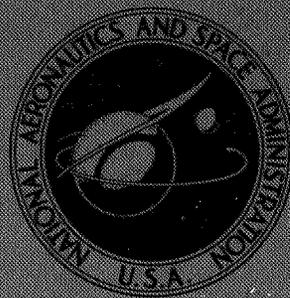


NASA CONTRACTOR
REPORT



NASA CR-2487

NASA CR-2487

EVALUATION OF WET TANTALUM CAPACITORS
AFTER EXPOSURE TO EXTENDED PERIODS
OF RIPPLE CURRENT

Volume II

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1.0 INTRODUCTION

A wet tantalum capacitor test program was initiated by the Viking Project Office (VPO) - Langley Research Center (LRC) in 1973 to obtain additional information on characteristics of the wet tantalum capacitor (slug and foil types) in selected Viking ripple current applications. This test program was conducted jointly between Flight Instrument Division (FID) at LRC, and Martin Marietta Corporation (MMC). This report is Volume II of a two-volume set which reports the results of all testing. Volume II refers specifically to the silver migration analysis effort.

1.1 Background

In late 1972 NASA MSFC organized a team to investigate wet slug capacitor failures experienced in the Apollo Telescope Mount (ATM) system tests. Representatives from VPO and MMC were invited to participate in this investigation. Due to the similarity of ATM circuit applications in which the wet slug capacitor failed and Viking wet slug circuit applications, several concerns were identified relative to the wet slug capacitor in Viking applications. The concerns related specifically to the part were:

A. The electrical performance characteristics of the Viking wet slug capacitor over extended periods of time in a ripple current application. This data is presented in Volume I.

B. The possibility of a "memory" effect within a wet slug capacitor, i.e., a capacitor subjected to long-term operating conditions at a low dc bias level and subsequently failing to perform correctly at a dc bias level approaching or equal to the rated level due to the extended low level operating level. This data is presented in Volume I.

C. The internal silver migration characteristics within the capacitor as a function of ripple current applications. This analysis is the subject of this volume.

The decision to acquire an insight into these concerns through additional testing of the wet tantalum capacitor was motivated by the following key factors:

- o Significant usage of the wet tantalum slug capacitor in the Viking Lander design: 118 circuit applications, 77 of these under some magnitude of ripple current.
- o Very limited data and considerable theory and opinion in the aerospace industry on the application of wet tantalum capacitors (slug and foil) under extended time periods of ripple current.

1.2 Test Objective

The specific objective in the Volume II MMC portion of the wet tantalum capacitor test program is to conduct an internal analysis of wet slug capacitors selected from the test specimens utilized in the electrical performance testing for an evaluation of silver migration characteristics.

1.3 Summary

The major observations from the internal silver analysis are as follows:

A. All wet tantalum capacitors contain some silver in the electrolyte and in the porous anode.

B. The silver present in the capacitor in general exceeds that predicted by the solubility constant of the principal silver salt, silver sulfate Ag_2SO_4 .

C. A constant positive dc bias on the anode will reduce the silver levels in the electrolyte.

D. The Viking ripple levels did not generally raise the silver content. While some large increases were noted, they do not correlate well with the ripple current exposure.

E. Capacitors with silver on the anode were indistinguishable electrically from other units.

2.0 SAMPLE DESCRIPTION

Samples were as follows:

A. The main samples for this analysis primarily were General Electric MIL-C-39006 CLR65 style capacitors. These are non-solid electrolyte units with a porous tantalum anode procured in two sizes, Case II and III. There were 69 units in this group. Vibration failures were experienced on some of the test samples as noted in section 2.1, Volume I. During the analysis of these failures, seven case size I capacitors were analyzed and their data is also included.

B. Two bathtub capacitors from MSC. One a failed unit and the other an unused stock unit. These units contained 16 capacitors, each in a parallel bank. These were approximately size II units.

C. Ten ST90D-38A Sprague units from a long-term reliability test Titan Inverter, plus five identical lot date units from MMC stock. The application in this case involved a substantial ripple level.

3.0 INTERNAL SILVER ANALYSIS

3.1 Description of Test Article

The capacitors utilized in this testing program were General Electric wet slug tantalum capacitors in case sizes II and III. These were procured to the CSV39006 Drawing.

The heart of this component is a porous sintered tantalum anode. When a very thin insulating film is grown over the high surface area of the anode and contact is made using a conductive electrolyte, a capacitor with very high capacitance per unit volume/weight is possible. Anodes are produced by pressing a mixture of powdered high purity tantalum powder and an organic binder into a cylindrical shape and inserting a piece of tantalum wire into one end. The units are then prefired to drive off the binding agent in a vacuum oven. Sintering is done in a special furnace to fuse at their contact points all the grains of tantalum. This produces a porous slug with high surface area.

Numerous variables determine the degree and size of porosity achieved and desired. To achieve a given capacitance, voltage and anode size, the anodes are made using different grades of tantalum powder with differing particle size distributions; different sintering temperatures and time durations to vary the degree of sintering achieved, and finally, differing anode weights and press densities, and hence, size to vary the surface area achieved.

Sintered anodes are placed in an electrolyte bath and voltage slowly applied, keeping below a certain voltage level to oxidize the tantalum into tantalum pentoxide (Ta_2O_5). The voltage is increased to the desired level and current allowed to flow until the Ta_2O_5 has formed to the point where insulation resistance on each unit is in the megohms and resultant leakage currents are in the order of pico amps. The resultant Ta_2O_5 film is quite thin, resulting in an optical thin film interference color which is characteristic of the forming voltage and temperature used. As the film is formed electrochemically it is very uniform in thickness and hence color providing a useful indicator of the surface conditions. Where the film is thin, a different color appears and provides an easy method to detect the location of "breakdown" or damage sites. The actual leakage on each sample varies constantly, due to an effect known as "sintillation", in which the film is dynamically reforming in minute locations. Over a long period of time the film becomes more stable and reformation sites less numerous, leading to a slow but steady reduction in the average leakage current.

The form of the grown film is amorphous. A crystalline form is possible but not desired, due to higher leakage currents inherent to the material. Crystalline areas are colorless due to the optical interference of the grain boundaries, and the phenomenon of crystalline oxide growth is known as "grey-out". Oxide formation conditions of current density, electrolyte composition and temperature, are carefully controlled to prevent this crystalline growth.

Once the anodes are formed, a lot sample is placed in a test cell and the capacitance measured. Where capacitances are above the desired value, further oxide growth is accomplished at a higher voltage as a thicker film gives a lower capacitance due to $C = \frac{\epsilon A}{D}$. The ultimate properties of the capacitor are due to the quality of the film. Consequently, materials of the highest purity are used and careful control of cleanliness and process are essential.

Finished, formed anodes are then cleaned of the forming electrolyte and vacuum backfilled with a 30-40% solution by weight of sulfuric acid. Anodes are assembled into the case with a vibration containment Teflon spider on the base and a gelled 30-40% solution of sulfuric acid almost filling the remaining volume.

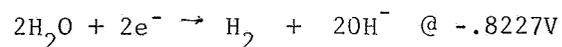
The case itself is made of fine Ag, approximately .020 inch thick. To increase the cathode surface area the interior of the case is etched and platinum black is electrochemically deposited on the inside.

Final assembly involves installation of a seal which prevents leakage of electrolyte from the anode area and from the case in general.

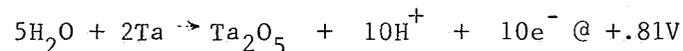
3.2 Internal Chemistry

The wet tantalum capacitor is basically a simple electrochemical cell. With a positive potential on the anode, electrons enter the electrolyte and form negative ions which migrate to the Ta₂O₅ surface charging that side of the capacitor. Where the conductivity of the Ta₂O₅ is high the negative ion gives up its electron and the oxygen present is used to oxidize tantalum to tantalum pentoxide which then further insulates the surface. In this manner the capacitor chemically builds up an insulative layer which reduces the leakage at a given voltage to a very low level. In an ideal system using a platinum cathode and pure water as an electrolyte, the two predominate reactions would be:

At the Cathode



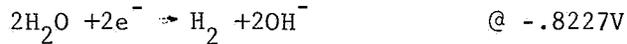
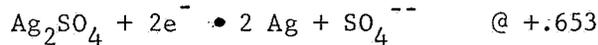
At the Anode



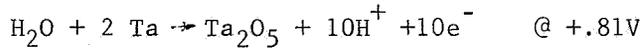
In an actual capacitor with a silver case and a 30% H₂SO₄ electrolyte the system becomes more complex.

The sulfuric acid acts to ensure that plenty of ions are present in the form of OH⁻, H⁺, and SO₄²⁻, and very little H₂O exists. During a charging cycle with the anode positive the following reactions are most probable:

At the Cathode



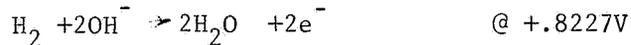
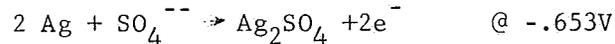
At the Anode



Those reactions involving the highest positive potential are the most probable so at the case the first reaction to occur would be $\text{Ag} + \text{e}^- \rightarrow \text{Ag}$, the next most likely being $\text{Ag}_2\text{SO}_4 + 2\text{e}^- \rightarrow 2\text{Ag} + \text{SO}_4^{2-}$. $2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$ can occur but at a much less likely level. High charging currents would create a voltage crowding and tend to promote the H₂ generation, however.

At the anode the tantalum reaction would predominate until no tantalum was available, at which point the net electrical leakage of the film would force the production of O₂.

For the case (cathode) silver reactions to occur there must be silver present in the electrolyte. An explanation for this silver presence lies in the discharge properties of the capacitor. During discharge the negative ions present at the tantalum oxide film must give up their electrons to the cathode. Here three possible reactions are:

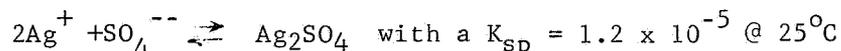


In both cases the silver reaction is to inject silver ions into the electrolyte. The hydrogen reaction is the most probable, but requires the presence of hydrogen. When insufficient hydrogen is available the silver reactions would be the next most probable ones. Again a rapid discharge would cause a voltage crowding effect and further promote silver injection into the electrolyte.

This analysis then indicates that a dynamic silver exchange is occurring during charge/discharge cycles, such as in a ripple condition, and that high charging and discharging currents, such as square wave ripple, promote the silver reactions.

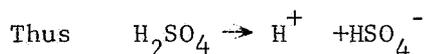
There is no guarantee that from one cycle to the next the silver reactions would balance. It is quite probable that some silver ions diffuse away from the case and become unavailable for immediate replating thus forming the basis for the silver analyzed in this analysis.

As noted in the charging analysis, both Ag^+ and Ag_2SO_4 can be formed. In the presence of sulfate ion these two materials interact to reduce the silver ion content by



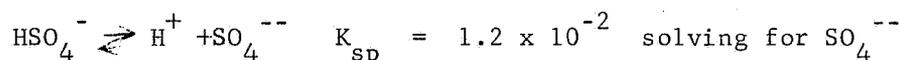
The maximum Ag^+ content of a 30% H_2SO_4 solution can be calculated as follows:

30% H_2SO_4 from a 3.7 molar solution



$$\boxed{[\text{H}^+]} = 3.7\text{M}$$

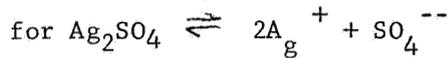
$$\boxed{[\text{HSO}_4^-]} = 3.7\text{M}$$



$$\frac{\boxed{[\text{H}^+]} \boxed{[\text{SO}_4^{--}]}}{\boxed{[\text{HSO}_4^-]}} = 1.2 \times 10^{-2}$$

$$\frac{\boxed{3.7\text{M}} \boxed{[\text{SO}_4^{--}]}}{\boxed{3.7\text{M}}} = 1.2 \times 10^{-7}$$

Therefore $\boxed{[\text{SO}_4^{--}]} = 1.2 \times 10^{-2} \text{ M in a } 30\% \text{ H}_2\text{SO}_4 \text{ solution}$



$$\left[\text{Ag}^+ \right]^2 \left[\text{SO}_4^{--} \right] = 1.2 \times 10^{-5}$$

$$\left[\text{Ag}^+ \right]^2 = \frac{1.2 \times 10^{-5}}{1.2 \times 10^{-2}} = 10^{-3}$$

$$\left[\text{Ag}^+ \right] = .032\text{M}$$

The atomic weight of silver is 108 gm/M so 3.5 gms Ag^+ /L is the maximum allowable.

For a GT3 capacitor of fill volume .033cc the maximum allowable silver ion content would be 114 μg . Any silver content above this value would require that silver be present in a compound form such as Ag_2SO_4 . Large crystals of Ag_2SO_4 have been observed inside tantalum wet slug capacitors. Other forms, such as Ag_2O , are possible, but were not observed in this testing.

Some implications can be drawn from the above analysis. First, silver would be expected in the electrolyte in either sulfate or ionic forms. Since some silver is diffusing away at an undetermined rate would anything limit the amount of silver present? In a properly biased condition a leakage current exists due to the conductivity of the Ta_2O_5 film and scintillation effects. This would act to create the equivalent of a constant charging condition which would promote the reduction of silver at the case and in a non-ripple condition would be expected to decrease the silver concentrations over a long period of time. A comparison of the -10 old and -8 old capacitors versus the new parts displays precisely this condition with the units having 2000 hours of burn-in being almost an order of magnitude lower in silver concentrations.

Some form of balance might be expected between leakage current, ripple current, and the silver concentrations. An attempt was made in the discussion section of this report to look for this, but no correlations were found.

Secondly, since hydrogen and oxygen are being produced by the reactions involved, what prevents the overpressuring of the case after extended periods of operation? An observed fact is that after 3000 hours of operation, capacitors in this testing program did not exhibit any evidence of internal pressure. It is believed that recombination of the oxygen and hydrogen is preventing this. In the case of a broken down dielectric, where high leakage currents are being exhibited, the amount of hydrogen and oxygen being generated may be sufficient that the release of energy occurring upon recombination is the factor which causes tantalum wet slug capacitors to violently explode.

A third implication from the analysis is that no way is apparent which would explain the formation of silver flowers on the anode due to ripple current. The electrochemistry of the anode in a properly biased condition would require that any silver in the area be converted to Ag^+ and the anode charge would repel these ions away. A possible explanation exists by considering what would happen if all potentials were removed from the capacitor by shorting it out and letting it sit. Since silver ions and Ag_2SO_4 are present in the electrolyte, there would be no factor on the anode which would prevent these materials from forming crystals on the case, in the electrolyte or on the anode. As there is no way of detecting silver flowers on the anode, there is no way to tell if the flowers existed on a particular anode prior to ripple testing and it is possible that silver flowers are primarily a shelf life phenomenon. Examination of the data will show that older parts which had just seen shelf storage displayed significantly greater probability of silver sulfate crystals and silver flowers on the anode and case.

In a reverse bias condition, silver on the anode is easily explained because not only is the situation similar to discharge, but the electron exchange at the anode surface is favoring conversion of silver ions into metallic silver. A silver plating bath for deposition on the anode has been formed.

3.3 Test Technique

The test technique employed on the majority of samples was as follows:

A. X-Ray in Two Perpendicular Axes - .005 in. and .001 in. tungsten wire was used to establish exposure in hopes that silver flowers might be seen. None were observed.

B. Visual Inspection - Each sample was examined at 20X for external case anomalies and mechanical damage.

C. Seal Leakage - Phydriion 1.0 to 2.5 pH paper and deionized water were used to check for electrolyte leakage through the seal. A small strip of paper was dipped in the water and immediately applied to the seal surface. Minute traces of a strong acid led to an obvious red coloration on the strip. None of the ripple tested samples were observed to have seal leakage.

D. D.C. Leakage - Each sample was tested electrically for the DC leakage. Charging was from a Fluke Precision DC Power Supply through a one K series resistor. Leakage current was recorded using Viking rated voltage at 30 sec, 1 min, 1.5 min, 2, 3, 4, and 5 min. The recording of leakage was an attempt to see if leakage and healing rate could be used to detect potentially defective units. Throughout this portion of the test no significant changes were observed that could be attributed to the presence of silver.

E. Open Case and Electrolyte Flush - The opening method for the samples was of extreme importance because particles from the case would seriously disturb the silver content readings. Each sample was chucked up on a tooling lathe and a .016" deep cut made directly behind the spacer crimp. This cut did not penetrate the case and if it had, it would have hit the Teflon and not the cavity. The unit was then cleaned and the case flexed to crack the cut open and the case removed. Immediately upon opening, the case interior and the gel were inspected at up to 50X. As near as could be determined no opening silver from the case was getting into the cavity. Using deionized water all electrolyte and gel present was flushed into a clean flask. The anode was then examined and all adhering electrolyte also flushed into the flask. These became the samples for electrolyte analysis.

F. Microscopic Examination - All anodes were examined at 50X and 100X magnification for the presence of silver. A photograph was taken of all anodes which exhibited silver flowers.

The case was similarly examined and photographs made as required to show silver sulfate crystals and redeposited silver. Results of the visual exams are noted in the data sheets.

G. Removal of the Anode - The anode was carefully removed by cutting the tantalum tube weld and pushing the anode out of the seal. All anodes were submitted for analysis of the silver content within the slug.

H. Wet Chemical Silver Analysis - The electrolyte sample was prepared for analysis by adding 10 drops conc. H_2SO_4 and evaporating the solution to SO_3 fumes on a hot plate. The residue was dissolved in 5 ml of 0.5 N H_2SO_4 and heated to a boil, then filtered and diluted to a standard volume (10 or 100 ml) for analysis.

The tantalum slug was prepared for analysis by boiling the slug in 5 ml conc. HNO_3 and 10 drops of conc. H_2SO_4 . Boiling was continued until fumes of SO_3 were visible. The residue and slug were cooled and silver salts were extracted from the slug with several warm 2 ml portions of 0.5 N H_2SO_4 . The extractant was diluted to a standard volume (10 or 100 ml) with 0.5 N H_2SO_4 for analysis.

Silver was detected by a standard colorimetric analysis using a 0.001% dithiazone in CCl_4 solution. The absorption of the silver dithiazonate was analyzed on a dual beam Beckman DB-GT spectrophotometer at 495 $m\mu$. The samples were always run in duplicate and were compared daily with standards.

The analysis was carried out as follows: Ten milliliters of 0.5 N H₂SO₄, two milliliters of sample, and five milliliters of dithiazone solution were added to a small separatory funnel and shaken for 30 seconds. The lower dithiazone layer was drained off and placed in a dry 1 cm quartz spectrophotometer cell. The sample was scanned from 530 to 480 mu and its absorbance was calculated at 495 mu and adjusted for the absorbance of a blank sample run over the same region. The amount of silver in the sample was calculated from the absorbance of the sample versus standard silver samples by a Beers law relationship.

To verify that all the silver was being analyzed in the anodes, some were completely crushed at the end of the analysis and re-analyzed. Residual silver was less than 1 μ gram.

The results of all the above testing are in the data sheets attached to this report.

3.4 Discussion

The data section of this report contains two summaries of the data derived during this testing. Documented in the first group is the silver visual and chemical data compared to the electrical parameters reported in Volume I and measured in the laboratory immediately prior to dissection. The Min-Max Ripple Test leakage values are somewhat perturbed by the vibration failures. It will be noted that most of the higher values occurred subsequent to vibration and that the lower value represents the more nominal reading for the test.

All silver levels are detailed in micrograms, being reported for both the anode and the electrolyte. Since the anode is highly porous, silver from the electrolyte contained in the anode is expected in the reported anode data. Consequently, silver levels measured on the anode are not necessarily from metallic silver flowers; a point verified by visual examinations of the anodes which disclosed very few visual silver flowers. In the entire ripple test only four anodes had visual levels of silver and their electrical data is indistinguishable from the rest of the samples in all but one case.

In most cases the anodes appeared normal. After vibration, some anodes displayed Teflon impressed into the base structure due to anode movement and one evidenced a slight crack. Insides of the silver cases routinely showed two distinct phenomena. First, many cases displayed redeposited silver on the surface of the platinum. These redepositions appeared as needles or sprays running parallel to the surface, loops outward from the surface and large patches up to .010 inch diameter. Electron beam microprobe found these redepositions to be primarily silver with few impurities, while microscopic exam showed a normal silver crystalline structure. The other case effect was exceedingly small transparent crystals which analyzed to be primarily silver and platinum. The crystal structure, while quite distinct, could not be related to any compounds and has been left unidentified. Laboratory investigations outside this report have occasionally shown this same effect and it does not appear to be related to ripple.

The silver sprays and redeposition are probably related to ripple as it is believed that silver is in a dynamic exchange into and out of solution due to current flow at the case. When silver replates on the case it would easily tend to form around existing silver particles and lead to large redeposited areas. These redepositions have been previously reported along with large silver sulfate crystals¹.

It was noted that some apparent correlation existed between the case silver redeposition, the electrolyte silver, and the ripple current exposures. The second group of data sheets is an effort to understand this correlation. ESR measurements from Volume I on individual devices were used to divide the applied ripple level between the individual capacitors on the basis of parallel resistance; these devices having been connected in parallel banks. A ripple level was thus calculated for each capacitor and multiplied by the exposure time to give an amp hour figure. The total amp hour figure was then compared to the measured total silver content (anode + electrolyte). Since the total amp hour figures vary greatly in some cases, a good sampling of conditions was obtained. The data for case size three capacitors is plotted in Figure #1. No obvious trends are apparent that would seem to relate to the ripple level. At any amp hour level where large silver contents were noted, another sample with nearly the same level can be found which has a very low content (-7's on sink #5 and -10's on sink #1). In general, the total silver levels did not deviate too significantly from the initial values.

Those units with high silver contents can not be clearly explained by the ripple current levels. This may be due to insufficient sample size to give meaningful data or the cause of high silver contents may lie somewhere other than just ripple current. The evidence does not show that the Viking ripple levels will in themselves lead to increased electrolyte silver contents or silver flowers on the anodes.

The principal salt detected in tantalum capacitors is silver sulfate Ag_2SO_4 . It has a handbook solubility product (K_{sp}) of 1.2×10^{-5} at 25°C which is high enough to allow some silver to be dissolved in the electrolyte possibly explaining the existence of silver in all the tested units. A GT-3 size capacitor normally contains about .31 to .33 cc of a 30% sulfuric acid gelled electrolyte which, by the analysis presented in Section 3.2, could contain a maximum of 114 mg of silver in an ionic form.

This value is below the normally observed concentration of silver in the capacitors and would require that the additional silver be in a compound state. Some factor other than solubility may account for these increased levels.

¹ W. J. Moore, Jr. and A. W.H. Smith, Ripple Current and Silver Migration in Non-Solid Electrolytic Sintered Anode Tantalum Capacitors, Proceedings 1972 of the 22nd Electronic Components Conference, Washington, D.C., May 15-17, 1970, pp 313-315.

One significant point is indicated by the silver data: Those units which saw no ripple, only DC bias have lower silver contents than parts out of stock. This could be predicted because without ripple injecting silver into the electrolyte the basic electrochemistry would tend to redeposit silver on the case by the action of the leakage current and negative polarity of the case. The best examples of this effect are the -10 and -8 old units which had seen 2000 hours of burn-in prior to inclusion in the ripple test program. After these units had been subjected to ripple, their silver concentrations returned to the apparent norm for their case size.

Outside of the specific samples from the ripple test program, other wet slug capacitors were analyzed for their silver contents. This data is presented in the data section for additional information and completeness, as these analyses were performed in support of this program.

The first of these additional samples are -8's which were used in the vibration failure analysis program. These are case size GT-1 and came directly from stock being similar to the units used in the memory test of Volume I.

The second group is comprised of -21 capacitors, case size GT-2, which were part of the vibration failure analysis and the ripple test program. Three samples were from VPO. Most of these samples had never seen ripple and yet displayed the case anomalies and silver on the anode.

The third group is comprised of MMC components from a Titan inverter which had undergone a long-term life test that applied substantial ripple levels to the components. The capacitors are Sprague 140 D non-hermetic units purchased under MMC specification ST90D-38A. Ten units from the inverter and five units from stock were analyzed. All are case size 3.

The final group is made up of two units from the Apollo Telescope Mount (ATM) Charger Battery Regulator Modules (CRBM's) which experienced failures thought to be due to silver growths on the anodes of wet tantalum capacitors caused by high ripple levels. Two General Electric SC155CN441MP3 capacitor assemblies were analyzed. One designated as the "B" group was a failed component and the other, designated "VS", was from stock. Each assembly is composed of 16 non-hermetic wet tantalum capacitors in a parallel bank. Both banks were dissected and examined with some units being returned to VPO for their analysis. The surprising data point from this analysis is that the good unused stock part shows more silver anomalies than the failed component. A complete report on the history of these parts is contained in an IBM, Federal Systems Division, Electronics Systems Center/Huntsville, Alabama report IBM No. 73W-00050, entitled: "Investigation of Tantalum Wet Slug Capacitor Failures in the Apollo Telescope Mount Charger Battery Regulator Modules," prepared under Skylab Contract NAS8-20899.

4.0 DATA

LAB. DATA SHEET 102

Table 4.1

TITLE	AG Summary Pre-Ripple Testing										DATE	IDENT.
	DASH NO.	S/N	IDC	E/A LEAKAGE @ 5 MIN uA	ELECTROLYTE AG uG	SLUG AG uG	CASE VISUAL	SLUG VISUAL	TOTAL G SILVER	4/18/73		
-21	2	7238A	.1	231	105	No Anom.	No Ag	336				
-21	1	7238A	.13	117	95	PT Sloppy	Cracks	212				
-21	107	7238A	.07	531	186	Ag on Case	No Ag	717				
-10	168	7236A	.12	213	148	No Anom.	No Ag	361				
-10	167	7236A	.13	23	2	No Anom.	No Ag	25				
-10	156	7236A	.12	250	75	No Anom.	No Ag	325				
-8	11	7236A	.022	38	14	No Anom.	"	52				
-8	18	7236A	.022	75	38	Ag on Case	"	113				
-8	12	7236A	.024	27	35	No Anom.	"	62				
-7	134	7240C	.13	338	76	No Anom.	"	414				
-7	149	7240C	.13	475	112	No Anom.	"	587				
-7	164	7240C	.17	429	112	No Anom.	"	541				
-24	46	7237A	.14	396	96	No Anom.	"	492				
-24	48	7237A	.15	254	85	No Anom.	"	339				
-24	50	7237A	.13	433	135	No Anom.	"	569				
-10 old	212	7220	.12	34	19	No Anom.	"	53				
-10 old	224	7220	.3	57	27	Ag on Case	"	84				
-10 old	238	7220	.2	28	19	"	"	47				
-8 old	351	7223A	.08	17	18	"	"	35				
-8 old	375	7223A	.05	11	3	No Anom.	"	14				
-8 old	388	7223A	.02	10	4	Ag on Case	"	14				

LAB. DATA SHEET 102

Table 4.2

TITLE	Ag Summary Post 300 Hr Ripple & Vib										DATE	4/18/73	IDENT.	
DASH NO.	S/N	IDC SINK #	E/A LEAKAGE @ 5 MIN	MIN-MAX LEAKAGE DURING TEST	ELECTROLYTE	SLUG AG	CASE VISUAL	SING VISUAL	POST 300 HR REPLE-PRE VIB LEAKAGE	Test #6				
-10	355	7236A #3	.06	22nA-90nA	114	33	PT Vari- ations	No Ag		22 nA				
-10	273	7236A #3	.07	21nA-70nA	168	126	No Anom.	No Ag		21 nA				
-7	18	7240C #5	.1	4nA-100uA	323	155	No Anom.	No Ag		7 nA				
-7	252	7240C #5	.04	2nA- 90nA	302	98	PT Vari- ations	No Ag		7 nA				
-7	374	7240C #5	20uA-1.5 min.	2nA-40uA	570	265	PT-Ag Crys fals-pt Variations	AG Flower		5 nA				
-21	51	7238A #7	3mA-30sec	13nA-23mA	430	229	No Anom.	Ag on end		15 nA				
-21	35	7238A #7	1.8mA - 30 sec	13nA-15mA	832	---	Ag Spots	2 Ag Flowers		16 nA				
-21	81	7238A #7	.04	14nA-27A	262	107	No Anom.	No Ag		19 nA				
-10 old	194	7220A #11	15 uA - 2 min	28nA-70uA	64	61	PT-AG Crystals	No Ag		Not Tested				
-10 old	199	7220A #11	1 Erratic	25nA-30uA	86	53	Destroyed			Not Tested				
-10 old	215	7220A #11	.08	28nA-160nA	46	78	PT Vari- ations	No Ag		Not Tested				

LAB DATA SHEET 102

Table 4.3

TITLE	Ag Summary Post 1000 Hr Ripple/Vib		DATE	4/18/73	IDENT.				
DASH NO.	S/N	IDC #	E/A LEAK- AGE @ 5 MIN	MIN-MAX RIPPLE TEST	ELECTRO- LYTE AG	CASE VISUAL	SLUG VISUAL	POST 300 HR PRE-VIB Test #9	
-7	303	7239A #5	.17	2nA-400nA	2500	415	Silver Sprays	No Ag	8 nA
-7	265	7239A #5	.21	22nA-210nA	1893	401	.010 Ag Area on End Cap	No Ag	8 nA
-7	320	7240C #5	6.3/.12*	5nA-860nA	240	111	Vib Abrasion Marks	Teflon in Slug-No Ag	7 nA
-7	264	7240C #5	76/37*	2nA- 18uA	55	37	Vib Abrasion Marks	Teflon in Slug-No Ag	7 na
-24	061	7237A #9	.21	8nA- 80nA	172	55	No Anomalies	No Ag	30 nA
-24	058	7237A #9	.19	9nA- 80nA	454	238	Spotty PT-No Ag	No Ag	36 nA
-24	062	7237A #9	.16	6nA- 90nA	1289	298	No Anomalies	No Ag	26 nA
-10 Old	210	7220A #11	2.0/.16*	9nA-150nA	111	55	Vib Abrasion Marks-Ag Sprays	Teflon in Slug-No Ag	Not Tested
-10 Old	213	7220A #11	.09	30nA- 90nA	41	51	Ag Spots	Fine Crack No Ag	Not Tested
-10 Old	220	7220A #11	.10	28nA-100nA	52	81	Ag-PT Crystals	No Ag	Not Tested
-10 Old	222	7220A #11	15/13*	25nA- 2uA	141	61	Ag-PT Crystals	Teflon in Slug-No Ag	Not Tested
-10	107	7236A #1	.12	30nA-105nA	22	9	No Anomalies	No Ag	40 nA
-10	236	7236A #1	.11	31nA-140nA	2015	409	Ag Spray	No Ag	36 nA
-10	244	7236A #1	.083	29nA-125nA	2380	432	Ag Spray	No Ag	34 nA
-10	278	7236A #3	.11	19nA- 65nA	388	91	No Anomalies	No Ag	19 nA

*Additional 5 minutes.

LAB DATA SHEET 102

Table 4.4

TITLE		AG Summary Post 1500 Hr Ripple										DATE	IDENT.			
DASH No.	S/N	EDC SINK #		E/A LEAKAGE @ 5 MIN.		MIN-MAX RIPPLE TEST		ELECTROLYTE		SLUG AG		CASE VISUAL	SUC VISUAL 100X	POST 300 HR RIPPLE PRE-VIB	TEST #6	POST 1500 HR REPORTED LEAKAGE
		#	#	uA	uA	uA	mA	EST	EST	EST	EST	AG	AG	TEST #9	TEST #8	
-7	44	7240C #4	7240C #4	60 uA	1.1nA-14uA	168	56	Abrasion marks on Case	Ag Flowers	8 nA	1.3 uA					
-7	104	7240C #4	7240C #4	.12 uA	80 nA-2nA	229	120	No Ag	No Ag	11 nA	8 nA					
-21	60	7238A #7	7238A #7	700 uA	14 nA-1 mA	853	165	Ag Sprays	No Ag	19 nA	1.4 uA					
-21	39	7238A #7	7238A #7	760 uA	12 nA-500uA	181	29	Ag-PT Crystals	No Ag	15 nA	150 uA					
-7	101	7240C #4	7240C #4	15	8.4uA-1 nA	203	77	No Ag	No Ag	150 nA	8 nA					
-7	086	7240C #4	7240C #4	9.2	3 uA-3 nA	73	35 est	Ag-PT Crystals Abrasion marks	No Ag	10 nA	7 nA					
-7	082	7240C #4	7240C #4	22	1.3 uA-1 nA	311	148	No Ag	No Ag	9 nA	6 nA					
-21	097	7238A #7	7238A #7	200	4 uA-15 nA	98	65	Abrasion marks No Ag	No Ag	20 nA	25 nA					
-21	011	7238A #7	7238A #7	.25	2.5uA-15 nA	65	41	Small Ag crystals	No Ag	17 nA	18 nA					
-21	036	7238A #7	7238A #7	10	100uA-14 nA	154	100	Abrasion marks No Ag	No Ag	18 nA	110 nA					
-21	023	7238A #7	7238A #7	21	5.4uA-12 nA	62 est	27	No Ag	No Ag	16 nA	20 nA					

LAB. DATA SHEET 102

Table 4.5

TITLE		RIPPLE CURRENT DISTRIBUTIONS				Sink #1	-10	DATE	IDENT.
S/N	I at 100 Hr	Total AH X100	I at 300 Hr (+200)	AH X200	I at 1000 Hr (+700)	AH X1700	Total AH	Total Ag uG	
184	.172	17.2	.170	33.9	.164	114	166		
188	.168	16.8	.152	30.3	.160	112	159		
189	.169	16.9	.157	31.5	.167	117	165		
203	.169	16.9	.167	33.4	.161	113	163		
207	.170	17.0	.157	31.5	.130	91.2	140		
236	.171	17.1	.158	31.7	.168	118	166	2424	
237	.141	14.1	.135	27.1	.137	96.0	137		
257	.141	14.1	.148	29.6	.151	106	150		
244	.144	14.4	.165	32.9	.164	115	162	2812	
117	.143	14.3	.164	32.8	.174	122	169		
111	.140	14.0	.135	27.0	.141	99.0	140		
107	.140	14.0	.097	19.4	.089	62.5	95.9	31	
092	.141	14.1	.151	30.3	.127	88.9	133		
071	.142	14.2	.149	29.9	.157	110	154		
069	.143	14.3	.170	34.0	.169	118	167		
170	.141	14.1	.152	30.4	.142	99.6	144	180	
171	.143	14.3	.154	30.8	.163	114	159		
173	.141	14.1	.151	30.3	.156	109	153		
178	.138	13.8	.112	22.3	.109	76.9	112	28	
181	.143	14.3	.154	30.9	.169	118	163		

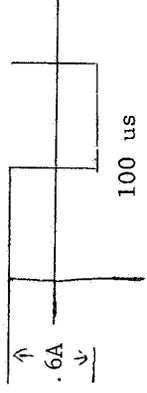
$I_{\text{individual}} = .150A \text{ RMS}$

GT-3

LAB. DATA SHEET 102

Table 4.6

TITLE		RIPPLE CURRENT DISTRIBUTIONS					Sink #3 -10		DATE	IDENT.
S/N	I at 100 Hr.	100 AH	I at 300 Hr.	200 AH	I at 1000 Hr	700 AH	Total AH	Ag		
385	.256	25.6	.269	53.8	.274	192	271	287		
358	.302	30.2	.319	63.7	.315	220	314			
356	.302	30.2	.265	53.1	.213	149	232	88		
355	.302	30.2	.280	56.0			86.2	147		
354	.307	30.7	.323	64.6	.310	217	312	264		
338	.298	29.8	.304	60.7	.311	217	307			
309	.308	30.8	.286	57.2	.297	208	296			
308	.305	30.5	.311	62.1	.300	210	303			
302	.299	29.9	.305	60.9	.302	212	303			
278	.306	30.6	.321	64.3	.318	223	318	479		
273	.305	30.5	.300	60.1			90.6	294		
271	.309	30.9	.315	63.1	.360	252	346	2095		



$I_{RMS} = .300A$ per device
 $F = 10$ KHz

GT-3

LAB. DATA SHEET 102

Table 4.8

TITLE	Ripple Current Distributions Sink #5 -7				DATE	IDENT.		
S/N	I at 100 Hr	AH X 100 Hr	I at 300 Hr	AH X 200 Hr	I at 1000 Hr	AH X 700 Hr	Total AH	Total Ag
	$V = .36V$							
	$V = .168$							
	$V = .166$							
018	.270	27.0	.275	55.0			82	478
320	.267	26.7	.264	52.8	.276	193.2	272.7	351
314	.269	26.9	.287	57.4	.284	198.8	283.1	
306	.266	26.6	.285	57.0	.274	191.8	275.4	
303	.292	29.2	.290	58.0	.296	207.2	294.4	2915
286	.292	29.2	.290	58.0	.278	194.6	281.8	
265	.275	27.5	.281	56.2	.285	199.5	283.2	2294
264	.272	27.2	.262	52.4	.259	181.3	260.9	92
252	.269	26.9	.290	58.0			84.9	400
471	.269	26.9	.289	57.8	.294	205.8	290.5	
445	.268	26.8	.272	54.4	.275	192.5	273.7	
421	.273	27.3	.285	57.0	.289	202.3	286.6	
418	.269	26.9	.290	58.0	.294	205.8	290.7	1216
404	.266	26.6	.271	54.2	.266	186.2	267.0	
390C	.272	27.2	.262	52.4	.277	193.9	273.5	
377	.266	26.6	.271	54.2	.254	177.8	258.6	
374	.233	23.3	.172	34.4			57.7	835
367	.276	27.6	.280	56.0	.292	204.4	288.0	
353	.267	26.7	.245	49.0	.255	178.5	254.2	
357	.271	27.1	.275	55.0	.288	201.6	283.7	

$I_{RMS} = .270A$

LAB. DATA SHEET 102

Table 4.13

TITLE		Vibration Analysis Summary -21										DATE	IDENT.
Dash No.	S/N	Case Size		Leakage @ 5 min		Electrolyte		Slug Ag		Case Visual		Slug Visual	
		ug	ug	ug	ug	ug	ug	ug	ug				
-21	2	2	60V .1ua	231	105	OK	OK	OK	OK	OK	Ripple Test		
-21	1	2	60V .13ua	117	95	PT Sloppy	Corner Broken				Baseline		
-21	107	2	60V .07	531	186	Ag Clusters	OK				Units		
						Top of Case							
-21	51	2	40V 3 ma	430	229	OK	1000u tree				Ripple Test		
-21	35	2	40V 1.8ma	832		Many Ag Spots	Ag Flowers				Failures		
-21	81	2	40V .04ua	262	107	OK	200u Ag Tree						
-21	9	2	40V 20ua		97	OK	Broken End - OK				Stock Failures		
-21	58	2	40V 1.2ma		126	Spider Outline	OK				" "		
-21	65	2	40V 2.5ma		240	Ag Spray Patterns	OK				" "		
-21	78	2		719	146	Many Ag Patterns	Broken End - OK						
-21	L2	2	.1ua	78	50	OK					VPO Samples 7307A		
-21	L4	2	.1ua	88	92	OK					" "		
-21	L6	2	.1ua	859	176	Ag Spray Patterns	OK				" "		

LAB. DATA SHEET 102

Table 4.14

TITLE		Inverter Samples			ST90D-38A			DATE	IDENT.
Sprague 140D 82 ufd - 50 VDC LDC 6821									
S/N	Leakage at 5 min ug	Electrolyte Leakage	Electrolyte Content ug	Slug Content ug	Ag Content ug	Ag Content ug	Ag Content ug	Ag Content ug	Ag Content ug
305	.14	None	1184	500	Two Ag spot on inside of case-electrolyte dry				
306	.155		724	454	One Ag spot on inside of case "				
307	.16		1116	425	Many Ag spots on inside of case "				
308	.1		780	234	1 Ag sppt on inside of case "				
309	.17		620	342	1 Ag sppt on inside of case "				
310	.14		926	500	1 Ag flwver on Slug, Cu (Prbb) spot on slug				
311	.15		596	475	OK				
312	.13		924	380	OK				
313	.17		Damaged during opening - No Data						
314	.13		646	454	Many Ag spots on side of case* "				
427	+ 1 ua		286	572	Many Ag spots on case				
428	.2 ua		860	500	Many Ag spots on case				
429	.3 ua		860	476	Many Ag spots on case				
430	+ 3 ua		360	368	Very small Ag spots on case				
431	.3 ua		440	500	OK				
*Microprobe displays metallic Ag in the spots.									

LAB. DATA SHEET 101

Table 4.15

TITLE	ATM Capacitor Summary				DATE	IDENT.	
General Electric SCL55CN441MP3 440 ufd - 100 VDC LDC 8-67							
Silver Analysis				Visual			
S/N	Electrolyte	Anode	Leakage @ 5 Min	Anode	Case		
			uA				
VS1	458	262	.18	No	Ag	2 Silver Redeposition Areas	
VS2	362	310	.1	"	"	3 Silver Areas	
VS3	444	310	.1	"	"	1 Silver Area	
VS4	548	262	.14	"	"	Several Small Spots	
VS5	456	274	.8	"	"	"	"
VS6	375	274	.11	"	"	"	"
VS7	285	285	.09	"	"	2 Ag Areas	
VS10	360	322	.16	"	"	5 Areas	
VS11	172	262	.12	"	"	3 Areas	
VS12	402	286	.1	"	"	"	"
VS13	724	262	.15	"	"	"	"
VS14	415	262	.12	"	"	1 Ag Area	
VS15	388	273	.13	"	"	1 Ag Area	
VS16	504	262	.13	"	"	5 Ag Areas	
B1	252	208	.2	"	"	OK	
B2	298	263	1.5	"	"	"	
B3	310	240	.17	"	"	"	
B6	228	263	1.1	"	"	"	
B7	183	153	.18	"	"	"	
B8	204	191	1.7	"	"	"	
B9	*41		14	Burnt and			
				Destroyed			
B10	2853	.63 mA	.63 mA	"	"		
B11	297	*116	.28 uA	"	"		
B12	286	274	.25	"	"		
B13	366		.16	Large Ball of Silver		"	
B14	303	218	.58	"	"	"	
B15	232	156	3.4	"	"	"	
B16	286	286	.18	"	"	"	
	*Bump Loss						

5.0 PHOTOGRAPHS

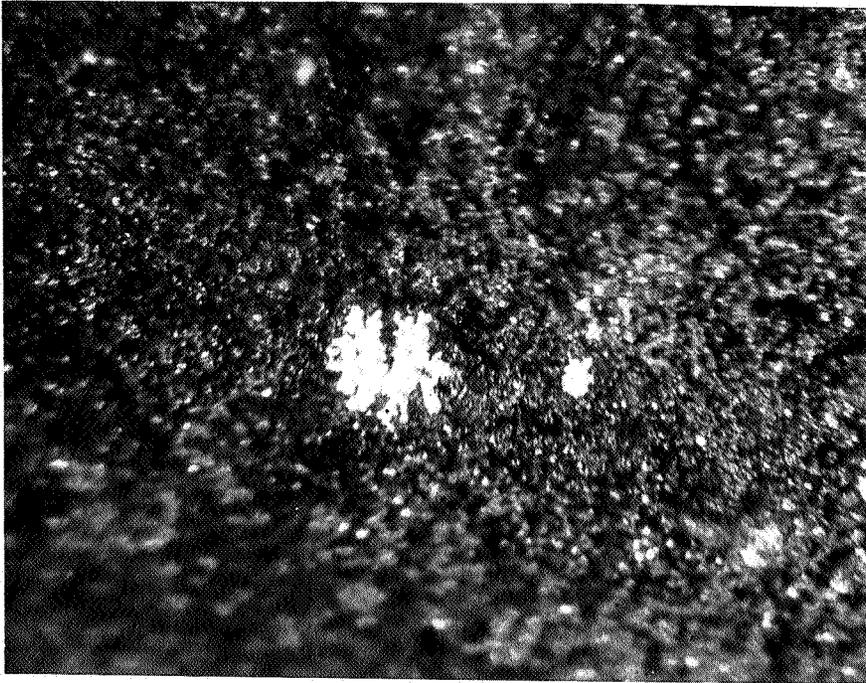


Photo #1
Silver flower on S/N 374
-7. 300 hr. (140X)



Photo #2
Silver area on bottom of
Anode S/N 51. -21
300 hr. (70X)

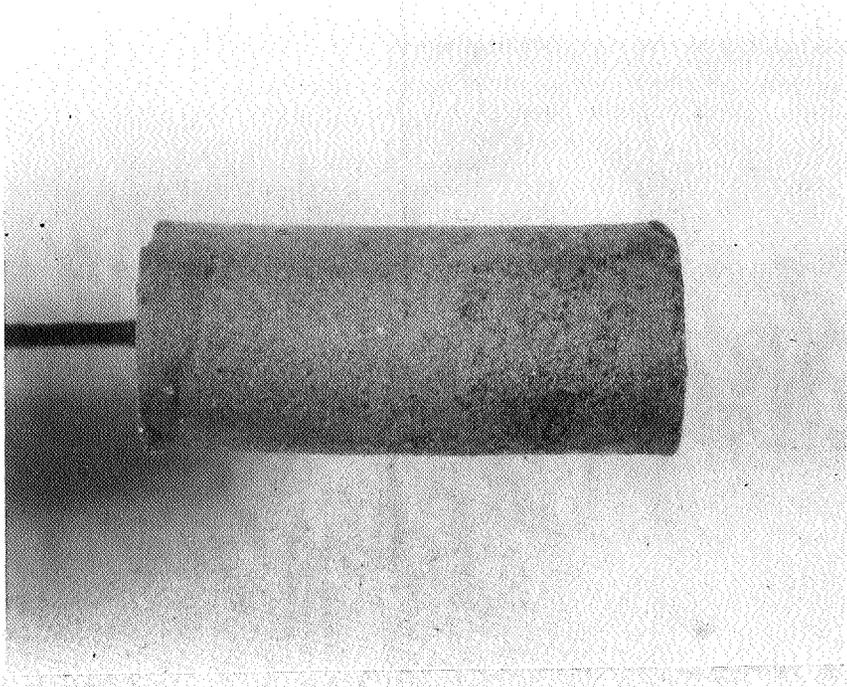


Photo #3
Anode of S/N 35 showing
location of silver flower
-21. 300 hr. (8X)



Photo #4
Silver flower on S/N 35.
(280X)
These crystals were not
identified, however, were
composed of Ag₂S.
(100X)

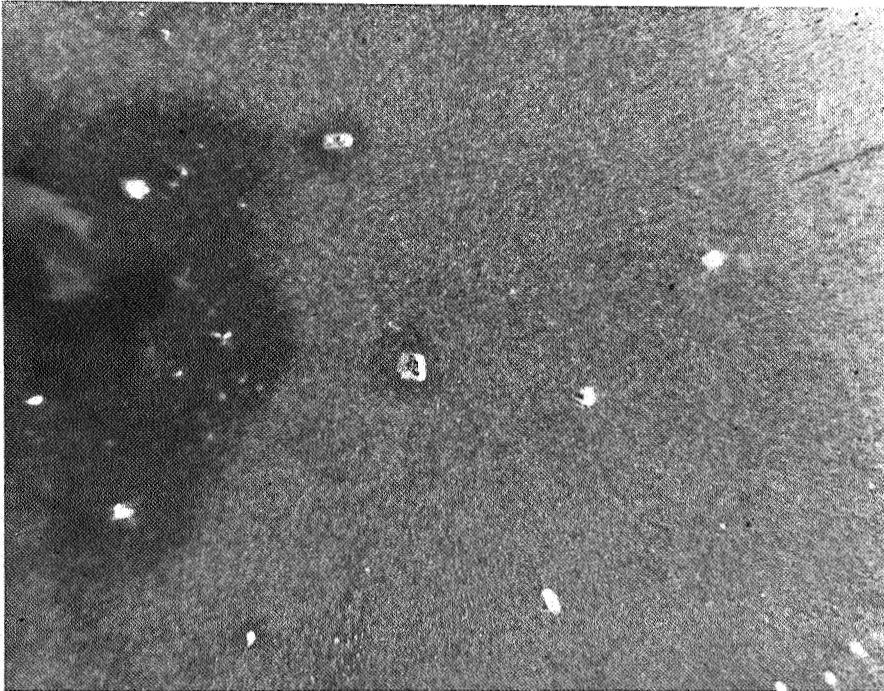


Photo #5

Inside of case on S/N 194
showing crystals. (50X)



Photo #6

Magnified view on crystal
from case of S/N 194.
These crystals were not
identified; however, were
comprised of Ag-PT.
(140X)



Photo #7

Typical inside of case
which displayed the
silver redeposition.
S/N 78 -21.

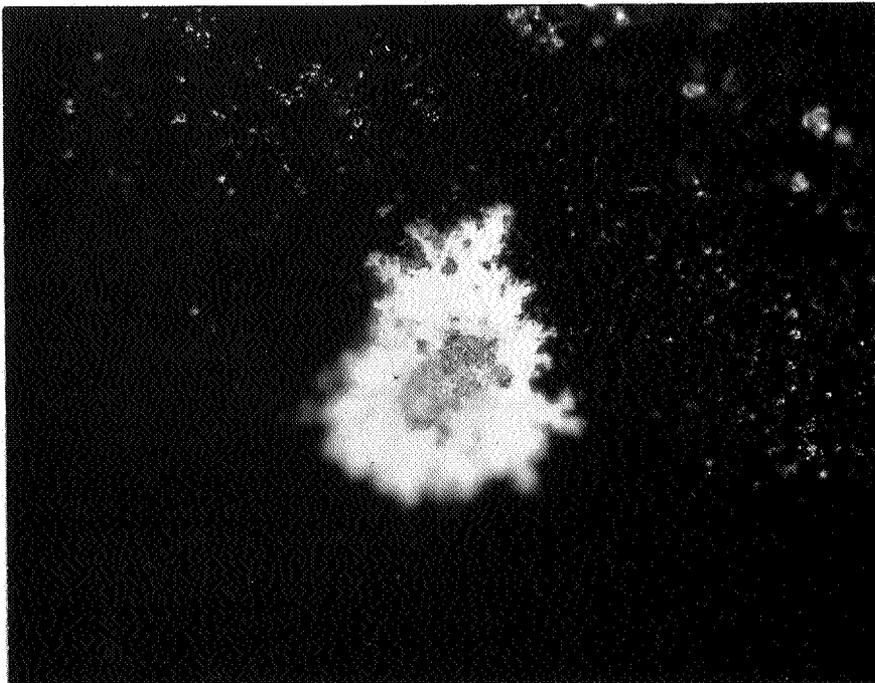


Photo #8

Silver flower on S/N 44
-7. 1500 hr. (150X)