0 Micron  
Semitransparent  
Illumination**

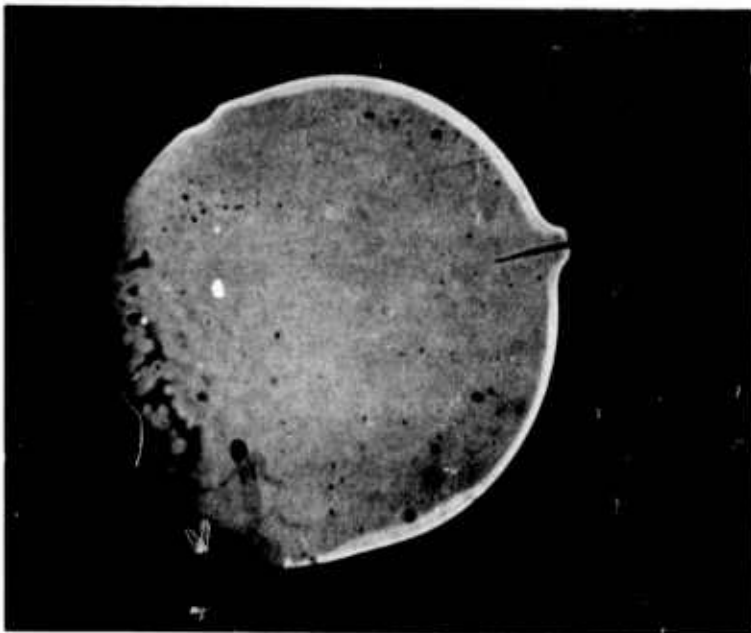


**(b) 1.0 Micron  
Opaque  
Illumination**

Figure 4. Photographs of the Output Image of INV #56 With Uniform Input Illumination



(a) 1.0 Micron  
Semitransparent  
Illumination



(b) 1.0 Micron  
Opaque  
Illumination

Figure 5. Photographs of the Output Image of INV #58  
With Uniform Input Illumination

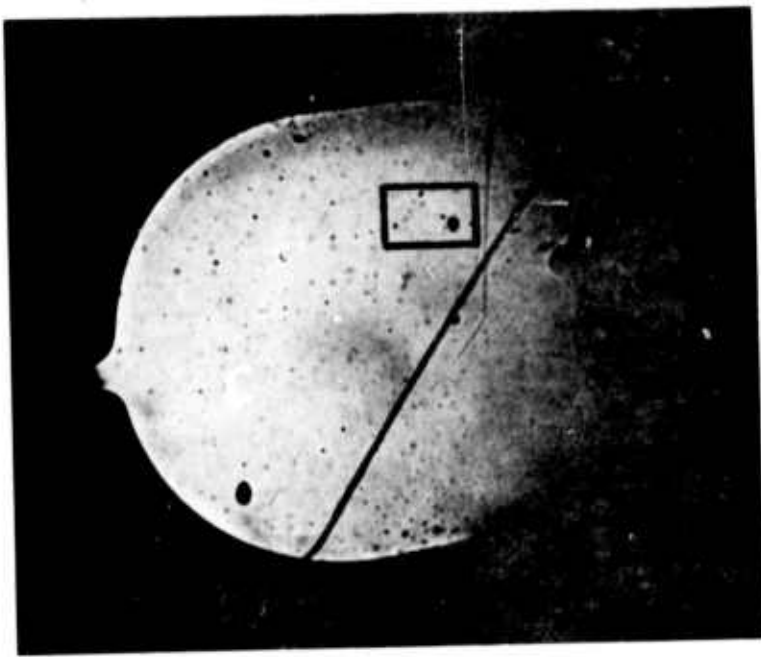
(1) Scratches on the front surface which appear with both semitransparent and front-surface illumination. These result from post-growth handling, including the transfer operation, and are not believed to be a fundamental problem.

(2) Scratches on the back side of the cathode which appear with semitransparent illumination only. These are scratches in the  $\text{SiO}_2$  antireflection coating and should be eliminated when apparatus now on order, for chemical vapor deposition of the oxide, is installed in the next reporting period.

(3) Thickness nonuniformities which appear as shaded regions with semitransparent illumination, and, as discussed in previous reports, result from temperature nonuniformities in the small-bore furnaces used for growth to date. Although no cathodes grown in the new large three-zone furnaces have been transferred into image tubes, very uniform thickness across 18 mm has been observed. Thicknesses varying between 3 and 3.5 microns across the diameter appear typical compared to 3 to 6 microns using the small-bore furnaces. Cathodes grown in the new furnaces should be transferred into image tubes in the next week.

(4) Defects which are believed to be at the interface and appear as gray, roughly circular blemishes with semitransparent illumination only, see Fig. 5. These are due to some, as yet undetermined, variation in the details of the growth procedure because only about one-third of the cathodes are observed to have this type of cosmetic problem. Note, for example, that this defect is not observed in Fig. 4.

(5) Finally, the very black blemishes; these result from at least two problems, both of which are made apparent with the aid of Fig. 6. Figure 6(a) is the usual semitransparent photograph of a cathode in an image tube. Figure 6(b) is a micrograph of the region of the cathode outlined in Fig. 6(a) obtained after the cathode was removed from the tube. The large black



(a) Photograph of the Output of INV #54 With Uniform Input Illumination



(b) Microphotograph (50X) of the Area Outlined in (a)

Figure 6.

blemish is due to melt which was not removed from the cathode after growth. The smaller blemishes, six of which are visible in the right-half of Fig. 6(b), are particles believed to be chips from the edge of the cathode broken off during transfer to the image tube. These particles were removed quite easily by ultrasonic agitation in a dilute Alconox solution. Methods are under investigation to more effectively remove the melt and minimize the number of particles bound to the surface.

2.1.2.3 Quaternary Surface Texture Dependence on Growth Temperature. It is well known that the surface defect density is a function of the substrate quality. It has been observed, however, that it is an even stronger function of the growth temperature. (For a defect-free substrate, one would expect no dependence of the surface texture on growth temperature.) Our layers are grown at 650°C; however, when the growth originates at 600°C we observe a marked increase of the surface defect density; conversely, growth at 700°C results in practically mirror-smooth surfaces. Growth at higher temperatures involves other problems, such as control of the liquid composition due to the volatility of the components. This, however, is not considered a serious problem. At present we are investigating the growth of InGaAsP at 700°C lattice-matched to InP.

The effect of the growth temperature on defect density can be seen in the Fig. 7(a,b,c) series of microphotographs.

### 2.1.3 Shelf Life and Operating Life

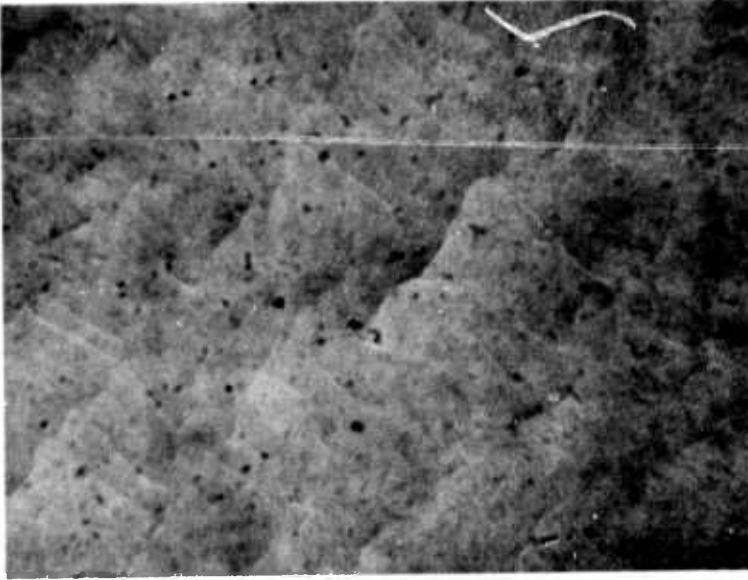
2.1.3.1 Short-Term Shelf-Life Instabilities (Slumpage). The most serious obstacle to a successful completion of this contract is the reversible degradation of cathode sensitivity with time (slump) which necessitates periodic oxygen treatments of the tubes. Considerable progress has been made on this problem, and it is believed that a solution will be found in the near future. Early in the work on sealing 1.06-micron sensitive III-V cathodes



(a) 600°C



(b) 650°C



(c) 700°C

Figure 7. Microphotographs (50X) of InGaAsP Layers Grown at Three Different Temperatures



in devices the typical shelf life was that of D#5, Fig. 8(a). This degradation was irreversible in that the sensitivity could not be restored with Cs and/or oxygen. With the development of the EPIC process now used to fabricate image tubes the shelf life was improved, taking the form of a reversible decrease or slump (Fig. 9), periodic oxygen treatments being required to maintain the high sensitivity. Improvements in the precesiation of the tube and the activation geometry resulted in better shelf life, fewer oxygen treatments being required, as shown in Fig. 8(b).

One tube sealed off during the quarter, INV#58, exhibits the best stability observed to date--see Fig. 8(c). No oxygen treatments have been administered to this tube in the 60 days since pinch-off. Three things were slightly different in the fabrication of INV#58. First, the oxygen inlet to the pre-processor was modified to eliminate the black shade (see Sec. 2.1.2.1). Second, a 100°C bakeout was inserted into the processing after the precesiation of the tube to, hopefully, more uniformly distribute Cs in the tube envelope. Several tubes have been made since INV#58 with these two modifications however, and none have the stability of INV#58. Third, due to an error in the tube assembly the spring which holds the cathode in the tube was poorly aligned and the cathode is extremely loose, essentially only lying on the input window. Although it seems like a very remote possibility that spring tension could cause the slump, an experiment is planned to test the effect stress has on the yield of the quaternary.\* The mere fact that the procedures used to fabricate 1.06-micron sensitive image tubes produced a tube with the stability of INV#58 seems to indicate that a solution to the slumpage problem is quite near.

---

\* The effect of stress on Cs<sub>2</sub>O-activated GaAs was reported by James et al., at the PSEE Conference, U. of Minnesota, August 1971. In this experiment a decrease in sensitivity was observed when a stress was applied to an activated GaAs photocathode. The sensitivity could not be restored with Cs and/or oxygen while the stress was applied, the sensitivity did not recover when the stress was removed, but the sensitivity could be restored to the original yield with Cs after the stress was removed.

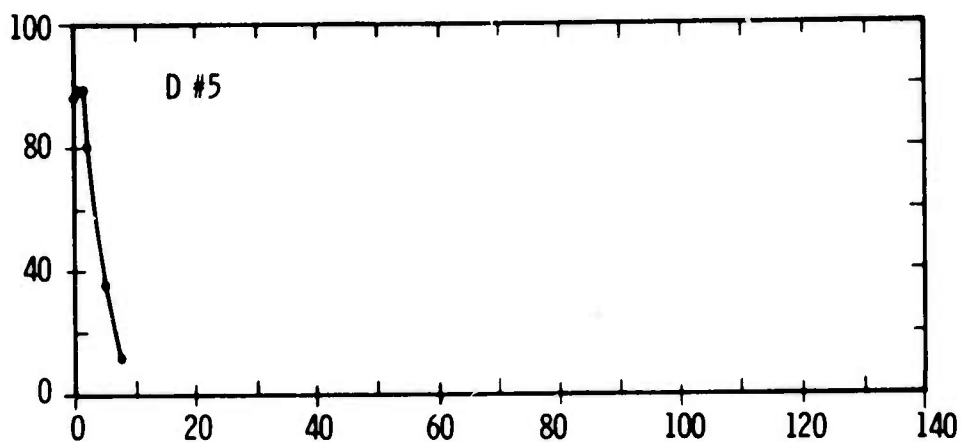


Figure 8(a)

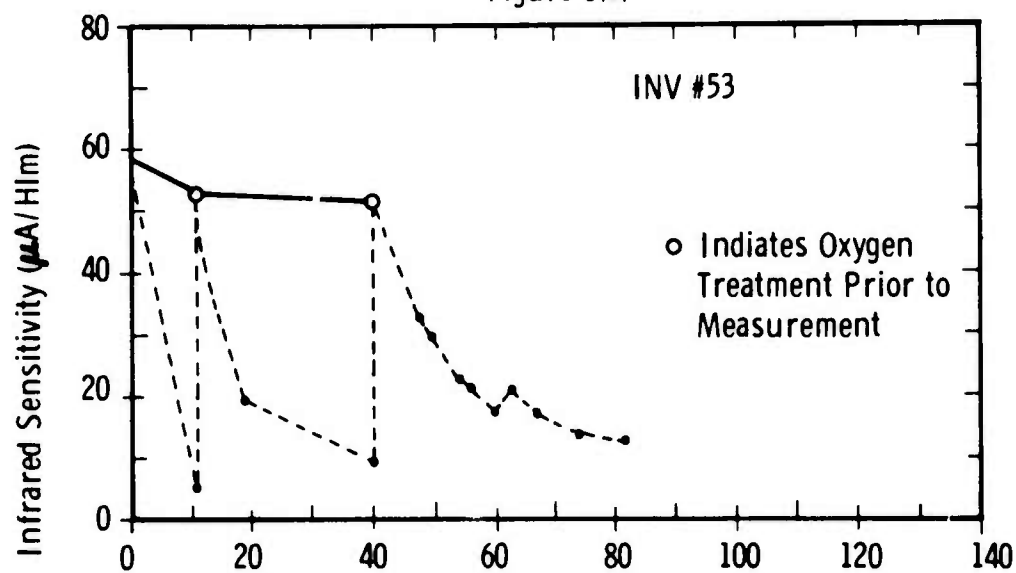


Figure 8(b)

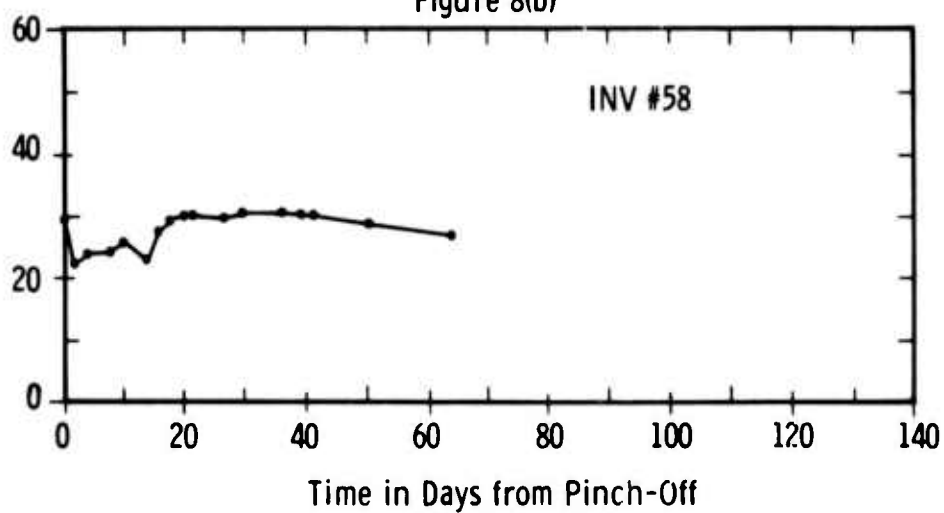


Figure 8(c)

Figure 8. Shelf life of three tubes illustrating slump.

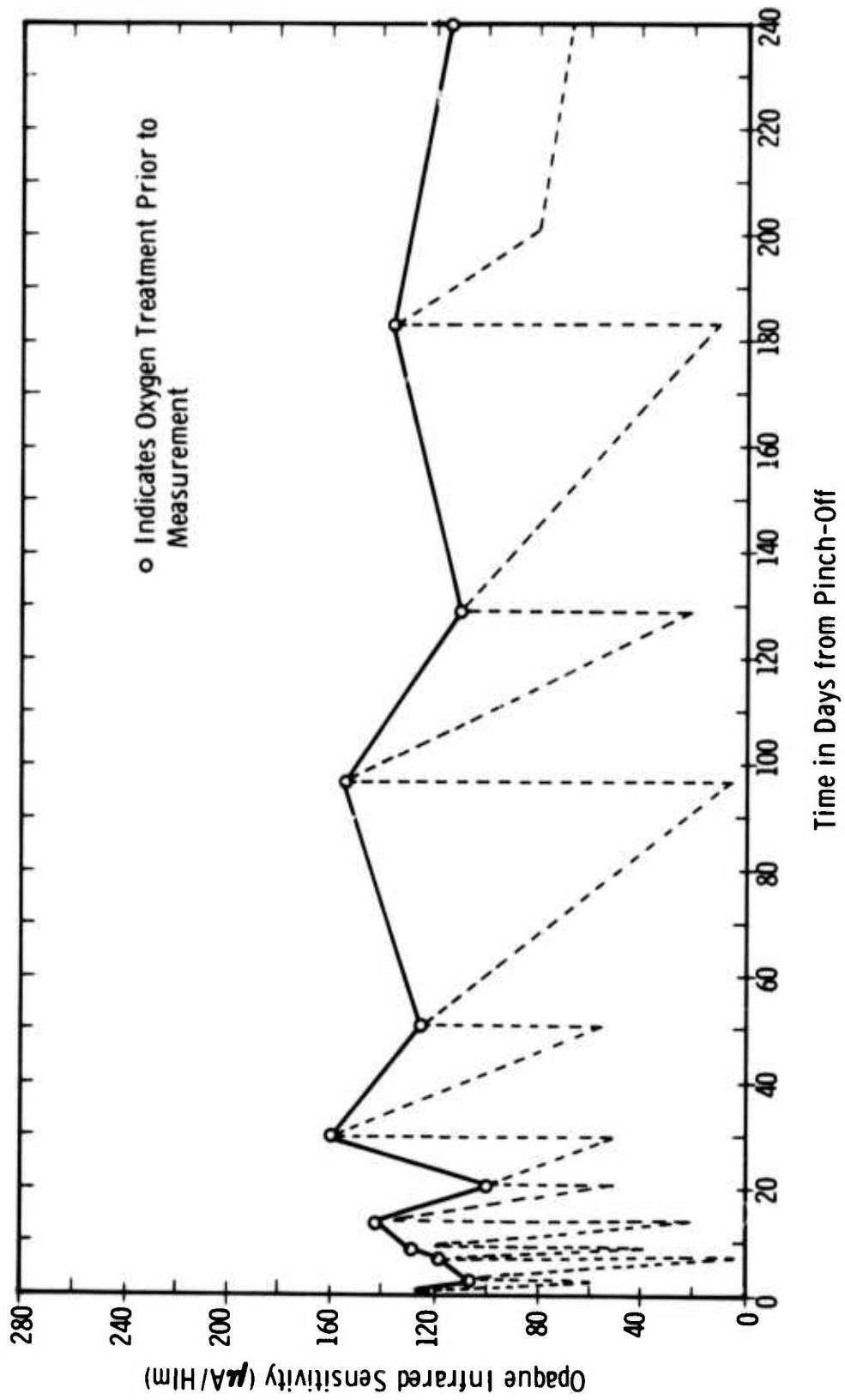


Figure 9. Detailed shelf life of D#18.

Several other characteristics of the slump have been uncovered:

(1) When a cathode slumps the sensitivity and dark current change by roughly the same factor. This, coupled with data reported earlier on the behavior of a very low bandgap cathode sealed off in a tube indicates that the slump is due to an increase in the work function, and not, for example, due to changes in the heterojunction barrier.

(2) The sensitivity and dark current change in a reasonably predictable manner with successive oxygen treatments (Fig. 10). It seems impossible to explain the loss in sensitivity as simply due to an increase in Cs-oxide thickness since the dark current would have to increase by a factor  $\geq 10$  (see Fig. 13 of Quarterly Report No. 2) and this is not observed.\*

(3) No change in the observed slumpage behavior is noted when the tubes are stored in vacuum. This seems to eliminate from consideration small leaks in the tube envelope and diffusion of gases through the walls, for example, hydrogen diffusion through Kovar.

(4) It has been determined that the slump is accelerated by heating and eliminated by storage at  $-15^{\circ}\text{C}$  indicating that the slump is the result of a thermally-activated process. Although cold storage and operation is a solution to the problem, it is not considered acceptable. Heating the entire tube, although accelerating the slumpage, does not produce any permanent improvement in stability. Heating too hot ( $\sim 100^{\circ}\text{C}$ ) or too long can cause permanent degradation.

---

\* One way of explaining the observed data is that the slump is due to excess Cs within the tube and that the longer-term gradual decline in sensitivity with successive oxygen treatments is due to residual gas contamination of the activator. The amount of Cs required to produce the observed slump is experimentally known to be  $\sim 0.1$  monolayer, thus many oxygen treatments would be required to produce any observable change in sensitivity or dark current.

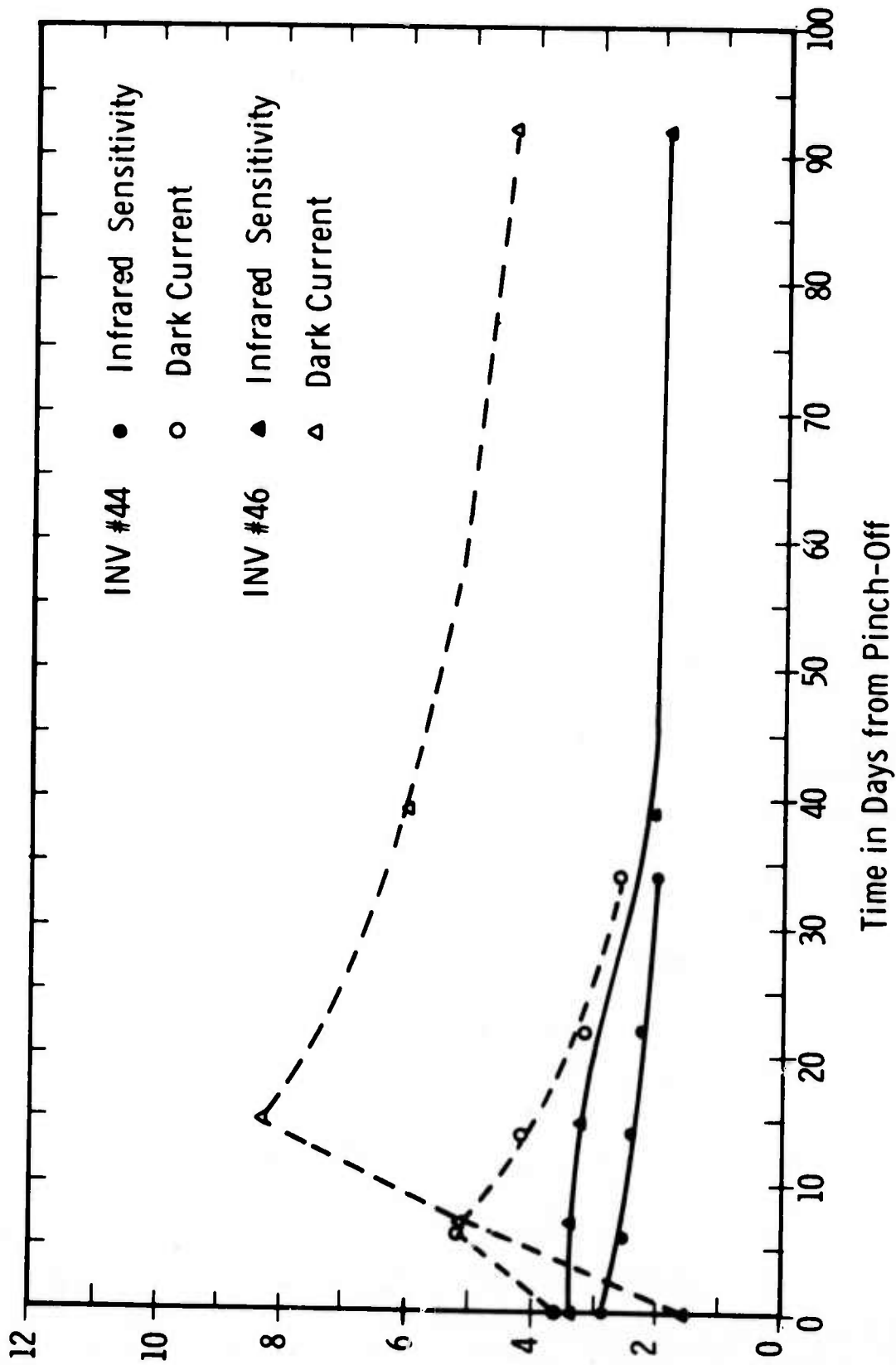


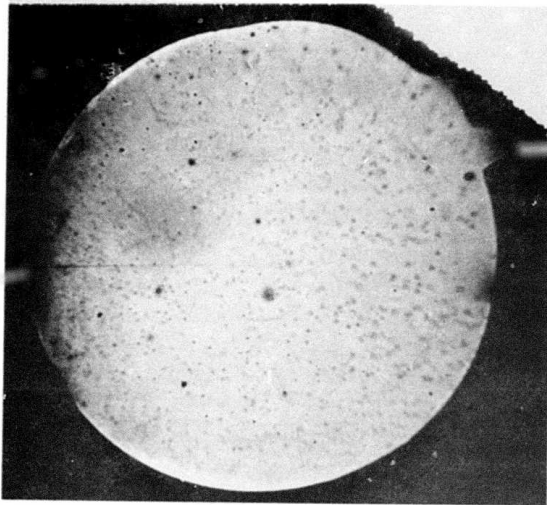
Figure 10. Infrared sensitivity and dark current as a function of time from pinch-off. Cathode peaked with oxygen prior to making measurements.

(5) Cathodes have been re-peaked by preferentially heating the input of the tube on a hot plate (75-180°C), but the sensitivity obtained has always been less than would have been obtained with a normal oxygen treatment. Also, no permanent improvement in stability has been observed.

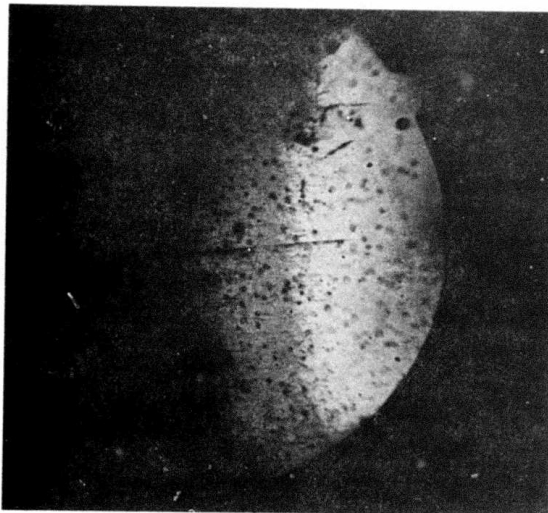
Two sizes of quaternary cathodes are routinely used in fabricating image tubes at this laboratory, 1/2-inch and 18-mm diameter. To somewhat complicate the picture, the slump observed in the two different tubes is not the same. A uniform decrease in sensitivity is observed in the case of 1/2-inch diameter tubes, whereas a gradual preferential shading of the cathode, always from the same side, is observed in 18-mm diameter tubes (as required on this program)--see Fig. 11. Besides the size difference the only other difference is the processing system in which the cathodes are activated, in particular, the way in which the oxygen is admitted to the system. Hence, this unusual behavior is believed to result from nonuniform oxygen deposition on the cathode during activation. All systems have now been made the same and incorporate the latest innovations in oxygen and Cs sources to obtain the best uniformity. Tubes with 18-mm diameter cathodes will be fabricated on the modified systems shortly to test the validity of this argument.

2.1.3.2 Long-Term Gas Evaluation in III-V Tubes. With the hope of shedding some light on the slumpage problem as well as the long-term shelf life, a flash-filament experiment has been initiated. This experiment (see the photograph in Fig. 12) consists of a standard inverter tube envelope in which is mounted a tungsten filament, a quadrupole residual gas analyzer (RGA) mounted close to the tube, and a valve for isolating the tube from the vacuum system and RGA. The tube was baked and outgassed in the usual manner and data have been obtained before and after the precesiation procedure. Residual gas buildup on the filament is monitored by closing the tube isolation valve for a period of





(a) Just After Pinch-off



(b) Three Days After Pinch-off

Figure 11. Photographs of the Output Image of INV #57 With Uniform Input Illumination

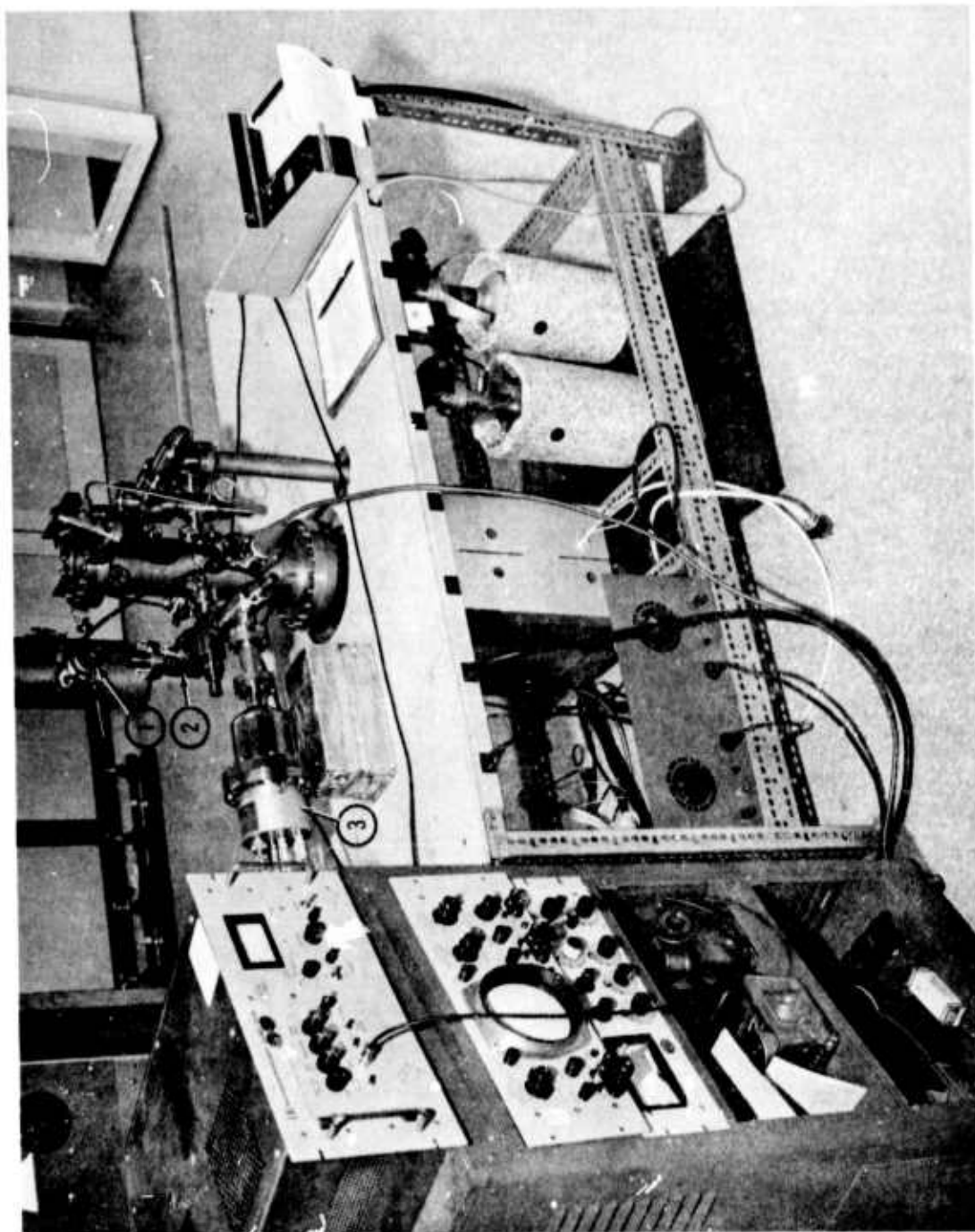


Figure 12. Photograph of the Flash-Filament Experimental Setup.  
(1) Standard inverter tube containing filament.  
(2) Valve for isolating the tube from the vacuum system.  
(3) Quadrupole residual gas analyzer.

time, then opening the valve and flashing the filament while monitoring the gas of interest. Methane data were obtained simply by monitoring the mass 15 peak while opening the isolation valve, since flashing the filament cracks any methane present on the filament.

Prior to cesiating the tube, the prominent residual gases observed after the valve had been closed for a few days were  $H_2$ , CO, and  $CH_4$ . The system pressure also increased momentarily to  $\sim 5 \times 10^{-9}$  torr when the isolation valve was opened. After cesiating the tube the prominent gases were  $H_2$  and CO. The efficiency of Cs as a getter was apparent in that pressure increases of only  $\sim 10^{-10}$  torr were observed upon opening the isolation valve. Peak partial pressures observed when the filament was flashed are shown in Fig. 13. Cesium apparently getters methane at a very high rate, essentially eliminating it as a residual gas. Calculations indicate that 30 nA represents roughly one monolayer (an accurate calibration will be obtained when the experiment is completed). Experimentally, the effect that various gases have on the sensitivity of the III-V photocathodes is known--see Table I. On the basis of the previously-measured sensitivity of the quaternary to  $H_2$  and CO, it is difficult to interpret the results of the present experiment. From the results presented in Fig. 13 and Table I, one would expect image tubes with quaternary cathodes to die irreversible in a few hundred hours and, as discussed in Sec. 2.1.3.1, this is clearly not the case.

2.1.3.3 Preliminary Operating Life Data. Preliminary operating life data were obtained during the quarter but the results are difficult to interpret because of the slumpage problem. The amount of decrease or increase in cathode sensitivity during operation depends in detail on the activation state of the cathode. For example, the cathode in INV#40 dropped 5% with 3.5 mC of charge passing through the tube at 7 kV just after an oxygen treatment. A few days later when the cathode was slumping, the observed

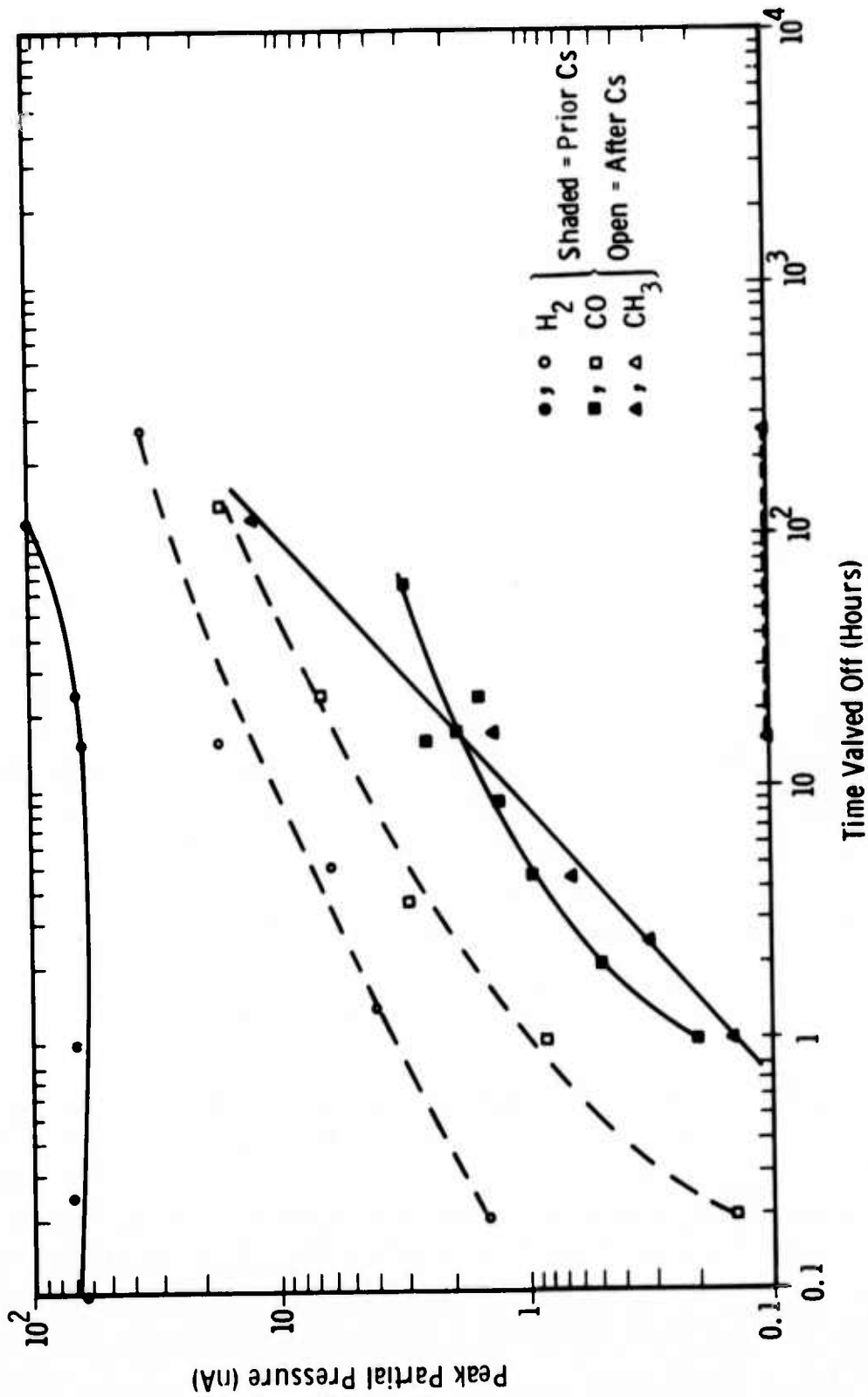


Figure 13. Results obtained in flash-filament experiment.

TABLE I

Approximate Langmuirs exposure for a drop in luminous sensitivity of 20% (exponential drop-off assumed). The InAsP and InGaAsP tested had bandgaps of 1.18 eV, while the InGaAs tested had a bandgap of 1.3 eV.

	Gas Material	H <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub> O	CO <sub>2</sub>	CO	CH <sub>4</sub> <sup>*</sup>	O <sub>2</sub>
As-activated surface	GaAs	no effect <sup>†</sup>	1500	0.32	0.035	0.73	6.9	
	InAsP			0.43	0.029	0.10	4.5	0.025
Re-ciesiation to peak	InGaAs					0.12		0.074
	InGaAsP	0.43 <sup>‡</sup>		1.8	0.073	1.68	13.8	1.0
Heat-cleaned surface at 500°C	GaAs			0.60	0.386?	0.13	6.4	0.7
	InAsP					0.42		
	InGaAsP	0.43 <sup>‡</sup>			> 20	11.3		
	GaAs							

\* Gauge Langmuirs, all others true Langmuirs.

† S. Garbe, private communication.

‡ 20% decrease in infrared sensitivity.

degradation was 25% with 5.6 mC of charge passing through the tube. An increase in cathode sensitivity of 20% was observed in another tube, INV#46, with 6.3 mC of charge passing through the tube at 7 kV (this can occur, for example, when the cathode is slightly over-cesiated and oxygen is desorbed during operation). Further operating life tests are planned when the shelf-life problem is improved. It is encouraging to note that the deterioration in %/mC found here is about the same as results obtained on GaAs image tubes some time ago, both here and at Night Vision Laboratory. Although, the above results are subject to confirmation, it is our opinion that operating life will not be a serious problem provided the imaging system incorporates some sort of bright source protection.

#### 2.1.4 Background in the Gated-Off Mode

In the previous quarterly report, field emission from the gate was reported to be the mechanism responsible for limiting the background in the gated-off mode (focus electrode at  $\sim -2$  kV). During this quarter several tubes were tested in the OFF mode and the results are presented in Fig. 14. The variation in magnitude and shape of the curves obviously indicates that the simple field-emission mechanism is not the complete story. This is not believed to be a serious problem but techniques will be considered for minimizing the OFF background.

## 2.2 BROADBAND INFRARED PHOTOCATHODES

The performance of InGaAsP sealed to glass as a broadband semitransparent photocathode has been calculated and the results are presented in Fig. 15. The assumptions used in the calculations are zero optical reflection, a bandgap of 1.19 eV, a diffusion length of 4 microns, and an escape probability about 15% less than the best accomplished to date. The beneficial effects of a thin layer of InP is apparent, and excellent passive and 1.06-micron performance is indicated. In the coming quarter, cathodes will be fabricated to experimentally verify these calculations.



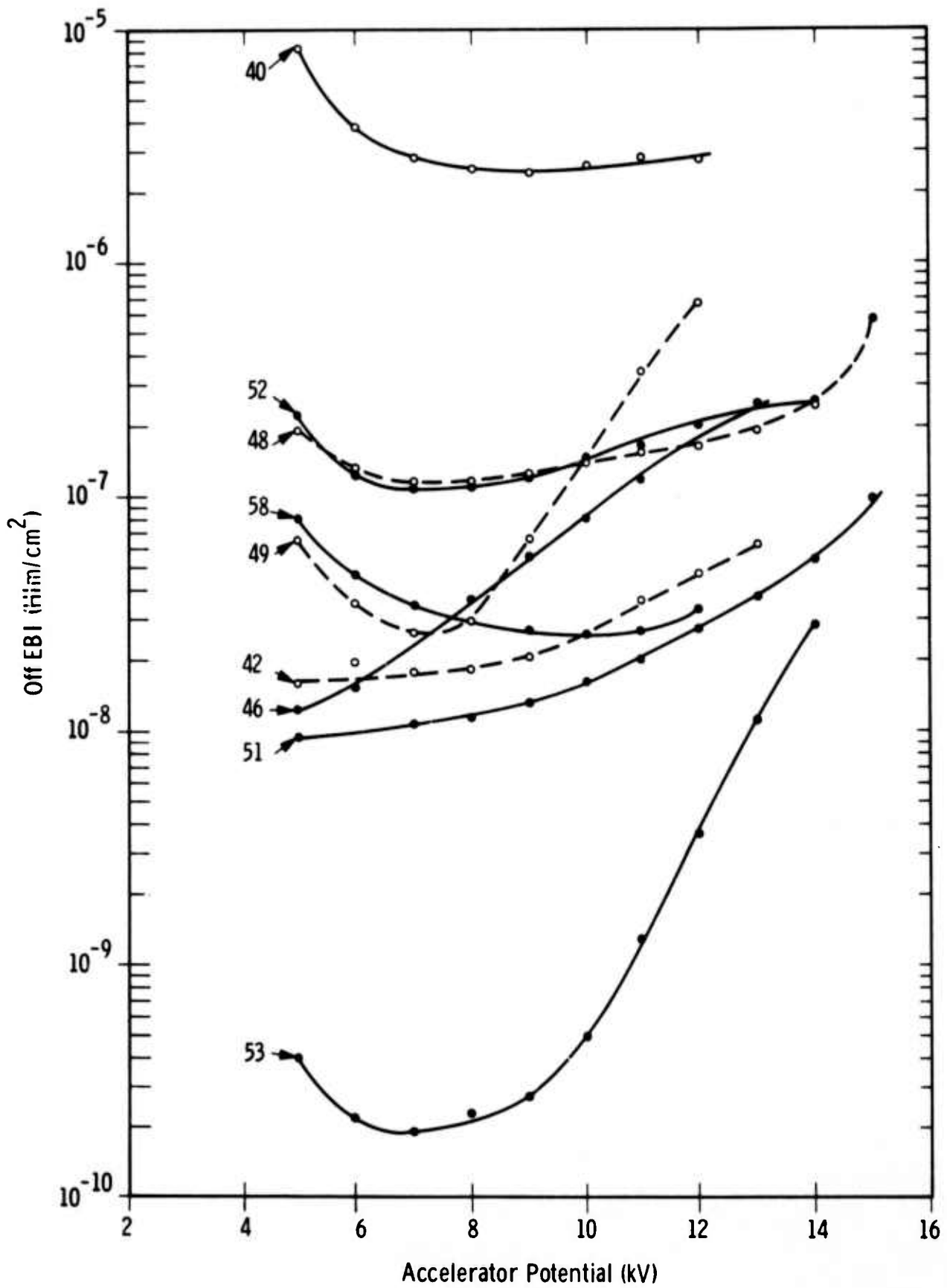


Figure 14. Background in the gated-off mode (focus electrode at -2 kV) vs accelerator potential for several inverter tubes with InGaAsP cathodes.

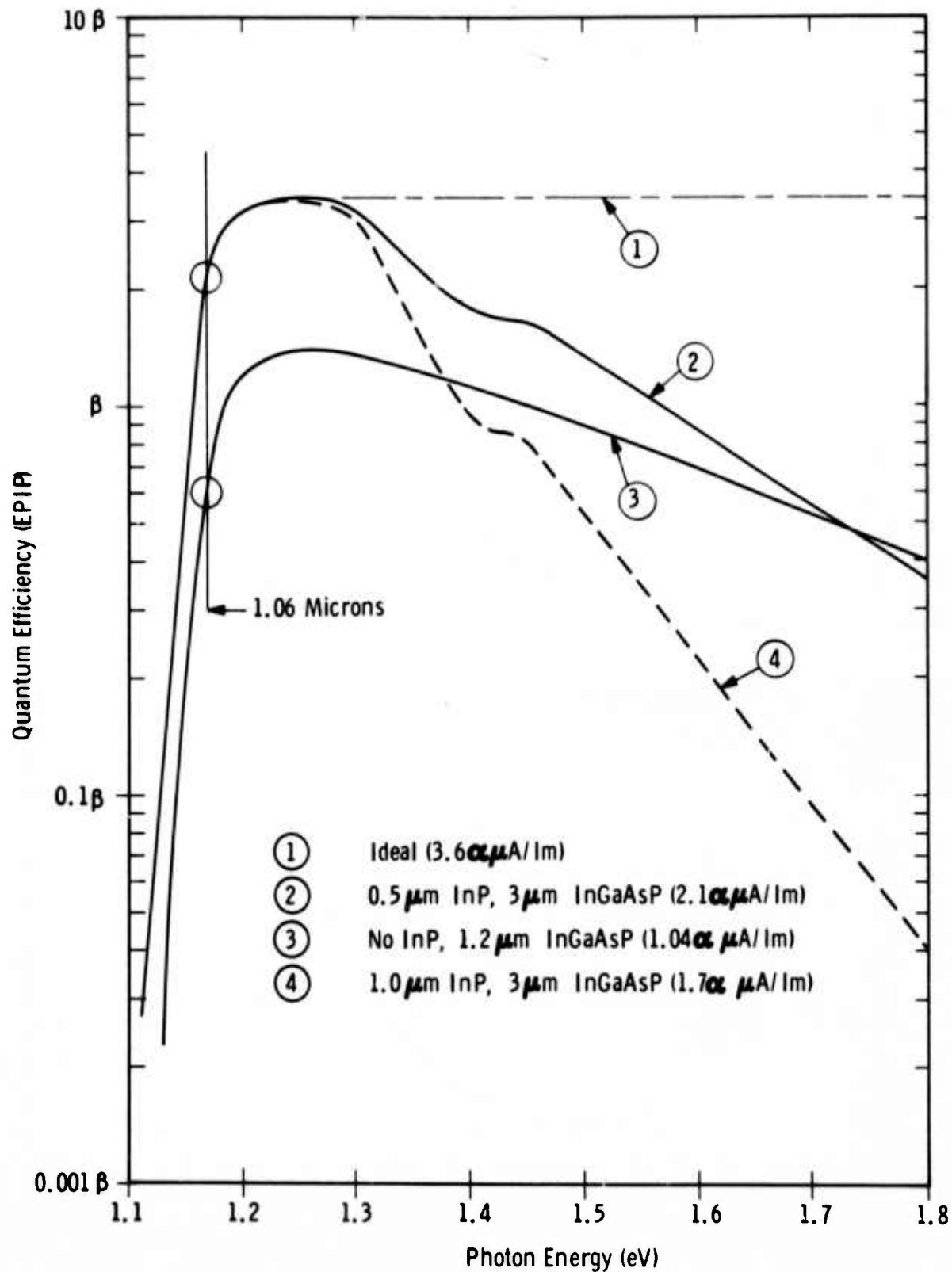


Figure 15. Calculated broadband performance of InGaAsP sealed to glass assuming zero optical reflection, a 1.19 eV bandgap, and a 4-micron diffusion length.

### 3. CONCLUSIONS AND RECOMMENDATIONS

Considerable progress has been made on this program. Many of the parameters associated with 1.06-micron sensitive image tubes incorporating III-V photocathodes are understood, and are within the specifications of the contract. Among these characteristics are 1.06-micron sensitivity (in the narrowband case), background, resolution and MTF, uniformity of activation (not including cosmetic appearance), operating life, and gating. The major problems remaining are shelf life (the slumpage problem), cosmetic appearance, and the development of a cathode with a broadband spectral response.

Essentially all the effort on this contract has now been directed toward solving the slumpage problem (a tube problem) and the cosmetic appearance problem (a materials problem). In the case of the slumpage problem, it is important to emphasize that the procedures used to fabricate tubes have produced one tube, INV#58, during the quarter which has excellent shelf life. This seems to indicate that a solution to the problem is near, and each step in the processing will now be critically evaluated for variation. With regard to the cosmetic problem, a similar argument applies, namely, the procedures used have yielded excellent-looking cathodes at times. Again it is a question of scrutinizing each step in the procedures. Progress on both of these problems has been somewhat hampered by poor control of attainable cathode sensitivity; this, of course, the result of our incomplete understanding of this technology, which is just in its infancy.

Since the pressing problems of cathode cosmetics and slumpage have received the bulk of the effort during the quarter, little has been done on the development of a broadband 1.06-micron photocathode. Calculations indicate that excellent performance is possible with InGaAsP sealed to glass and experimental verification is expected in the coming quarter.

The MTF machine is now operational and routine measurements on 1.06-micron image tubes will start next month.

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13. ABSTRACT

During the quarter, the majority of the effort was devoted to the slumpage and cosmetic problems. The characteristics of the slumpage problem are reviewed. Although the mechanism responsible for the slump has not been determined, it is shown that the fabrication procedures have been developed to the degree that good shelf life is possible.

The new LPE growth facility, solely for growing InGaAsP cathodes for tubes, has been completed and reoptimization of the doping has nearly been accomplished. Several pre- and post-growth procedures for improving surface cosmetics have been tested with encouraging results. Implementation of these procedures will commence as soon as the cathode sensitivities are at an acceptable level.

Calculations have been made which indicate excellent broadband performance potential using InGaAsP sealed to glass. Experimental verification is expected in the next reporting period.

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14 KEY WORDS	LINK A		LINK B		LINK C	
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Operating life						
Gated-off mode						
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