SELENIUM IMMERSED THERMISTOR BOLOMETER STUDY

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16. Abstract
Thermistor bolometers, optically immersed in arsenic/selenium glass have previously become noisy after varying intervals of time and particularly when exposed to elevated temperatures. This study examined the noise characteristics of thermistor bolometers immersed in layers of arsenic/selenium glass which were uniform in composition. Using a controlled deposition technique, layers of glass were deposited, thermistor bolometers immersed and their electrical characteristics measured after various thermal treatments. Markedly improved stabiity of the detector noise was observed using this new technique.

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List of Abbreviations and Symbols

Ge	Germanium
S.O.W.	Statement of Work
As	Arsenic
Se	Selenium
S.E.M.	Scanning Electronic Microscope
V_p	Peak Voltage
Al	Aluminum
R	Flake Resistance
N/No	Normalized Noise
τ	Time Constant
S	Detector Signal
V_B	Bias Voltage (0.6 V_p)
V_n	Noise voltage with bias
V_{no}	Noise voltage without bias
T_a	Ambient Temperature

1.0 SUMMARY

The noise characteristics of thermistor bolometers immersed in As/Se glass were considerably improved using a new deposition technique. A study of the As/Se glass itself was carried out using "partial" bolometers which were solely Ge flats with a layer of the glass on one surface. To study the noise properties of the thermistors, 24 germanium flats were coated with As/Se glass and 2 thermistor flakes were immersed into the glass on each flat. Twelve of the flats had 8% As/Se glass on them and the other twelve 12% As/Se glass. The glass was deposited using glow discharge, a shutter and crystal monitor to put down glass layers of uniform composition and controlled thickness.

These 48 flakes were measured for peak voltage, resistance, time constant, signal and noise after baking at various temperatures. After 168 hrs at 50°C, 168 hrs. at 60°C and 500 hrs at 70°C the average noise increase was less than 20%. No failures were recorded despite the rigorous thermal treatment given to the detectors.

2.0 OBJECTIVE

Improve and refine current manufacturing techniques and processes of selenium immersed thermistor bolometers to the extent that they will reliably perform at elevated temperatures up to at least 50°C.

3.0 INTRODUCTION

The theoretical basis upon which this project was based involved the inhomogeneous nature of the deposited arsenic selenium glass. Pure selenium, in contact with the flake gradually lost its glassy state and became crystalline at a rate dependent on the device temperature. This arsenic free crystalline layer produced a noisy resistance in parallel with the thermistor flake. Since the presence of impurities is known to inhibit this change of state, a new method of depositing the arsenic/selenium glass was proposed to ensure that the deposited glass was uniform, so that the glass in contact with the flake contained the proper fraction of arsenic.

A number of germanium (Ge) flats were covered with a film of glass to enable NASA to study the glass properties. Subsequently, 24 more germanium flats were prepared and two 0.1mm x 0.2mm thermistor flakes were immersed on each flat. The bolometers were subjected to a number of elevated temperature bakes and electrical tests. The results of these tests, their interpretation and recommendations, follow the body of this report.

4.0 ARSENIC/SELENIUM GLASS DEPOSITION

4.1 Initial Deposition As a basis for comparision, the first set of Ge flats (Item 1 of NASA S.O.W.) had a layer of As/Se glass deposited upon them in the usual manner, i.e., without a glow discharge, shutter or thickness monitor. This was described previously in the monthly report 9/12/78 - 9/30/78 and is reproduced below.

The equipment and material used to deposit the selenium-arsenic mixture is as follows:

1. Evaporation Source Boat - molybdenum with an iron-constantan thermocouple attached to the external underside
2. Selenium-Arsenic Mixture - 92.25% Se, 7.75% As by weight, 99.999% pure, mixed in molten state and solidified in a prior operation
3. Weight of Charges Placed in Boat - 4.48 grams and 0.5 grams
4. Distance from Source to Substrates - 18 cm
5. Substrates - 6 polished germanium flats, optical quality, 15mm diameter, 1mm thick
6. Germanium Cleaning Procedure - boiled in trichloroethylene for 15 seconds, removed and immediately immersed in methyl alcohol, gently swabbed in methyl alcohol, removed, placed in evaporation fixture, and placed in bell jar

A log of the events during the vapor deposition of the BEC standard mixture of 92.25% Se, 7.75% As follows. Note that this was done without a shutter, thickness monitor, or glow discharge. Date: 9/19/78.

Time	Operation	Remarks
9:05 a.m.	Start evacuation	Mechanical pump
9:06	Switch to diffusion pump	Liquid nitrogen cooled
9:10	Evacuating bell jar	1×10^{-5} torr
9:13	Evacuating bell jar	2×10^{-6} torr
9:16	Apply power to source	1.6×10^{-6} torr
9:27	Source temperature 380°C	1.4×10^{-6} torr
10:12	Source temperature 390°C	1.0×10^{-6} torr
	Source Power Off	
10:42	Bell jar opened	Source refilled with 0.5 grams of 92.25% Se, 7.75% As
10.47	Start evacuation	Mechanical pump
10:50	Switch to diffusion pump	Liquid nitrogen cooled
10:53	Evacuating bell jar	2×10^{-6} torr
10:54	Apply power to source	1.4×10^{-6} torr
10:58	Source attains 180°C, turned off	1.1×10^{-6} torr
11:30	Bell jar opened, 6 samples removed	1.0×10^{-6} torr

The six sample substrates that were removed from the bell jar constitute Item 1 in the NASA Statement of Work. These were inspected under a 100 power microscope prior to shipment. A number of small mounds were noticed on the vapor deposited Se-As surface. They are thought to be spatterings from the material in the source boat. It is hoped that NASA can analyze these mounds and check on the material.

The bell jar was modified after the previous run and the following equipment was installed:

1. Manually operated shutter
2. Glow discharge - D.C. system, CVC type SDC-100
3. Thickness monitor - Sloan type DTM-3

A photograph is included to show this equipment after installation. Figure 1. A frequency change of 60KHz was calculated to produce a 10 micron thick layer of Se-As. A trial run was made using glass slides as substrates. The new controls listed in Paragraph 1.3

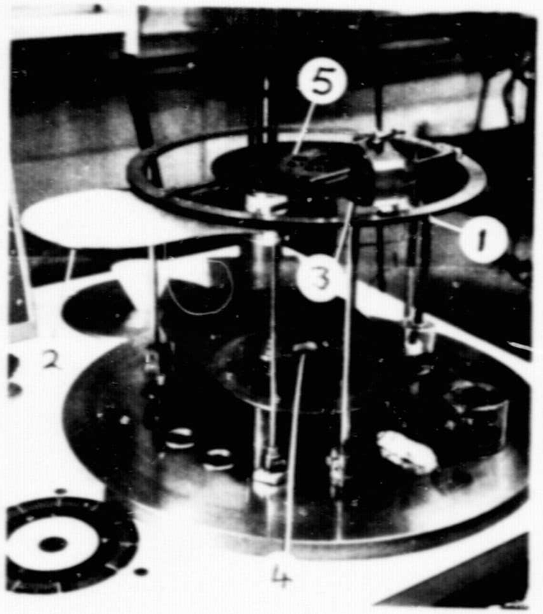
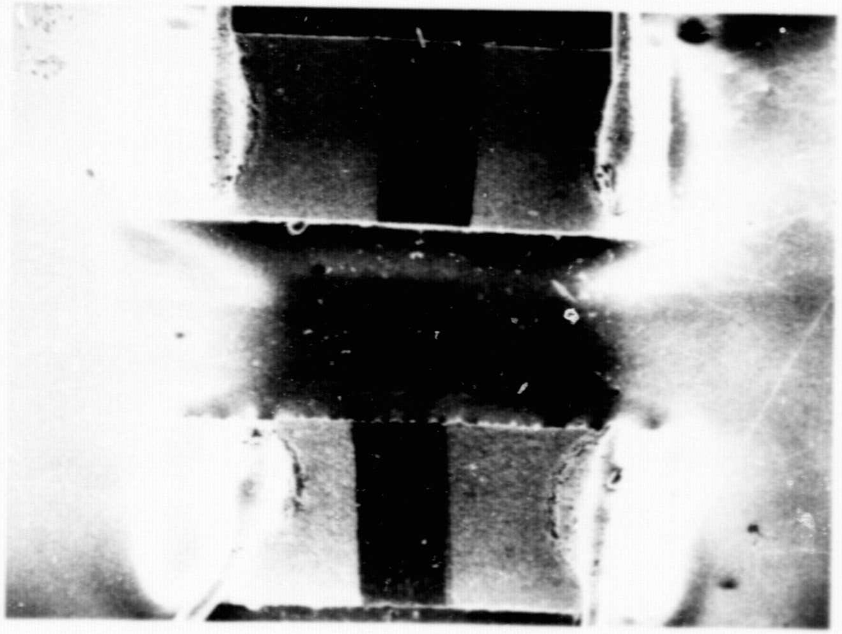


Figure 1.

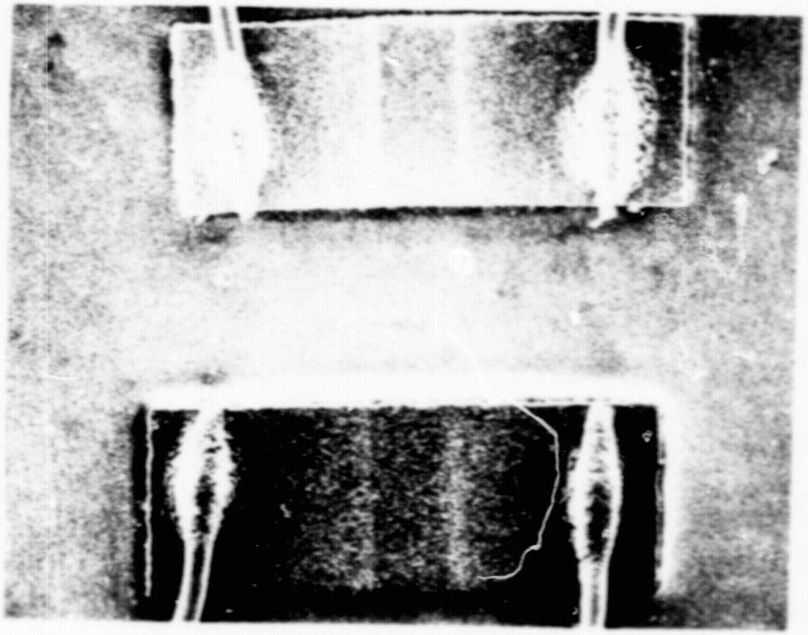
Evaporation Equipment with New Features

1. Glow Discharge
2. Shutter
3. Thickness Monitor
4. Source with Thermocouple
5. Substrate Holder



#34 Typical flake. No top coat

Figure 26 S.E.M. X130



#32 Typical flake with top coat

Figure 27 S.E.M. X130

of the NASA Statement of Work were followed during the trial run. After the run, the thickness of the deposited Se-As layer was determined by SEM analysis to be 13.3 microns.

4.2 <u>System Calibration</u> The thickness monitor, Sloan DTM-3, used in the initial calibration runs (Table 1) was replaced by the more accurate and versatile Sloan 1000 system. Evaporation runs 4 → 7B were used to correlate the measured Angstrom count on the Sloan 1000 to the layer thickness, which was measured on both the Unitron microscope and the Scanning Electron Microscope (S.E.M.).

4.3 <u>New Glass Preparation Method</u> In section 4.1 the deposition of 8% As/Se glass onto 6 germanium flats was described. These layers were examined at NASA by J. L. Tarpley using Auger analysis and found to contain copper. Two possible sources of the copper were considered: evaporation from the copper conductors to the evaporation source and from the As/Se glass source. The copper conductors were capped with glass covers and the source preparation charged as described below. Previously the preparation of the Se/As glass had been carried out using a quartz flask with a rubber stopper through which a copper pumping tube was passed. This was pumped to less than 10^{-5} torr and then pinched off prior to reacting the two components. It was felt that the source of copper in the evaporated layers could be caused by fragments of copper or copper oxide from the copper pinch off tube. To eliminate this possible source, an all quartz system was set up. The materials are loaded through a wide tube on the side of the flask which is sealed prior to evacuation (Figure 2).

When the pressure is less than 10^{-5} torr the flask is sealed and the material reacted as before.

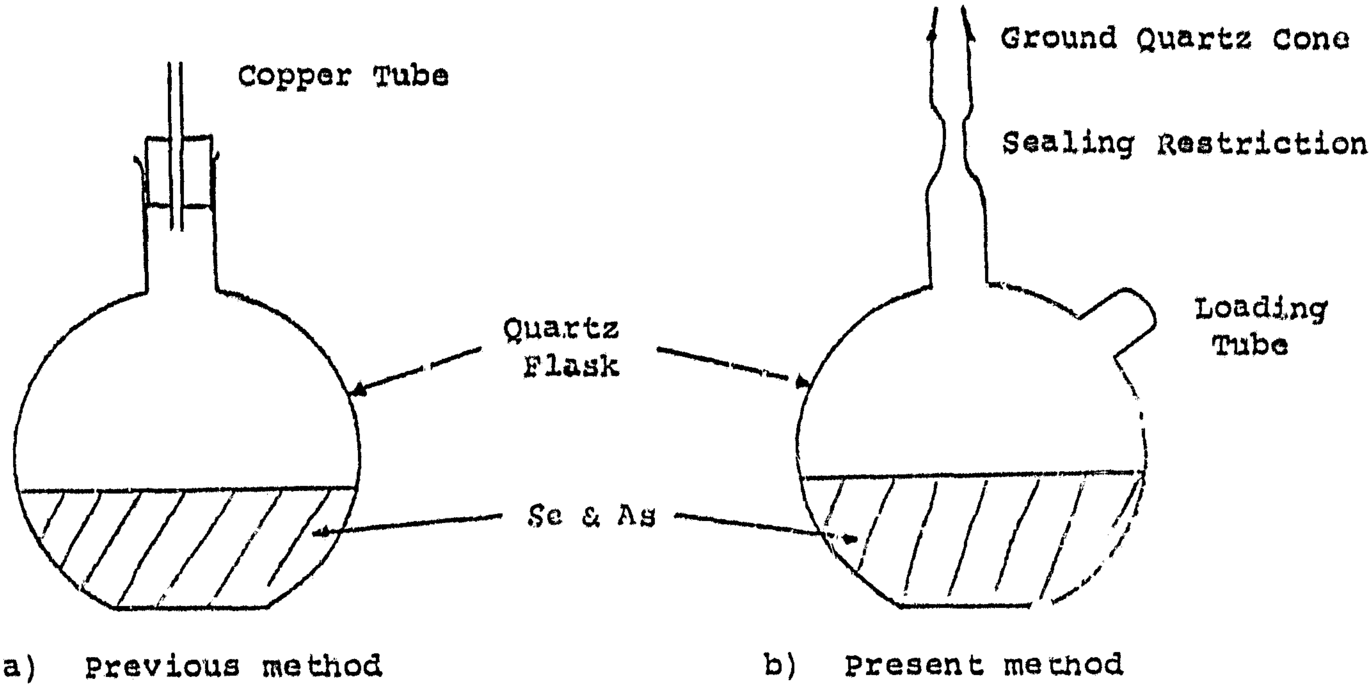


Figure 2
Comparison of Glass Preparation Methods

4.4 <u>Detector Preparation</u> As shown in Table 1, six partial bolometers using 8% As/Se glass and six partial bolometers using 12% As/Se glass were prepared. At this time the thickness calibration was complete and only glass prepared according to section 4.3 was used as a source material. These partial bolometers were designed to allow analysis of the glass. Further layers were evaporated and thermistor bolometers immersed to provide the detector elements used in the noise study. Figure 3 shows a representation of the physical construction of the bolometer. Approximately 7μm of As/Se glass is evaporated onto the Ge flat. The thermistor flake, which has a 3μm layer of glass upon its surface, is immersed into the 7μm layer. Finally, a top coat of 3um of the glass is deposited. In this study the bolometers were immersed in pairs to give two detectors on each Ge flat. Table 1 gives a list of all evaporations carried out using the

Run #	% As Batch #	t (μm) S.E.M.	t(μm) Unitron	Surface Features	Thick 2 (Sloan 1000) Å	Mean Rate Å/sec	Comments
1 2934	8% 45	13.3	13	Mounds Dimples etc.	-	-	Sloan DTM-3 used to calibrate thickness.
2 2935	8% 45	11.7	11.5	"	-	-	Unsuitable as accurate monitor and all subsequent runs use Sloan 1000 System.
3 2937	8% 45	8.1	8.6	"	-	-	
4 2939	8% 47	9.3	10.5	"	16,140	90	- Calibration of
5 2940	12% 47	6.6	7.7	"	9,885	62	- Sloan 1000 System
6 2941	12% 47	8	10.3	"	13,180	100	-
7A 2942	12% 47	9.7	9.4	"	13,070	50	-
7B 2943	12% 47		1.1	"	1,634	-	-
8A 2945	12% 47	7.9	8.8		15,110	60	6 Ge Flats Item 2 (part)
8B 2946	12% 47	2.8	3.0	Many mounds	5,510	110	6 Ge Flats from #8A
8C 2947	12% 47	(500Å)	/		150	~ 100	6 Ge Flats from #8B
9A 2948	8% 48	10.0	10.1	10 mounds per flat	16,610	105	6 Ge Flats Item 2 (part)
9B 2949	8% 48	(500Å)	/		112	80	6 Ge Flats from #9A
10A 2950	8% 48	7.0	7.5	1 mound per flat	12,760	105	6 Ge Flats 3/32" aperture. Test only.
11A 2958	8% 48	Not measured			5,200	60	~ 20 0.1mm x 0.2mm flakes

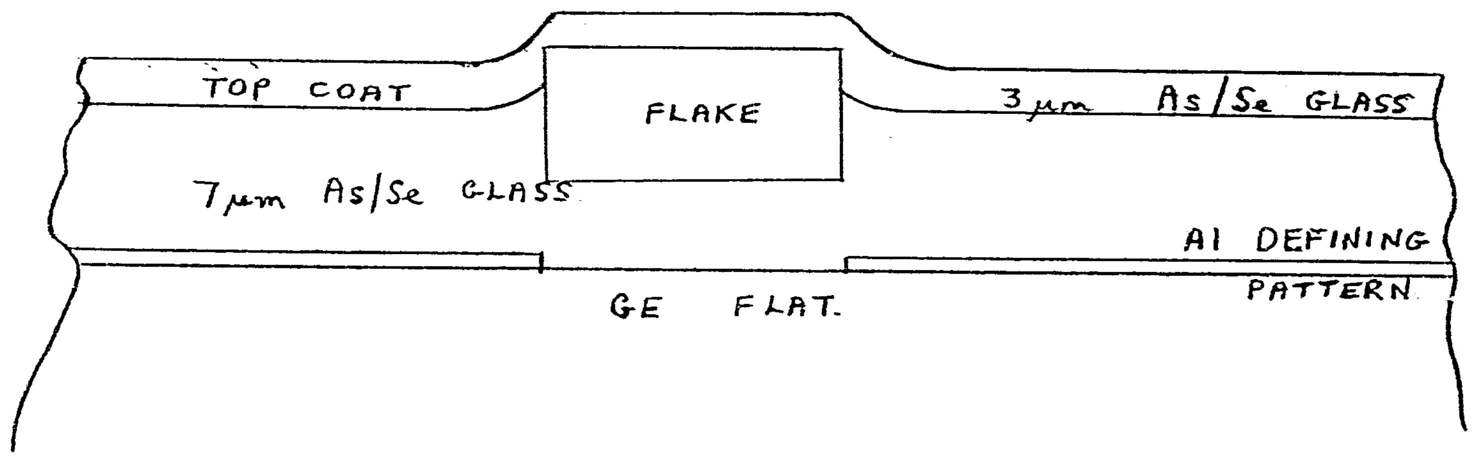
Table of Evaporation Data

Table 1

Run#	% As Batch #	t (μm) S.E.M.	t(μm) Unitron	Surface Features	Thick 2 (Sloan 1000) Å	Mean Rate Å/sec	Comments
11B 2959	8% 48	6.5	7.0	Free of mounds	9,885	50	6 Ge Flats Al pattern 3/32" aperture
11C 2962	8% 48	6.2	7.0	None	11,230	95	6 Ge flats Al pattern
11D 2966	8% 48	2.5	2.45	None	4,378	116	Top coat on 11B
11E 2970	8% 48	2.8	3.1	None	4,608	100	Top coat on 11C
12A 2974	12% 47	Not measured			5,632	40	0.1mm x 0.2mm Flakes
12B 2975	12% 47	7.4	7.25	A few mounds	11,270	65	6 Ge flats Al pattern
12C 2980	12% 47	8.6	7.5	A few mounds	11,260	60	6 Ge flats Al pattern
12D 2986	12% 47	2.8	3.0	A few mounds	4,688	50	12B top coat
12E 2989	12% 47	3.1	3.0	A few mounds	4,733	60	12C top coat
13A 2994	8% 48	6.8	7.1	None	12,110	90	7 Ge flats Al pattern
13B 2997	8% 48	-	-	None	4,600	115	.1 x .2mm^2 flakes
13C 3006	8% 48	6.3	7.5	None	12,520	100	1 Ge flat Al patterr
13D 3008	8% 48	Not measured		None	4,768	65	To coat on 13A and 13C
14A 2990	12% 47	Not measured		None	4,789	80	0.1mm x 0.2mm Flakes
14B 3005	12% 47	7.13	7.5	A few mounds	12,420	75	2 Ge flats Al pattern
14C 3010	12% 47	Not measured		A few mounds	4,741	100	Top coat on 14B

Table of Evaporation Data

Table 1 (Cont'd)



Detector Construction

Figure 3

new calibrated/controlled system. The immersions were performed with Ge flat temperatures ranging from 119°C to 153°C as shown in Table 2. In general the 8% As/Se glass required a higher immersion temperature than the 12%.

5.0 DETECTOR MEASUREMENTS

5.1 Measurement Schedule The following electrical tests were performed on all flakes at various intervals during the thermal soak cycle.

1) Time constant. Table 3.
2) Signal voltage with 0.6 peak voltage bias. Table 3.
3) Peak voltage. Table 4.
4) Noise voltage in the 5-100Hz bandwidth with 0.6 peak bias voltage and with no bias voltage. Table 5.
5) Bandwidth.
6) Electrical resistance between the two flakes and from each flake to the Ge flat with 0.6 peak voltage applied in dual polarity. Table 6.

After manufacture, the 12 units of each composition were divided into groups 5-5-2. The first five were soaked at temperatures with no bias, the second five were soaked at temperatures with 0.6 peak voltage bias (temperature adjusted - Figure 4) and the remaining two kept at room temperature and with no bias. All the results are shown in Tables 3-6.

5.2 Discussion of Electrical Measurements Each parameter measured will be discussed and related to the appropriate figure. The temperature bake was performed in the oven shown in Figure 5.

5.2.1 Isolation Resistance The isolation resistance between flakes and between each flake and the Ge flat are for the most part in the $10^{13}\Omega$ range (Table 6). Only in the case of 4 flakes with high noise was there a correlation between the isolation resistance and noise. Here the isolation resistance had dropped to $\sim 10^{6}\Omega$ in all cases.

5.2.2 Peak Voltage - Flake Resistance The peak voltage (V_p) and the flake resistance (R) (as measured on the equipment shown in Figure 6) show an increase with time in all cases, including those held at room

Date	Lens No.	Flake Size	Tamping Wt.	Final Temp. °C	% As
12/4/78	1	.1 x .2	57 g	153	8
12/4/78	2	"	"	145	8
12/4/78	3	"	"	146	8
12/11/78	4	"	"	141	8
12/11/78	5	"	"	141	8
12/11/78	6	"	"	144	8
12/12/78	7	"	"	136	8
12/12/78	8	"	"	137	8
12/12/78	9	"	"	133	8
12/13/78	10	"	"	133	8
12/13/78	11	"	"	128	8
12/13/78	12	"	"	130	8
1/3/79	13	"	"	138	12
1/4/79	14	"	25 g	135	12
1/4/79	15	"	"	132	12
1/5/79	16	"	"	134	12
1/5/79	17	"	"	133	12
1/8/79	18	"	"	129	12
1/9/79	19	"	"	123	12
1/10/79	20	"	"	130	12
1/10/79	21	"	"	25	12
1/12/79	22	"	"	119	12
1/12/79	23	"	"	120	12
1/16/79	24	"	"	129	12
1/24/79	25	"	"	143	8
1/23/79	26	"	"	130	8
1/24/79	27	"	"	136	8
1/24/79	28	"	"	134	8
1/24/79	29	"	"	127	8
1/25/79	30	"	"	139	8
2/1/79	31	"	"	124	8
2/14/79	32	"	"	127	12
2/14/79	33	"	"	126	12
2/14/79	34	"	"	123	8

Glass Immersion Temperatures
Table 2

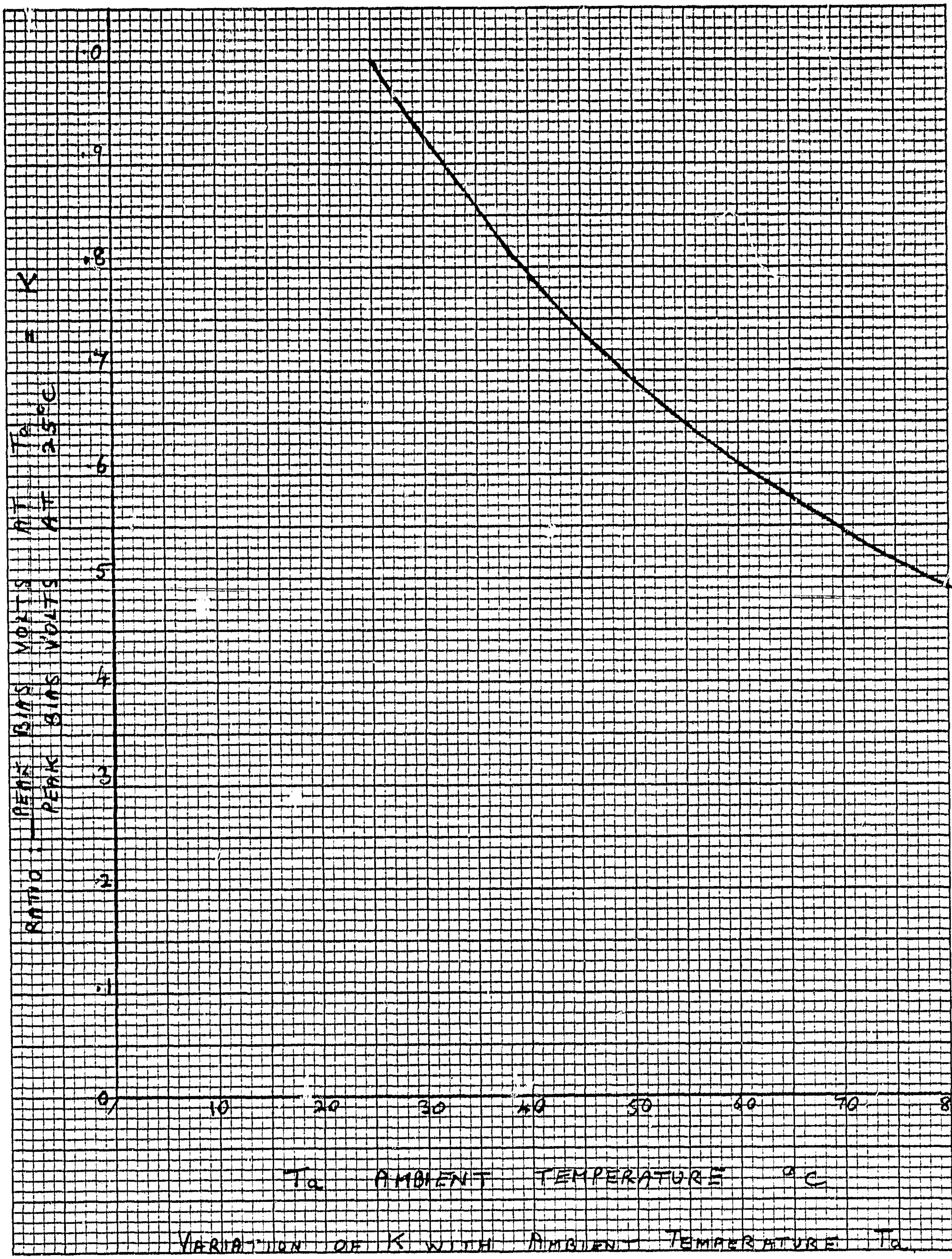

RATIO : MEAN BIAS VOLTS AT Ta / PEAK BIAS VOLTS AT 25°C = K
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
10
20
30
40
50
60
70
80
Ta AMBIENT TEMPERATURE °C
VARIATION OF K WITH AMBIENT TEMPERATURE Ta

Figure 4

Flat	Flake	Initial		Post 50°C		Post 60°C		Post 70°C		Post 70°C		Post 70°C	
		S (μV)	τ(mS)	S (μV)	τ(mS)	S (μV)	τ(mS)	S (μV)	τ(mS)	S (μV)	τ(mS)	S (μV)	τ(mS)
2	1	5.5	2	9.1	2	8.4	2	8.6	2	8.6	2	8.6	2.1
	2	5.4	2	8.6	2	8.1	1.7	8.0	1.7	8.0	1.8	8.2	1.7
5	1	7.4	1.5	8.8	1.5	8.5	2	8.6	2	8.5	2	8.6	2
	2	7.6	1.5	9.6	1.5	8.8	2	9.0	2	9.2	2	9.2	2
8	1	5.7	1.5	7.2	1.5	6.9	1.6	7.5	1.6	6.9	1.7	6.8	1.8
	2	6.0	1.5	7.7	1.5	7.1	1.8	7.8	1.8	7.1	1.8	7.2	1.8
9	1	6.2	1.5	7.9	1.5	8.0	1.8	8.2	1.8	8.0	1.8	8.4	1.8
	2	5.2	2	7.0	2	6.8	1.8	6.8	1.8	6.9	1.9	6.7	1.8
10	1	6.5	1.5	8.6	1.5	7.7	1.8	7.6	1.9	7.8	2	7.8	2
	2	7.3	1.5	10.5	1.5	8.6	1.8	9.4	1.8	8.4	1.9	9.3	1.8
25	1	7.8	1.5	7.9	1.9	7.8	1.8	8.2	1.8	8.1	1.8	8.3	1.7
	2	7.9	2	9.7	2	8.2	1.8	8.8	1.8	8.2	1.9	8.9	1.8
26	1	11.0	2	1.6	1.8	14.5	2.1	14.2	2	14.0	2.1	13.5	2
	2	10.5	2	10.8	1.9	7.8	2	8.0	1.9	8.3	2	8.1	2
28	1	9.5	2	11.0	2	9.8	2	10.0	2	9.4	1	9.9	2
	2	9.1	2	11.2	2	9.6	2	10.2	2	9.7	2	9.4	2
29	1	8.2	1.5	8.6	1.9	10.5	2	10.5	2	10.2	1.9	10.5	1.9
	2	19	2	22	2	20.5	1.9	12.5	1.9	13.8	1.8	12.5	1.8
30	1	7.0	1.5	7.7	1.9	8.0	2	8.2	2	8.0	2.1	8.0	2
	2	7.7	1.5	8.6	1.7	9.5	1.9	9.8	2	10.0	2	10.0	2
31	1	10.5	1.8									7.1	1.6
	2	10.0	1.7									7.4	1.4
32	1	3.9	1.3									10.0	1.9
	2	4.2	1.2									9.8	1.8

Detector Signal and Time Constant

Table 3

Flat	Flake	Initial		Post 50°C		Post 60°C		Post 70°C		Post 70°C		Post 70°C	
		S(μV)	τ (mS)	S(μV)	τ(mS)	S(μV)	τ(mS)	S(μV)	τ(mS)	S(μV)	τ(mS)	S(μV)	τ(mS)
15	1	9.2	2	9.7	2	9.2	2	8.5	2	8.8	2	8.6	2
	2	8.2	1.8	9.0	2	8.5	1.9	7.5	2	8.2	2	8.2	1.8
16	1	9.2	1.8	9.5	1.9	10.7	1.9	11.5	2	11.2	1.9	11.0	1.9
	2	9.0	1.8	9.4	2	10.0	2	10.5	2	10.0	2	10.0	1.9
22	1	8.6	2	8.7	2.4	9.0	2.4	10.5	2.5	11.0	2.4	12.5	2.3
	2	8.7	2	10.0	2.5	10.0	2.5	11.5	2.5	12.5	2.5	13.0	2.4
23	1	8.7	2	8.6	2.6	10.0	2.5	10.0	2.5	9.6	2.5	8.2	2.5
	2	8.5	1.5	9.3	2.5	9.0	2.3	9.0	2.5	9.1	2.4	9.5	2.2
24	1	9.0	2	9.2	2.5	8.9	2.5	8.5	2.5	8.6	2.6	8.6	2.5
	2	8.6	2	9.4	2.6	-	-	8.5	2.5	8.9	2.5	8.9	2.5
14	1	9.5	2	9.1	2	10.8	2	10.0	2	10.0	2	-	-
	2	9.5	2	3.5	2	11.5	2	11.0	2	11.0	2	-	-
17	1	9.3	2.1	9.2	2.3	12.5	2.4	12.0	2.4	11.0	2.4	11.5	2.6
	2	9.0	2	8.2	2.1	10.8	2.2	10.0	2.1	10.0	2.1	10.0	2
18	1	10.8	2.2	9.8	2.4	9.6	2.4	9.5	2.4	9.3	2.5	9.6	2.4
	2	11.8	2	10.0	2.2	10.0	2.2	9.8	2.3	10.0	2.3	10.0	2.2
20	1	8.8	1.8	8.2	1.9	9.2	1.9	9.3	2	9.2	2	8.7	2
	2	8.3	1.8	7.2	2.1	8.8	2	8.0	2	8.3	2.1	8.2	2
21	1	8.3	1.5	8.4	2.3	9.0	2.2	9.1	2.2	8.9	2.3	-	-
	2	8.0	1.8	7.7	2.3	8.4	2.2	8.4	2.2	8.5	2.2	-	-
33	1	8.6	1.9									8.2	2
	2	7.8	1.8									7.3	2
34	1	9.8	1.9									9.2	2
	2	10.0	1.9									9.2	2

Detector Signal and Time Constant

Table 3 (Cont'd)

Flat	Flake	Initial R(kΩ)	Initial V_B(V)	Post 50°C R(kΩ)	Post 50°C V_B(V)	Post 60°C R(kΩ)	Post 60°C V_B(V)	Post 70°C R(kΩ)	Post 70°C V_B(V)	Post 70°C R(kΩ)	Post 70°C V_B(V)	Post 70°C R(kΩ)	Post 70°C V_B(B)
2	1	118	17									119.5	17.4
	2	119.5	18.3							123	18.6	123	18.8
5	1	121	17.2									123	17.5
	2	119.5	17.1							122	17.3	122	17.4
8	1	121	18.9									123	18.8
	2	121.5	18.0							123	18.2	123	18.3
9	1	123	18									132.5	17.7
	2	119.5	16.8							123	17.1	123	17.2
10	1	121.5	17.8									126	18.0
	2	118.5	17.8							121	18	122	18.0
25	1	123	17.3	122.5	17.5							128	18.7
	2	123	17.4	122.4	17.5					128	18.2	128	18.3
26	1	121.5	17.0	121.8	17.0							129	17.5
	2	122.5	17.6	122.4	17.6					126	17.7	127	17.8
28	1	121.5	16.7	122.3	16.9							127	17.2
	2	124	17.7	124.5	17.9					126	17.7	131	18.4
29	1	123	17.5	123	17.6							126	17.9
	2	123	17.2	124	17.3					130	18.1	123.5	17.3
30	1	123.5	18.2	123.7	18.4							128	17.3
	2	123.5	18.0	124.3	18.1					127	17.7	128	17.8
31	1	125	17.8									126	19.7
	2	125	18.2									125	20.1
32	1	123	19.3									128	18.1
	2	122	19.8									127	18.5

Detector Resistance and Bias Voltage
Table 4

Flat	Flake	Initial		Post 50°C		Post 60°C		Post 70°C		Post 70°C		Post 70°C	
		R(kΩ)	V_B(V)	R(kΩ)	V_B(V)	R(kΩ)	V_B(V)	R(kΩ)	V_B(V)	R(kΩ)	V_B(V)	R(kΩ)	V_B(B)
15	1	127	17.7	127	17.9							131	18.1
	2	125	18.5	126	18.3					130	18.4	131	18.5
16	1	131	18.0	131	18.0							133.5	18.1
	2	126	18.4	127	18.4					131	18.5	131	18.6
22	1	127	16.3	129	16.5							132	16.7
	2	127	16.1	128	16.4					131	16.4	132	16.6
23	1	120	16.0	124	16.2							127	16.3
	2	127	17.0	128	17.1					132	17.2	132	17.4
24	1	123	15.8	124	16.0							128	16.3
	2	124	15.9	125.5	16.0					129	16.2	129.5	16.3
14	1	125	17.3	126	17.7							–	–
	2	122.5	17.8	126.5	17.9					130	17.4	–	–
17	1	133	17.4	134	17.9							129	17.6
	2	128	17.3	128	17.5					132	18.2	132	18.2
18	1	127	17.1	129	17.2							133	17.3
	2	126	17.5	126	17.5					129	17.5	129	17.5
20	1	129	17.9	132	18.1							135	18.1
	2	128	17.8	129.6	17.9					133	18.0	134	18.1
21	1	127	17.0	128	17.0							–	–
	2	130	17.3	132	17.5					134.5	17.7	–	–
33	1	116.5	16.5									117	16.7
	2	105.5	15.2									106	15.5
34	1	124	17.2									125	17.5
	2	125	17.3									125	17.5

Detector Resistance and Bias Voltage

Table 4 (Cont'd)

Flat	Flake	Vn (μV) Initial Vn	Vno		Vn (μV) 2nd Measure 50°C			Vn (μV) 3rd Measure 60°C			Vn (μV) 4th Measure 70°C			Vn (μV) 5th Measure 70°C			Vn (μV) 6th Measure 70°C	
11B2	1	.59	.43		.66	.41		.66	.42		.64	.39		.64	.39		.65	.38
#2	2	.66	.43		.65	.42		.65	.41		.64	.40		.66	.40		.65	.39
11B5	1	.63	.41	N	.64	.41	N	.65	.41	N	.65	.39	N	.66	.39	N	.66	.40
#5	2	.63	.42	O	.61	.40	O	.63	.40	O	.63	.39	O	.66	.40	O	.64	.40
11C2	1	.60	.41		.61	.40		.63	.40		.64	.40		.64	.39		.64	.39
#8	2	.68	.44	B	.66	.40	B	.66	.42	B	.68	.40	B	.64	.40	B	.66	.41
11C3	1	.58	.43	I	.62	.40	I	.59	.40	I	.65	.40	I	.66	.39	I	.66	.40
#9	2	.60	.42	A	.61	.41	A	.62	.42	A	.64	.38	A	.66	.40	A	.70	.39
11C4	1	.56	.42	S	.58	.40	S	.62	.41	S	.62	.40	S	.62	.39	S	.63	.40
#10	2	.65	.44		.61	.41		.62	.41		.62	.39		.65	.40		.66	.40
13A1	1	.60	.43		.59	.41		.60	.42		.65	.40		.64	.39		.64	.39
#25	2	.68	.44	11V	.62	.42	10V	.62	.42	9V	.64	.40	9V	.66	.40	9V	.67	.41
13A2	1	.65	.44		.58	.44		.60	.42		.64	.40		.66	.39		.66	.39
#26	2	.60	.43	B	.64	.46	B	.58	.42	B	.62	.40	B	.66	.39	B	.66	.40
13A4	1	.58	.41	I	.58	.40	I	.62	.42	I	.67	.40	I	.66	.39	I	.70	.40
#28	2	.65	.43	A	.58	.41	A	.63	.42	A	.68	.40	A	.68	.40	A	.70	.40
13A5	1	.64	.43	S	.54	.40	S	.60	.41	S	.60	.40	S	.60	.39	S	.64	.39
#29	2	.55	.43		.55	.42		.61	.41		.62	.40		.62	.39		.64	.39
13A6	1	.60	.43		.61	.43		.64	.41		.67	.40		.70	.39		.70	.40
#30	2	.66	.44		.66	.46		.66	.41		.68	.40		.72	.39		.70	.39
13A7	1	.62	.46	R.T.													.54	.40
#31	2	.64	.48	NO													.54	.40
13C1	1	.55	.41	BIAS													.52	.39
#32	2	.54	.41														.54	.40

Noise Measurements with and without Bias

Table 5

Flat	Flake	Vn (μV) Initial Vn	Vno		Vn (μV) 2nd Measure 50°C			Vn (μV) 3rd Measure 60°C			Vn (μV) 4th Measure 70°C			Vn (μV) 5th Measure 70°C			Vn (μV) 6th Measure 70°C	
12B3	1	.63	.45		.54	.40		.66	.47		.59	.39		.64	.40		.63	.40
#15	2	.55	.40		.54	.42		.70	.48		.62	.39		.62	.39		.65	.40
12B4	1	.61	.41	N	.59	.39	N	.72	.47	N	.67	.40	N	.67	.39	N	.66	.40
#16	2	.56	.43	O	.54	.41	O	.68	.48	O	.61	.39	O	.63	.40	O	.64	.40
12C4	1	.51	.41		.54	.40		.65	.46		.60	.39		.60	.39		.65	.40
#22	2	.51	.41	B	.56	.41	B	.68	.47	B	.63	.39	B	.64	.40	B	.63	.40
12C5	1	.65	.42	I	.54	.39	I	.72	.47	I	.65	.39	I	.66	.39	I	.62	.39
#23	2	.56	.42	A	.55	.41	A	.69	.48	A	.65	.39	A	.64	.42	A	.66	.41
12C6	1	.52	.42	S	.52	.40	S	.65	.47	S	.57	.39	S	.60	.39	S	.60	.40
#24	2	.53	.41		.52	.41		.68	.48		.57	.39		.60	.42		.60	.40
12B2	1	.55	.41		.53	.39		.66	.48		.62	.40		.61	.39			
#14	2	.54	.42		.55	.42		.66	.47		.62	.40		.62	.40			
12B5	1	.58	.41		.56	.40		.70	.48		.65	.40		.67	.39		.68	.40
#17	2	.60	.43	11V	.56	.41	10V	.70	.48	9V	.65	.39	9V	.66	.40	9V	.68	.40
12B6	1	.56	.41	B	.54	.40	B	.68	.48	B	.64	.40	B	.66	.39	B	.64	.39
#18	2	.60	.43	I	.57	.41	I	.70	.48	I	.64	.40	I	.65	.40	I	.66	.41
12C2	1	.54	.44	A	.54	.40	A	.67	.46	A	.63	.40	A	.61	.39	A	.60	.39
#20	2	.55	.41	S	.56	.41	S	.68	.47	S	.66	.40	S	.64	.40	S	.68	.41
12C3	1		.42		.53	.40		.65	.46		.60	.40		.60	.39			
#21	2	.54	.41		.54	.40		.66	.47		.58	.40		.62	.40			
14B1	1	.58	.41	R.T.													.54	.39
#33	2	.52	.40	NO													.52	.38
14B2	1	.55	.42	BIAS													.54	.40
#34	2	.55	[illegible]														.54	.40

Noise Measurements with and without Bias

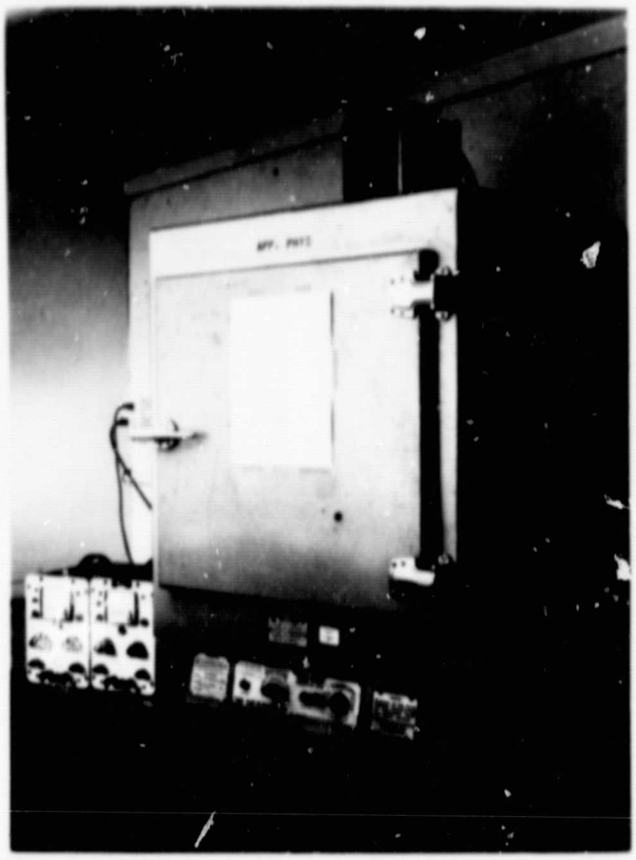
Table 5
(Cont'd)

	Initial 10^{13} (Ω)			Initial 10^{13} (Ω)			Initial 10^{13} (Ω)			Initial 10^{13} (Ω)			Initial 10^{13} (Ω)			Initial 10^{13} (Ω)		
Flat	R_{12}	R_{1c}	R_{2c}	R_{12}	R_{1c}	R_{2c}	R_{12}	R_{1c}	R_{2c}	R_{12}	R_{1c}	R_{2c}	R_{12}	R_{1c}	R_{2c}	R_{12}	R_{1c}	R_{2c}
2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
5	5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
8	1	1	1	1	1	1	.2	.1	.1	.2	.2	.1	.2	3	1	.7	.5	1
9	1	1	1	1	1	1	.3	.1	.1	.4	.4	.4	.4	.4	.4	.5	.5	1
10	.04	.5	.5	8x 10^{-4}	.08	1	3x 10^{-4}	3x 10^{-4}	.02	4x 10^{-4}	3x 10^{-4}	.03	3x 10^{-4}	4x 10^{-4}	4x 10^{-4}	.3	.2	.07
25	7	.08	.04	.1	.1	.5	.1	.1	.6	.1	.2	.5	.2	.3	.6	.08	.06	.5
26	.4	.1	.1	.1	1	.4	1	1	.5	1	1	.5	1	1	.5	1	1	1
28	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
29	.8	.6	.8	.1	.1	.05	.15	.1	.05	.2	.2	.06	.2	.2	.05	.2	.2	.1
30	1	1	1	1	1	1	1	.1	1	1	.2	1	1	.2	1	1	1	.2
31	1	1	.5													.4	.4	.3
32	1	.5	1													1	.05	1
15	1	.5	.5	.5	.5	.2	.5	.6	.1	.5	.6	.1	.5	.5	.2	.5	1	1
16	1	.03	.03	.8	.5	.5	.8	.6	.5	.8	.6	.6	.8	.6	.5	1	1	1
22	.07	.3	.07	.2	1	.03	.2	.1	.04	.2	1	.05	.2	1	.02	.5	.8	.3
23	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	.5	.5	1
24	.8	.5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	.03
14	1	.12	.25	.1	.2	.1	.1	.1	.1	.1	.2	.1	.1	.1	.1			
17	1	1	1	1	.2	.2	1	.2	.2	1	.3	.3	1	.3	.4	1	1	.4
18	.8	.8	1	.8	.7	.2	.8	.6	.3	.8	.7	.7	.8	.8	.2	1	.5	1
20	.5	.03	.03	.2	.1	1	.3	.2	1	.3	.3	1	.3	.3	1	1	1	1
21	1	.02	.02	.2	1	1	.3	1	1	.4	1	1	.4	1	1			
33	1	1	1													.5	.3	1
34	1	.4	1													.5	.6	1

R_{12} Resistance from Flake 1 to Flake 2
R_{1c} Resistance from Flake 1 to Case
R_{2c} Resistance from Flake 2 to Case

Isolation Resistance Values

Table 6

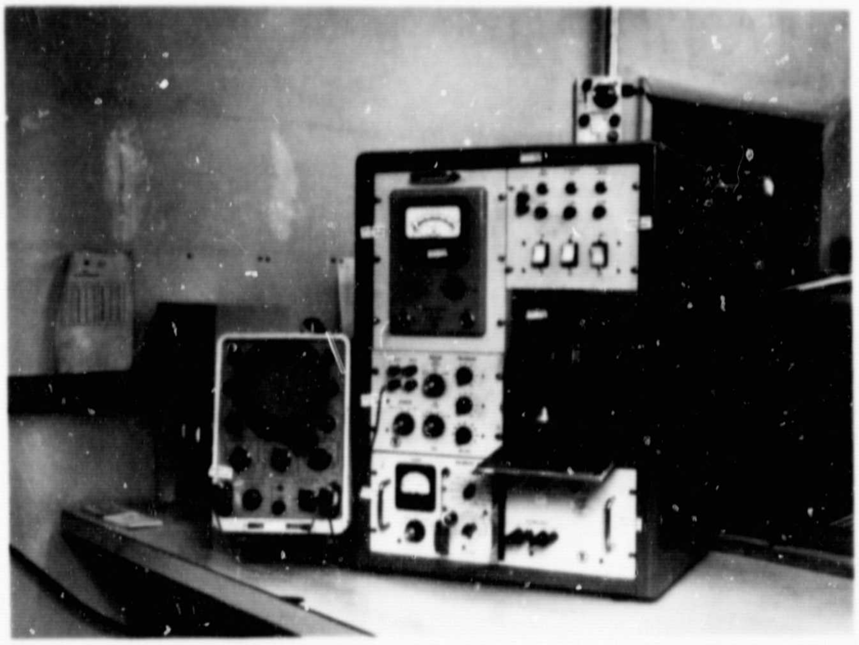


Thermal Soak Oven

Figure 5

Equipment for measuring Vp & R

Figure 6



Signal & Noise Measurement Equipment

Figure 7

temperature (R.T.) and with no applied bias. This increase in resistance has been observed also with flakes kept at R. T. for an extended period of time.

5.2.3 <u>Signal Voltage and Time Constant</u> Variations in the signal voltage and time constant were observed during the testing although not in a consistent fashion. In most cases there is a trend towards higher signal levels and longer time constants. This increase in time constant is most likely caused by the relocation of the glass which will increase the distance of the flake from the surface of the flat.

5.2.4 <u>Detector Noise</u> Since the main purpose of the contract was to establish a procedure for producing quiet bolometers, the noise measurements are analyzed in greater detail than the other measurements. Table 5 shows the raw data measured on all 24 bolometers using the equipment shown in Figure 7. To eliminate variations in the gain of the test set from test to test, the noise with bias was compared to the unbiased Johnson noise values. (Normalized.) This is shown in Table 7. Also shown in Table 7 are the ratios of the normalized noise values obtained at the various test points. For the 12% glass samples the number 1 flake of detector #21 appears to be anomalous in the initial noise value. Subsequent values are consistent with the remainder of the bolometer flakes. However, in order to ensure consistent data, this flake was not included in the mean values of N_x/N_1 (x = 1-6). Due to the accidental destruction of detectors #14 and #21, the final data points for these two were unavailable. For this reason a revised mean of the remaining 6 flakes was calculated. All mean values of noise increase are plotted in Figures 8 and 9. It can be seen that in all cases the noise increase is less than 22% and increases with rise in temperature. Only a small increase is observed for the 50°C bake with larger increases on going to 60°C and then to 70°C. Having been at 70°C for the initial 168 hrs. however, further baking at the same temperature does not result

Flat	Flake	N_1	$\frac{N_1}{N_1}$	Mean	N2	$\frac{N2}{N1}$	Mean	N3	$\frac{N3}{N1}$	Mean	N4	$\frac{N4}{N1}$	Mean	N5	$\frac{N5}{N1}$	Mean	N6	$\frac{N6}{N1}$	Mean
2	1	1.37			1.61	1.18		1.52	1.11		1.64	1.20		1.64	1.20		1.71	1.25	
	2	1.53			1.55	1.01		1.58	1.04		1.66	1.05		1.65	1.08		1.67	1.09	
5	1	1.50			1.56	1.04		1.58	1.05		1.67	1.11		1.69	1.13		1.65	1.10	
	2	1.54			1.53	0.99		1.58	1.03		1.61	1.05		1.65	1.07		1.60	1.04	
8	1	1.46	1.0	1.0	1.53	1.05	1.06	1.58	1.08	1.06	1.60	1.10	1.13	1.64	1.12	1.13	1.64	1.12	1.14
	2	1.55			1.65	1.06		1.57	1.01		1.70	1.10		1.60	1.03		1.61	1.04	
9	1	1.35			1.55	1.15		1.48	1.09		1.63	1.20		1.69	1.25		1.65	1.22	
	2	1.43			1.49	1.04		1.48	1.03		1.79	1.25		1.65	1.15		1.79	1.25	
10	1	1.33			1.45	1.09		1.51	1.14		1.55	1.16		1.59	1.19		1.58	1.19	
	2	1.48			1.49	1.01		1.51	1.02		1.59	1.07		1.63	1.10		1.65	1.11	
25	1	1.40			1.44	1.03		1.43	1.02		1.63	1.16		1.64	1.17		1.64	1.17	
	2	1.55			1.48	0.95		1.48	0.95		1.60	1.03		1.65	1.06		1.63	1.05	
26	1	1.40			1.32	0.94		1.42	1.02		1.60	1.14		1.69	1.21		1.69	1.21	
	2	1.48			1.39	0.94		1.38	0.93		1.55	1.05		1.69	1.14		1.65	1.11	
28	1	1.41	1.0	1.0	1.45	1.03	0.98	1.48	1.05	1.03	1.68	1.19	1.12	1.69	1.20	1.17	1.75	1.24	1.17
	2	1.51			1.41	0.94		1.50	0.99		1.70	1.13		1.70	1.13		1.75	1.16	
29	1	1.49			1.35	0.91		1.46	0.98		1.50	1.01		1.54	1.03		1.64	1.10	
	2	1.30			1.31	1.01		1.48	1.14		1.55	1.19		1.59	1.22		1.64	1.26	
30	1	1.40			1.42	1.01		1.56	1.11		1.68	1.19		1.79	1.28		1.75	1.25	
	2	1.50			1.43	0.96		1.61	1.07		1.70	1.13		1.84	1.23		1.79	1.20	

$$N = \left[\frac{\text{Noise measured with 0.6Vp bias}}{\text{Johnson Noise}}\right]$$

N1	Initial value of N
N2	After 168 hrs @ 50°C
N3	After 168 hrs @ 60°C
N4	After 168 hrs @ 70°C
N5	After 168 hrs @ 70°C
N6	After 168 hrs @ 70°C

Noise Values (Normalized)

Table 7

Flat	Flake	N1	N1/N1	Mean	N2	N2/N1	Mean	N3	N3/N1	Mean	N4	N4/N1	Mean	N5	N5/N1	Mean	N6	N6/N1	Mean
15	1	1.40			1.35	0.96		1.40	1.00		1.57	1.08		1.60	1.14		1.58	1.13	
	2	1.38			1.29	0.93		1.46	1.06		1.59	1.15		1.59	1.15		1.63	1.18	
16	1	1.49			1.57	1.02		1.53	1.03		1.68	1.12		1.72	1.15		1.65	1.11	
	2	1.30			1.32	1.01		1.42	1.09		1.56	1.20		1.58	1.21		1.60	1.23	
22	1	1.24	1.0	1.0	1.35	1.09	1.004	1.41	1.14	1.077	1.54	1.24	1.13	1.54	1.24	1.18	1.63	1.31	1.18
	2	1.24			1.37	1.10		1.42	1.17		1.61	1.30		1.60	1.29		1.58	1.27	
23	1	1.55			1.38	0.89		1.53	0.98		1.67	1.07		1.69	1.09		1.59	1.03	
	2	1.33			1.34	1.01		1.43	1.08		1.67	1.25		1.52	1.15		1.61	1.21	
24	1	1.24			1.30	1.05		1.38	1.12		1.46	1.18		1.54	1.24		1.50	1.21	
	2	1.29			1.27	0.98		1.41	1.10		1.46	1.13		1.43	1.11		1.50	1.16	
14	1	1.31			1.36	1.04		1.38	1.05		1.55	1.18		1.56	1.19		-	-	
	2	1.29			1.31	1.02		1.40	1.09		1.55	1.20		1.55	1.20		-	-	
17	1	1.41			1.40	0.99		1.46	1.03		1.63	1.15		1.72	1.22		1.70	1.21	
	2	1.40			1.37	0.98		1.46	1.04		1.67	1.19		1.65	1.18		1.70	1.21	
18	1	1.37	1.0	1.0	1.35	0.99	1.017	1.42	1.03	1.067	1.60	1.17	1.13	1.69	1.24	1.20	1.04	1.20	1.21
	2	1.40			1.39	0.99		1.41	1.04		1.60	1.14		1.63	1.16		1.61	1.15	
20	1	1.23			1.35	1.10		1.46	1.18		1.60	1.28		1.56	1.27		1.54	1.23	
	2	1.34			1.37	1.02		1.45	1.08		1.65	1.23		1.60	1.19		1.66	1.24	
21	1	1.79			1.33	0.74		1.41	0.79		1.50	0.84		1.54	0.86		-	-	
	2	1.32			1.35	1.02		1.40	1.06		1.45	1.10		1.55	1.17		-	-	

$$N = \left[\frac{\text{Noise measured with 0.6Vp bias}}{\text{Johnson Noise}}\right]$$

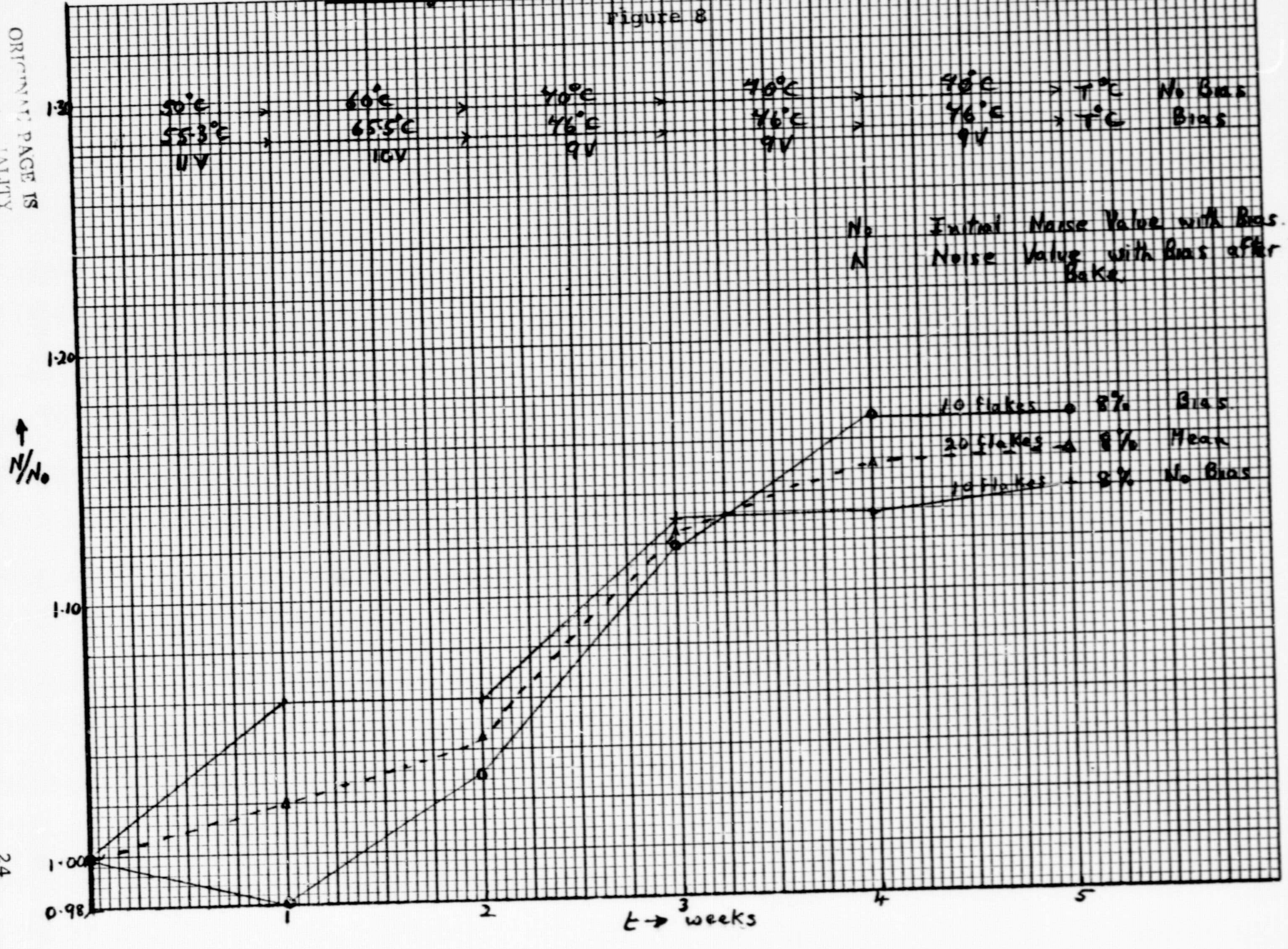
N1 Initial value of N
N2 After 168 hrs @ 50°C
N3 After 168 hrs @ 60°C
N4 After 168 hrs @ 70°C
N5 After 168 hrs @ 70°C
N6 After 168 hrs @ 70°C

Noise Values (Normalized)

Table 7
(Cont'd)

Average Noise Increase N/N_o vs Temperature Bake

Figure 8



Average Noise Increase N/No vs Temperature Bake

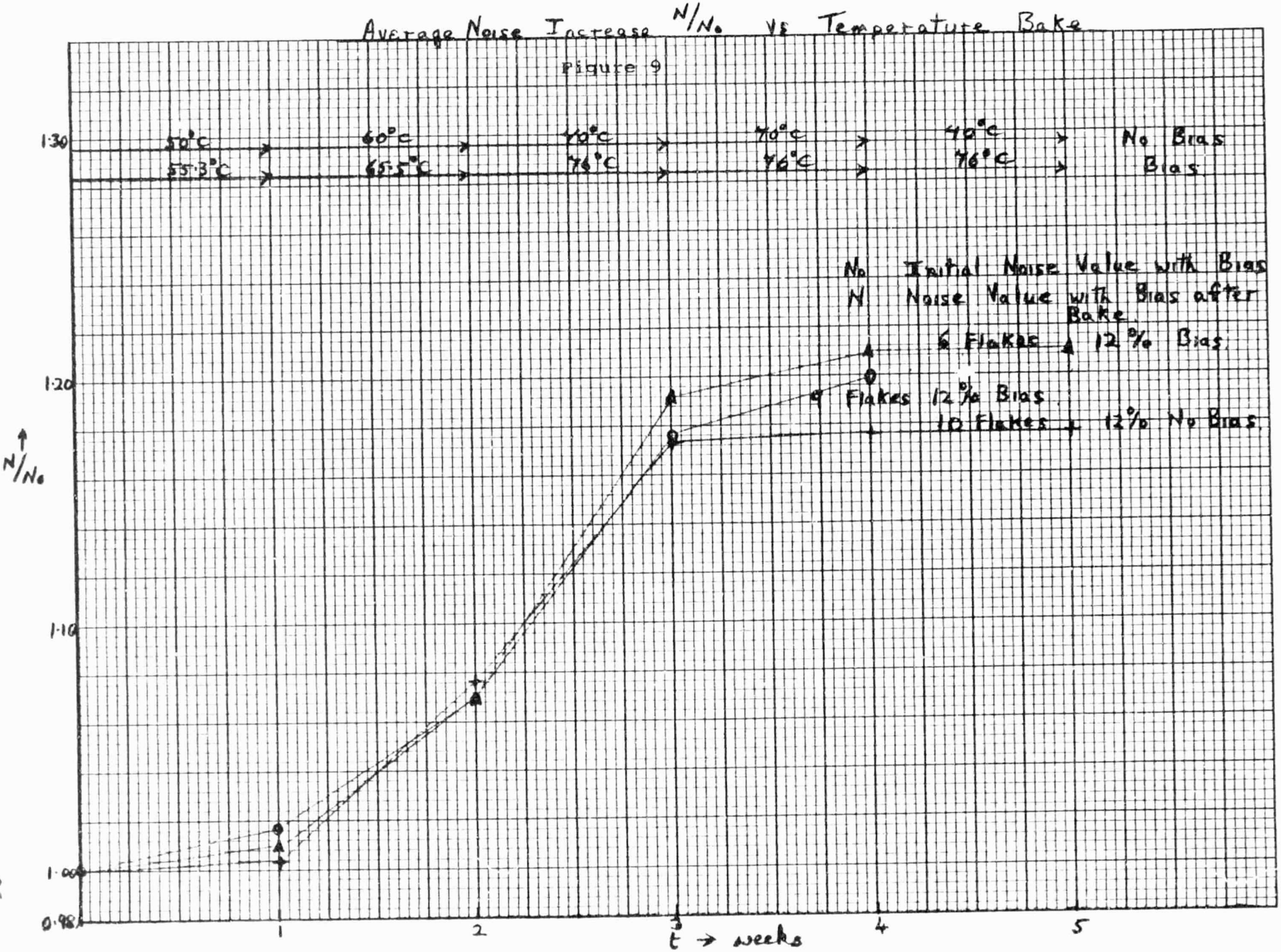


Figure 9

in such a large increase. Note that in Figure 9 the results for biased flakes are closely similar regardless of the sample size (6 or 9). The mean values of both the Johnson Noise and the noise with $0.6V_p$ applied are plotted for each group of detectors in Figures 10-11.

6.0 OBSERVATIONS - DISCUSSIONS

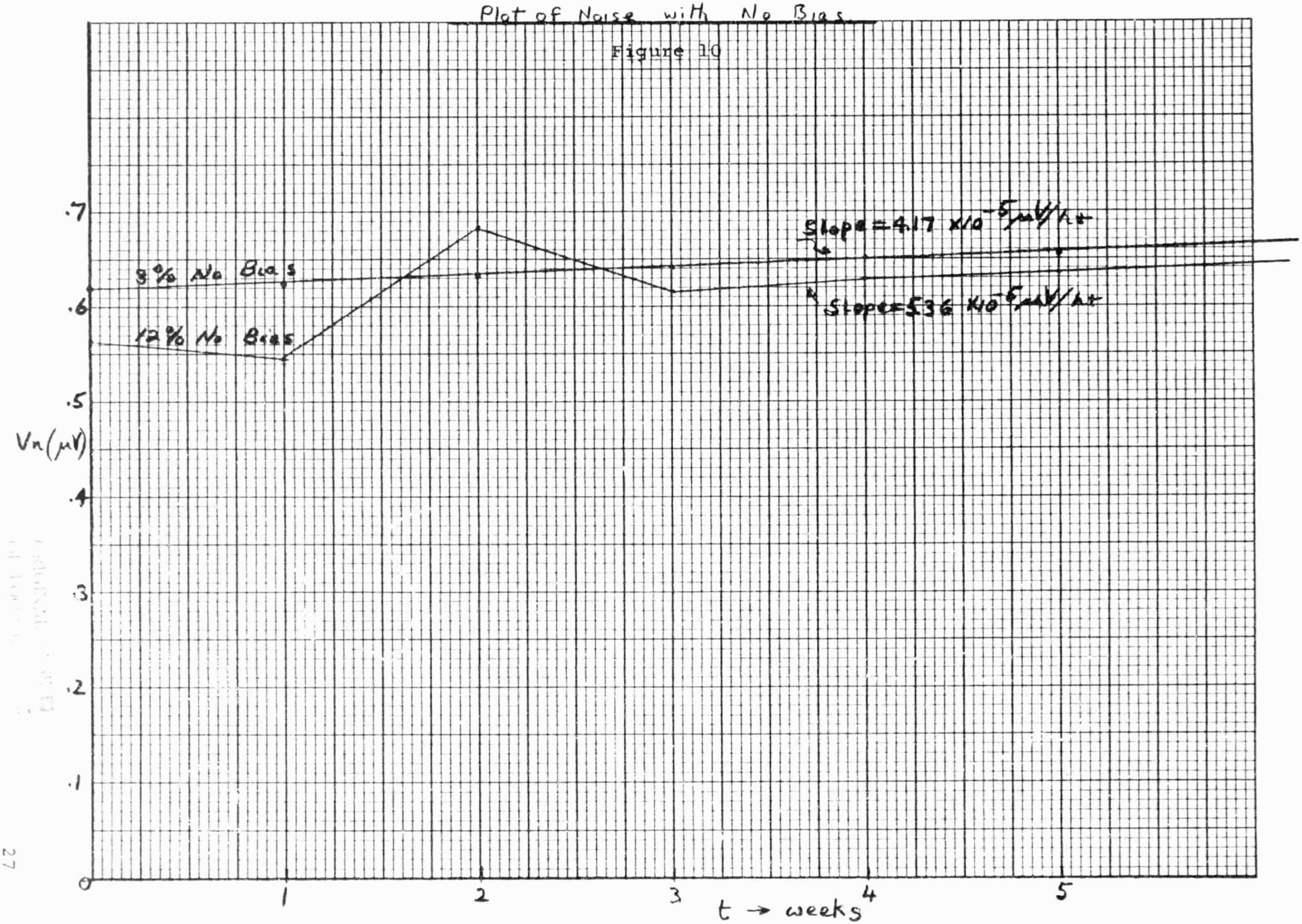
6.1 Surface Features The evaporations were all carried out using the same source for each composition. It was noted that the surface of the glass contained a number of "balls." These were analyzed and found to contain selenium and oxygen with little or no arsenic. A larger number occurred with the 12% As/Se glass than with the 8% As/Se glass. The source of these features may be selenium oxide in the original glass melt which are evaporated sporadically in the form of small pellets. None of the bolometers were immersed over or immediately adjacent to a selenium/oxygen ball.

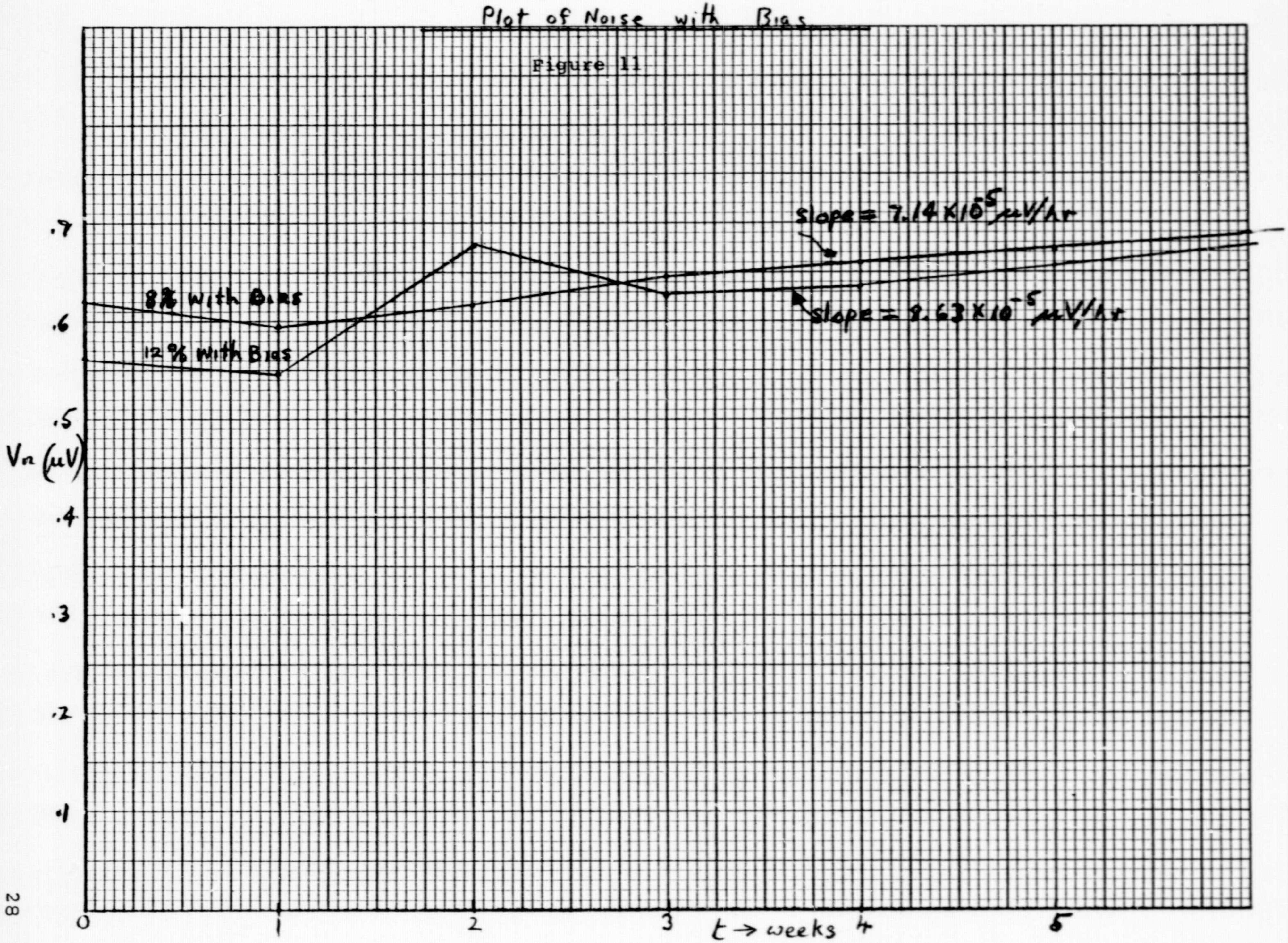
6.2 Bulk Changes After deposition, the glass is quite transparent and the aluminum defining the mask can clearly be seen. (Figure 3.) After immersion, and in the case of the top coat after temperature soaking, the layer becomes opaque to varying degrees. This may be seen in the photographs of the flakes. (Figures 12-21) Transmittance curves of As/Se glass layers on both Ge flats and Corning glass failed to show any change in transmittance upon heating the samples. A similar experiment performed at NASA Greenbelt using a flat with the "old technique" glass did show an increase in scattering. This may be the most sensitive way of detecting a change in the "glassy" properties of the layers.

6.3 Detector Losses A total of 34 flats were used to complete the 24 deliverable bolometers. Of the 10 lost during manufacture, 4 were rejected for noise. In all cases the noisy flake had a low isolation resistance ($10^6 \Omega$) between the flake and the Ge flat. The remaining 6 (12 flakes) were biassed at too high a voltage during the initial 50°C bake cycle and at least one of the two flakes was destroyed.

Plot of Noise with No Bias

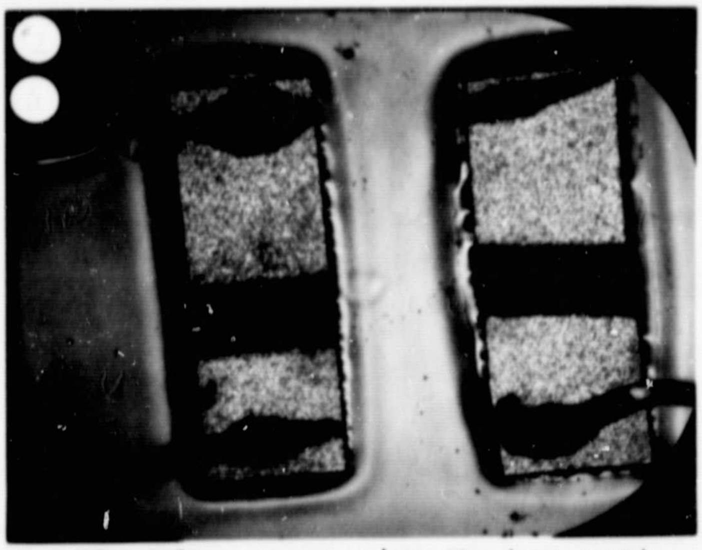
Figure 10



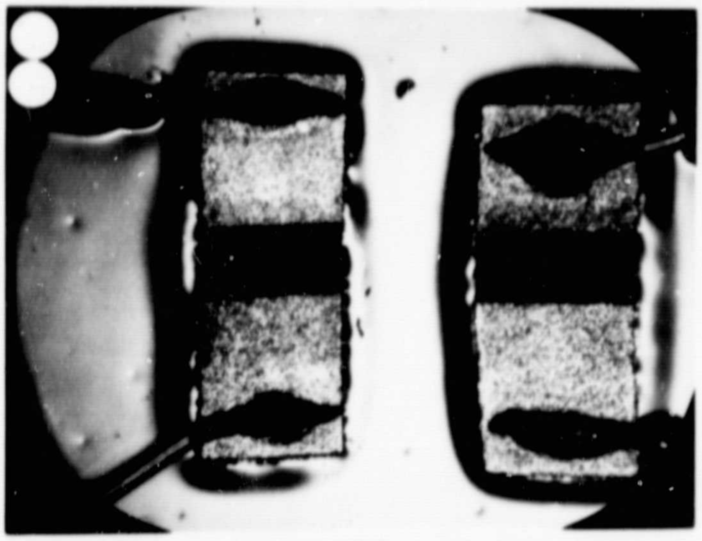


Plot of Noise with Bias

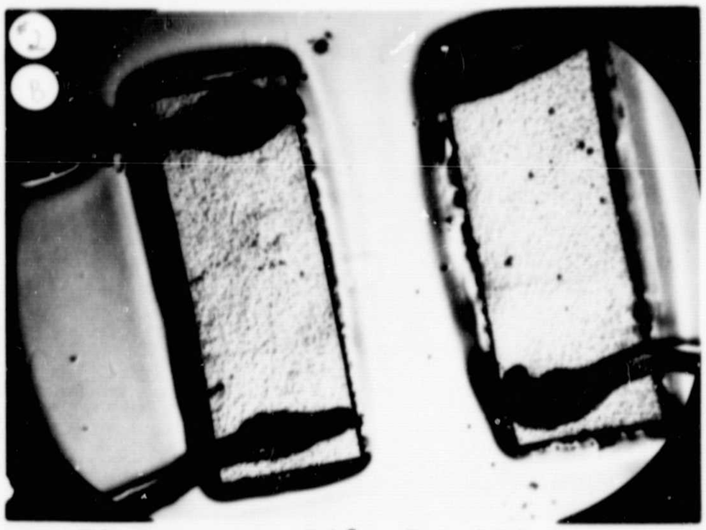
Figure 11



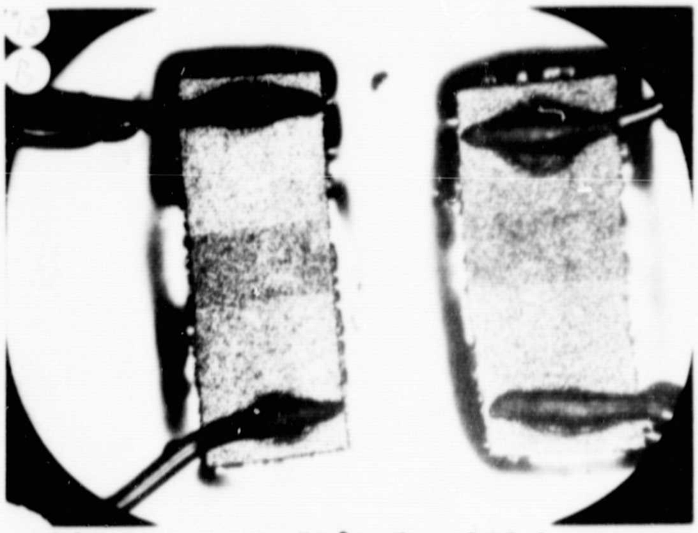
#2 After Immersion No top coat
8% As/Se X100
Figure 12



#15 After Immersion No top coat
12% As/Se X100
Fiqure 15

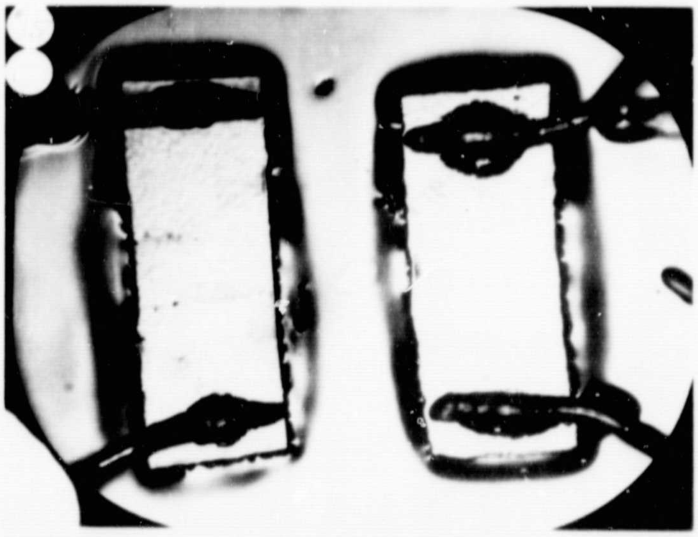


#2 Top coat 50°C for 168 hrs.
Figure 13 X100



#15 Top coat 50°C for 168 hrs.
Figure 16 X100

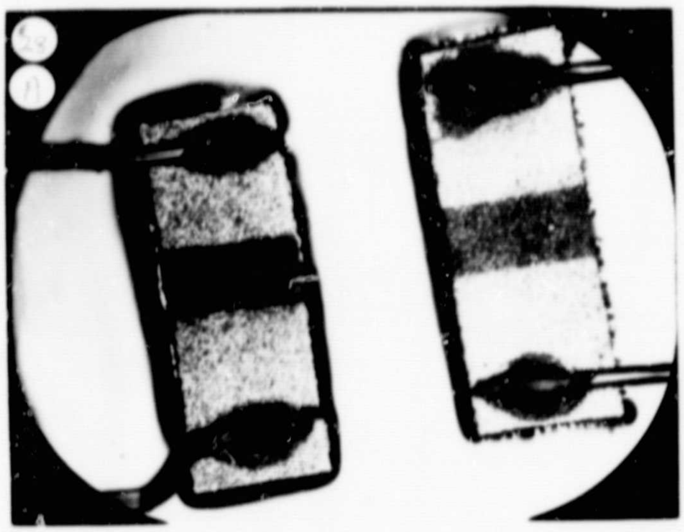
#3 After 500 hrs @ 70°C
Figure 14 X100



#15 After 500 hrs @ 70°C
Figure 17 X100

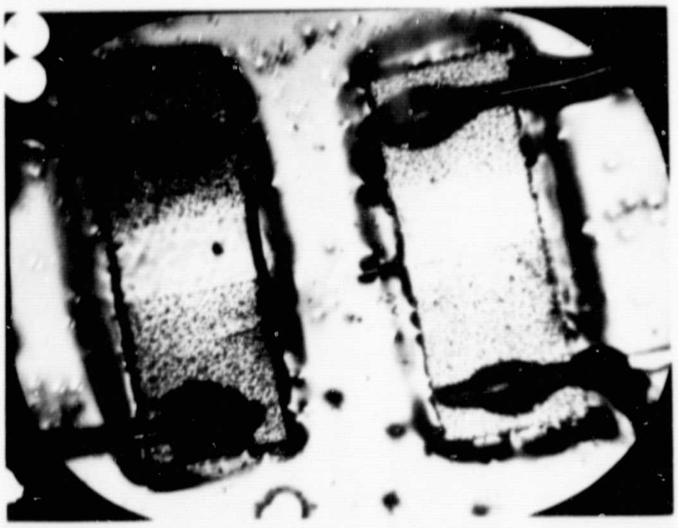
#17. Ater Immersion. No Top Coat.
12% As/Se x100

Figure 18



#28 After Immersion. No Top Coat.
8% As/Se x100

Figure 20



#17 After 500 hrs. at 70°C
12% As/Se x100

Figure 19

#28 After 500 hrs at 70°C
8% As/Se x100

Figure 21

6.4 S.E.M. Analysis All flakes were observed on the S.E.M. after immersion and prior to putting on the top coat. A number of typical photographs were taken of the flakes at both low and high powers. (Figures 22-27.) Nothing was observed that would suggest a failure mechanism during processing and testing. This is in line with the observed electrical results.

6.5 Glass Shrinkage During immersion the diameter of the glass layer decreases. It has also been noted that holes appear in the glass layer such that the Ge flat may be seen. These processes may be in part due to poor adhesion between the glass and substrate. It is suggested that the Se/O balls also contribute to the occurrence of holes (Figure 20.) These balls are readily removable and do leave craters in the glass. However, the electrical results indicated that at least within the scope of the present experiment the "balls" do not induce noise in the bolometers.

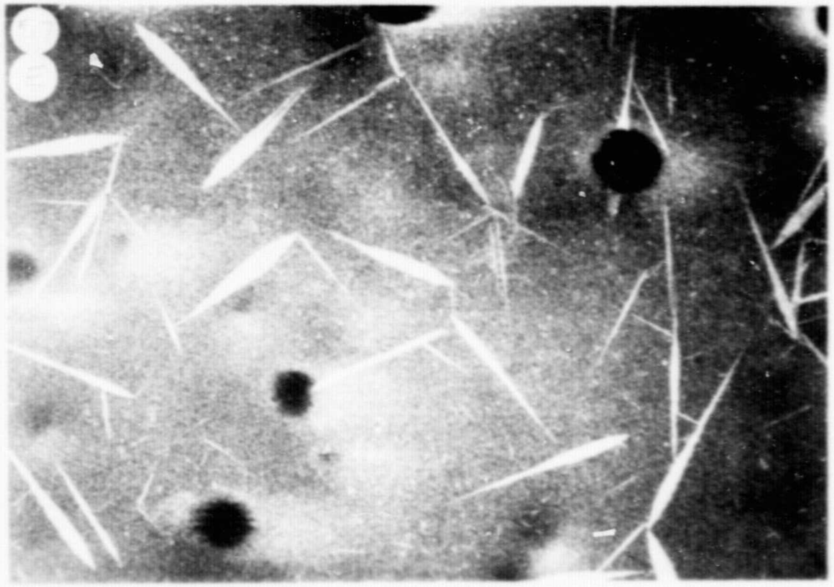
7.0 CONCLUSIONS

1) The evaporation schedule is controllable and has resulted in the consistent evaporation of As/Se glass layers.

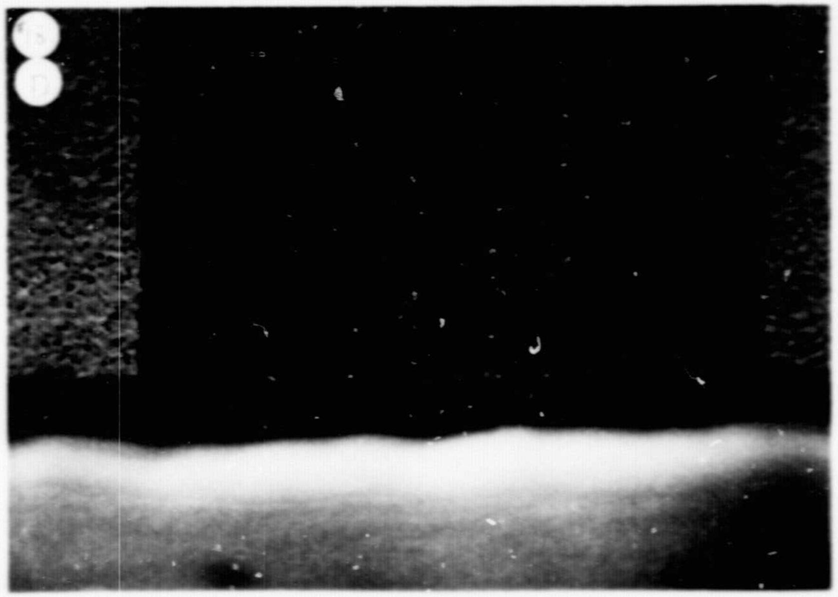
2) The 8% As/Se glass appears to be preferable to the 12% As/Se glass on two counts· namely that (a) the 8% glass has fewer mounds, balls, etc. on the surface, and (b) the noise increase is less (15% compared to 19.5%, Figures 8-9).

3) Some increase in device resistance is normal for the type of firing cycle used to produce the flakes. The change is possible due to the subsequent diffusion of oxygen or other impurity into the flake after firing.

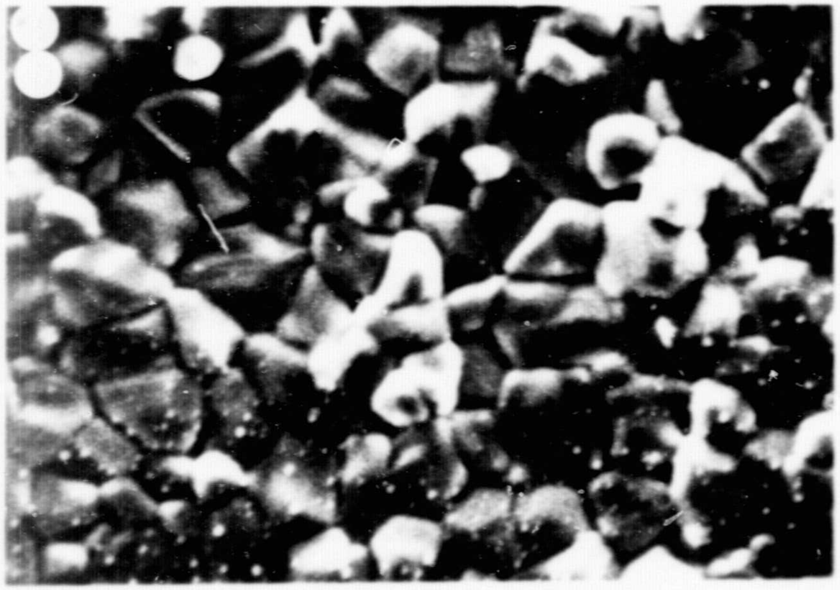
4) The small increase in noise from measurement to measurement makes it difficult to determine precisely whether the noise is still increasing slowly or whether it has stopped increasing totally. Presentation of the data in the form of "normalized" noise indicates the latter whereas the plot of directly measured noise shows some increase (Figures 8-11). However, no failures have occurred, where a failure is defined as an excessive increase in noise. A noise voltage of greater than 1.5μV



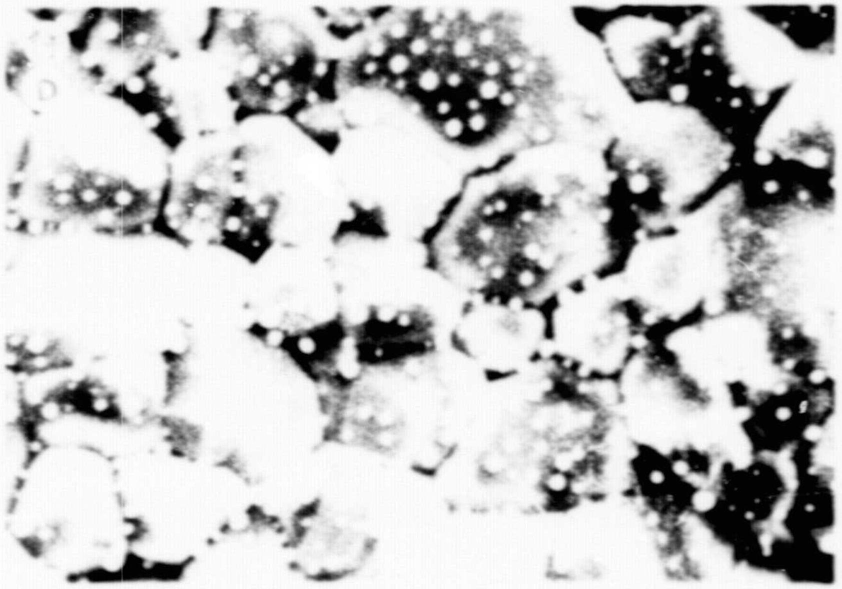
#17 Atypical As/Se Glass Surface
Figure 22 S.E.M. X575



#18 Immersed flake No top coat
Figure 23 S.E.M. X820



#15 Typical Flake showing crystal grains
Figure 24 S.E.M. X5950



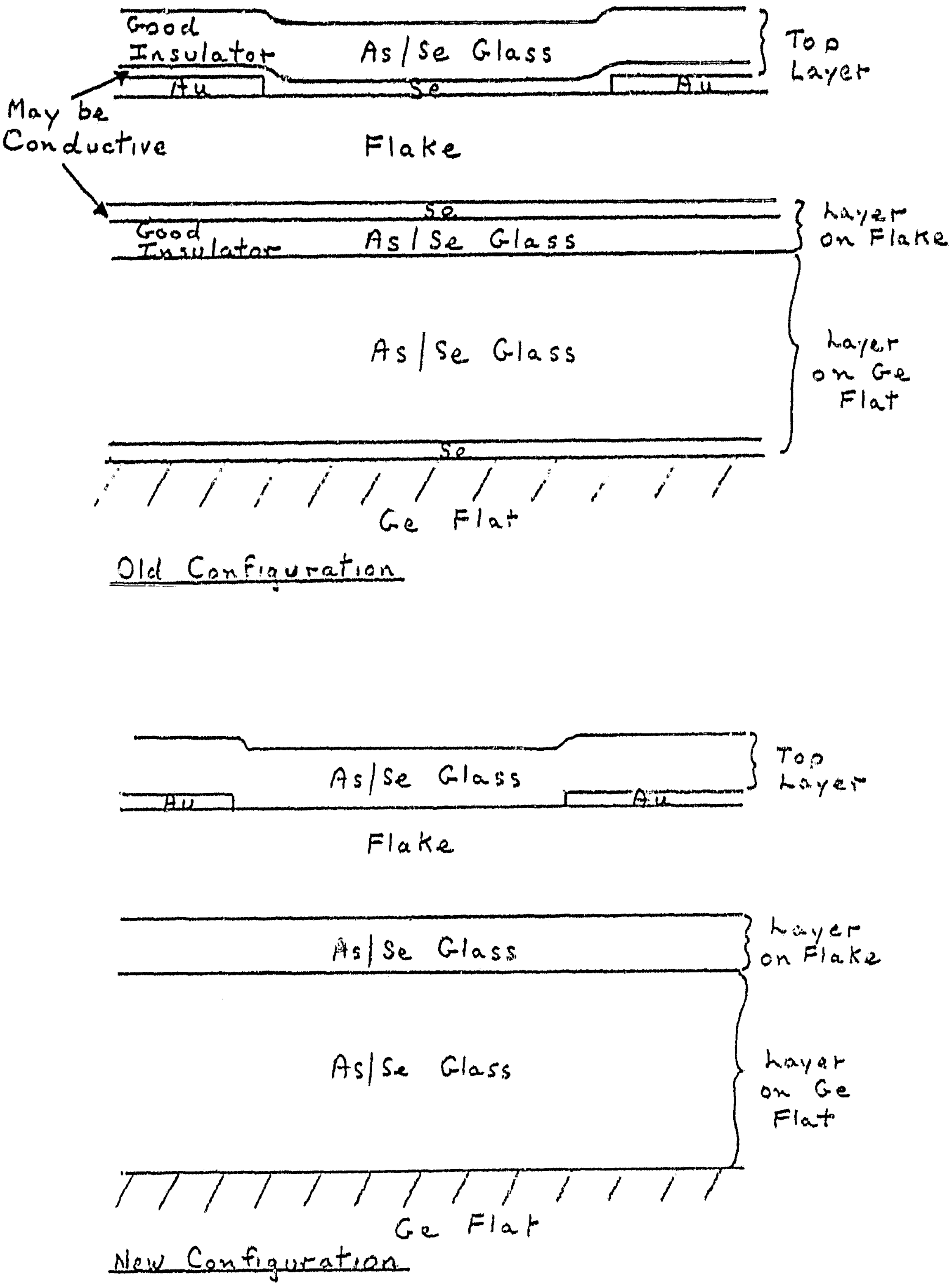
#9 Typical flake
Figure 25 S.E.M. X 15,000

would be considered excessive. These values of noise increase are less for the 8% than the 12% and the rate of increase of biassed flakes greater than that for unbiassed flakes. Since the temperature of bake does not appear to change the rate of noise increase, it seems unlikely that it is the higher flake temperature caused by bias which is responsible for the higher rate of noise increase for the biassed flakes. Either a field or current effect is responsible. Further bake cycles must be implemented to obtain more data on the flakes in order to be more definitive as to the trend in noise increase. Comparison of these data with previously measured As/Se glass immersed detectors shows that the rate of noise increase is at least 10-15 times less than those obtained using the unshuttered glass deposition technique.

5) The isolation resistance values do not have a good correlation with the noise of the flake. Only in the case of a "short" of $10^6\Omega$ do we see a large noise increase. This is not surprising either in the light of previous observations on the lack of correlation between noise and isolation resistance or on the basis of the proposed model. The formation of a conducting layer of selenium in contact with the flake would not show on the isolation resistance unless the total thickness of glass became crystalline. Since all deposited layers (unless extremely thin) contain arsenic in part of the glass between the flake and substrate we would not expect this to happen. Figure 28 shows the ideal (non mixed) configuration for previously unshuttered layers and the new configuration. The latter avoids the problem of the original deposition schedule by ensuring that noisy shunt resistance cannot be formed.

8.0 RECOMMENDATIONS

The project has demonstrated that the use of a shuttered source has enabled immersed detectors to be produced which have not shown an excessive increase in noise. This has been attributed to the film uniformity which now always contains an appreciable percentage of arsenic. To verify the validity of this assumption and to reaffirm the device stability, the following tasks are recommended:



Old and New Flake Configurations

Figure 28

1) Composition profiles of deposited layers (partial deliverables).

2) Continued thermal soaking at 50°C for up to 2,000 hrs. with and without bias for the complete bolometers.

3) Comparison of transmittance data before and after temperature soaking on both "old" (unshuttered) and "new" (shuttered) layers.