

XUV FILTER DEVELOPMENT  
FOR THE  
EXTREME SOLAR ULTRAVIOLET MONITOR

Contract No. NAS-5-23082

Goddard Space Flight Center  
National Aeronautics and Space Administration

By -

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THE EXTREME SOLAR ULTRAVIOLET MONITOR  
(Sigmatron, Inc., Santa Barbara, Calif.)

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## I. INTRODUCTION

The use of ultrathin aluminum foil. XUV filters for rocket and satellite applications has been broad enough that a certain confidence exists on the part of fabricators and users of these delicate components that they may be designed to function as desired for long periods in satellites. This has been achieved, however, with some failures and considerable learning.

Such confidence did not exist at the outset of this program with respect to certain filter materials of interest in the Acmospheric Explorer series. These materials are typical of tin, indium and titanium. Filter foils nominally 1600 Å thick, supported on 70-LPI nickel screen, are physically delicate for the rigors of rocket launch and are much more susceptible to corrosion than their film-on-glass counterparts. This corrosion is known to be dominated by moisture in the gaseous environment and is evidenced by generating pinholes. Most foils this thin have some pinhole leakage, but high humidity can increase this leakage by a factor of  $10^4$  in one day.

Thus this program has been carried out to provide quickly the confidence in tin, indium and titanium that usage and development have provided over many years for aluminum. In addition to this, Sigmatron's participation has included an advisory capacity with respect to instrument design to incorporate experience into filter design.

## II. PROGRAM

Tests in this laboratory have shown highly beneficial effect of alloying a small amount of silicon in aluminum foils in increasing the moisture resistance (aging) and improving the microstructure.

The problems anticipated with tin, indium and titanium could be overcome, possibly, by either the alloying or a tandem structure wherein the pure metal film resides on a foil of another material. In the tandem solution the base foil must have a compatible XUV transmission, and the complex transmission is expected to be a product of the component layers.

The particular problems which the alloying or tandem approaches would solve vary with each material. At the



outset, tin was expected to be the least troublesome. Tin is a very soft metal, melting at 232°C.; therefore, there was some concern, although misplaced, regarding strength to endure the rocket launch stress and thermal recrystallization. These same problems were felt to be much more severe in the case of indium because of its extreme softness as a metal and the low melting point of 156°C. Although titanium melts much too high to present a thermal problem, thin foils prepared by vacuum deposition are noted for their brittleness. There was, therefore, some concern regarding the strength of these filter foils.

It was of vital interest to characterize all three with respect to aging. The program plan to explore these problems and their possible solutions lay in the following sequence of investigations and milestones:

- (1) Evaluate the aging and thermal recrystallization character of pure metal foils.
- (2) Evaluate possible tandem structure bases.
- (3) Determine the effect of binary alloying on the XUV transmission and aging of Sn:Al alloy over the complete range of compositions.
- (4) Use the transmission, aging and recrystallization information determined from the alloy series to make valued judgment of alloy foils which might provide the desired characteristics.
- (5) Use the base foil evaluation to make valued judgment for tandem foils.
- (6) Fabricate and deliver filters of the best materials.

### III. METHOD

Aging was determined as a parameter of pinhole leakage of white light in a sensitive photometer whose lower limit of detectability was a transmittance (expressed as the fraction of incident light transmitted) of  $10^{-9}$  and the practical upper limit was  $10^{-4}$ . Calibration was achieved by neutral density filters, and the accuracy, which was



not particularly important in the series, was probably  $\pm 5\%$ . Reproducibility was quite good, being  $\pm 5\%$  in repetitive measurements.

Exposure of pairs of filters to various relative humidities was achieved by storing the filters in the water vapor in equilibrium with certain chemicals. Humidity was determined periodically by measuring the dewpoint within the container. A 20-percent R.H. ambient was provided by controlled concentration of sodium hydroxide solution. The 75-percent R.H. ambient was provided by a saturated solution of sodium-potassium carbonate.

Thermal recrystallization was detected via pinhole transmission by heating a test filter at a rate of  $2^{\circ}\text{C. per minute}$  in a position within an oven built into the photometer. Alloy compositions used in this work were accurately controlled in the preparation of bulk alloys. This was usually achieved by melting the components sealed in quartz, carbon-lined and evacuated ampoules, or vacuum-melting in an alumina crucible. The aluminum-titanium alloy was too reactive chemically to be made by these techniques and was, therefore, formed in place on the evaporation filament prior to the deposition.

It is recognized that the foil composition does not necessarily correspond to the composition of the starting material because of the differences of vapor pressures of the alloy components. A table of pertinent vapor pressures compiled and used in this laboratory is provided in Appendix I. The technique employed in this laboratory provides for a tungsten filament which evaporates a small volume of the melt wicked from a comparatively large reservoir of the melt. This ratio is estimated to be about 50:1. The filament temperature is controlled to provide a constant rate of deposition as detected by a quartz microbalance. In a typical alloy deposition, therefore, a melt of given alloy composition is drawn by surface tension from a reservoir into the evaporating zone, where in the initial stages of evaporation, the vapor is richer in the more volatile component.

The mole fraction of the less volatile component increases in the evaporating zone, becoming a larger fraction of the vapor composition until its vapor fraction just equals the melt feed composition from the reservoir. This equilibrium is, for all practical purposes, established after



a number of replenishments in the evaporating zone, depending on the ratio of the vapor pressures at the evaporating temperature. This could be a large number when the ratio is large, except that the control system demands that the evaporation rate be kept constant at about 1500 Å per second. With this added power input, the source tends to adapt to the least volatile component, and the composition of the film is felt to approximate within a few seconds of time. This is considerably different from wire-fed E.B. sources, requiring about one-half hour for this compositional equilibrium due to the large volume of the melt. No analytical evidence is available supporting this Sigmatron source contention at this point in time.

Foils were all prepared by a process developed at Sigmatron specifically for aluminum foil filters for the ATM instruments. This process will ultimately be described in detail elsewhere, but it briefly entails condensing the metal vapor at a deposition rate of 1500 Å/sec over a film of fluorescein on a 2x11-inch glass substrate, and subsequently cementing a fine screen to the metal film while on the substrate. The screen-film structure is released from the substrate in an acetone bath. Selected areas of foil are used to make filters.

#### IV. RESULTS

The program at Sigmatron for development has come to an end prior to comprehensive evaluation at GSFC for mechanical (vibration) durability and XUV transmission of the materials. There has been, however, considerable data generated on the thermal and moisture resistance (aging) effect of the metals and alloys resulting from this program. Below is a summary of that data. Unless indicated otherwise, alloys will be considered on a weight-percent (w/o) basis. The other compositional basis used is atomic percent (a/o), which is preferred for a binary alloy series involving the same two metals.

##### A. Aluminum

The standard 'aluminum' foil has derived from past developments, in which superior aging and microcrystalline structural characteristics have been found, exist in the alloy of 1-percent silicon in otherwise high-purity aluminum. The base line for comparative analysis of other metals and alloys, therefore, will be 99.0 Al:Si alloy. Thickness of this and other foils discussed are summarized in Table 1.



TABLE 1 .

Foil Type	Thickness Angstroms	Foil Type	Thickness Angstroms
99 Al:Si	1240	97 Sn:Au	1590
100 Sn	1600	97 Sn:Ge	1700
97.5 Sn:Al	1300	100 In	1700
95 Sn:Al	2260	97 In:Al	2200
87.5 Sn:Al	1620	97 In:Au	1170
75 Sn:Al	2220	97 In:Ge	1300
50 Sn:Al	1520	99 In:Ti	1730
25 Sn:Al	1450	96 Al:Ti	1800
12.5 Sn:Al	950	Al/100A Ti**	1590/100
2.5 Sn:Al	1550		



Aging of aluminum, which is shown in Figure 1, is negligible at R.H.'s of 25 percent or less. Within 35 days, the net change in pinhole transmittance was a  $4 \times 10^{-9}$  increase for a filter stored at less than 1-percent R.H., and after 106 days was a  $7 \times 10^{-9}$  increase. The filter stored at .25-percent R.H. underwent a zero change after 35 days' exposure and increased  $1 \times 10^{-9}$  after 106 days. Such changes are photometrically accurate but insignificant.

At an R.H. of 75 percent, 99.0 Al:Si shows a typical aging curve for foils which depend on an oxide layer for protection from corrosion. This consists of an induction period during which there is little change, followed by nearly stepwise increases in pinhole transmittance. It is felt that a weakness is developed during the induction period which then gives way. Attack is at vulnerable points such as existing pinholes, and thus frequently the transmittance often decreased in the early stages of exposure due to fill-in of pinholes by the corrosive product. Following the one-decade increase at five days, a second one-decade increase occurred at 47 days. These, coupled with the drift, resulted in an increased transmittance of about  $3 \times 10^{-5}$ .

Thermal recrystallization test of this aluminum alloy shows less than  $10^{-9}$  change in transmission up to the limit of the oven at  $300^{\circ}\text{C}$ . The temperature excursion was of two hours' duration. On cooling, however, considerable pinhole leakage developed due to differential thermal expansion, wherein the foil-screen material was cooler than the frame, and thereby stretched. It is estimated that the temperature of an aluminum or magnesium filter frame should not be more than  $35^{\circ}\text{C}$ . greater than the foil when the foil is near room temperature or more than  $15^{\circ}\text{C}$ . when the foil is near  $100^{\circ}\text{C}$ . This data is shown in Curve I of Figure 2.

Conclusion here is that 25 percent or lower R.H. is quite acceptable for 99.0 Al:Si filters for a minimum of three months. Exposure to 75-percent R.H. for even a few days is to be avoided.

## B. Tin Type Filter

### 1. Pure Tin

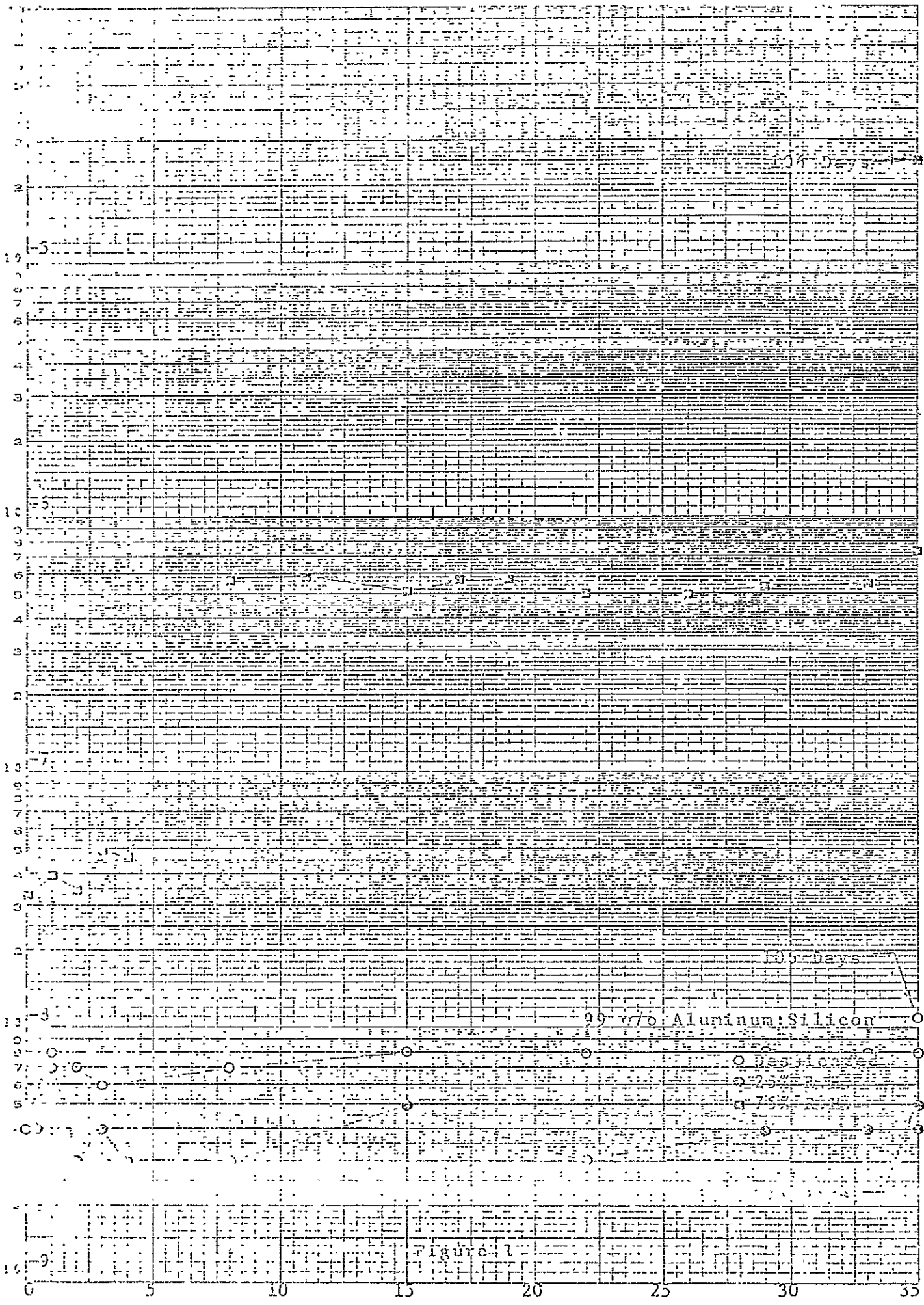
High-purity tin (4N Min.) foil shows aging



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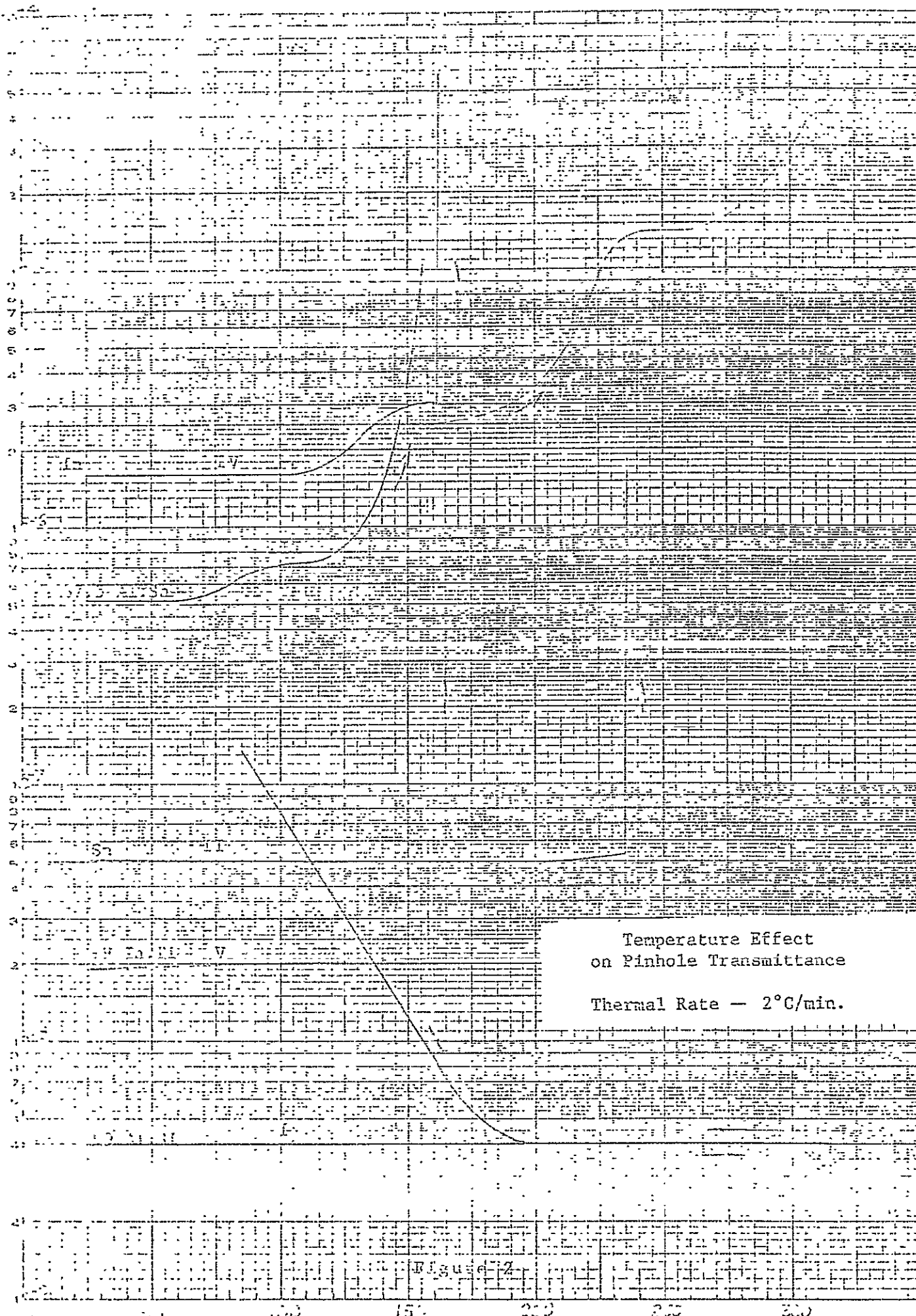
TRANSMITTANCE





5 CYCLES PER MIN. PER H.B.

Transmittance



Temperature Effect  
on Pinhole Transmittance  
Thermal Rate — 2°C/min.



Characteristics, illustrated in Figure 3, indicating extreme sensitivity to moisture. At R.H. less than 1 percent (desiccated), the data shows an increase in transmittance of  $3 \times 10^{-9}$  after 35 days, with no further change out to 106 days. At 25-percent R.H. one filter increased transmittance by  $10^{-4}$  in 22 days but it is felt that this was not representative of pure tin. A second filter at that same R.H. is also graphed showing an increase of  $5 \times 10^{-7}$  at 35 days and  $6 \times 10^{-7}$  at 106 days. At the higher humidity, pure tin increased by  $10^{-4}$  in five days. The companion filter, not graphed, increased by that amount in six days, demonstrating that the rapid aging of pure tin foil is real.

The effect of the temperature on pinhole transmittance of pure tin is shown in Curve II of Figure 2. Up to  $200^{\circ}\text{C}$ ., no pinhole degradation of pure tin is observed. At higher temperatures, some increase is observed and it must be assumed that long exposure in this range up to the melting point would have a degenerative effect. Although the melting point is affected by crystal size, the equipment employed here is not accurate enough to detect any lowering due to the  $0.1\text{-}\mu$  crystal size. The melting point of tin is  $232^{\circ}\text{C}$ . Catastrophic pinhole transmission occurred at  $235^{\circ}\text{C}$ , but interestingly, to the unaided eye, there was no apparent damage. Under the microscope it was clear that melting had taken place, and surface tension had drawn the metal into a fernlike structure within the intact oxide layer. Pinhole transmittance was estimated to be about  $10^{-2}$ .

The XUV transmittance of pure tin is shown in Figure 4. The data indicates that the transmission is comparable to results obtained on Sigmatron filters measured elsewhere.

## 2. Tin:Aluminum Alloys

The binary phase diagram for the aluminum-tin system is given in Appendix II. It shows no inter-metallic compounds and an infinite mutual solubility. In spite of this, the 50 atomic percent (a/o) alloy transmitted an estimated  $10^{-3}$  in the red. This characteristic is usually associated with



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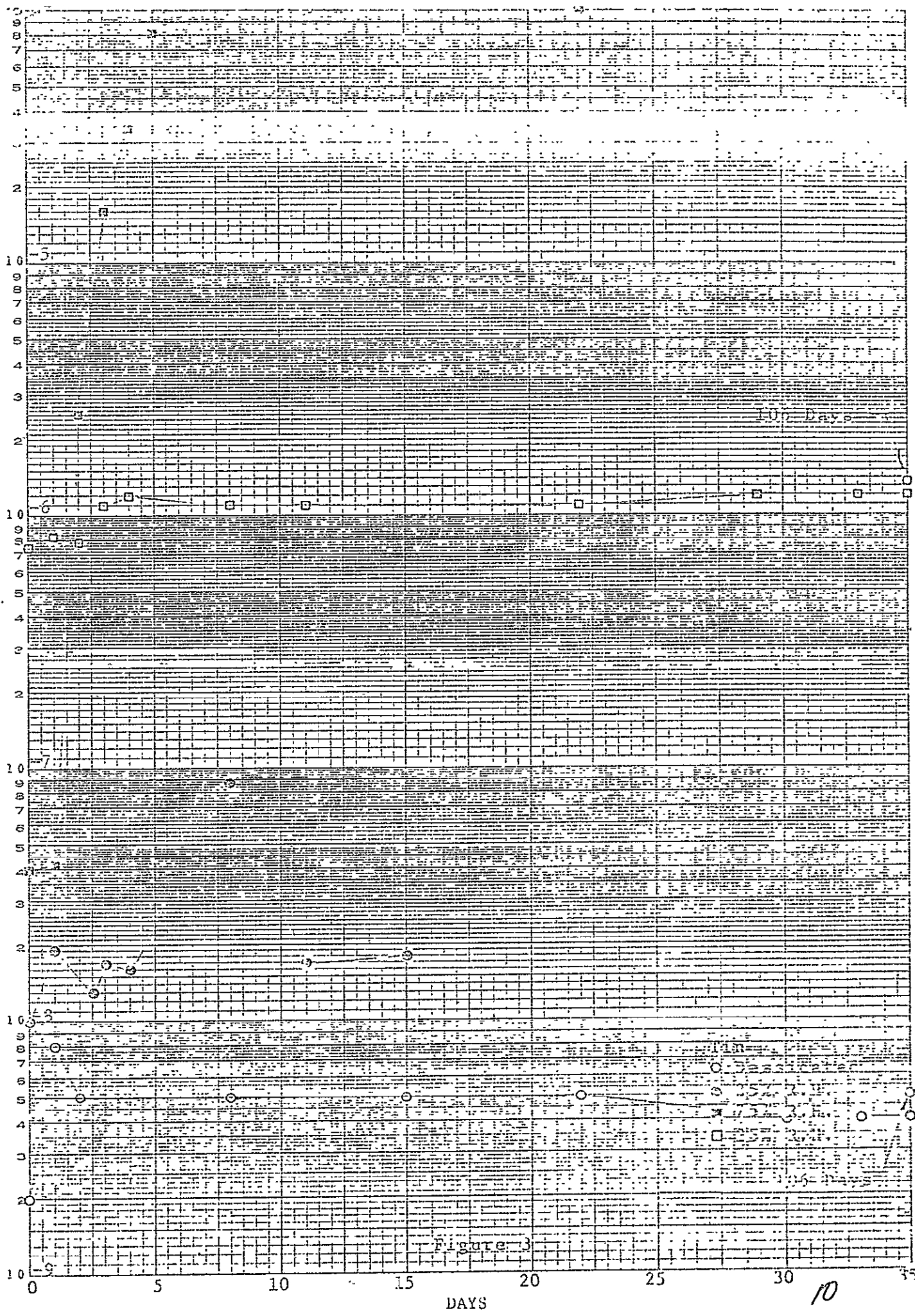
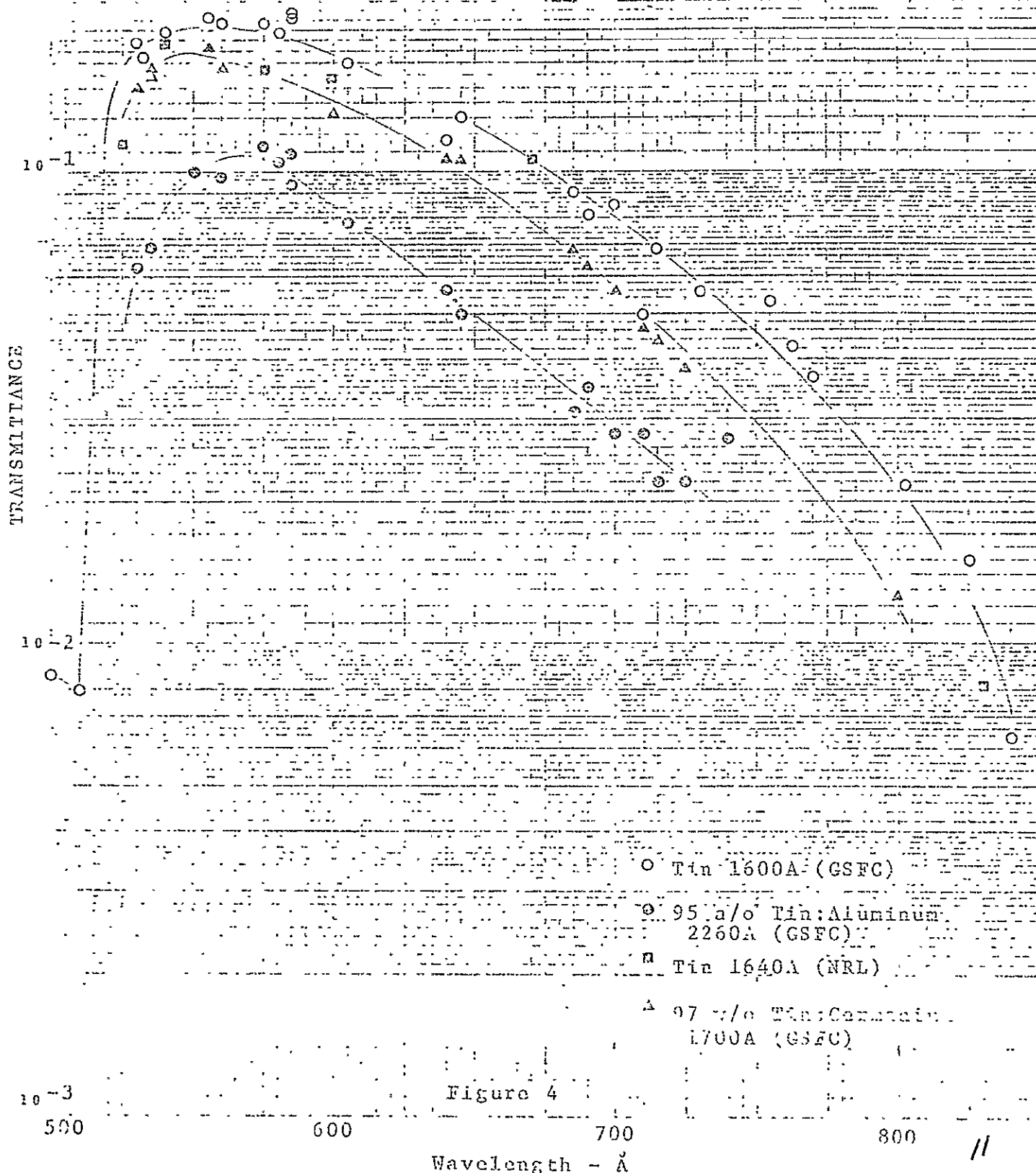


Figure 3







inter-metallic compounds. The alloys with compositions 25 a/o, and 75 a/o did not possess this feature.

The aging characteristics of the alloys, in general, showed increasing sensitivity to moisture with increasing aluminum content. Alloys were made with the compositions 97.5 a/o Sn:Al, 95 a/o Sn:Al, 87.5 a/o Sn:Al, 75 a/o Sn:Al, 50 a/o Sn:Al, 25 a/o Sn:Al, 12.5 a/o Sn:Al and 2.5 a/o Sn:Al. Of these alloys, the last three were so reactive toward moisture that they could not be made in a quality satisfactory for aging tests. The 50 a/o Sn:Al alloy transmitted too strongly in the red to qualify, and thus no data is offered on these four. The aging curves for the remainder are shown in Figures 5, 6, 7, 8 and 9, and are discussed here in the aggregate.

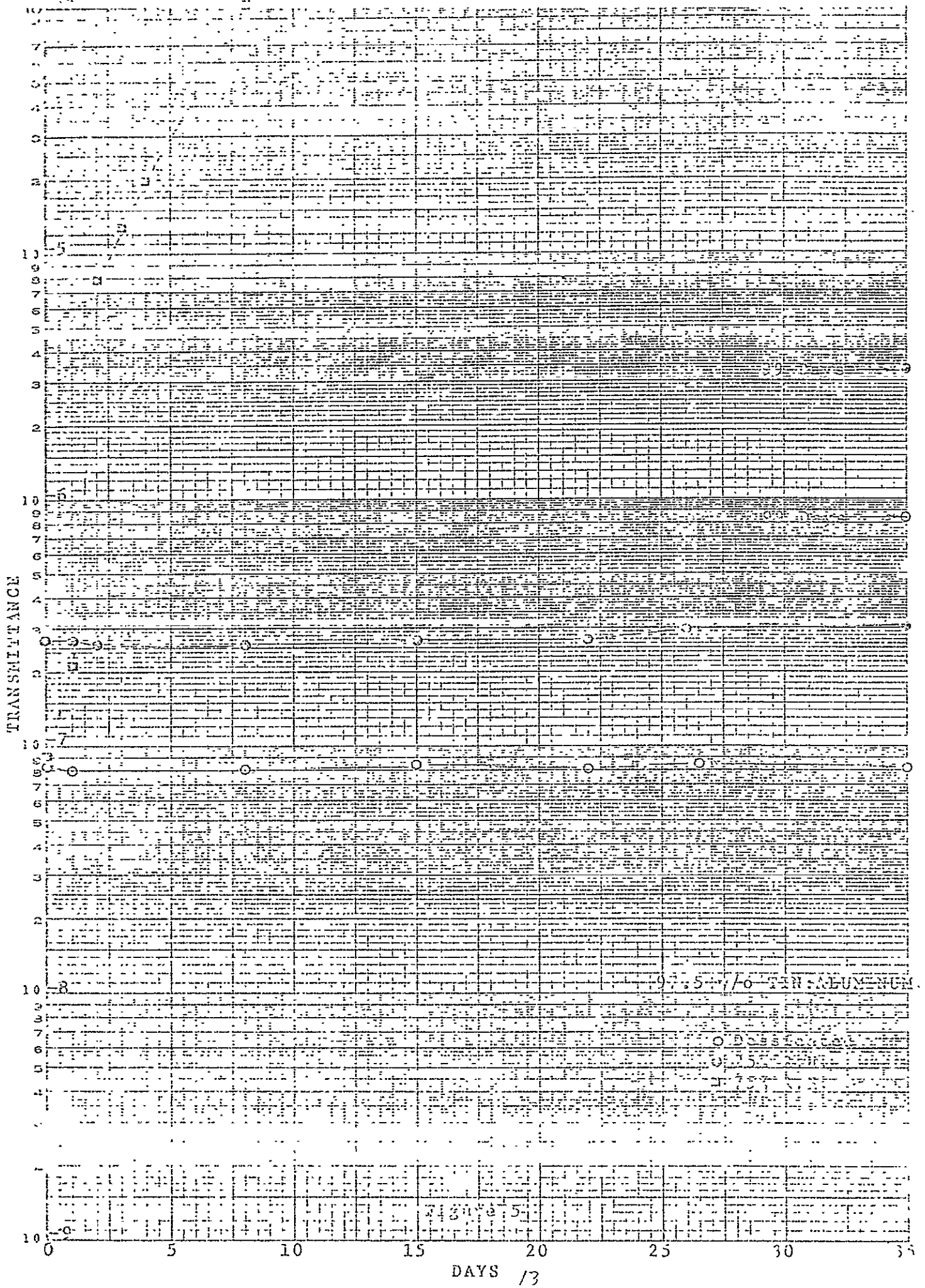
It is a point of convincing evidence that a R.H. of 25 percent or lower was not degrading to any of these chemically active alloys when tested out to an exposure of 100 days. The greatest net change observed in 35 days was less than  $3 \times 10^{-7}$ , which occurred in the relatively poor filter specimen available from the 75 a/o Sn:Al foil. Exposed to the 75-percent R.H., filters reached the terminal transmittance of  $10^{-4}$  in one day to three weeks.

Thermal recrystallization tests on these alloys are characterized by Curve III of Figure 2. Pinhole transmission increases slightly at temperatures above 60°C., followed by catastrophic destruction considerably below the incipient melting point of 228.3°C. Within the scope of this program, it was not possible to accurately determine the cause of this effect, but it is believed to be due to increased chemical reactivity toward the atmospheric moisture present during these tests. This is qualitatively substantiated by the observance that the alloys lower in aluminum content tended to degenerate more like pure tin.

The XUV transmission of this series at the two important wavelengths 740 Å and 504 Å are provided by measurements from GSFC and show rather unpredictable and remarkable results. A graph of the data uncorrected for thickness and uncorrected for .80 transmit-



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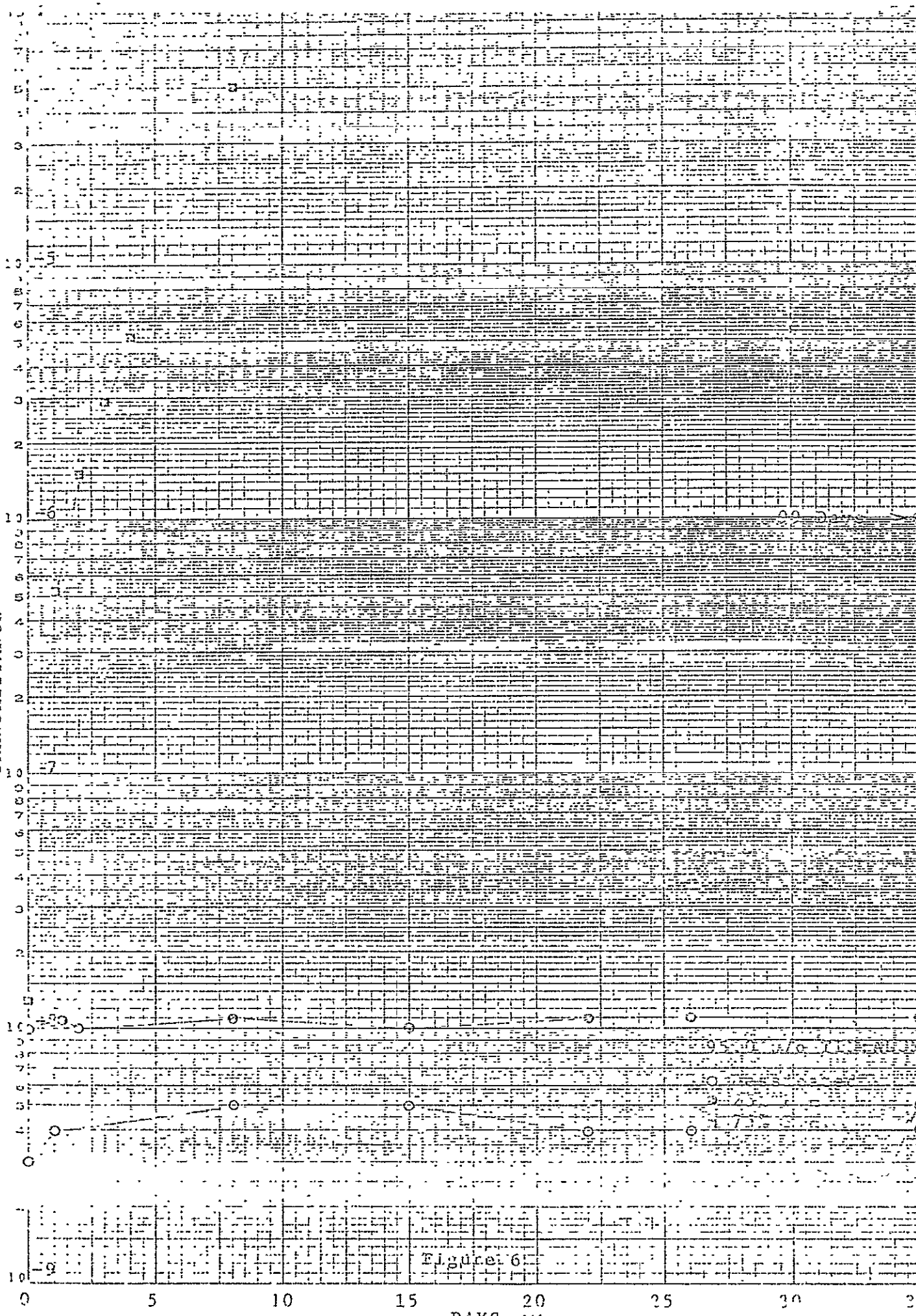


Figure 6







TRANSMITTANCE

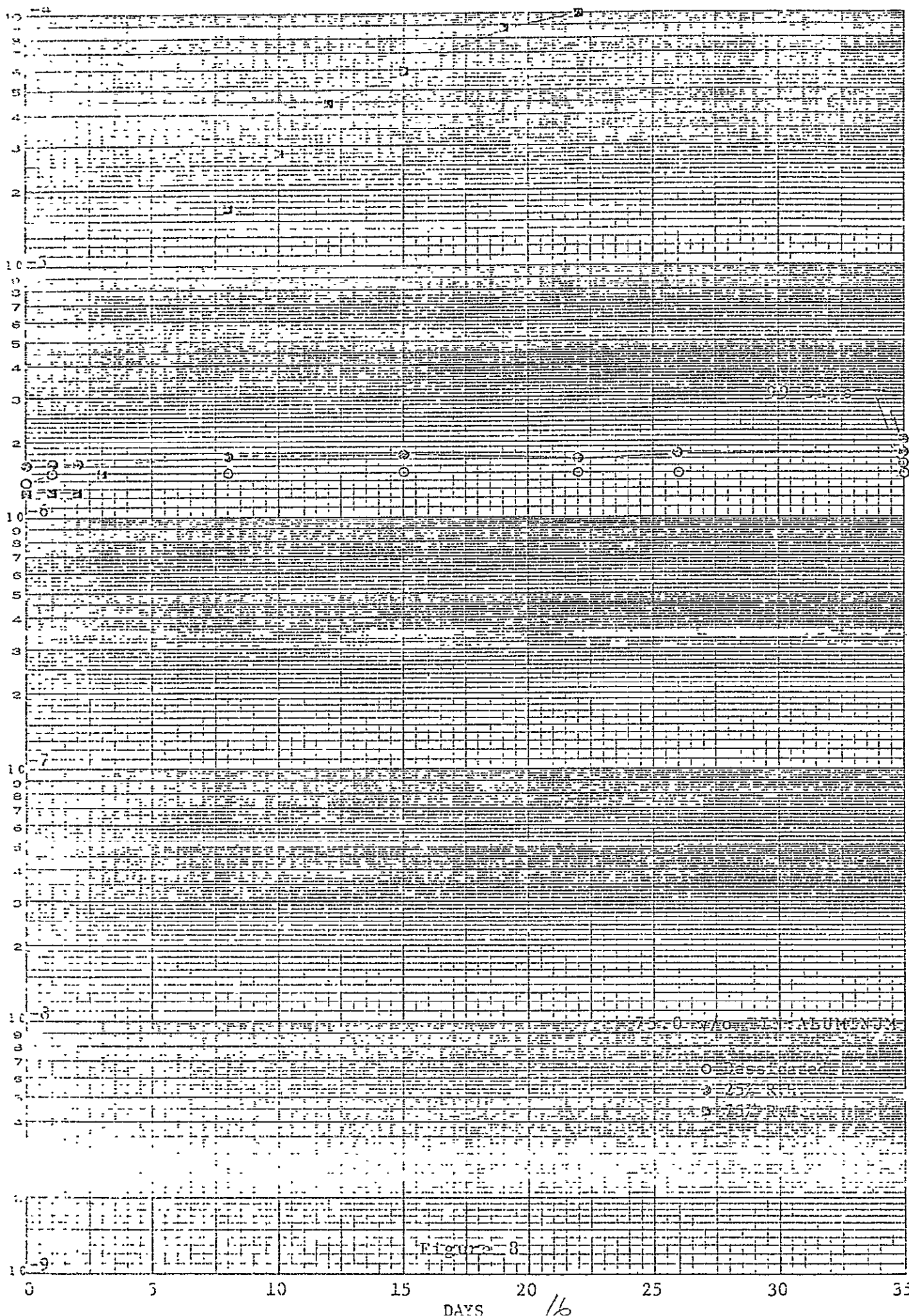


Figure 3



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TRANSMITTANCE

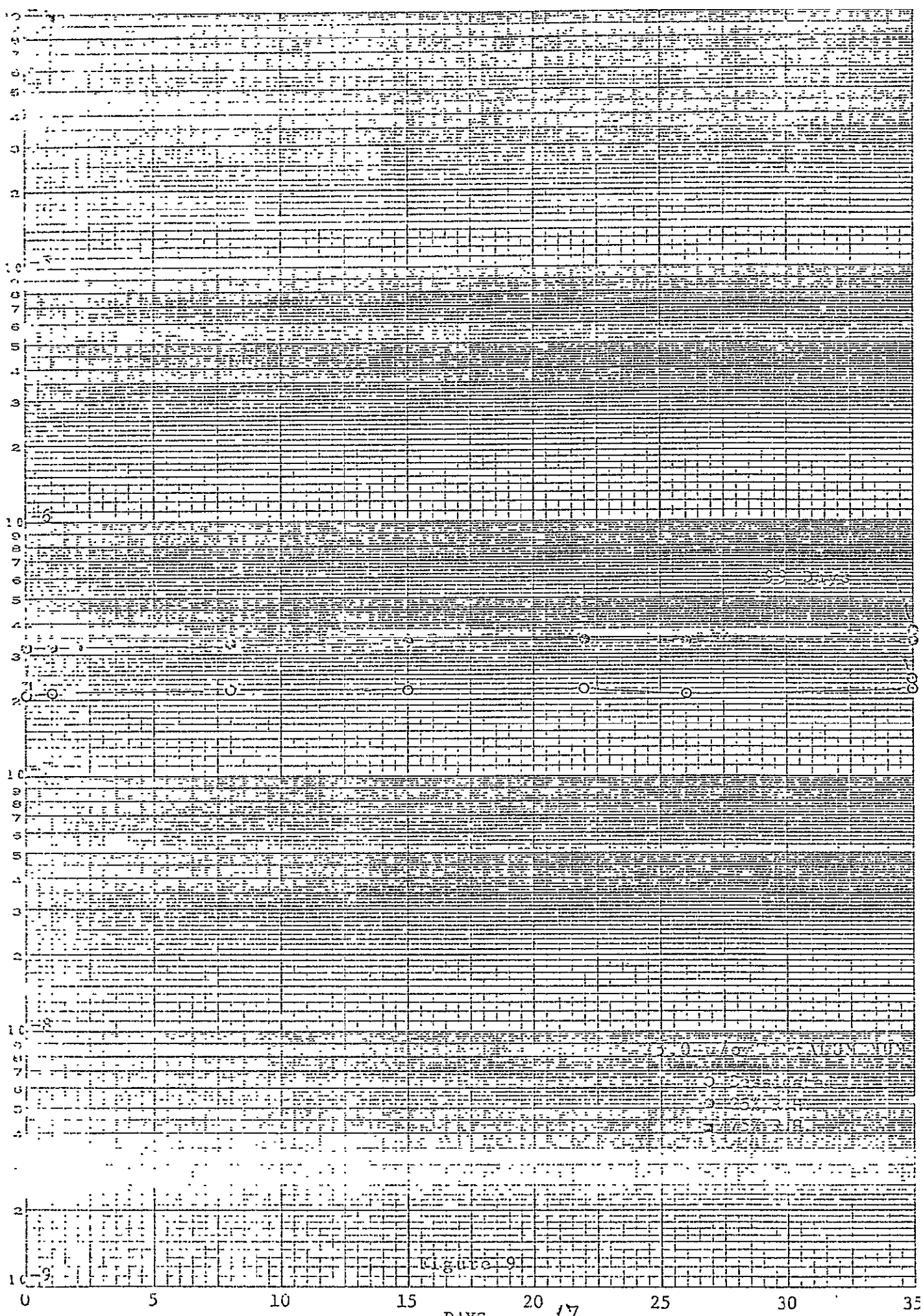


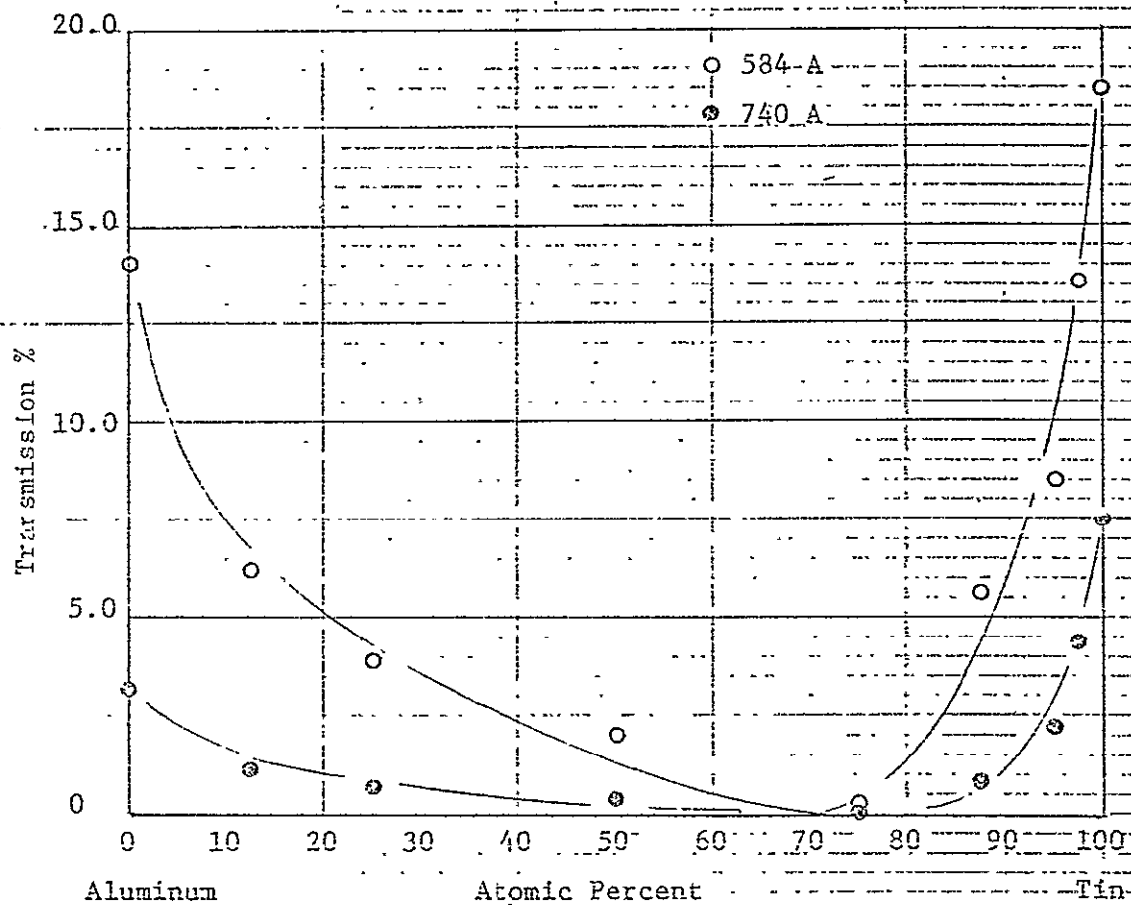
Figure 9



tance screen is shown in Figure 10. It would seem that when both tin and aluminum have appreciable transmission at these wavelengths as pure metal, that barring any inter-metallic compounds, their alloys should have corresponding transmissions without maxima and minima in the series. The data is not considered definitive, since it is believed that the transmission is effected by an oxide layer to the point that thickness corrections have meaningless effect. Individual foils are uniform but the series range in thickness from 1500 Å to 2260 Å. Actual thicknesses are provided in Table 1. More XUV transmission data, revealing the effect of alloying on the shape of the curve, is shown in Figure 4. The upper transmission curve is representative of pure tin and the lower is representative of 95 a/o Sn:Al. Treating the foil as a true alloy, one can estimate, based on the electron plasma density, that the onset of transmission should shift from the pure tin value of 886 Å to 880 Å for 95 Sn:Al alloy. It seems apparent, according to the data, the shift is at least 50 Å. Treating the alloy foil as a layer tandem type foil which calculations show to be approximately equivalent to an 80 Å aluminum layer over a 2180 Å tin layer, the peak transmittance of the alloy at the 584 Å radiation wavelength should be 0.23. Instead, the transmittance is measured to be considerably below this at 0.11.

It can be argued that the discrepancy between the anticipated XUV transmission of an alloy filter and the real transmission in this case is due to the strong tendency to oxidize. Although this is certainly a factor, the evidence of a minimum transmission in Figure 10 indicates some unrecognized relationship of metals in alloys or need for shoring-up the plasma density theory. In any case, the conclusion drawn in this laboratory, useful in the remainder of this work, is that constraints on alloying for improving structural quality of foils can be based on chemical and structural bases without concern for effect on XUV transmission. Such conclusion is acceptable as long as the alloy is composed principally of one metal with a few percent of the minor constituent.





XUV TRANSMISSION OF TIN-ALUMINUM ALLOY

Figure 10



### 3. Tin:Gold

On the basis that a principally tin alloy would be chemically inactive alloyed with gold, the 97 Sn:Au alloy was prepared in foil filters and tested. The phase diagram for this binary system is given in Appendix II.

At low humidities, 25-percent R.H. or less, no degradation of the test filters was detected. At the 75-percent R.H., surprisingly, the sensitivity to moisture aging was greater than pure tin or some of the tin-aluminum alloys. Aging data for this alloy is shown in Figure II. Thermal recrystallization of this alloy resulted in pinhole transmission degradation similar to Curve II of Figure 2. except that the onset of leakage was about 100°C. and the catastrophic temperature was 215°C. instead of 232°C. of pure tin. This is consistent with the 217°C. indicated in the phase diagram. No XUV transmission is available.

Conclusion from these tests is that there is no benefit to alloying small amounts of gold with tin to improve either the aging of tin type foils or suppress thermal recrystallization.

### 4. Tin:Germanium

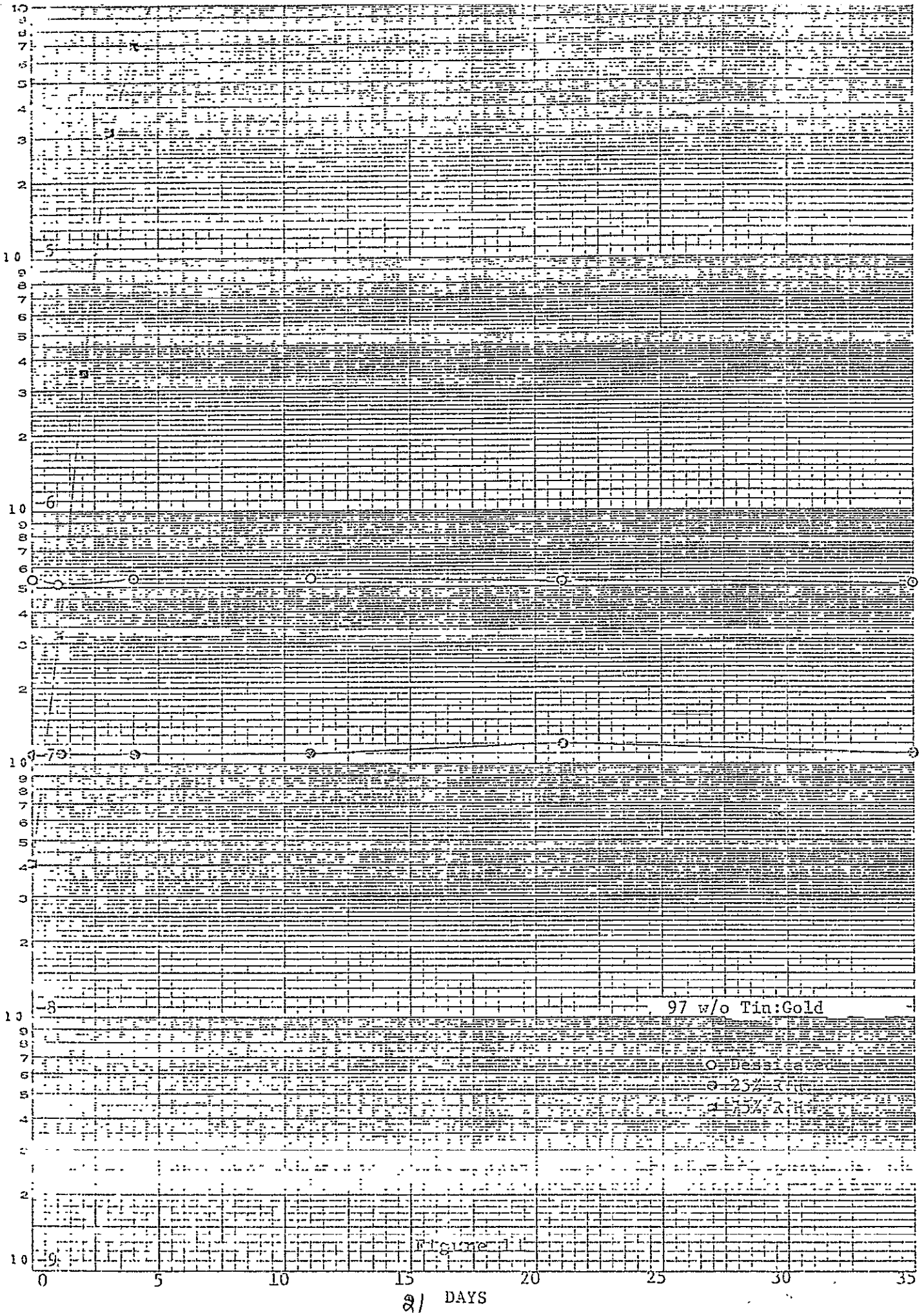
The alloy 97 Sn:Ge is believed to represent a new standard for tin filters with respect to moisture aging. Data are presented on this alloy in Figure 12. At the low humidities, 25-percent R.H. or less, basically no net degradation due to increased pinhole transmittance over a period of 35 days. At high humidity, 75-percent R.H., the pinhole leakage over the same period increased by  $2 \times 10^{-6}$ . The companion filter increased slightly more. At 54 days this alloy compares favorably with the aluminum. These would probably have been lower if a better quality of foil had resulted from this initial effort. It is appropriate to point out that pure tin increased in transmittance by  $10^{-4}$  in five days at the high humidity.



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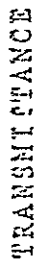
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97 w/o Tin:Germanium

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— 234 —

Figure 12

22 DAYS



The phase diagram for this binary is provided in Appendix II and shows an infinite mutual solubility with no intermetallics. There is no freezing point depression, and incipient melting temperature for alloys is the same as the melting point of pure tin. This is confirmed for this alloy, with the foil catastrophic temperature being measured at 233°C. Interestingly, zero change in pinhole transmittance is detected until melting. This indicates a structural stabilization of the tin by the germanium.

The XUV transmission, corrected for screen opacity and shown in Figure 4, indicates a slightly lower transmission when compared to the pure metal. The conclusion reached from these tests is that 97 Sn:Ge alloy in foils presents superior structural features and resistance to moisture aging over pure tin, with very little loss in XUV transmission.

### C. Indium Type Filter

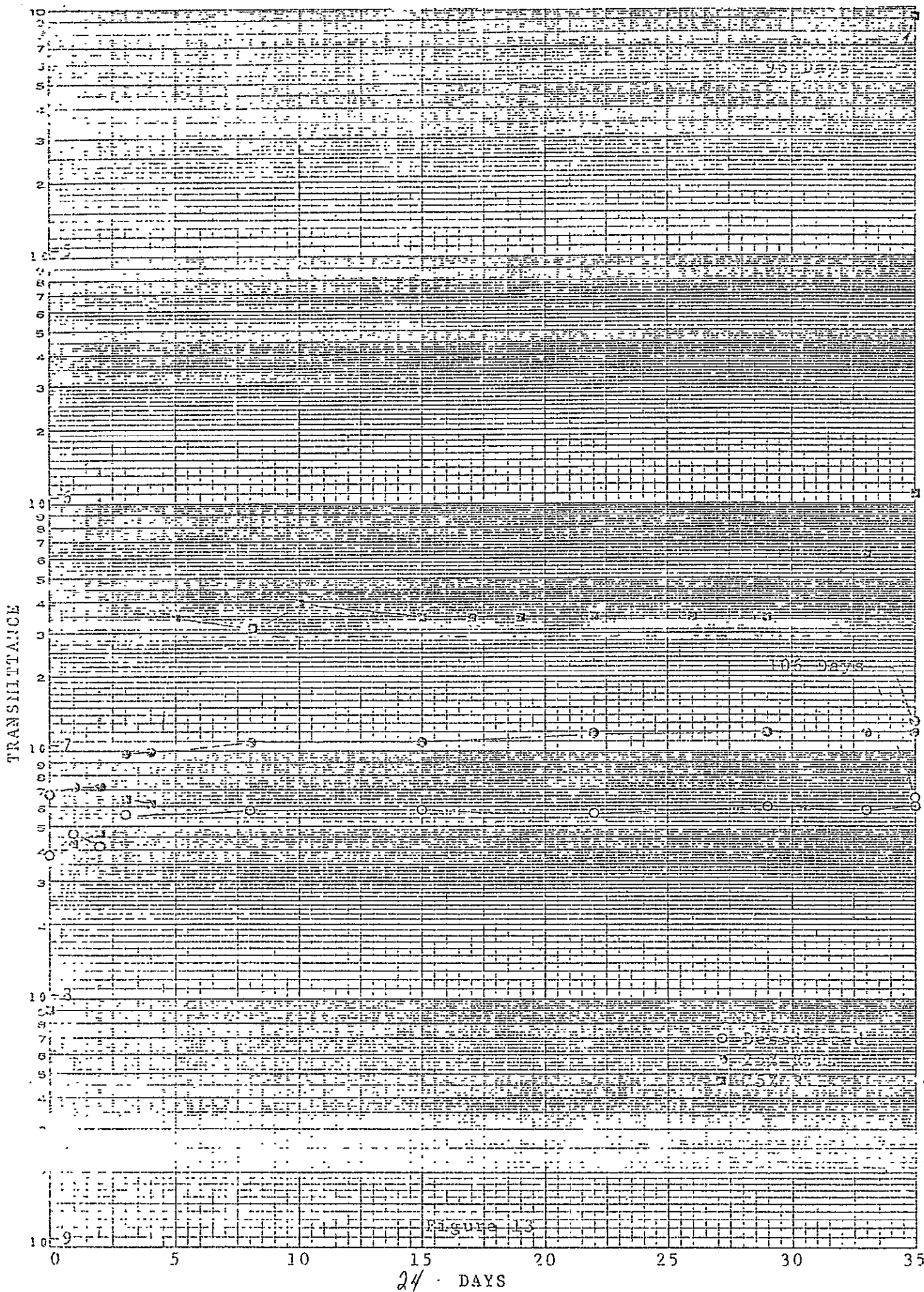
#### 1. Pure Indium

High-purity indium (5N) in foil form shows a resistance to moisture aging very similar to that described above for the 99 Al:Si alloy, except that it is slightly more reactive. Curves for these tests are shown in Figure 13. At 25-percent R.H. and lower, pinhole transmittances increased by  $5 \times 10^{-8}$  or less in the test filters over 35 days' exposure with no change thereafter out to 106 days. At high humidity, one pure indium test filter increased  $10^{-4}$  in 100 days, and the companion test filter reached the same in 43 days.

Pinhole transmittance as a consequence of thermal recrystallization of pure indium is shown in Curve IV of Figure 2. This indicates a tendency of ultrathin foils of indium to recrystallize at temperatures above 100°C. The temperature of complete degeneration agrees within the accuracy of the measurements with the melting point of that metal.

Conclusions drawn from these tests are that pure indium foil shows good aging characteristics with respect to moisture but has a poor resistance to thermal recrystallization. The temperature limit is about 100°C.







## 2. Indium:Gold

The phase diagram for the indium-gold system is included for reference in Appendix II. The aging characteristics of the alloy 97 Sn:Au is shown in Figure 14. No significant moisture degradation at low humidity is observed out to 57 days; any change being less than  $3 \times 10^{-8}$ . At high humidity, a transmittance of  $10^{-4}$  was reached in 15 days, and the companion filter was similarly degraded in five days.

Thermal recrystallization degradation characteristic is similar to pure indium shown as Curve IV of Figure 2. Catastrophic loss occurred near the melting point of indium.

It may be concluded that this alloy is inferior to pure indium with respect to moisture aging and no improvement with regards to thermal degradation.

## 3. Indium:Aluminum

The phase diagram for the indium-aluminum system is shown in Appendix II. Aging characteristics are shown in Figure 15. Foil made with the 97 In:Al alloy were of remarkably high pinhole quality for indium. This material in ultrathin foils, however, appears to be very moisture-sensitive, similar to the comparable tin-aluminum alloy. There is no apparent problem at low humidities, but at 75-percent R.H. an increase of  $10^{-4}$  in the pinhole transmittance occurs in one day. The companion test filter gave the same result.

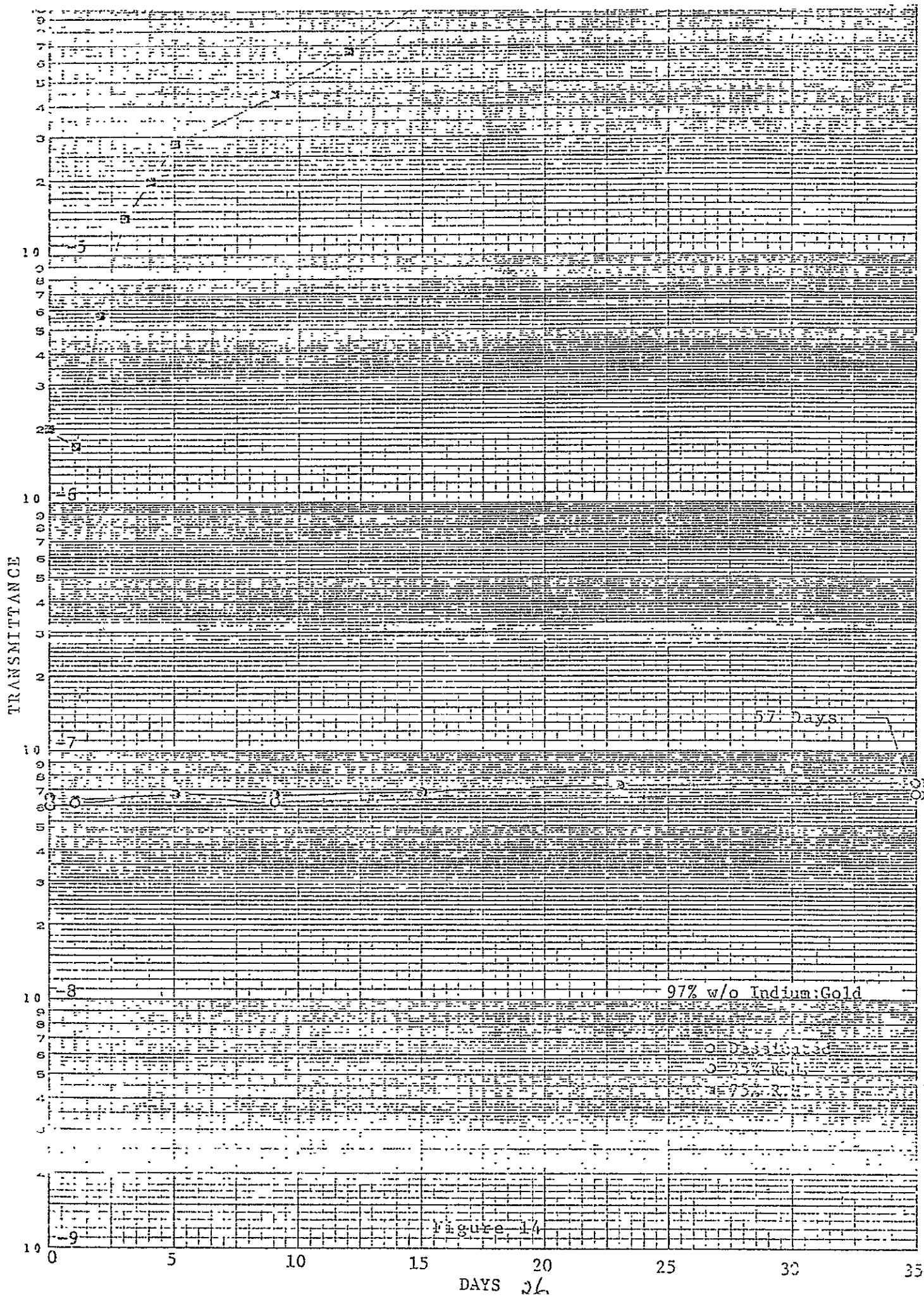
Thermal recrystallization of this alloy is approximated in character by Curve IV of Figure 2, with catastrophic increase in pinhole transmission occurring at a temperature approximating the melting point of pure indium.

## 4. Indium:Germanium

The phase diagram for the indium-germanium system, shown in Appendix II, is very similar to the tin-germanium system. As the aging curve for 97 In:Ge presented in Figure 16 shows, germanium does not have the same effect on indium that it has on tin.



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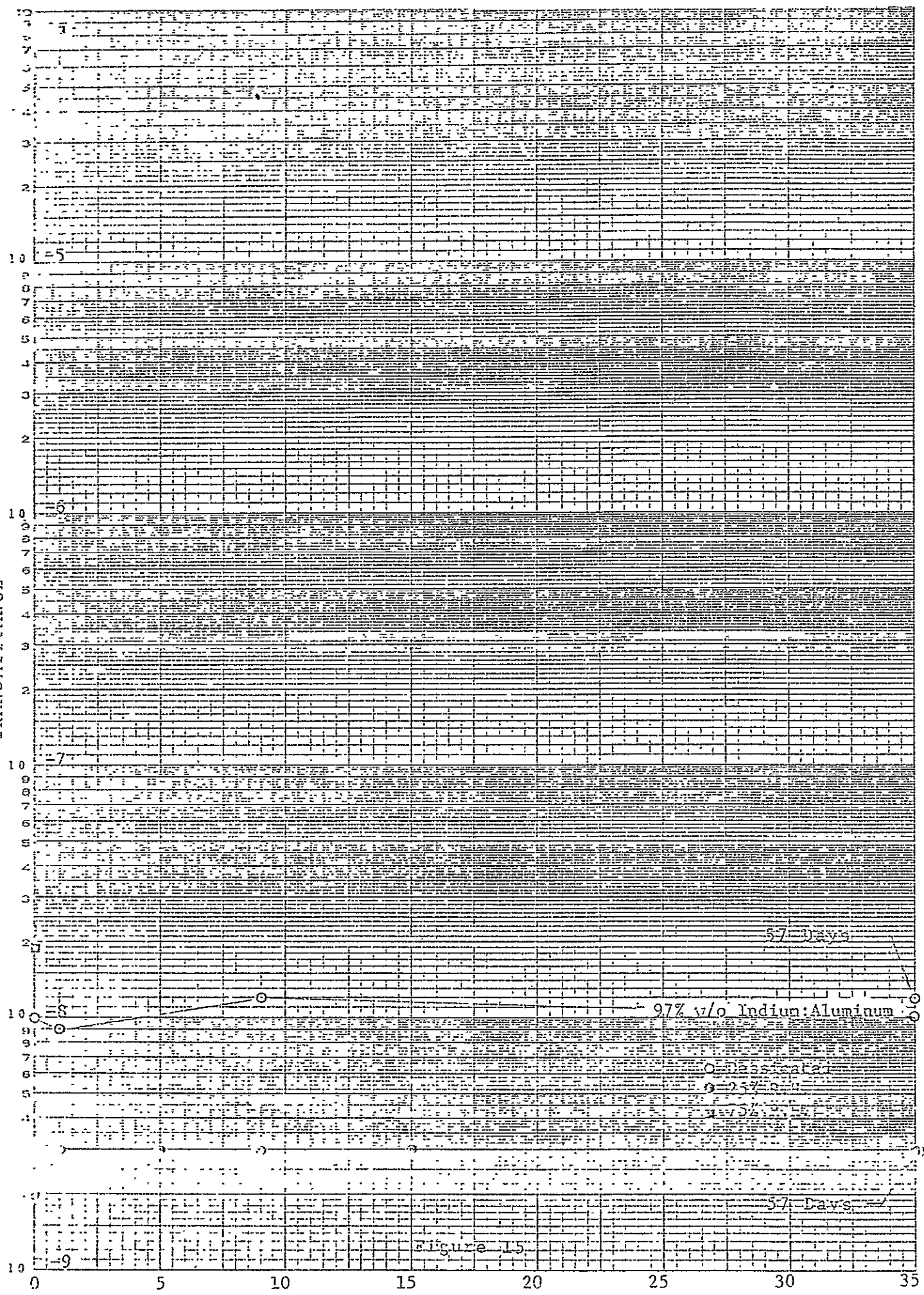


Figure 15



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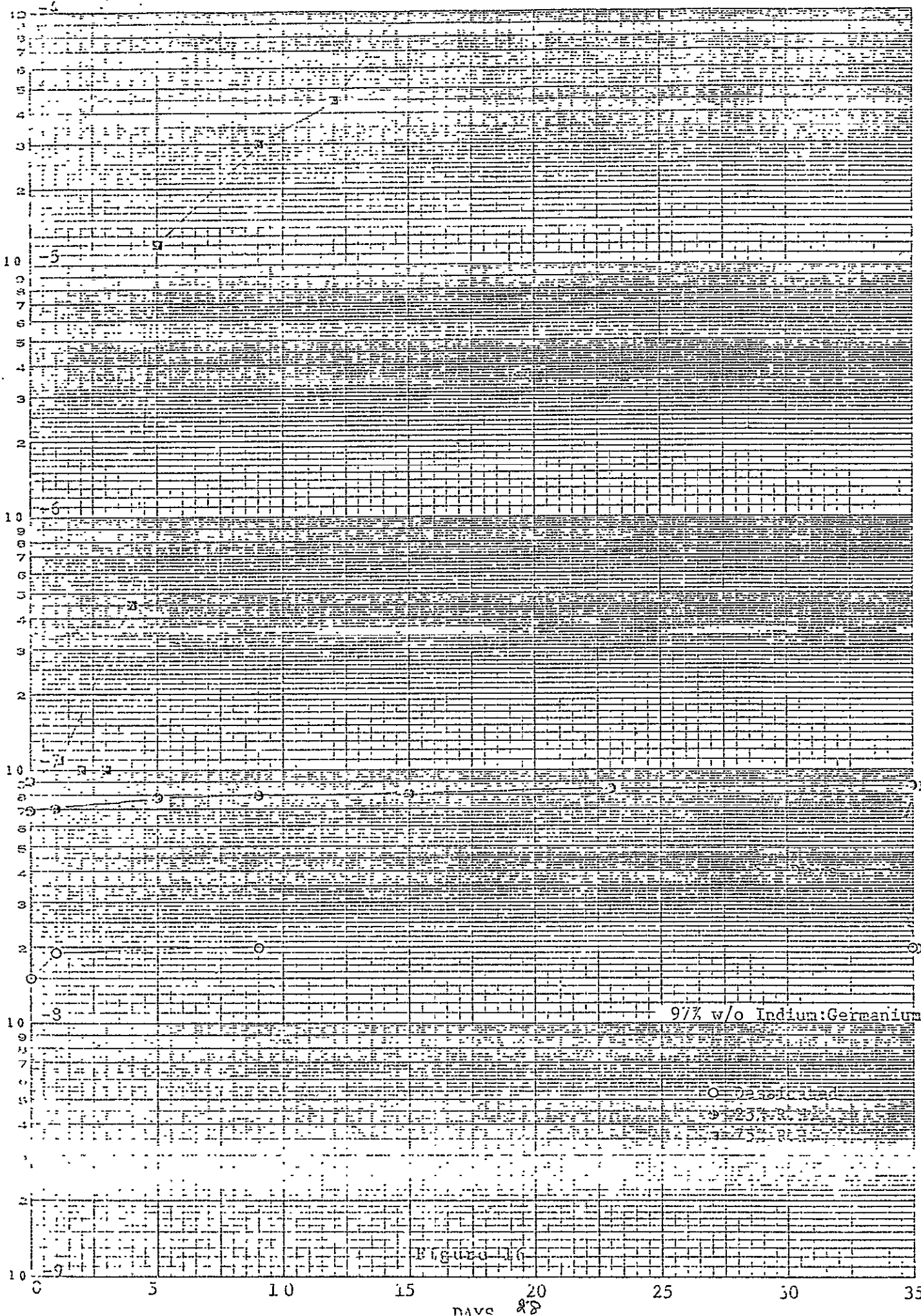


Figure 16

WAVELENGTH



At low humidity, again, there is no significant aging effect. At the higher humidity of 75% R.H., a transmittance of  $10^{-4}$  is reached after 15 days' exposure in the filter graphed, and 24 days for the companion filter, whereas it was 100 days for pure indium.

Degradation by thermal recrystallization was the same as that observed for indium and its alloys above; and long exposure to temperatures 100°C. and above can be expected to result in increased pin-hole leakage.

It may be concluded that no significant improvement in temperature or moisture resistance is offered by the 97 In:Ge alloy, as compared to pure indium.

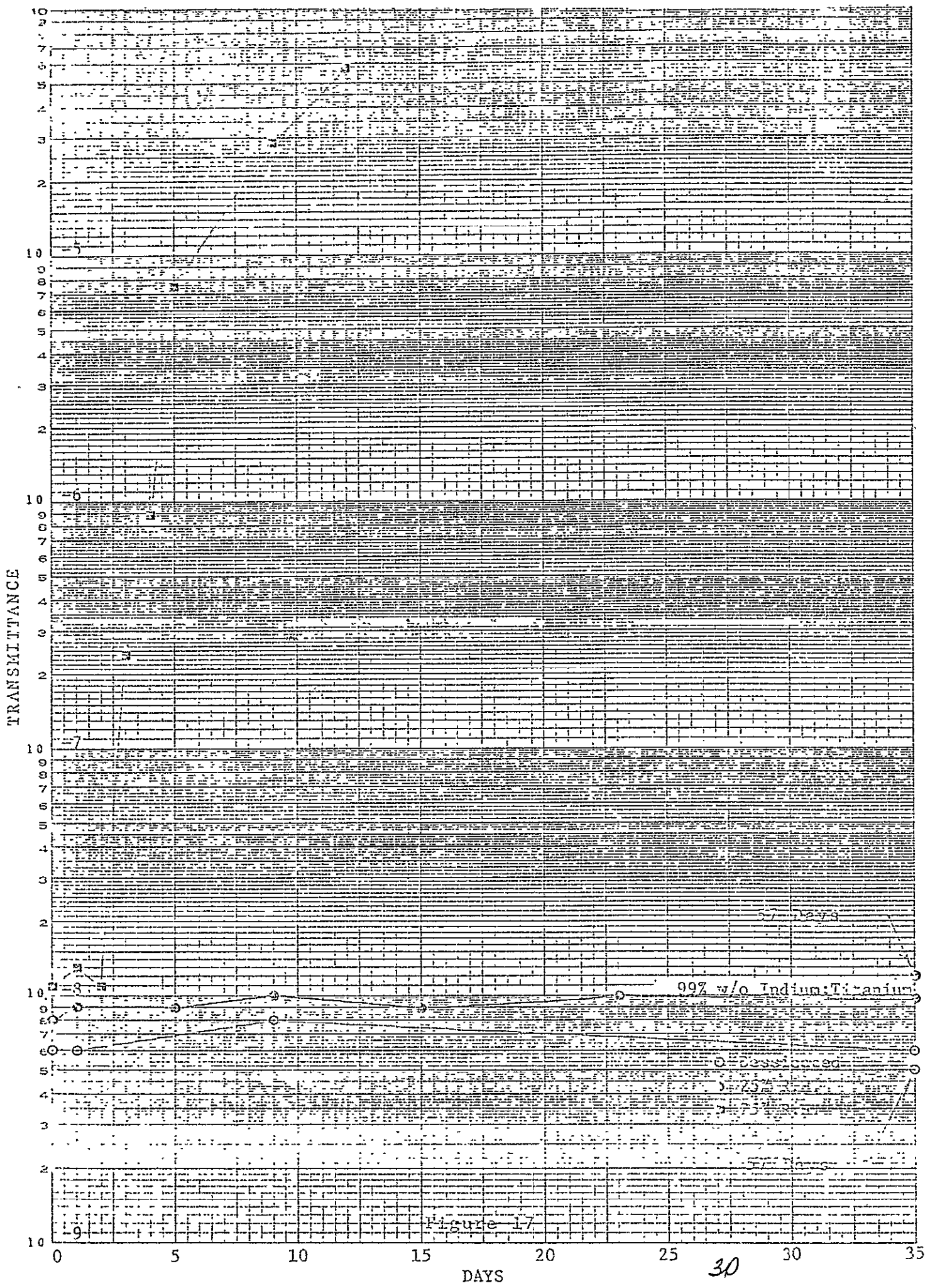
#### 5. Indium:Titanium

The phase diagram for the indium-titanium system is shown in Appendix IV. An alloy a little less rich in the minor component than used in other alloys was chosen to minimize an anticipated high chemical reactivity. The 99 In:Ti alloy was selected for that reason, and to keep the melting point low for formation of the bulk alloy.

Although this alloy turned out to be remarkable thermally, the aging characteristics are typical of the other indium alloys, as shown in Figure 17. At low humidity, no significant moisture aging was detected. The net change after 35 days at 25% R.H. was less than  $2 \times 10^{-9}$ . At 75% R.H., the pinhole transmittance increased  $10^{-4}$  in 15 days; the companion test filter increased that amount in 23 days.

The remarkable feature of the foil of this alloy lies in the recrystallization characteristic. Curve V of Figure 2 illustrates this feature, which shows a strong resistance to pinhole leakage due to thermal recrystallization right up to the melting point. One can expect increased resistance to elevated temperature of this alloy over pure indium or any of its alloys tested. This curve also illustrates that something happens at the melting point to further stabilize the filter such







that catastrophic degradation was not reached even at 300°C., whereas incipient melting point is 157°C. One must conclude that this filter is thus a liquid metal retained by two oxide films.

The XUV transmissions for the 99 In:Ti alloy and a Sigmatron pure indium foil are shown in Figure 18. As anticipated, the alloy transmission is everywhere some fraction of the pure metal. At the 770Å wavelength transmission peak, the reduction is accountable on a thickness basis since the absorption coefficient is  $1.55 \times 10^{-5}$  /cm for both materials. The calculated contributing thickness of titanium in a 1730Å thick 99 In:Ti foil is about 30Å. Considering a worst case situation, a tandem or layered foil would have a series transmittance, assuming an absorption coefficient for titanium of  $5 \times 10^{-5}$  /cm, of about 0.061 compared to the 0.063 measured. Thus if the 1% titanium is truly there and if XUV measurements are significantly accurate, a small addition of alloying element is demonstrated less absorbing to the XUV in solution with the major component than could be expected when present as a separate phase.

One may conclude that the structural stabilization of indium with a small amount of titanium is beneficial for applications requiring long term reliability. This is accomplished without loss of XUV transmission but is unfortunately accompanied by an increased moisture sensitivity. It does, however, represent a reasonable price to pay since the problem is transferred from a space environment control to atmospheric control.

#### D. Titanium Type Filter

##### 1. Pure Titanium - Parylene 'N' Base

One concept for 'pure' titanium XUV filters was to receive a film of this material on a thin foil of some other material such as Parylene 'N' or aluminum. Parylene 'C' has been previously evaluated elsewhere, but that material contains chlorine thought to be undesirable. Parylene 'N' foil,



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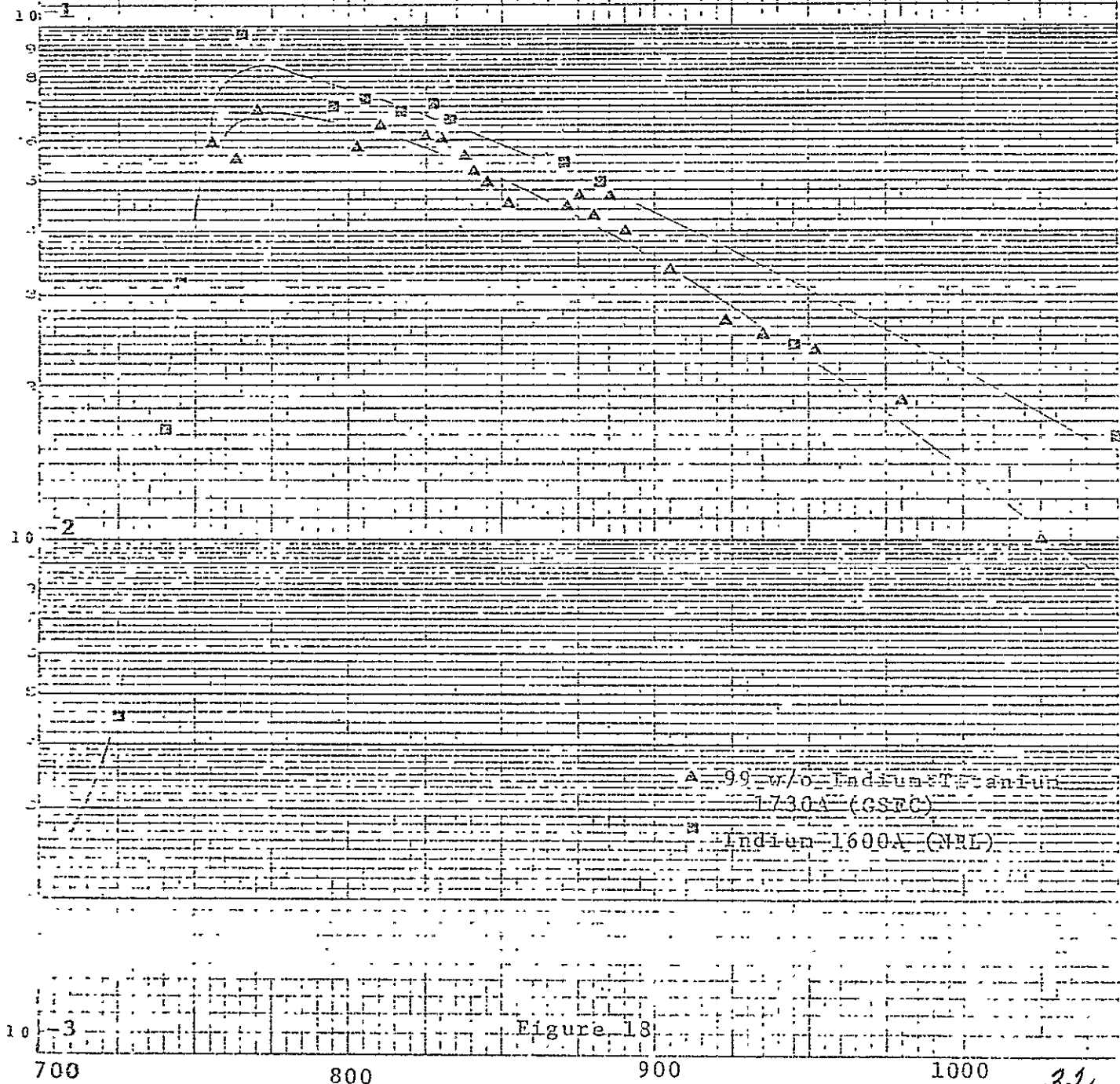


Figure 18



approximately 750Å thick, was obtained from Union Carbide Corporation, from whom it is available at their Bound Brook laboratory. Filters prepared on 90-percent transmitting screen were measured for XUV transmission at GSFC. The measurements corrected for screen transmission and reduced to absorption coefficients are provided in Table 2. This material was found to be unsuitable as a base for tandem filters for any of the metals of interest here on the basis that Parylene 'N' 750Å thick is strongly absorbing over the UV, except for an apparent peak of about 20 percent at 1600Å.

## 2. Pure Titanium - Aluminum Base

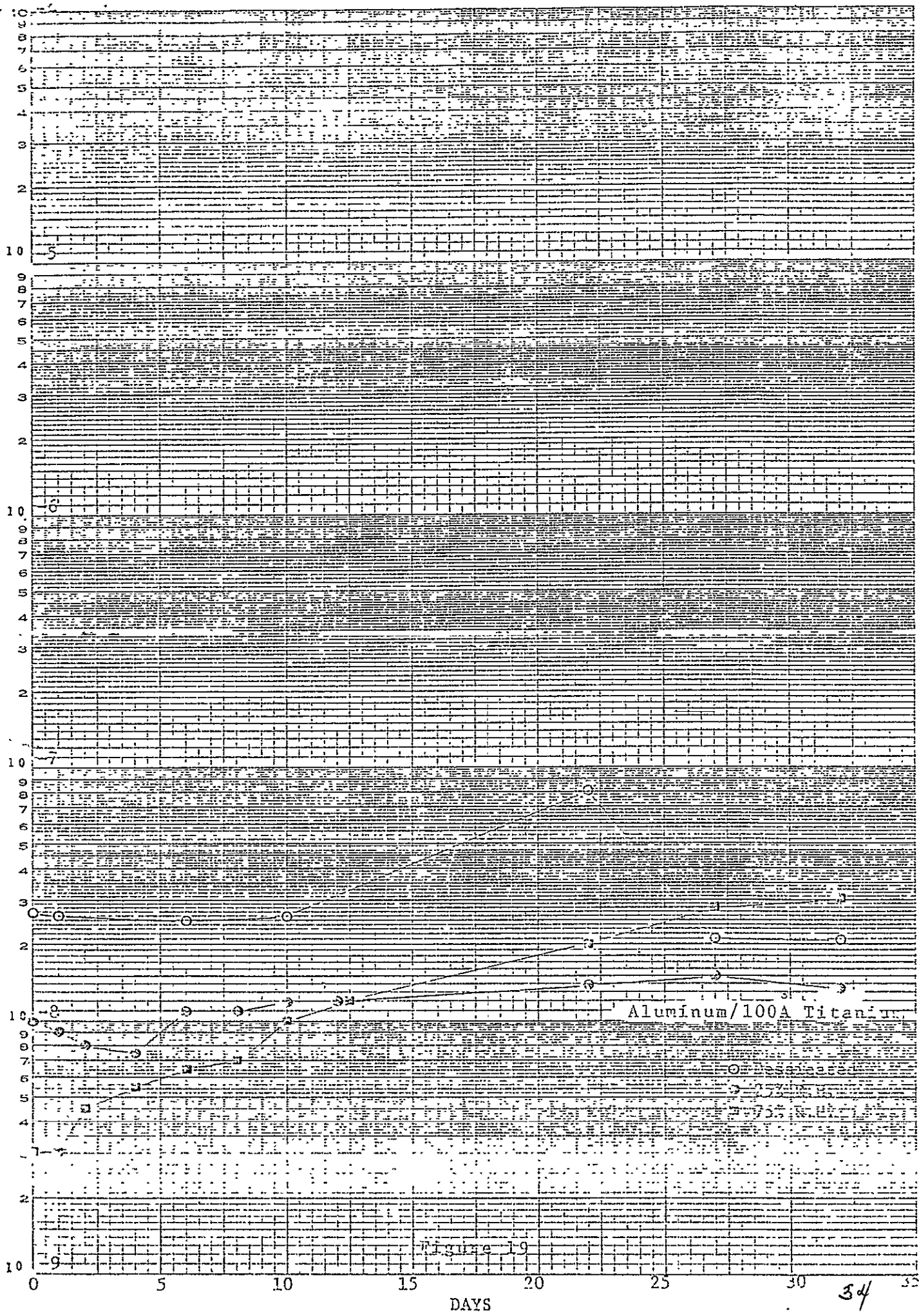
The peak transmission for titanium is at about the 400-Å wavelength. Transmission of Sigmatron's 1500-Å, 99 Al/Si filters is about 30 percent at this wavelength, and therefore offers a reasonably suitable tandem foil base. To achieve a series transmission approximating of a 525Å-thick titanium foil, a 100Å-thick layer of titanium over the aluminum is required. This was achieved wherein a conventional aluminum film received 100Å of titanium evaporated from a tungsten filament. —

The Al/100Å Ti test filters were completed too late in the program to have resulted in extensive aging data, but that which is available at this writing is shown in Figure 19. At low humidity, the test filters of this material graphed seem to change in a manner which could become significant within 100 days but the perturbation does not appear to be real and may be disregarded. The companion filters exposed to 25-percent R.H. and lower have shown none of this instability. The high-humidity tests indicate an aging characteristic similar to the 99 Al:Si foils.

No thermal degradation tests were performed on the Al/100Å Ti test filters since the melting point of these elements and their alloys are beyond the range of the photometer oven.



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Any conclusion regarding this type of filter is difficult at the present time. Certain suspicions regarding atomic diffusion between the two layers are difficult to allay when long-term stability is important. Then, too, with only 100Å of titanium exposed on one side of the aluminum, at least 50Å can be expected to oxidize before a protective layer has formed. Thus, this approach to a titanium type filter for satellite applications must be evaluated as needing further proof or to represent a last-resort solution. This could change if XUV transmission data, which should be very sensitive for this purpose, demonstrates stability.

### 3. Pure Titanium Foil

As a result of some of the more or less desparation work with titanium depositions, techniques developed for deposition of that difficult-to-deposit metal permitted preparation of pure metal foil (Commercial Grade 2N5). Within the time available the one foil prepared was only 1000Å thick. Although this was pinhole-free, it was not opaque to visible light. With that problem, it was out of range of the photometer for moisture aging or thermal degradation experiments. Therefore, no data on those features are available.

Conclusions regarding pure titanium are based on subjective considerations due to the lack of XUV and aging data. A reversal of opinion existing at the outset of this program with respect to the strength of ultrathin titanium alloys is in order. It is not clear what the XUV transmission would be, but it is our opinion that a 1600Å titanium foil would be opaque to the visible, could be made in reasonable quality (white light transmittance less than  $10^{-7}$ ) and would be structurally sound for launch environment. Moisture aging ought to be at least equivalent to pure indium but should be checked due to the high chemical reactivity of titanium. Also, the XUV transmission should be checked with aging for the same reason.



#### 4. Aluminum:Titaniun

The phase diagram for the aluminum-titanium system is shown in Appendix II. The alloy selected for a titanium type filter is based on 96Al/Ti, which in a 1800Å foil is equivalent to approximately 45Å of titanium. An alloy somewhat richer in titanium might have been selected; however, the experience gained from the aluminum-tin series tended to discount success with alloys with more than a few percent of any minor component. In retrospect, any alloy should have been primarily titanium with a few percent of some other element. This possibility did not present itself until some of the work above, undertaken after the 96 Al/Ti alloy work, was completed.

Moisture aging of the 96 Al/Ti filters is shown in Figure 20. This demonstrates that this material is not subject to pinhole degradation at low humidities. In high-humidity, this alloy appears to be very comparable to the aging of 99 Al:Si alloy, and after 57 day exposure the transmittance had increased  $2.7 \times 10^{-7}$ . The companion filter increased  $1.7 \times 10^{-7}$ .

No thermal degradation tests were performed on this alloy.

The XUV transmission for this alloy is shown in Figure 21 and compared there with similar data for the 99 Al:Si alloy. Considering that the titanium alloy is thicker than the silicon alloy absorption coefficients at 304Å wavelength for these are  $5.1 \times 10^4/\text{cm}$  and  $6.1 \times 10^4/\text{cm}$  respectively. Whether or not these values are accurate is not significant. What is important is that even though the alloy data spans the band pass of pure titanium, from onset to x-ray edge, there appears to be no strong departure from essentially pure aluminum transmission.

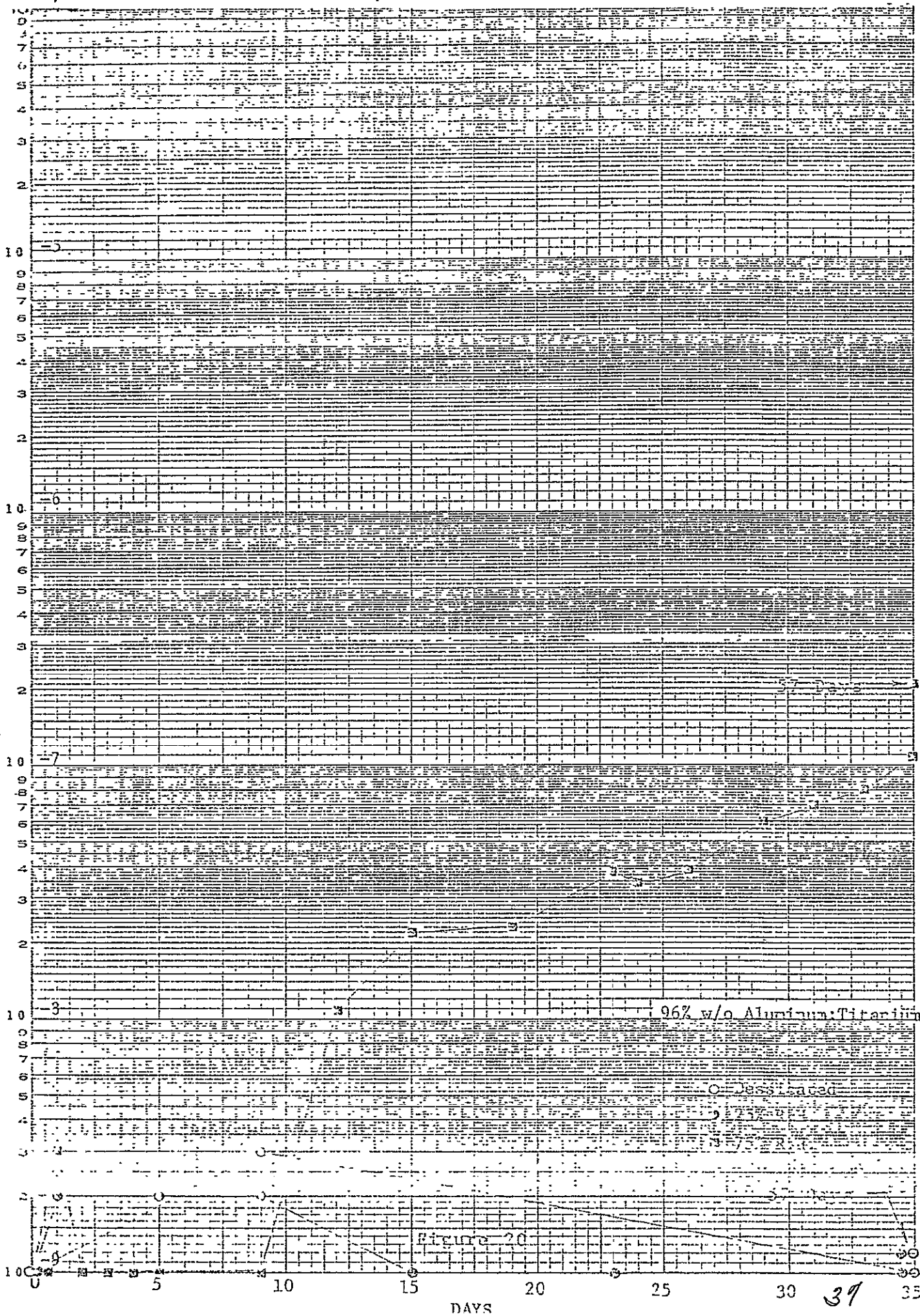
One may conclude that this alloy results in a foil which, from the standpoint of moisture aging, is rather superior. It does not, however, offer useful XUV transmission for a titanium type filter.



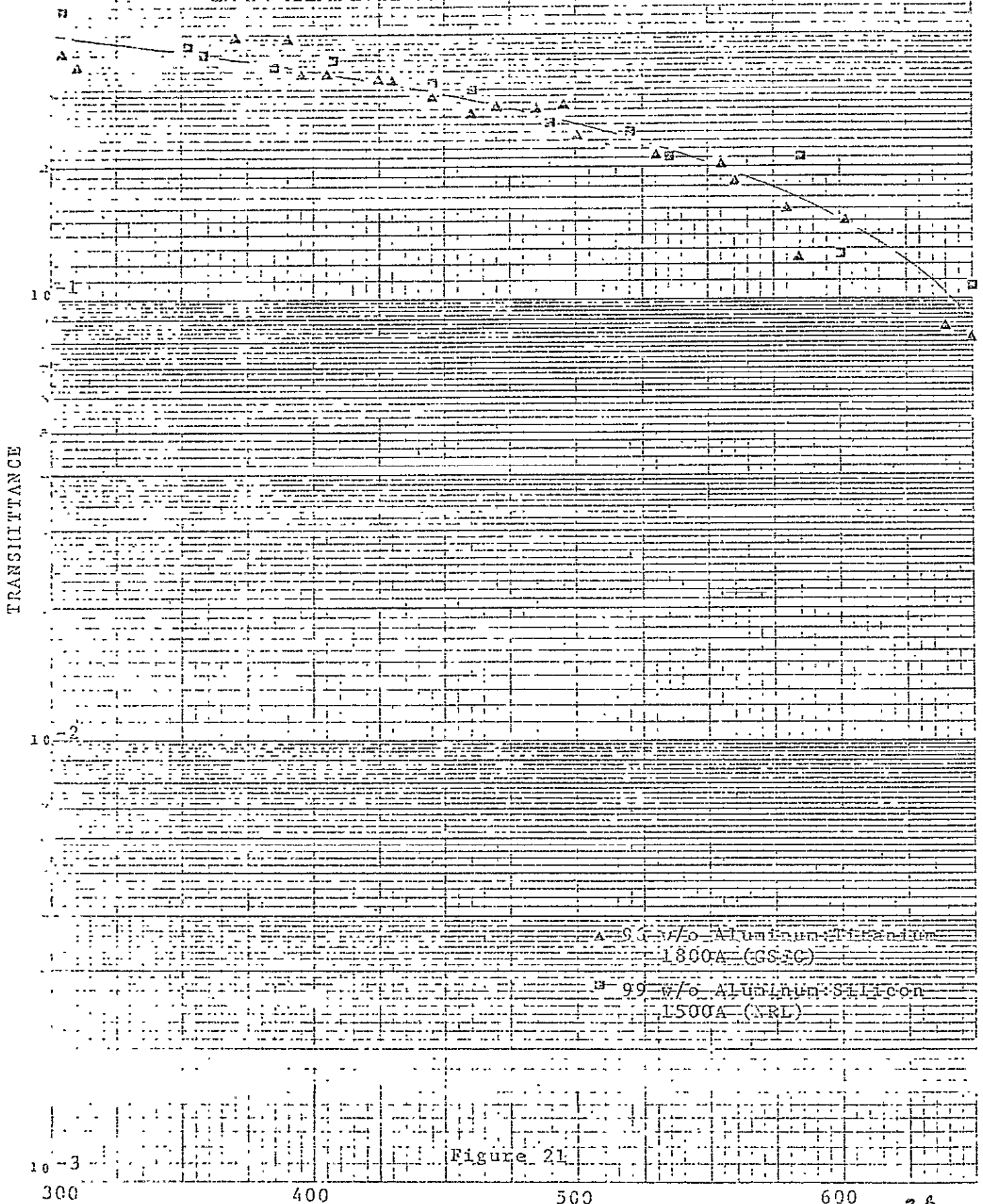
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## V. SUMMARY

It is felt that this work has added considerably to guidance in the problems associated with long-term reliance on ultrathin foil filters, particularly for satellite applications. In the atmosphere, one important problem is moisture degradation. In orbit, a major concern is thermal degradation.

Increasing the options for materials with suitable XUV band pass by alloying appears to be limited in scope but offer considerable merit.

Tin and aluminum have no intermetallic compounds and have fair transmissions individually at the 584Å and 740Å wavelengths, but it must be considered surprising that their binary alloy 75 a/o tin 25 a/o aluminum has by comparison essentially no transmission. Compositions on each side of the minimum have transmission monotonically increasing to the pure metal values. In this system, it appears that the pure metal transmission curve is not shifted by alloying, but perhaps uniformly suppressed, until at some mid-series composition is essentially zero. With further additions of the ad metal, the suppressed but unshifted transmission characteristics of that metal appear. This must be a tentative view, since more complete transmission data will be required for a firm conclusion.

Additions of small amounts of an alloying element have strong effect on the degrading effect of moisture and temperature. Wherein it was discovered that pure tin in ultrathin foil form is highly susceptible to moisture, alloying with a small amount of germanium provides a very satisfactory moisture resistance. On the other hand, all materials, pure metals and alloys, are sufficiently safe from degradation if exposed to environments of 25% R.H. or less.

Alloys are catastrophically degraded at the temperature of incipient melting. Pure metals develop increased pinhole transmittance by recrystallization below their melting points. Alloying with a few percent of another metal retards that recrystallization and results in an increased thermal stability.



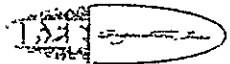
Anomalous thermal behavior of an alloy of indium with a small amount of titanium was observed wherein catastrophic loss did not occur at temperatures 100°C above the melting point of the indium. This may be accounted for by assuming that the indium was molten between two oxide layers.



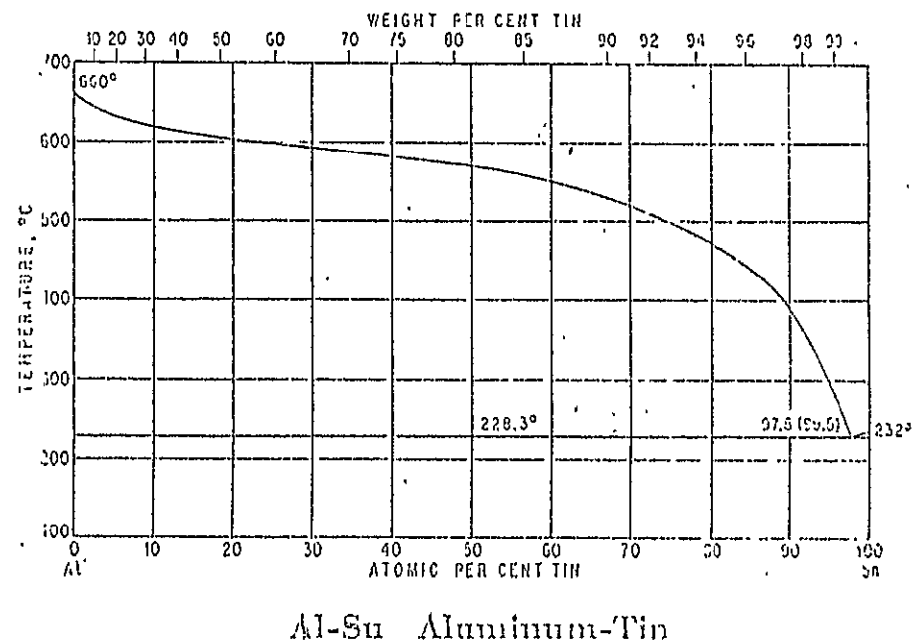
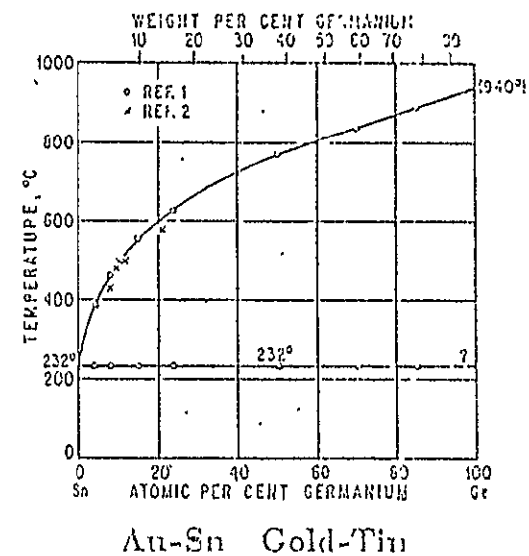
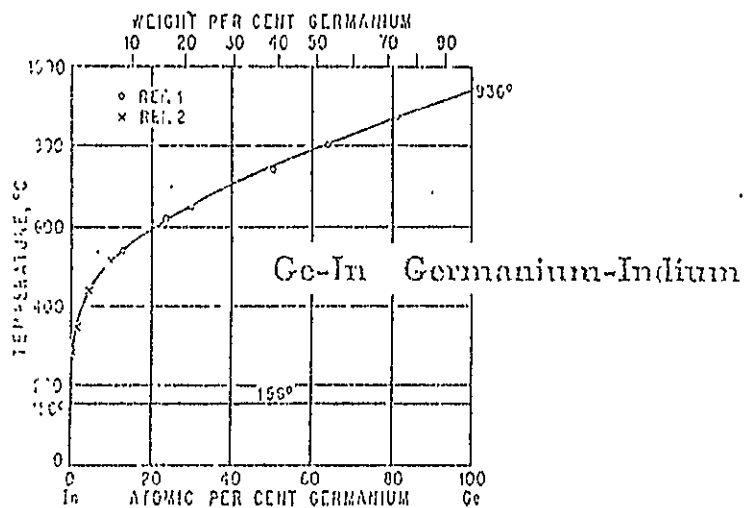
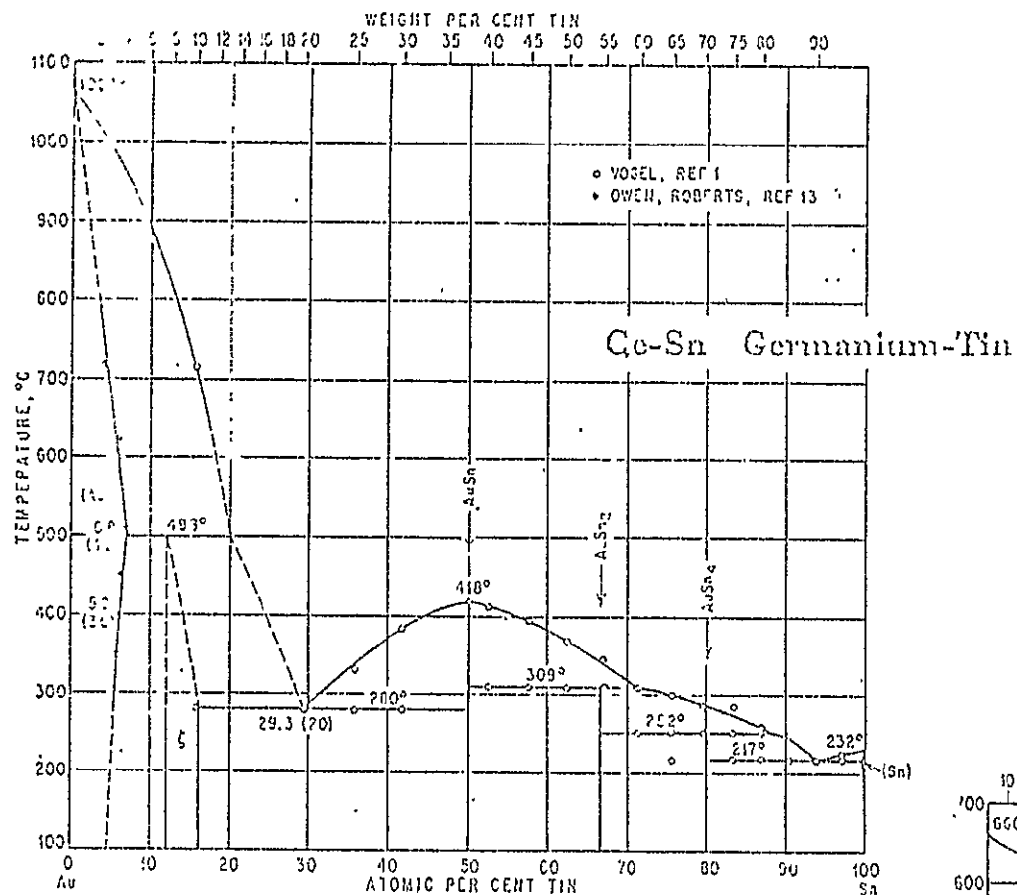
# APPENDIX T

## VAPOR PRESSURE OF ELEMENTS

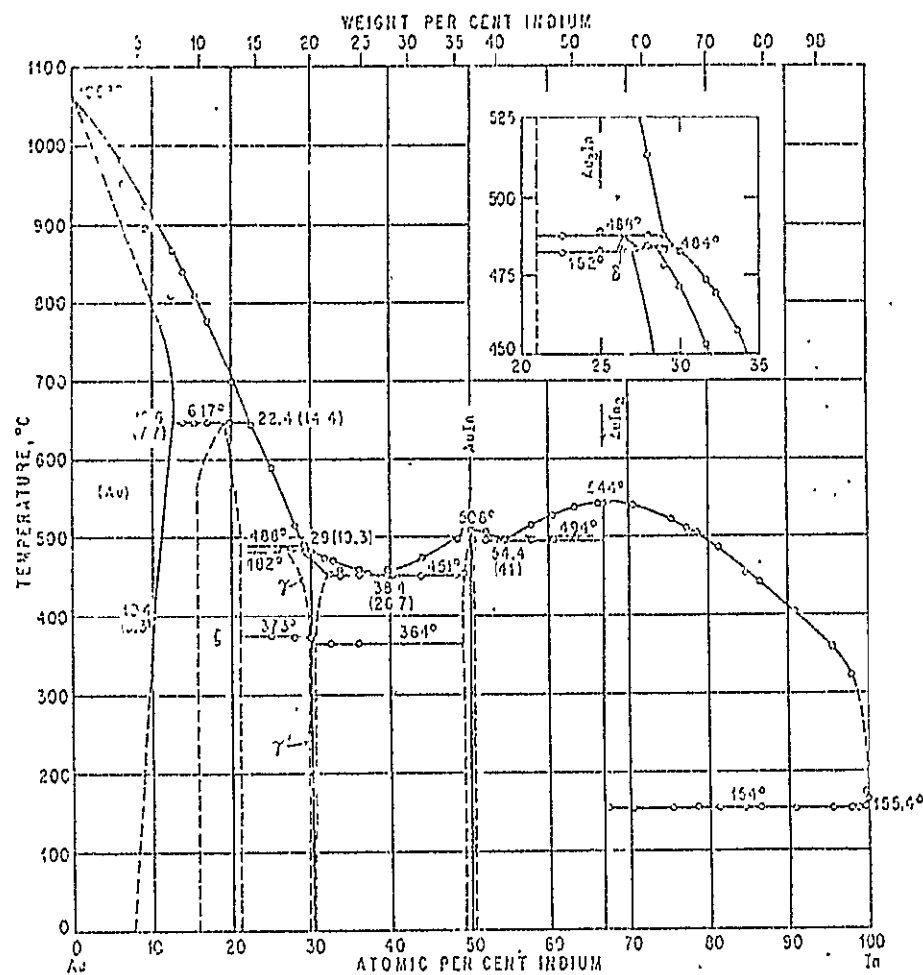
Metal	Melting Point °C	Temperatures (in °C) at which the Vapor Pressures are						
		0.001mm	0.01mm	0.1mm	1.0mm	10mm	100mm	760mm
Ag	961	936	1047	1184	1353	1575	1865	2212
Al	660	889	996	1123	1279	1487	1749	2327
As	817	237	277	317	362	437	517	613
Au	1063	1316	1465	1646	1867	2154	2521	2966
B	2000	1239	1355	1489	1648	3030	3460	2527
Ba	717	625	721	840	961	1049	1301	1638
Be	1284	1092	1212	1367	1567	1787	2097	2507
Bi	271	609	698	802	934	1136	1271	1627
Br	-7	-114	-99	-81	-59	-30	9	58
C		2471	2681	2926	3214	3946	4373	4552
Ca	810	528	605	700	817	983	1207	1482
Cd	321	220	264	321	394	484	611	765
Ce	785	1190	1305	1439	1599			2527
Cl	-101	-161	-150	-137	-120	-101	-72	-34
Co	1478	1494	1649	1833	2056			3097
Cr	1900	1090	1205	1342	1504			2222
Cs	29	110	153	207	277	387	515	690
Cu	1083	1141	1273	1432	1628	1879	2207	2595
Fe	1535	1310	1447	1602	1783	2039	2360	2727
Ga	30	965	1093	1248	1443	1541	1784	2427
Ge	959	1112	1251	1421	1635			2707
Hg	-40	18	48	82	126	184	261	361
I	114	-31	-11	12	39	72	115	183
In	157	840	952	1083	1260	1466	1756	2167
Ir	2454	2340	2556	2811	3118			4527
K	64	161	207	265	338	443	581	779
La	887	1242	1381	1549	1754			2727
Li	179	439	514	607	725	890	1084	1367
Mg	651	383	443	515	605	702	909	1126
Mn	1244	870	990	1100	1251	1500	1700	2000
Mo	2622	2295	2520		3102	3535	4109	4600
Na	98	238	291	356	437	548	696	914
Nb	2500	2539						5127
Nd	1297	1192	1342	1537	1775	2095	2530	3090
Ni	1455	1371	1510	1679	1884	2507	2364	2837
Os	2697	2451	2667	2920	3221			4627
P	597	160	190	225	265	310	370	431
Pb	328	625	718	832	975	1167	1417	1737
Pd	1555	1405	1566	1759	2000			3167
Po	254	263	314	385	475	590	750	965
Pt	1774	1904	2090	2313	2582	3146	3714	3827
Rb	38.5	123	165	217	283	387	519	679
Re	3180	2790	3060	3400				5630
Rh	1967	1971	2149	2358	2607			3877
Ru	2427	2230	2431	2666	2946			4227
S	119	66	97	135	183	246	333	444
Sb	630	595	678	779	904	1033	1223	1617
Sc	1397	1282	1423	1595	1804			2727
Se	217	200	235	280	350	430	550	685
Si	1410	1223	1343	1485	1670	1888	2083	2477
Sn	232	1042	1189	1373	1609	1703	1968	2727
Sr	771	475	549	639	750	898	1111	1384
Ta	2996	2820	3070	3370	3740			6027
Te	450	336	383	438	520	633	792	990
Th	1827	1999	2196	2431	2715			4227
Ti	1727	1384	1546	1742	1965	2180	2480	3127
Tl	304	527	606	702	821	983	1196	1457
U	1132	1730	1893	2098	2338			3527
V	1677	1725	1838	2079	2297	2570	2950	3527
W	3382	3016	3309	3640	3990	4507	5168	5677
Y	1477	1494	1649	1833	2056			3227
Zn	419	292	343	405	487	593	736	907
Zr	2127	1616	2001	2212	2459			3577



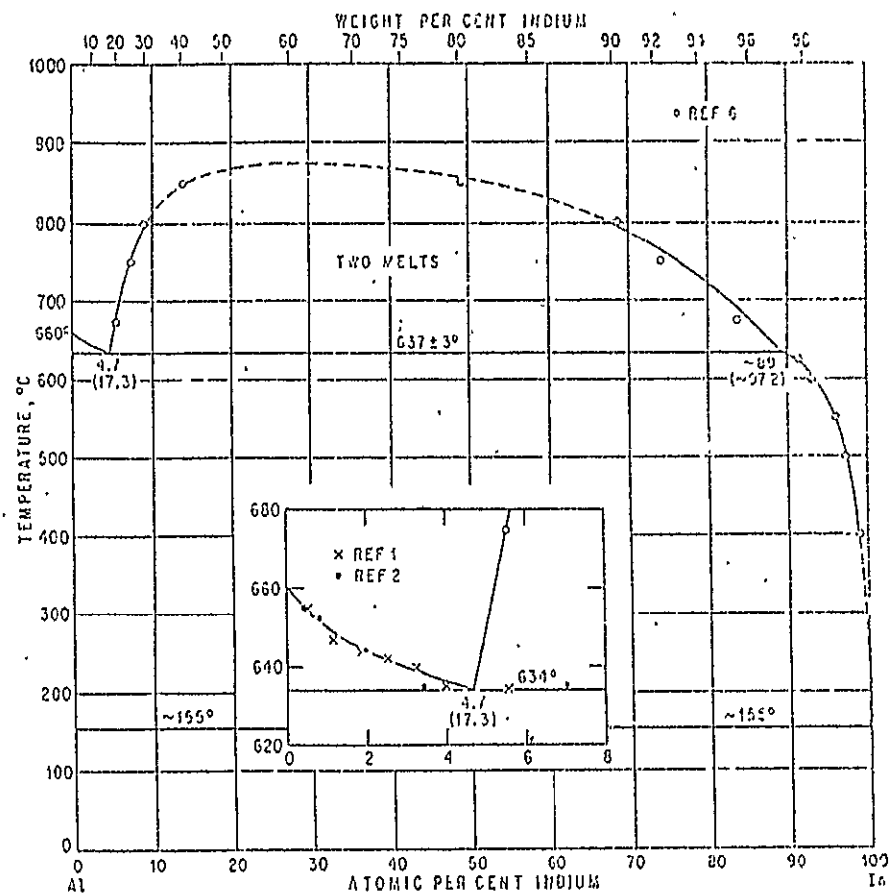






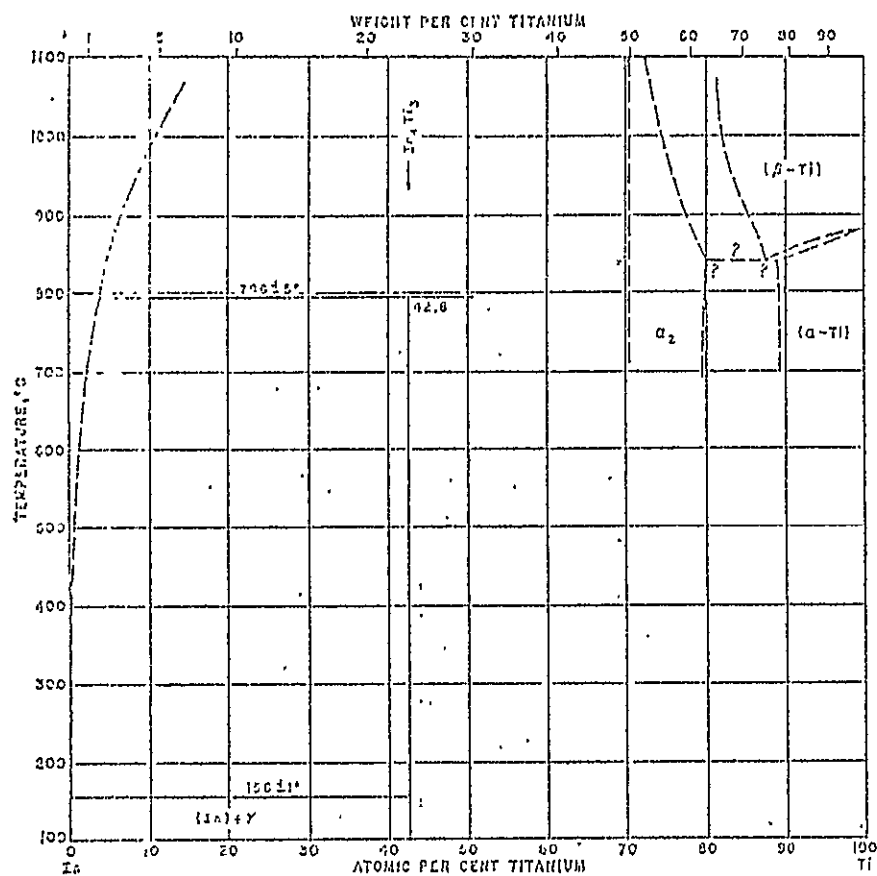


Au-In Gold-Indium

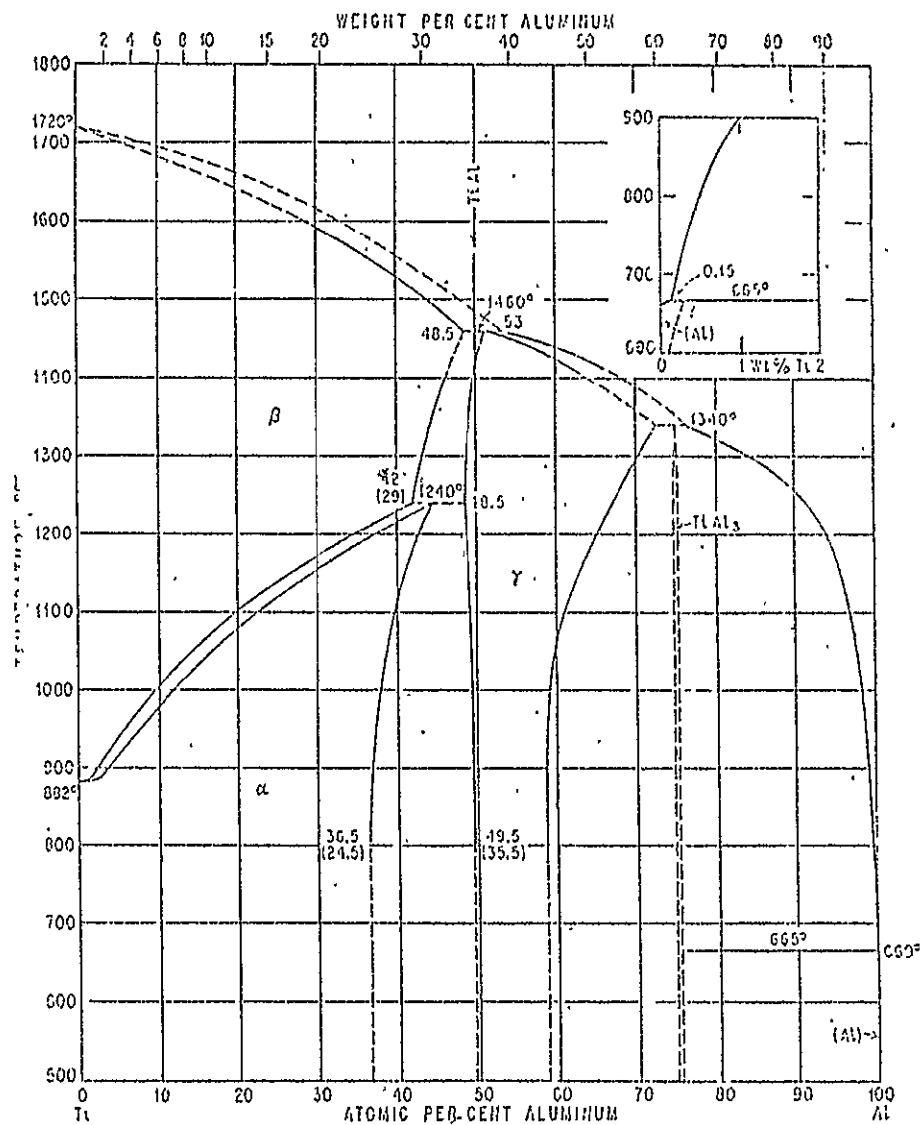


Al-In Aluminum-Indium





In-Ti Iodine-Titanium



Al-Ti Aluminum-Titanium