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IMPROVED CATALYSTS BY LOW-G PROCESSING

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CONTENTS

1. Discipline:
Electrochemical Processes

2. NASA Log No.:

PROPOSAL SUMMARY

SPACE PROCESSING INVESTIGATIONS FOR SPAR MISSIONS

3. Title: IMPROVED CATALYSTS BY LOW-G PROCESSING

4. Proposer's No.:
LMSC-D084883-392

5. Estimated Cost:
First Year: $135,527
Total: $189,400

6. Investigators:
Investigator: Dr. Philomena Grodzka
Consultants: Dr. Gabor A. Somorjai (University of California, Berkeley) and Dr. Paul H. Stonehart (Stonehart Associates)

7. Proposing Organization:
Lockheed Missiles & Space Company, Inc.
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8. Summary:

a. Data presented in this proposal do not contain "protectible data" as defined in NASA Procurement Regulation 9.201(d).

b. The specific objective of this study is to establish that elimination of convection accompanying metal electrodeposition at high overvoltages in one-g will result in surface topographies and particle sizes desirable for efficient catalysts.

c. Fine powders of silver and palladium will be electrodeposited in low-, one-, and two-g. These powders as well as one or two commercial catalysts will be characterized and evaluated for catalytic activity.

d. The results of the proposed investigations will establish the advantages of space for producing unique microcrystalline morphologies and structures. Should, as expected, the space grown morphologies show improved catalytic activities over those produced on the ground, the advantages of commercial catalyst production in space will be firmly established.

c. BACKGROUND AND JUSTIFICATION

Summary

The proposed space experiment is needed to verify the following hypothesis:

- A number of studies have shown that the catalytic activity of electrodeposited catalysts depends strongly on the potential at which the catalyst was deposited. Figure 1 shows the general nature of this dependence.

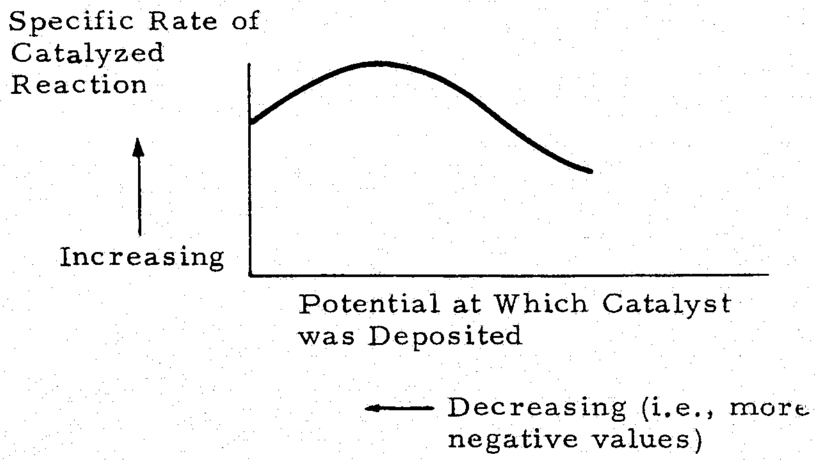


Fig. 1 - Typical Curve of Catalytic Activity as a Function of Deposition Potential

- Other studies have shown that catalytic activity depends on crystallite size, surface area, structure, and topography. These crystalline properties, however, are a function of the potential at which the crystals are deposited. The observed dependence of catalytic activities on deposition potentials, therefore, must be the result of the varying crystalline sizes, structures, and topographies produced at the various potentials.
- At more negative deposition potentials, convective currents become strong enough to alter the trend toward more powdery, dendritic, and twinned, and hence more catalytically active, crystalline morphologies.

- In low-g, the suppression of gravity-driven convection will allow the deposition of more powdery, dendritic, and twinned deposits and hence more active catalysts than are possible on the ground. It might be added that on the ground the only way to suppress convection emanating from growing, microcrystallite-dendrite arms is to use a gel. Subsequent separation of gel from microcrystallites of catalysts, however, would probably be difficult. Also, gel colloids would alter the crystal growth processes and catalytic activities because of strong adsorption. Furthermore, local heating effects at the high current densities required for powder formation would undoubtedly melt any gel to variable extents around growing dendrite arms. Convection would thus not be eliminated.
- If, as predicted, convection is the factor that interferes with deposition of catalytically favorable morphologies, it can be expected that the curve of specific rate of catalyzed reactions can be extended some distance, i.e., Fig. 2.

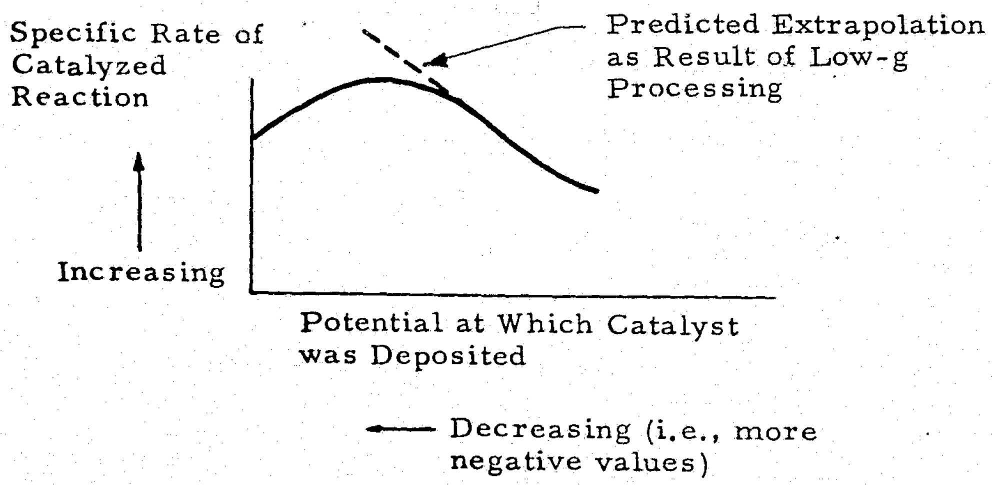


Fig. 2 - Predicted Extrapolation of Catalytic Activity as a Result of Low-g Processing

Verification of the preceding line of reasoning would be directly relevant to other methods for making catalysts (vapor deposition or solution precipitation) because much the same considerations are involved in all of these processes (Refs. 1 through 5). The economic value of catalysts, furthermore, is enormous. A description of catalysts role in our economy are given in Appendix A. (Appendixes and references are at the end of this proposal following the blue marker page.)

The background for the preceding rationale is presented in the following discussions.

Catalytic Activity as a Function of Deposition Potential and Hence of Crystalline Structure

Vitanov et al. (Ref. 6) found that the exchange current density in the case of silver electrodeposition by a special capillary method is strongly influenced by the surface profile and is proportional to the step density which is in turn a function of the deposition potential as shown in Fig. 3. The exchange current density may be viewed for present purposes as an indication of the catalytic activity of the electrode surface. The extrapolated value of the exchange current for zero step density is exactly the same as that measured on a dislocation-free plane (Ref. 7).

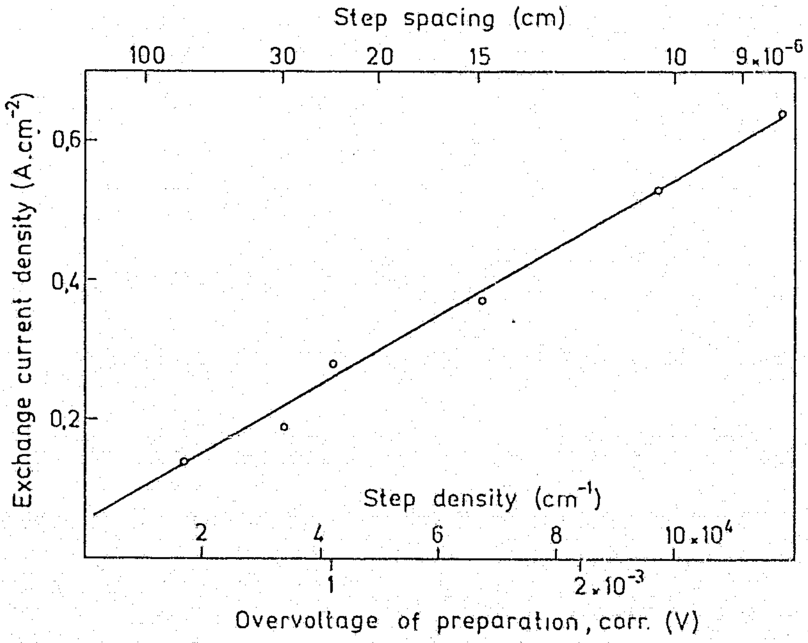


Fig. 3 - Overall Exchange Current Density as a Function of the Surface Profile (The abscissa gives the overvoltage used for the growth preparation of the face prior to impedence measurement (Ref. 9).)

Dr. G. A. Somorjai and colleagues at the University of California at Berkeley have accomplished a great deal of work establishing the role of surface steps and kinks in a number of chemical reactions. For example, in reactions where C-C, C-H, and H-H bonds are broken, atomic height steps and kinks in steps have been identified on platinum surfaces as distinct catalytic sites (Ref. 8). Figure 4 shows the dependence of the catalytic rates on step and kink density for one reaction.

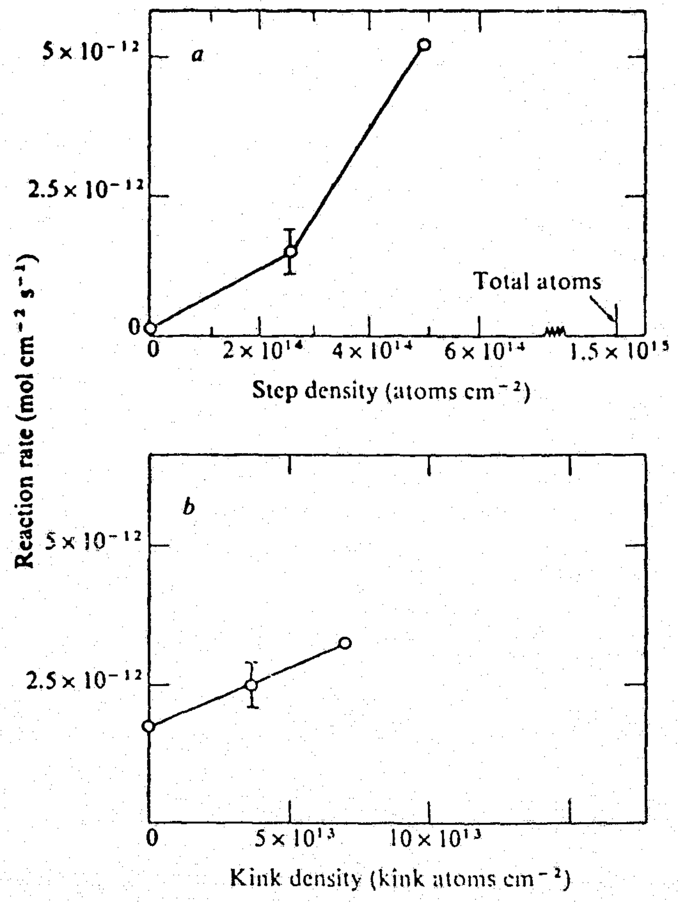
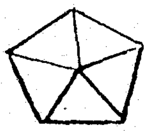


Fig. 4 - Initial Rate of Cyclohexene Dehydrogenation to Benzene on Platinum Single Crystal Catalysts with: (a) Increasing Step Density and (b) Increasing Kink Density at a Constant Step Density of 2.5 x 10^{14} Step Atoms cm^{-2}. The reaction conditions are 5 x 10^{-6} Pa of Cyclohexene, 1.0 x 10^{-4} Pa of Hydrogen and 423 K Catalyst Temperature. Hydrogen-Hydrocarbon ratio 20:1 (Ref. 8)

Other reports of catalytic activities of noble metals (Pd, Pt, and Rh) also show a strong dependence on deposition potentials (Refs. 10 through 14). For the case of silver, the catalytic activity for the epioxidation of ethylene was found to depend profoundly on its method of preparation and on specific crystalline morphologies (Ref. 15).

Other factors, in addition to step density, to be considered with regard to catalytic activity is that of size of catalyst particles and their particular morphologies. This consideration is required because "strained" surfaces characteristic of very small particles on certain crystal forces appear to be important for certain reactions. Dr. P. Stonehart has studied the role of particle size and surface morphology in electrocatalytic reactions and has recently reviewed the subject (Ref. 16). The crystallite morphologies in face-centered cubic metals that appear to offer the best promise of increased catalytic activity for certain reactions are as follows (Refs. 8 and 17):

Octahedron Bounded
by (111) Planes



Icosahedron (Five-
fold Symmetry)

The icosahedron morphology is apparently the stable morphology for very small microcrystals, although not for larger or macroscopic crystals. The octahedron morphology is a stable morphology for small and macroscopic crystals. Both morphologies possess (111) surface faces that have been shown to be the active faces for certain reactions (Refs. 18 through 20).

It will be shown in the next section that the higher the deposition potential the higher the proportion of twinned morphologies (i.e., icosahedrons) and finer or more powdery the crystalline deposit. Thus deposition at higher overvoltages (more negative deposition potentials) favors three crystalline features favorable for high catalytic activity: more surface steps and kinks, a higher proportion of twinned morphologies, and smaller particle sizes. It will also be shown in the next section that convection can become strong enough as deposition potentials and hence current densities increase to alter this favorable trend toward more catalytically active morphologies.

On the basis of the preceding evidence the following conclusions are drawn:

- Surface steps, kinks, twinned junctions, and small particles become more numerous in electrocrystallizations as the overvoltage of preparation increases.
- Surface steps, kinks, twinned junctions, and certain crystal faces are catalytically active sites for a number of structure sensitive reactions (See Appendix B for a discussion of structure-sensitive reactions.)

One can thus expect that crystallites deposited at higher overvoltages should be more catalytically active. Figure 5 shows that this expectation is partially fulfilled. The catalytic activity of electrolytically deposited palladium black increases sharply as the deposition overvoltage increases (the deposition potential becomes more negative). A maximum is then reached, after which a slow decline sets in. One explanation forwarded for the shape of the curves

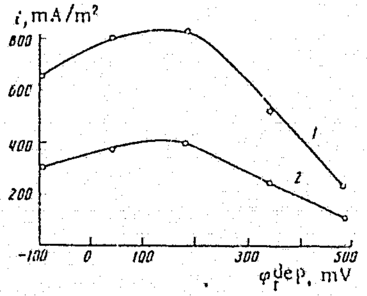


Fig. 5 - Dependence of the Specific Rate of Oxidation of 0.5 M N_2H_4 in 1 N KOH (1) at φ_r = 180 mV and of 1 M CH_2O in 1 N KOH (2) at φ_r = 360 mV on the Deposition Potential of Pd Black (Potentials versus standard hydrogen electrode (Ref. 14).)

shown in Fig. 5 is that the catalytic activity is influenced to a considerable extent by the structure of the palladium deposit. In the next sections we will consider the effects of electrodeposition potential and convection on crystalline morphologies.

Crystallite Morphologies and Sizes as a Function of Deposition Potential

A fairly substantial literature exists on the subject of powder formation as a function of deposition potential. Probably the review by N. Ibl (Ref. 21) represents the most comprehensive review of the subject to date although Refs. 9 and 22 through 25, also present large amounts of information. For purposes of this proposal the major findings of the research in this area are summarized in Table 1 (Ref. 21).

Table 1
FACTORS FAVORING POWDER FORMATION (REF. 21)

Electrolysis Conditions	Change in Character of Deposit*
Increase of Metal Salt Concentration	II → I
Increase of Concentration of Indifferent Electrolyte	I → II
Increase of Rate of Stirring	II → I
Increase of Current Density	I → II
Increase of Temperature	II → I
Increase of Viscosity	I → II

*I, coherent deposit; II, loose deposit.

Ibl has established that powder formation starts when the limiting current is reached, i.e.. when the rate of mass transport in solution becomes rate controlling (high deposition-overvoltages). Figure 6 illustrates the role of overvoltage in powder formation.

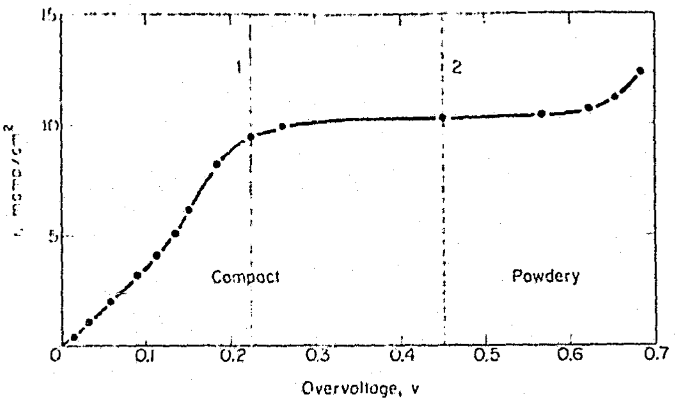


Fig. 6 - Overvoltage Characteristic of Transition from Compact to Powdery Deposit (Solution: $CuSO_4$ (0.1 M) + H_2SO_4 (0.5 M) (Ref. 21).)

A number of theories of powder formation have been advanced: one holds that the increased rate of nucleation (because of low interfacial concentrations) and instability of the crystal growth under mass transport control lead to a loose, finely divided deposit (Ref. 21). Another holds that metallic ions are discharged at some distance from the electrode at high enough overpotentials by means of a quantum mechanical tunnel effect (Refs. 21 and 26). More recently Ibl has presented evidence against the quantum theory (Ref. 27). Both theories agree, however, that powder deposition occurs under mass transport control and that stirring opposes the trend to more powdery deposits. The weight of the evidence presented by Ibl, furthermore, would favor the nucleation/dendrite theory of powder formation.

A rather large number of papers exist in the literature on the morphologies produced by electrodeposition in various metal systems. For example, for the case of zinc electrodeposition from alkaline zincate solutions, the following types of deposits were found (Refs. 28 and 29).

> Low overpotential (~50 to 100 mV): Epitaxial layer growth.
>
> Medium overpotential (~100 mV to 200 mV): "Boulders" suggested to be originated by nucleation.
>
> High overpotential (~over 200 mV): Flat dendrites

The boulder or mossy growths consist of large numbers of small crystals (Ref. 30). In still another study of zinc electrodeposition the boulder growth was established as being an activation-controlled (or kinetically-controlled), deposit, and the dendritic growth as a diffusion-controlled (or mass transport controlled) deposit (Refs. 31 and 32). The general conclusion of this latter work was: When a high local concentration of zincate exists, the reaction is activation-controlled, but when a condition of zincate depletion exists, the reaction is diffusion-controlled (i.e., transport-controlled).

Recent work by N. A. Pangarov and others (Refs. 33 and 37) has established that rates of nucleation increase at high overpotentials as does the proportion of twinned crystallites. The ratio of the nucleation rate of twinned to normal nuclei is given by the following equation (Ref. 36):

$$\frac{I_T}{I} = \exp - \frac{K_2 \gamma}{\beta(\beta - \gamma)}$$

where

I_T = rate of nucleation of twinned nuclei
I = rate of nucleation of normal nuclei
K_2 and γ = constants for a particular situation
$\beta = c\,\eta$ where c is another constant and η the overpotential

Effect of Convection on Crystalline Morphologies and Sizes

The potential, E, at which a metal is electrodeposited is made up of the equilibrium potential, E_e, and the overvoltage, , i.e.,

$$E = E_e + \eta$$

The overvoltage is in turn made up of an activation overvoltage, η_a, a concentration overvoltage, η_c, and a resistance overvoltage, η_r, (Ref. 38). Each of these overpotentials depend on the concentrations of active ions at the electrode surface, and these in turn are determined by the thickness of the diffusion layer, i.e., Fig. 7.

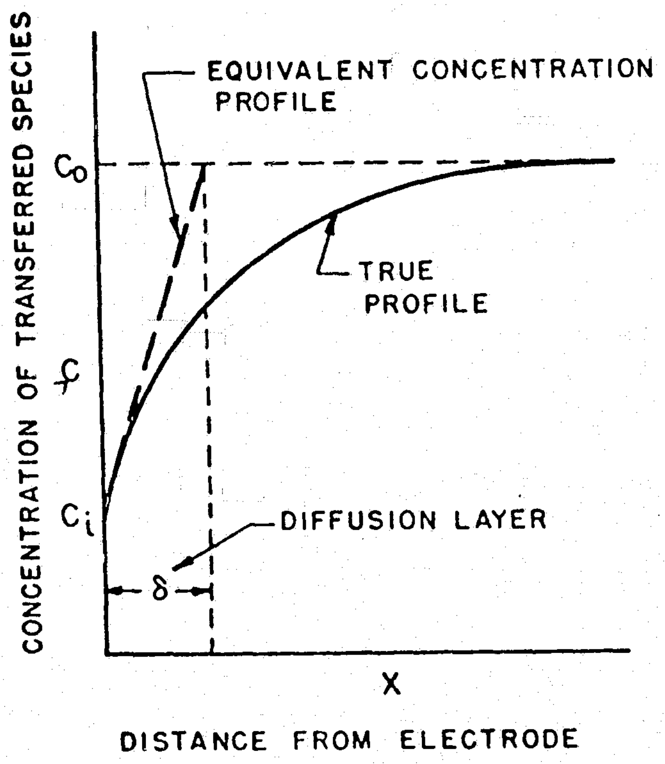


Fig. 7 - Illustration of Concentration Profile in the Vicinity of an Electrode During Electrolysis (Ref. 39)

A number of mathematical treatments of natural convection are available in the literature (Refs. 21 and 39 through 45). For present purposes it is sufficient to note that increased vigor of natural convection decreases the thickness of the diffusion layer and hence increases the concentration gradient at the electrode surface. A steep concentration gradient favors a shift of the overall rate from diffusion to activation control, i.e., non-dendritic morphologies. Or, "increasing the agitation or decreasing the viscosity of the solution; has to a first approximation, the same effect as decreasing the current density, because mass transfer is mainly affected" (Ref. 22). Decreasing the current density would be equivalent to decreasing overvoltage.

We should expect that at higher overpotentials convective effects would become increasingly more pronounced because faster rates of deposition of metal ions from solutions would result in greater density differences. Also, greater heating would be expected at higher overvoltages because of Joulean heating and reaction heats. On a local scale these heat effects could appreciably heat surrounding liquid and thus lead to reinforcing convection generated as the result of solute depletion because of the electrolysis (Refs. 33 and 40). In low-g, locally heated solution without convection should be a desirable condition because it would make the kinetics of the electron transfer reaction faster and hence shift the process control toward diffusion.

Figure 8 shows that forced fluid flow can indeed change crystallite morphology in the predicted direction.

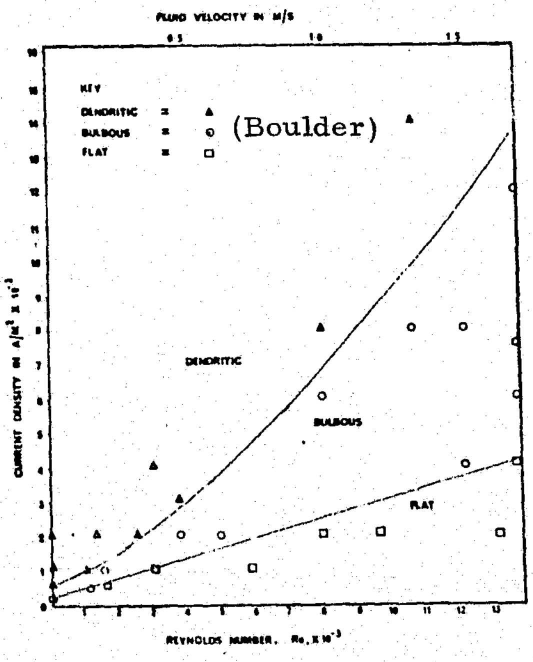
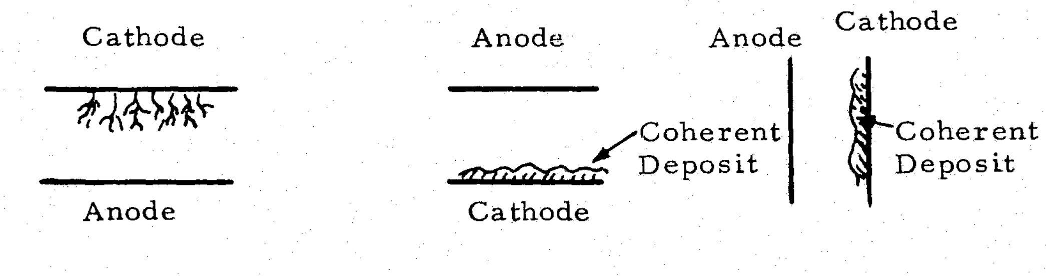


Fig. 8 - Graph of Zinc Morphology as a Function of Reynolds's number, Re, Flow Velocity, and Current Density for Zinc (Ref. 46)

In a study of convection accompanying zinc deposition, it was found that zinc dendrites grew if the cathode was orientated in the gravity field so as to minimize convection, i.e., depleted solution on top, as shown in Fig. 9, Case a, (Ref. 47).



Case a. Dendritic Deposit

Case b. Smooth Adherent Deposit

Case c. Smooth Adherent Deposit

Fig. 9 - Arrangement of Electrodes in One Convection Study

It might be mentioned that even though putting a cathode over the anode substantially changes the nature of the deposit, convection cannot be eliminated on the microscale at which the dendrites are growing. The silver crystal experimental data discussed in a following shortly will show this.

In a study of free convection accompanying electrolysis of copper at the limiting current (electrodes as in case (b), Fig. 9), it was found that the boundary layer was turbulent (Ref. 48). The data could be correlated at steady state by the expression:

$$\mathrm{Nu}' = \frac{I_L\,(1 - t_{cu^{+2}})\,x}{n\,F\,C_b\,D} = 0.19\,(\mathrm{Gr} \cdot \mathrm{Sc})^{1/3}$$

where

I_L = limiting current density
$t_{cu^{+2}}$ = transference number
x = characteristic dimension
n = number of electrons exchanged
F = Faraday
C_b = bulk concentration
D = diffusion coefficient
Gr = solutal Grashof number
Sc = Schmidt number

Numerous other works on convection during electrolysis exist including electrolysis at vertical electrodes. Reference 49 is a current review.

The strongest bit of evidence that low-g can alter both the particle sizes and micromorphologies of electro-deposited metal powders comes from a Skylab experiment in which silver crystals were electrolytically deposited by a displacement reaction (Ref. 50 and Appendix C). In this experiment the silver crystals grown aboard Skylab IV were compared to crystals grown similarly at 1 g and 5 g. The space grown crystals generally possess more perfect microcrystalline forms and are less coherent than the earth or centrifuge grown crystals. The observed degrees of dispersions is qualitatively in accord with the prediction of Ibl's theory that a more diffusive environment is favorable to powder formation. Furthermore, the observed micromorphologies showed a strong dependence on g level and hence on vigor of natural convection prevailing during the crystallization.

A final bit of evidence offered for the case that natural convection can profoundly affect crystallite size and morphology are the following set of microphotographs of samples prepared just recently (Figs. 10, 11 and 12).

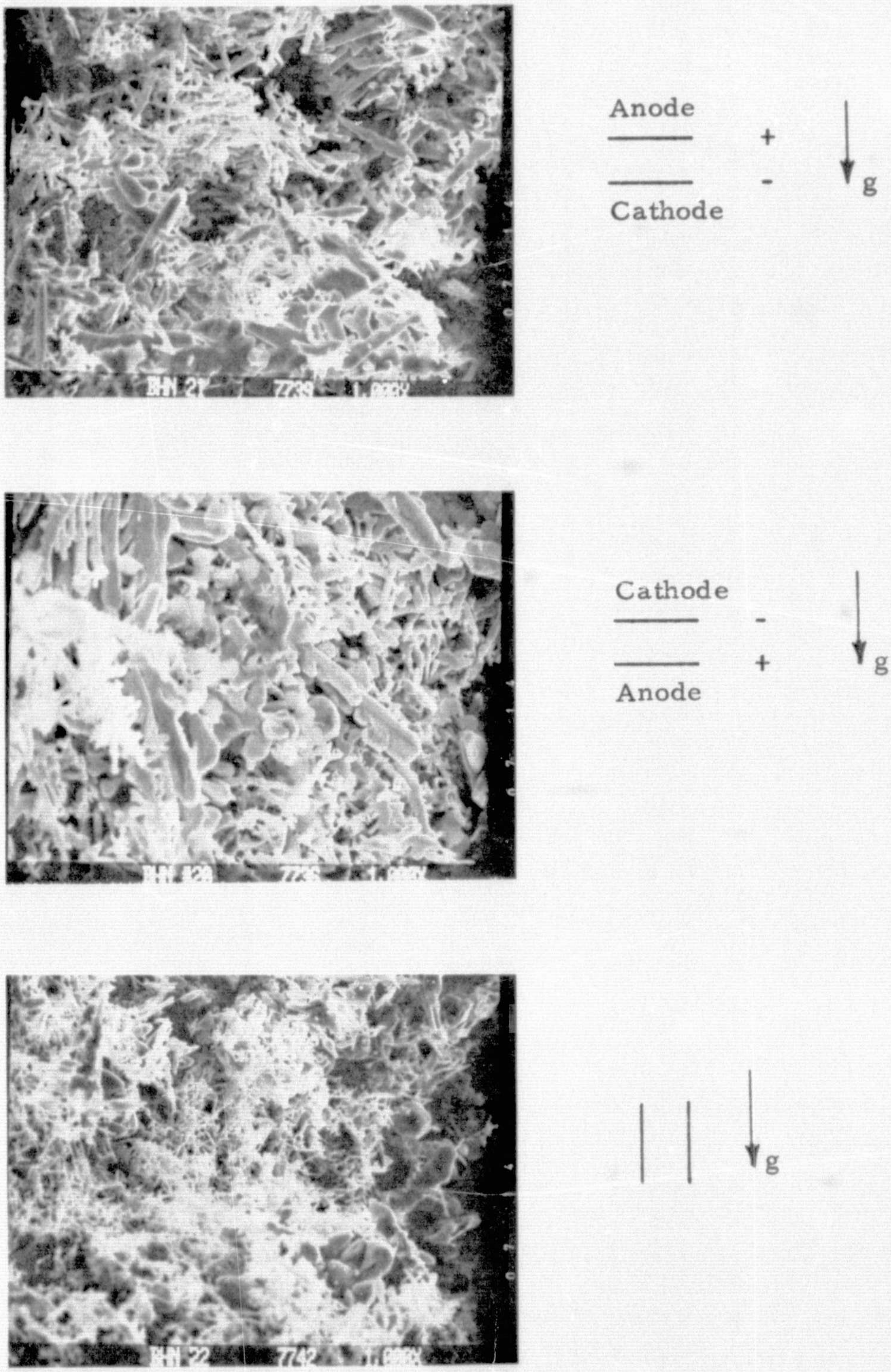


Fig. 10 - Morphologies of Silver Deposits as a Function of Electrode Orientation in Gravity Field (0.24 M $AgNO_3$, 0.88 M $NaNO_3$, 0.06 M HNO_3, 2 V Applied. Mag. x 1000)

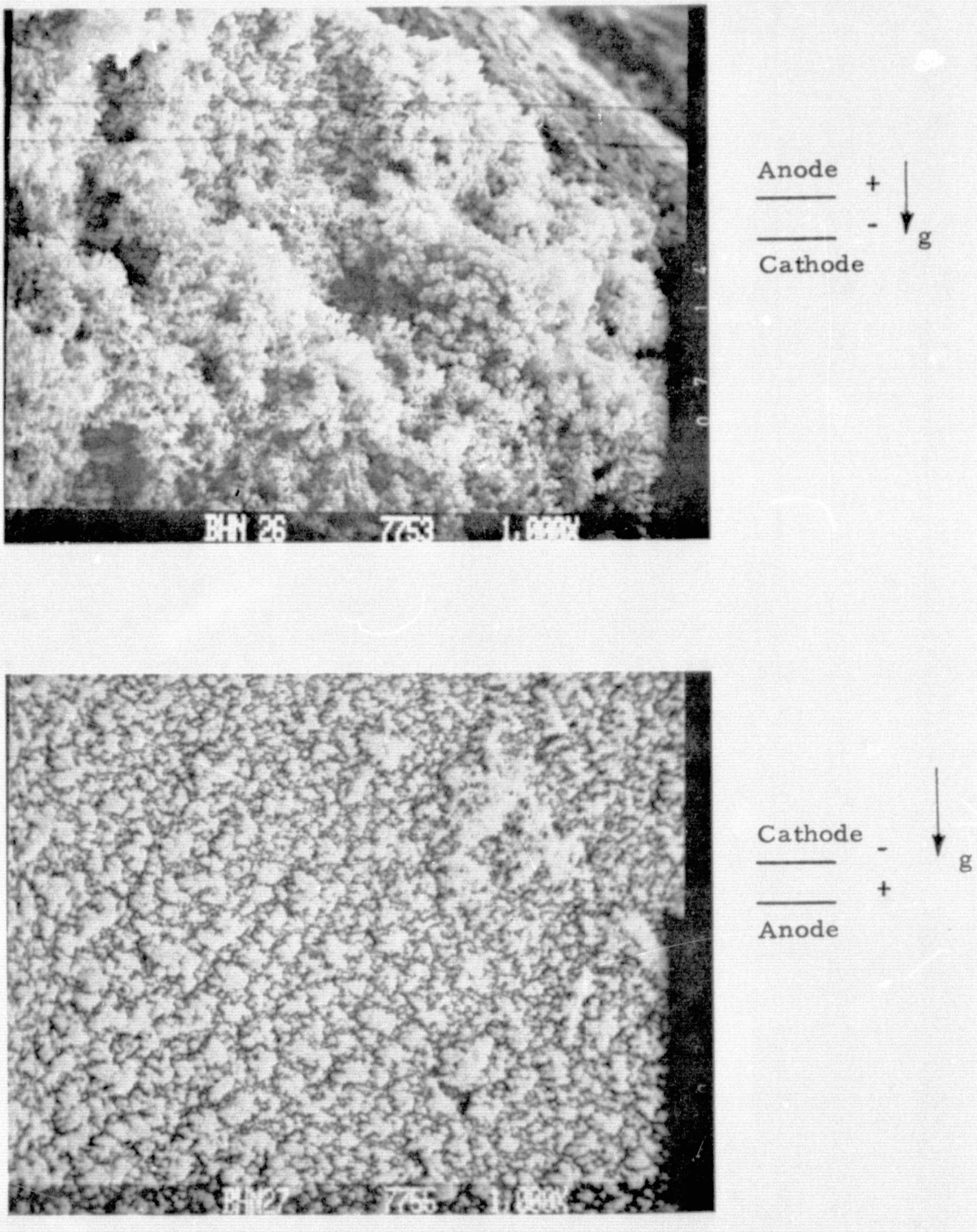


Fig. 11 - Morphologies of Palladium Deposits as a Function of Electrode Orientation in Gravity Field. (0.1 M Pd $C\ell_2$, 1 M NaCℓ, and 2.5 V Applied. Mag. x 1000)

Fig. 12 - Electrodeposit of Platinum. Curved Arms Indicate Convection Influence (Ref. 50) (0.024 M H_2 Pt $C\ell_6$, 0.1 M $HC\ell$, 1 x 10^{-3} M Pb Acetate, Applied Voltate 6.5 V).

As can be seen a change in the orientation of the electrodes with respect to the gravity field results in a different convective situation and so alters the morphologies of the deposits. The particular morphologies observed cannot at this point be correlated with level of convective vigor present during the electrolysis for a number of reasons. The experiments were performed with a very simple apparatus which did not possess the capability of potential or temperature control. Also, in the case of palladium the kinetics and roles of the electron transfer and the hydrogen ion reduction reaction need to be elucidated. Part of the present proposed work will be concerned with establishing a firm correlation of morphology and particle size on deposition potential and level of natural convection. This aspect of the study is discussed in the section on Ground Studies.

It might be added that Auger spectroscopy of the electrodeposited silver and palladium powders showed that they were very pure. Purity is a highly important consideration for catalysis studies and utilization.

Choice of Silver and Palladium as Model Materials

The electrodeposition of silver has been exhaustively studied and is well documented. Further advantages of silver include: The electron exchange reactions are very fast compared to the crystal growth processes. Morphology changes are thus readily correlated with mass transport conditions (Ref. 51). The hydrogen overvoltage is relatively high on silver. Thus generation of hydrogen bubbles is not a problem. Silver is utilized to an appreciable extent for catalyst applications, but its choice as a model material for the present study is primarily for what it will reveal about the crystal growth processes in low-g. It might be noted that the report of the Skylab silver crystal experiment was one of 70 reports chosen from more than 850 reviewed by The Silver Institute as "most likely to affect silver and silver products industries in the near future" (Ref. 52). The possibility of improved catalytic properties was particularly stressed.

Palladium is a more difficult material to deal with theoretically because the electron exchange reactions are not much faster than the crystal growth processes (Ref. 53). Also the hydrogen overvoltage is low on palladium. Thus hydrogen is liberated but fortunately is immediately absorbed by the palladium. From the viewpoint of electrode kinetics palladium is rather typical of the Group VIII metals, all of which have important catalytic applications. The particularly intriguing aspect of palladium electrodeposition in low-g is what the role of local Joulean heating might have on the electrodeposition processes. It is predicted that the local heating will raise the temperature of the solution near the electrode interface much higher in low-g than in one-g because of the absence of convection and thus promote faster electron exchange kinetics and lead to more dendritic deposits. A somewhat similar mechanism was proposed for dendritic deposition of cobalt and nickel from aqueous solutions (Ref. 53).

Addendum

A series of relevant abstracts* from the older literature was discovered after most of the present section was written. In these studies temperature rise at the cathode during electrodeposition of Cd, Cu, and Zn powders were experimentally determined, as was also the effect of stirring. The findings reported give added support to two of the important contentions of the present proposal — appreciable temperature rises occur at the deposition surfaces during powder electrodeposition and stirring increases particle sizes. (Ergo, natural convection increases particle size.)

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*The Temperature Effect on the Cathode During Electrodeposition of Cadmium Powder," D.N. Gritsan and A.M. Bulgakova (A.M. Gor'kii State Univ., Kharkov). Zhur. Fiz. Khim, Vol. 28, pp. 258-261 (1954).

"Effect of Anions on the Temperature Effect at the Cathode During Electrodeposition of Metal Powders," D.N. Gritsan, A.M. Bulgakova, and G.A. Zolotareva (A.M. Gor'kii State Univ., Kharkov). Zhur. Fiz. Khim. Vol. 28, pp. 337-344 (1954).

"Use of Thermobatteries in the Measurement of the Temperature Effect on the Cathode During the Electrodeposition of Metal Powders," D.N. Gritsan, A.M. Bulgakova, and N. Bagrov (A.M. Gor'kii State Univ., Kharkov). Zhur. Fiz. Khim. Vol. 29, pp. 345-349 (1955).

"Cathodic Polarization and Temperature Effect on a Cathode During Electrodeposition of Metal Powders," D.N. Gritsan and A.M. Bulgakova (A.M. Gor'kii State Univ. Kharkov). Zhur. Fiz. Khim. Vol. 29, pp. 450-454 (1955).

"Effect of the Electrolyte Concentration on the Temperature Effect at the Cathode During Electrodeposition of Metal Powders," D.N. Gritsan and A.M. Bulgakova (A.M. Gor'kii State Unit., Kharkov). Zhur. Fiz. Khim. Vol. 29, 649652(1955).

"Effect of Stirring Electrolyte on the Electrolytic Deposition of Powdered Copper," A.V. Pomosov and V.A. Branshtein. Zhur. Priklad. Khim., Vol. 30, pp. 1255-1258 (1957).

d. OBJECTIVES

The general objective of the proposed project is to evaluate the potential of low-g electrodeposition for producing unique silver and palladium catalysts. A more specific objective is to verify the following hypothesis:

- Convection accompanying electrodepositions at high overvoltage is deleterious to the production of surface topographies and particle sizes desirable for efficient catalysts.

e. PLAN OF INVESTIGATION

1. The proposed study will consist of the following elements:

- Prepare fine powders of silver and of palladium metals by electrodeposition at constant potential in low-g (SPAR rocket) and in one-g (ground) and two-g (centrifuge). Electrolyses with the apparatus in different orientations with respect to gravity in one-g will also be conducted.
- Characterize the morphologies, particle sizes, purity, and surface areas of the powders produced by means of SEM microphotographs, Auger spectrography, and an electrochemical technique or a gas adsorption technique or a SEM technique for surface areas.
- Evaluate catalytic activities of the space, ground, centrifuge, and commercial powders by conducting a structure-sensitive electrochemical test reaction.

2. The following primary ground support activities are envisioned:

- Design and construct an electrolysis cell that will allow electrolysis at low-, one-, and two-g at controlled potential and temperature.
- Determine morphologies obtained at various deposition potentials, temperatures, and concentrations in one- and two-g by means of SEM. Interpret data with regard to mechanism of electrodeposition. The electrodeposition of silver is well understood and documented. The electrodeposition of palladium has been less studied. Some questions regarding influences of temperature and concentration, therefore, need to be investigated further.
- Define and develop a method for obtaining surface area values.
- Define and develop a method for determining catalytic activities. It is foreseen that an electrochemical technique will probably be the most convenient.

In addition, some checkout tests of the apparatus and solution concentration on one or two KC-135 flights would be desirable. The design phase of the work will need some thermal analysis to make sure that in low-g, in the absence of convection, local regions of solution do not overheat and produce vapor.

3. Silver and palladium crystals will be electrodeposited in low-g from aqueous solution in especially designed electrochemical cells. During the deposition, current and potential data will be transmitted to the ground. After the flight the cell will be recovered and the deposited crystals removed for structure and catalytic characterizations. Ground tests prior to the flight tests will establish the appropriate current-potential ranges and will also provide data for the assessment of theoretical models.

The electrochemical apparatus will contain three cells in which three separate electrolyses can be conducted simultaneously. The apparatus will be designed to allow electrolysis at essentially controlled-potential. The cells would be reflyable and would readily be dismantled for easy access to the electrodes for removal of low-g deposited product and for refitting and refilling. The cells would also be provided with an automatic on/off switch that would start and stop the electrolysis at the beginning and end of the zero-g period.

The overall dimensions will be on the order of $7 \times 7 \times 21\ \mathrm{cm}^3$ and will weigh (at one-g) about 0.7 kilograms. Probably about 15 to 20 V at about 0.5 to 1.5 A will be required in the way of electrical power. As mentioned previously, telemetry for potential and current during eletrolyses will be required. Telemetry for temperature would also be desirable. A desirable feature on the apparatus would be magnified photography. The photography feature would add very little to the total cost. Considering the value of additional data that could be acquired, however, the photography option appears quite cost effective. Further engineering and design details are given in Appendix D.

The features of the cells proposed for this study are summarized as follows:

Operational Mode:	Constant, controlled electrode potential
Number of Cells in Unit:	Three

Transmitted Data:	Current, applied potential, and temperature
Other Features:	Automatic on/off switch
	Magnified photography of electrodeposition.

4. Three rocket flights and two or three KC-135 flights for checkout purposes are requested. The four month period required by NASA for payload integration and launch will be sufficient for turnaround for the proposed experiments.

5. A number of supporting studies will have to be conducted in conjunction with the cell design phase of the study. Variables such as choice of anode reaction, design of permeable barrier, total applied potential, working electrode potentials, current densities, degree of heating in electrolytic solution, and electrolytic concentrations will have to be studied and defined to ensure that the cells will operate without overheating or bubble generation and also to ensure that sufficient product is produced in the 5 to 6 minutes available for processing.

As indicated previously, the ground and flight data will be correlated with theoretical models. Also the returned crystals will be characterized by the instrumental means initiated previously. In addition, the crystals will be tested for catalytic effectiveness in the laboratory. Part of the supporting studies will be concerned with developing a suitable electrochemical reaction as a test of catalytic effectiveness.

f. EXPECTED RESULTS

It is expected that smaller silver and palladium crystals of more perfect micromorphologies will be produced in the low-g environment of the SPAR rockets. These unique low-g crystals, it is expected, will have enhanced catalytic properties, as will be demonstrated by a test reaction. The minimal acceptable criteria for the proposed study is that: the experiments be conducted as planned, i.e., no deterioriation of solutions, loose connections, leakage, air bubbles, etc.; and that the sizes and microcrystalline sizes actually obtained show some improvement of catalytic activity that can be correlated with the reduced convective environment of the SPAR rocket.

g. KEY PERSONNEL

Summary

The overall manning plan is indicated in Chart 1 on the next page. Detailed resumes, including addresses, education and experience of all personnel involved in the proposed program, are presented in the subsection following this summary.

Dr. Philomena G. Grodzka, the proposed Principal Investigator is well qualified to direct this study, having performed her doctoral thesis work in the area of electrochemical reactions. She has conducted a number of contracted studies in conjunction with the Space Processing Program on the nature of convection in low-g environments and has a number of papers in the area (shown in resume). She has acquired a broad background in materials stemming from a number of studies in electroanalytical chemistry, ablation, energy storage and conversion, and shock wave/materials interaction. Dr. Grodzka also has a broad experience in space experimentation, having been the Project Scientist for the successful Apollo 14 Heat Flow and Convection Experiments, and Co-Principal Investigator for the equally successful Apollo 17 Heat Flow and Convection Experiments. In addition, she was Co-Principal Investigator for the Skylab TV 106 Science Demonstration Deposition of Silver Crystals which is discussed in some detail in the preceding sections and a Co-Principal Investigator for the Science Demonstration Chemical Foams which was conducted aboard Apollo-Soyuz.

Professor G. A. Somorjai, who will provide general guidance and supervise the catalyst characterization studies under subcontract, is internationally recognized as an authority on surface science and mechanisms involved in catalysis.

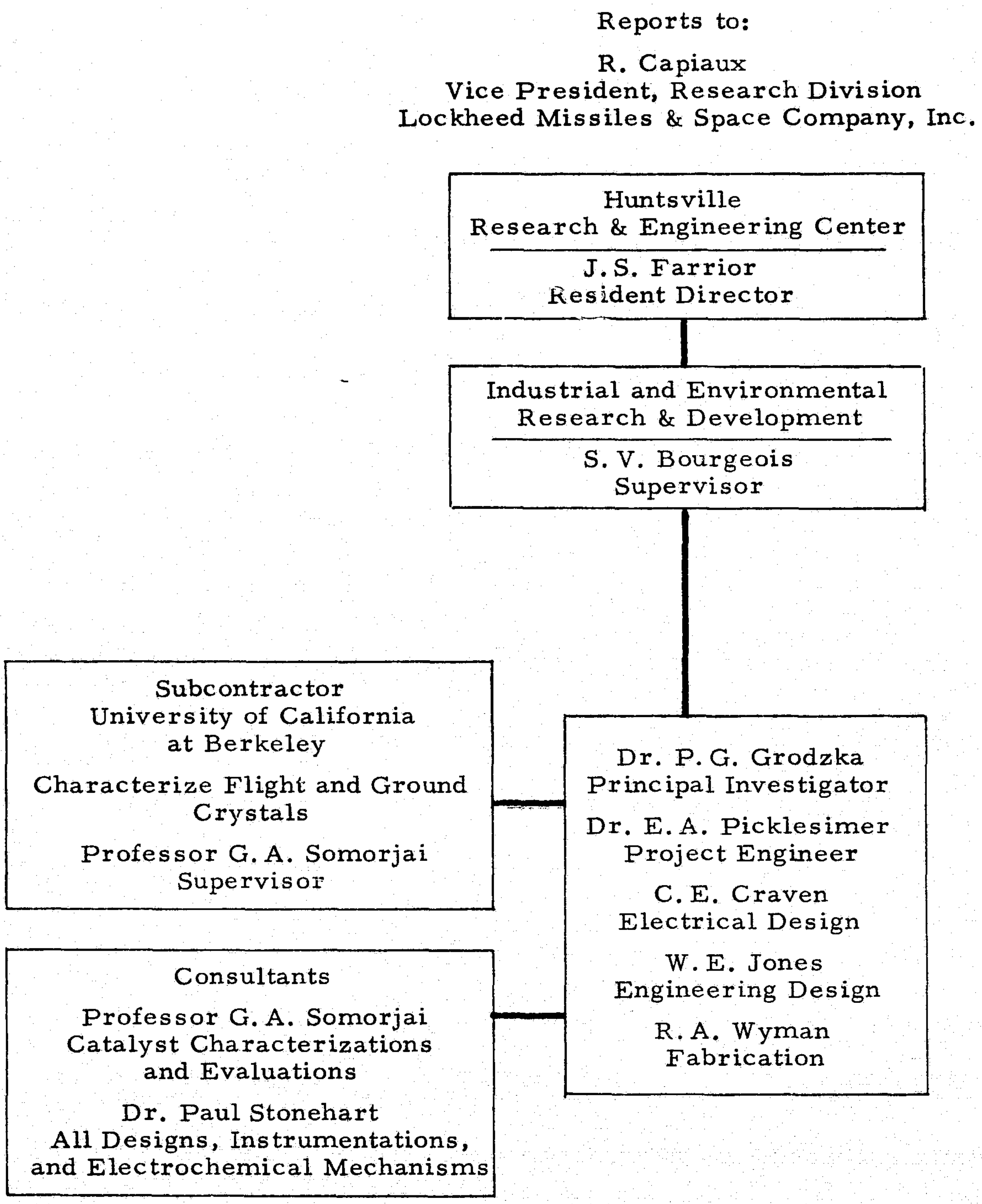

Chart 1 - Organizational Relationship for Proposed Study

Dr. Paul Stonehart, who will provide expert guidance and advice on test cell design, special instrumentation, and electrochemical mechanisms, is also internationally recognized as an authority on electrochemical reactions, electrochemical instrumentation, and electrocatalysis.

Dr. E.A. Picklesimer, who will assist Dr. Grodzka with the hardware fabrication portion of the study, is also well qualified. Dr. Picklesimer's background is in the area of mechanical engineering. He is also experienced in fluid mechanics and he has conducted a number of studies in the area of Space Processing. He is currently working on Space Processing programs involving cryogenic energy storage and electrodeposition in zero-g environment.

Mr. Charles E. Craven who will be responsible for the electrical designs, has been engaged for approximately 12 years in the design and testing of electronic systems for special applications.

Mr. William E. Jones who will be responsible for the engineering designs has for several years participated in the design of many and varied systems ranging from aircraft and missiles to the design of very accurate models used in testing reentry systems. Mr. Jones is ideally suited to provide the design support for the apparatus required for this program.

Mr. R.A. Wyman who will supervise the equipment construction has more than 20 years experience in the design, fabrication, and testing of many types of electrical and mechanical devices through prototype and production. He is presently Supervisor of the Development Laboratory at Lockheed-Huntsville. Thus he is responsible for the operation of machine shops, electronic assembly operation and model shop. He, therefore, is extremely well qualified to provide the fabrication support required for this program.

Resumes

J.S. Farrior
S.V. Bourgeois
P.G. Grodzka
G.A. Somorjai
Paul Stonehart
E.A. Picklesimer
C.E. Craven
W.E. Jones
R.A. Wyman

JAMES S. FARRIOR Resident Director

Education

Graduate Studies, Mathematics, Physics, Engineering, 1951-52, University of Alabama in Huntsville; Engineering, 1960, Stanford University; Summer Courses in Management and Technical Fields, 1957, 1960 and 1965, Massachusetts Institute of Technology; Institute for Management, 1968, Northwestern University

B.S.E.E., Communications, 1949, Auburn University

Experience

1963 to present - LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center. Resident Director. Established new LMSC facility; initiated staffing, facilities construction and organization to provide research, engineering, prototype fabrication and testing of flight hardware and support equipment for Government programs. Recruited managers and specialized staffs to perform analytical and experimental studies in aerospace electronics, guidance and controls, flight mechanics, aerodynamics, thermodynamics, systems engineering, and related fields. Secured contracts and exercised general management control efforts of scientific and engineering departments and supporting organizations. Established management liaison with other aerospace contractors in the Huntsville area, and projected facility and capability achievement plans based on existing and planned local competition, anticipated customer requirements and Lockheed interests.

1959 - 62 LOCKHEED MISSILES & SPACE COMPANY, Sunnyvale, Calif.
1961 - 62 Associate Director for Dynamics and Navigation, Research and Engineering. Responsible for management of organizations totaling about 400 people engaged in research, development, design, and prototype fabrication in connection with the Polaris and Agena programs. Responsibilities included analytical work such as trajectory and performance analysis, guidance and control equations, and preflight and postflight analyses, as well as technical and administrative control of numerous subcontractors to fulfill LMSC contractual obligations for their performance.
1959 - 60 Manager, Guidance Department, Polaris Missile System. Responsible for integration of the guidance system into the Polaris missile system.

1951 - 59 Army Ballistic Missile Agency, Redstone Arsenal, Alabama. Chief, Navigation Branch. Responsible for the development of guidance and control systems for the Redstone, Jupiter and Pershing missiles. Also responsible for the development of guidance and control techniques for space projects such as Jupiter C and Saturn.

1949 - 51 Auburn University, Auburn, Alabama. Instructor, Electrical Engineering Department.

Activities

Chairman of the Alabama Section of AIAA
Awarded Gold Knight of Management, 1968, National Management Association

S. V. BOURGEOIS, JR. Scientist Associate-Research

Education

Ph.D., Chemical Engineering, 1971, Louisiana State University. (Dissertation: Investigated a packed bed reactor involving the irreversible reactions of gaseous components with a metal distributed throughout the solid packing material — flue gas desulfurization.)

M.S., Chemical Engineering, 1970, Louisiana State University

B. S., Chemical Engineering, 1968, Louisiana State University

Experience

1971 - Present LOCKHEED MISSILES & SPACE COMPANY, Inc. Huntsville Research & Engineering Center. Supervisor, Industrial and Environmental R&E Section. Responsible for feasibility analysis of processes under NASA's Space Processing Program. Performing analysis of natural convection of fluids in space flight experiments. Also serving as task order manager on support services contract to provide quick reaction engineering in the area of pollution control technology to EPA's Industrial Environmental Research Laboratory.

1970 - 71 Louisiana State University, Baton Rouge, Chemical Engineering Dept. Graduate Assistant. Instructed courses in the undergraduate curriculum and performed dissertation research in power plant flue gas desulfurization.

1970 Ethyl Corporation, Baton Rouge. Research and Development Dept., Development Engineer. Responsible for long-range process evaluation projects. Processes studies include the mining and later reduction of metallic ores (bauxite and ilmenite).

1969 Esso Research Laboratories, Baton Rouge. Assistant Research Engineer. Conducted pilot plant studies involving simultaneous kinetics and transport phenomena in fixed-bed chemical reactor and related math modeling. This process research aimed at flue gas desulfurization.

1967 - 68 Shell Oil Company, Norco, La. Technical Services Dept., Assistant Process Engineer (Summers). Did debottlenecking studies of crude oil distillation plant; reactivated crude oil distillation unit; performed troubleshooting assignments.

Societies

Sigma Xi; Tau Beta Pi; Phi Kappa Phi; Phi Lambda Upsilon; American Institute of Chemical Engineers, National Management Association, and American Institute of Aeronautics and Astronautics.

Honors

Selected to Outstanding Young Men of America for 1975, 1st Place Short Story in 1965 Louisiana College Writer's Society Contest.

Publications and Reports

"Thermocapillary Convection in Microgravity Crystal Growth Melts of Indium-Antimonide," with L. W. Spradley, Lett. Heat Mass Trans., Vol. 3, No. 3, May-June 1976, pp. 193-204.

"Development Status and Environmental Hazards of Several Candidate Advanced Energy Systems," with M.M. Penny, EPA/IERL-Ci-092, February 1975.

"Buoyant and Capillary Natural Convection in Infinite Horizontal Liquid Layers Heated Laterally, Lett. Heat Mass Trans., May-June 1975, Vol. 2, p. 223.

"Liquid Drop Behavior in Weightlessness from Skylab," with T.C. Bannister, The Science of Liquid Drops and Bubbles, D. J. Collins, ed., Jet Propulsion Laboratory, Pasadena, Calif., 1975.

"Space Processing Convection Evaluation: G-Jitter Convection of Confined Fluids in Low Gravity," with L.W. Spradley and F.N. Lin, to be presented at AIAA 10th Thermophysics Conference, Denver, May 1975.

"Analysis of Skylab IV Fluid Mechanic Science Demonstrations," with M.G. Klett, to be presented at AIAA 10th Thermophysics Conference, Denver, May 1975.

"Analysis of Liquid Dynamics for M551 and M553 Skylab Space Processing Experiments," with M.R. Brashears and C. Fan, presented at International Colloquium on Drops and Bubbles, California Institute of Technology, Pasadena, August 1974.

"Fluid Motions in a Low-G Environment," with P.G. Grodzka, M.R. Brashears and L.W. Spradley, Third Space Processing Symposium: Skylab Results, NASA-Marshall Space Flight Center, Huntsville, Ala., 1 May 1974.

"Fluid Dynamics and Kinematics of Molten Metals in the Low-Gravity and Environment of Skylab," with M.R. Brashears, AIAA Paper 74-205, AIAA 12th Aerospace Sciences Meeting, Washington, D.C., 1 February 1974.

"Analysis of Fixed Bed Sorption Flue Gas Desulfurization," with F.R. Groves and A.H. Wehe, AIChE J., Vol. 20, No. 1, January 1974, pp. 94-103.

"A Numerical Solution for Thermoacoustic Convection of Fluids in Low Gravity," with L.W. Spradley, P.G. Grodzka and C. Fan, NASA CR-2269, May 1973.

"Analysis of Fixed Bed Reactor Involving Multiple Solid-Fluid Reactions: Glue Gas Desulfurization," with F.R. Groves and A.H. Wehe, presented as Paper 30b, 74th National AIChE Meeting, New Orleans, 13 March 1973.

Company Reports

"Convection Sensitivity and Thermal Analyses for Indium and Indium-Lead Mixing Experiment (74-18)," with J.P. Doty, LMSC-TR D496846, May 1976.

"Skylab M518 Multipurpose Furnace Convection Analysis," with L. W. Spradley, LMSC-HREC TR D496534, September 1975.

"Space Processing Applications Convection Evaluation," with J. H. McDermit and L. W. Spradley, LMSC-HREC TR D390825, May 1975.

"Analysis of Skylab IV Fluid Mechanic Science Demonstrations," with M. G. Klett, LMSC-HREC TM D390362, September 1974.

"Physical Forces Influencing Skylab Experiments M551, M552 and M553; Summary Report," LMSC-HREC TR D390056, January 1974.

"Solidification Theory for Skylab Experiments M551, M552 and M553: Summary Report," LMSC-HREC TR D390140, January 1974.

"Survey of Effects of Combustion Modifications on Particulate Emissions," with S. J. Robertson, LMSC-HREC TR D390900, October 1973.

"Apollo 17 Heat Flow and Convection Experiments Final Data Analyses Results," with T. C. Bannister, P. G. Grodzka, L. W. Spradley, R. O. Hedden and B. R. Facemire, NASA TM X 64772, 16 July 1973.

"Fluid and Particle Dynamic Effects in Low-G Composite Casting," with P. G. Grodzka, LMSC-HREC TR D306402, February 1973.

"A Numerical Solution for Thermoacoustic Convection of Fluids in Low Gravity," LMSC-HREC TR D306140, January 1973.

"Convection in Space Processing (M512) – Phase A Report," with P. G. Grodzka, LMSC-HREC D306065, July 1972.

"Analysis of Parameters for Molten Braze Allow Flow in the Skylab M552 Experiment," LMSC-HREC TN D306111, 25 August 1972.

"Environmental Conditions and Physical Properties for Skylab Experiments M551, M552, M553 and M554," LMSC-HREC TN D306001, July 1972.

"Report on Phase A Progress for Skylab Experiments M551, M552, M553 and M554," with P. G. Grodzka, LMSC-HREC TN D306014, June 1972.

"Recommendations for KC-135 Aircraft Testing (M553)," LMSC-HREC TN D225983, June 1972.

"Physical Forces and Solidification Theory for Skylab Experiments M551, M552, M553 and M554," LMSC-HREC TN D225975, June 1972.

PHILOMENA G. GRODZKA Staff Scientist

Education

Ph.D., Analytical Chemistry 1961, University of Michigan

M.S., Physical Chemistry, 1953, Wayne University

B.S., Chemistry, 1951, Wayne University

Experience

1965 - Present LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center. Space Processing Group.

1968 - 77 Co-Principal Investigator on Chemical Foams Demonstration Experiments conducted aboard Apollo-Soyuz. Co-Principal Investigator on Skylab Silver Crystal Growth and Surface Tension Effects Science Demonstrations and on Apollo 17 Heat Flow and Convection Demonstration Experiments. Project Scientist on Apollo 14 Heat Flow and Convection Demonstration.

Provided consultation for Skylab M512 experiments. Research on natural convection as a function of gravity level and new concepts for thermal energy storage. Directed and conducted studies of ion beam processing, crystal growth, chemical syntheses, and separation processes in space environments.

1965 - 68 Defined high heats of fusion materials for use in thermal control devices. Conducted studies of high pressure equations of state and thermodynamics of solids. Also conducted studies in the areas of reacting gas flows, ablation, and frozen free radicals.

1962 - 64 Avco Corporation, Wilmington, Mass. Staff Scientist. Directed a study of characteristics of charred Apollo heat shield material and other materials. Performed research on formulation of magnetic fluids. Coordinated the collection and evaluation of data pertaining to carbon cloth characterization. Analyzed instrumentation and measurement requirements for a space vehicle to be used as a cometary probe.

1959 - 61 Smith College, Northampton, Mass. Instructor. Taught undergraduate general, analytical and physical chemistry, and graduate instrumental and physical chemistry. Directed graduate student research on hydrolysis mechanism of the compound thenoyltrifluoracetone.

1953 - 59 University of Michigan, Ann Arbor. Research Assistant and Teaching Fellow. Developed analytical procedures for inorganic ions in solution. Carried out research in the field of organic polarography. Performed doctoral research on the mechanism of electrochemical reduction of conjugate double-bond systems. Taught a number of analytical chemistry laboratory courses.

1952 - 53 Wayne University, Detroit. Instrumental Methods Laboratory. Teaching fellow during masters degree work.

1951 - 52 Nelson Chemical Co., Detroit. Chemist. Conducted product evaluation and development of testing procedures for fabrics, dyes and surface active agents.

Publications

"Thermal and Solutal Convection and Dendritic Solidification," with M. H. Johnston and C. Griner, to appear in AIAA J., November 1977.

"Process Chemistry for L5," Space Manufacturing Facilities, Editor Jerry Grey, published by AIAA, 1 March 1977.

"Natural Convection in Low-g Environments," with T. C. Bannister, AIAA Progress in Astronautics and Aeronautics, Vol. 52, Editor Leo Steg, 1977.

"Clusius-Dickel Separations (CDS): A New Look at an Old Technique," with B. R. Facemire, Separation Science, Vol. 12, No. 2, 1977.

Contributed section on Space Processing in the AIAA publication "Using the Space Environment: an AIAA Review," 1976.

"Electrochemical Deposition of Silver Crystals Aboard Skylab IV," with B. R. Facemire, M. H. Johnston and D. W. Gates, J. Crystal Growth, Vol. 35, 1976, pp. 177-184.

"Tracking Transient Temperatures with Liquid Crystals," with B. R. Facemire, Letters in Heat and Mass Transfer, Vol. 2, 1975, pp. 169-178.

"Heat Flow and Convection Experiments Aboard Apollo 17," with T. C. Bannister, Science, Vol. 187, 1975, pp. 165-167.

"Selection of Solid-Liquid Phase-Change Materials for Spacecraft Thermal Control," Progress in Astronautics and Aeronautics, Vol. 24, 1972, pp. 547-565.

"Heat Flow and Convection Demonstration Aboard Apollo 14," with T. C. Bannister, Science, Vol. 176, 1972, pp. 506-508.

"Polarographic Reduction of the Phenyl-Substituted Ethenes I. Relation of Postulated Mechanisms to Theoretical Behavior Patterns," with P. J. Elving, J. Electrochem. Soc., Vol. 110, 1963, pp. 225-230.

"Polarographic Reduction of the Phenyl-Substituted Ethenes II. Electrochemical Kinetic Parameters and Mechanism in Dimethylformaide," with P. J. Elving, J. Electrochem. Soc., Vol. 110, 1963, pp. 231-236.

"Thenoyltrifluoracetone: Polarographic and Spectrophotometric Behavior and Dissociation Equilibria, with P. J. Elving, Anal. Chem., Vol. 33, No. 2, 1961.

"The Effect of Temperature on the Density and Surface Tension of Monofluoroacetic Acid," with J. J. Jasper. J. Am. Chem. Soc., Vol. 76, 1954, p. 1453.

Papers Presented at Meetings

"Expanding NASA's Charter to Facilitate Space Utilization," with J. A. Tevepaugh, The Industrialization of Space, American Astronautical Society 23rd Annual Meeting, San Francisco, Calif., 18-20 October 1977

"Cryogenic Temperature Control by Means of Energy Storage Materials," with E. Picklesimer and L. E. Connor, AIAA 12th Thermophysics Conference, Albuquerque, N. M., 27-29 June 1977.

"Convection and Dendritic Crystallization," with M. H. Johnston and C. Griner, AIAA 15th Aerospace Sciences Meeting, Los Angeles, 24-26 January 1977.

"A Space Utilization Authority (SUA) Could be the TVA of the Future," The Future Society Conference, Southeast 2001: The Next 25 Years, 12-13 November 1976, Urban Life Center of the Georgia State University, Atlanta.

"Extraterrestrial Mining and Industrial Processing," with R. L. Nichols, AIAA/MSFC Symposium on Space Industrialization, 26-27 May 1976, Marshall Space Flight Center, Ala.

"Processing Lunar Soil for Structural Materials," Special Session of the Seventh Lunar Science Conference entitled "Utilization of Lunar Materials and Expertise for Large Scale Operations in Space," 16 March 1976, Johnson Space Center, Texas.

"Three Model Space Experiments on Chemical Reactions," with B. R. Facemire, Colloquium on Bioprocessing in Space, 10-12 March 1976, sponsored by NASA-Johnson Space Center, Houston, Texas.

"Electrochemical Deposition of Silver Crystals Aboard Skylab IV," with B. R. Facemire and M. H. Johnston. Third American Conference on Crystal Growth, American Association for Crystal Growth, Stanford University, 13-17 July 1975.

"Process Chemistry for L_5," The Princeton University Conference on Space Manufacturing Facilities," Princeton University, 7-9 May 1975.

"Some Practical Aspects of Thermal Energy Storage," Workshop on Solar Energy Storage Subsystems for the Heating and Cooling of Buildings, Co-sponsored by NSF and ERDA, 16-18 April 1975, Charlottesville, Va.

"Highlights in Heat Pipes and Space Processing," with R. Kosson, AIAA 11th Annual Meeting and Technical Display, Washington, D. C., 24-25 February 1975.

"Fluid Motion in a Low-G Environment," with S. V. Bourgeois, M. R. Brashears and L. W. Spradley, Third Space Processing Symposium, Marshall Space Flight Center, Ala., 30 April - 1 May 1974.

"Natural Convection in Low-G Environments," with T. C. Bannister, AIAA Paper No. 74-156, AIAA 12th Aerospace Sciences Meeting, Washington, D. C., 30 January - 1 February 1974.

"Heat Flow and Convection Demonstration Experiments Aboard Apollo 14 and Apollo 17," with T. C. Bannister, XXIVth Congress of the International Astronautical Federation, Baku, U.S.R.R., October 1973.

"Melting Freezing Phase-Change Materials Systems: Report on Current Research and Development," AIAA 6th Thermophysics Specialists Conference, Open Forum Session, University of Tennessee Space Institute, Tullahoma, 26 April 1971.

"Surface Tension Convection During Float-Zone Refining," with T. C. Bannister, AIAA Paper 70-12, 4th International Conference on Electron and Ion Beam Science and Technology, 137th Annual Meeting of the Electrochemical Society, Los Angeles, 10-15 May 1970.

"Spacecraft Thermal Control by Use of Solid-Liquid Phase-Change Materials," AIAA 8th Aerospace Sciences Meeting, New York City, 19-21 January 1970.

"Gravity-Driven and Surface Tension-Driven Convection in Single Crystal Growth," Space Processing and Manufacturing Meeting, NASA-Marshall Space Flight Center, Ala., 21 October 1969.

"Thenoyltrifluoroacetone: Polarographic and Spectrophotometric Behavior and Dissociation Equilibrium: Mechanisms of Electrochemical Reduction," American Chemical Society 137th National Meeting, Cleveland, Ohio, 11-14 April 1960.

Invited Talks and Lectures

Speaker for the Space Processing Session in the Space Institute of the University of Tennessee Short Course on "Space Shuttle," 16 December 1976, Tullahoma, Tenn.

Lecture on "Exotic Energy Storage Techniques," Solar Heating and Cooling Course at the University of Alabama, Huntsville, 11 October 1976.

"Electrodeposition of Silver Crystals Aboard Skylab IV," National Bureau of Standards, Gaithersburg, Md., 29 April 1975.

"Some Science and Technology and Technology Aspects of Space Manufacturing," Graduate Seminar, Department of Mechanical and Industrial Engineering, University of Illinois at Urbana, 28 November 1973.

Awards

1977 Aerospace Engineer of the Year, presented by the Alabama Section of the AIAA.

1977 Outstanding Service Award, AIAA.

1975 Group Achievement Award for ASTP experiments, NASA-Johnson Space Center.

1975 Lockheed Missiles & Space Company Publication Award for "Heat Flow and Convection Experiments Aboard Apollo 17" paper.

1973 Lockheed Missiles & Space Company Publication Award for "Heat Flow and Convection Experiments Aboard Apollo 14" paper.

1971 Lockheed Missiles & Space Company Publication Award for "Selection of Solid-Liquid Phase-Change Materials for Spacecraft Thermal Control" paper.

1968 Award of Honor in recognition of extraordinary services and leadership. Lockheed Management Association (affiliated with The National Management Association).

Patents

"Energy Efficient Regenerative Liquid Dessicant Drying Process," S. M. Ko, P. G. Grodzka and P. O. McCormick, Patent application filed 7 June 1977, Lockheed Disclosure D-0306581 (ERDA Docket S-48, 492).

Professional Societies

Director, Alabama Chapter AIAA, 1977-79.

Member AIAA Technical Committee for Space Processing, 1977.

Organizer and Chairman of the AIAA Technical Committee for Space Processing, Term 1975-76.

Member of the AIAA Technical Committee for Thermophysics, Term 1972-74.

Organizer and Chairman of the Solar Energy Applications Session of the AIAA Eleventh Thermophysics Conference, San Diego, 15 July 1976.

Technical Program Chairman of the AIAA 10th Thermophysics Conference, Denver, 27-29 May 1975.

Organizer of two Space Processing Sessions, Chairman of one, at the AIAA/ASME Thermophysics and Heat Transfer Conference, Boston, 15-17 July 1974.

Organizer of two Space Processing Sessions, Chairman of one, at the AIAA 12th Aerospace Sciences Meeting, 30 January - 1 February 1974, Washington, D. C.

Secretary, 1967-68, Lockheed Management Club of the National Management Association.

Program Chairman, 1964-65, Analytical Chemistry Group of the Northeastern Section of the American Chemical Society.

Secretary, 1963-64, Analytical Chemistry Group of the Northeastern Section of the American Chemical Society.

Member; American Institute of Aeronautics and Astronautics (AIAA), American Chemical Society (ACS), Sigma Xi, North American Thermal Analysis Society, New England Association of Chemistry Teachers, American Association for the Advancement of Science, National Management Association, The World Future Society, The L-5 Society.

Other Activities

Member of an evaluation team for the Alabama A&M University project, "A Comprehensive Institutional Program to Initiate, Support, and Improve the Quality and Effectiveness of Research Among Young Scientists," sponsored by the National Science Foundation, December 1976.

Testimony on Space Processing before the U. S. Senate Subcommittee on Aerospace Technology and National Needs of the Committee on Aeronautical and Space Sciences, Washington, D. C., 17 June 1976.

Participant in the ERDA Workshop on Advanced Thermal Energy Storage Technologies for Solar Applications, Baton Rouge, 23-24 April 1976.

Reports

"Space Processing Applications of Ion Beam Technology," LMSC-HREC TR D497370, 15 August 1977.

"Thermal Energy Storage — Second Interim Report," with L.E. Connor, LMSC-HREC TR D497048, 29 October 1976.

"Electrochemical Deposition of Silver Crystals Aboard Skylab IV," with B. Facemire, M.H. Johnston and D.W. Gates, NASA TN D-8277, July 1976.

"Thermal and Convection Analyses of the Dendrite Remelting Rocket Experiment; Experiment 74-21 in the Space Processing Rocket Program — Final Report," with J.E. Pond and L.W. Spradley, LMSC-HREC TR D496848, 31 May 1976.

"Thermal Energy Storage — Interim Report," LMSC-HREC TR D496600, November 1975.

"Clusius-Dickel Separations (CDS): A New Look at an Old Technique," LMSC-HREC TR D496521, September 1975.

"Apollo 17 Astronaut/Ground Control Conversations Relevant to Space Processing," with R. Onyeabo, LMSC-HREC TM D390139, March 1974.

"Apollo 17 Heat Flow and Convection Experiments: Final Data Analyses Results," with T.C. Bannister, L.W. Spradley, S.V. Bourgeois, R.O. Hedden and B.R. Facemire, NASA TM X-64772, 16 July 1973.

"A Numerical Solution for Thermoacoustic Convection of Fluids in Low Gravity," with L.W. Spradley, C. Fan and S.V. Bourgeois, NASA CR-2269, May 1973.

"Fluid and Particle Dynamic Effects in Low-G Composite Casting," with S.V. Bourgeois, LMSC-HREC TR D306402, January 1973.

"Types of Natural Convection in Space Manufacturing Processes: Summary Report," LMSC-HREC TR D306350, January 1973.

"Convection in Space Processing (M512) — Phase A Report," with S.V. Bourgeois, LMSC-HREC D306065, July 1972.

"Space Thermal Control Development — Final Report," with M.J. Hoover and M.J. O'Neill, LMSC-HREC D225500, December 1971.

"The Apollo 14 Heat Flow and Convection Demonstration Experiments," with C. Fan and R. O. Hedden, LMSC-HREC D225333, September 1971.

"Natural Convection in Space Manufacturing Processes — Interim Report," with C. Fan, LMSC-HREC D162926, July 1971.

"Mass Diffusion of Gallium Arsenide in a Gallium Solution," with C. Fan, LMSC-HREC D225149, June 1971.

"Zero-Gravity Solidification," LMSC-HREC D148619B, March 1970.

"Space Thermal Control by Freezing and Melting: Second Interim Report on Space Thermal Control Study," LMSC-HREC D148619A, May 1969.

"Thermal Control by Freezing and Melting: Interim Report on Space Thermal Control Study," with C. Fan, LMSC-HREC A791342, March 1968.

"Equation of State Study," LMSC-HREC A784903, Technical Brief, December 1967.

"Grüneisen Parameter Study, Final Report," LMSC-HREC A784868, October 1967.

"Grüneisen Parameter Study, Interim Report," LMSC-HREC A784074, March 1967.

"Physical and Chemical Models of Soot Formation in Rocket Engine Combustion," Technical Brief, LMSC-HREC A783071, August 1966.

"Effects of Chemically Reactive Gaseous Products of Ablation on Heat Transfer to a Charring Ablator," Technical Brief, LMSC-HREC A712400, October 1965.

"A Theoretical-Experimental Study of Moon Volcanoes," Technical Brief, LMSC-HREC A712107, October 1965.

"Study of the Sub-Surface Reaction Between Silica and Graphite During Ablation," Technical Brief, LMSC-HREC A710938, July 1965.

"Apollo Heat Shield Ablation: Thermal Degradation Studies (U)," Avco TR RAD210-5-64-194, 1 December 1964 (Confidential).

"Char Characterization for Elevated Temperature Conductivity Measurements," Avco TR RAD210-5-64-193, 27 November 1964 (Confidential).

"Chemical Characterization of Avcoat 5026-39HC and Its Constituents," with T.S. Light, J.S. Perkins and B.W. Burrell, Avco TR R210-5-64-10, 16 April 1964.

"Creation of a Magnetic Fluid: Report of Present Status and Proposed Future Work on Two Promising Fluids," Avco Memo R260-4-64-48, 12 March 1964.

"Determination of Chlorine in Organic Compounds," Avco Procedure No. C1-2, 18 April 1963.

"Elemental Analysis of Char Layers from Samples along Cylinder (MK 4, Mod. 5B, SN-26)," Avco TR R210-5-62-89, 5 October 1962.

"Purification of Harcure A," Avco TR R210-5-62-70, 21 June 1962.

"Determination of Analytical Procedures for Iron, Chromium, Sulphate and Nitrate in a Mixed Inorganic Salt Solution," Engineering Research Institute, University of Michigan, Ann Arbor, Reports 2121:1-4-T-5, -5-T, -7-T, and -8-T, February 1954, Atomic Energy Commission Contract No. AT (10-1)-773.

EDISON A. PICKLESIMER Research Specialist

Education

Ph.D., Engineering Science and Mechanics, 1969, University of Tennessee

M.S.M.E., 1966, Georgia Institute of Technology

B.M.E., 1965, Georgia Institute of Technology

Experience

1976 - Present LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center. Industrial and Environmental R&E Section, Lead Engineer. Performed laboratory and theoretical analyses of cryogenic phase change materials. Currently working on ground based research for the electrodeposition of catalytic materials. Development of KC-135 aircraft, low-g flight test hardware and procedures for electrodeposition of Ag, Ni and Pd are presently underway.

1973 - 75 Carolina Power & Light Company, Raliegh, N.C. Project Engineer. Responsible for the construction of meteorological towers, and collection, reduction and evaluation of meteorological data for the operation of nuclear power plants as required by the National Regulatory Commission. Developed analytical models used to estimate atmospheric dispersion, concentration-to-source ratio estimates, and to perform frequency analysis of meteorological data. Responsible for the analysis of hydrological and geophysical data used to select site for future nuclear and fossil fuel power plants. Performed preliminary design work on evaporative cooling systems for nuclear power plant heat dissipation systems. Developed computer programs simulating the performance of fossil fueled power plants as a function of varying coal characteristics.

1971 - 73 Oak Ridge National Laboratory, Oak Ridge, Tennessee. Thermal Hydraulics Consultant. Developed finite difference models for predicting atmospheric dispersion of effluents from evaporative cooling towers serving large nuclear power plants. Evaluated cooling systems serving nuclear power plants and prepared heat dissipation sections of Environmental Impact Statements on Nuclear Power Plants for the National Regulatory Commission.

1969 - 71 General Motors Research Laboratories, Warren, Mic. Senior Research Engineer. Responsible for the research and development of static and dynamic sealing devices (face seals, lip seals, "O" rings and packing) for automotive applications.

1967 - 69 University of Tennessee, Department of Mechanical Engineering, Instructor. Taught basic courses in Mechanical Engineering Department. Performed basic research in buffered bushing seals for turbine applications in Boiling Water Reactors under contract to DoD.

1967 LOCKHEED AIRCRAFT COMPANY, Marietta, Ga. Propulsion Engineer. Designed inter-lock mechanism for cargo bed on C5A aircraft. Performed inlet test studies on VTOL aircraft to measure pressure distorsions in the inlets of lift engines.

1965 - 67 Georgia Institute of Technology, Atlanta. Graduate Teaching Assistant. Taught laboratory courses in Mechanical Engineering Department. Designed air-conditioning systems in the Plant Engineering and Maintenance Department.

1960 - 65 General Motors Corporation, Detroit, Michigan. Co-Op Student. Performed engineering design of mechanical and electrical systems associated with the production of automotive component parts.

Professional License

Registered Engineer, North Carolina (No.PE7022)

Professional and Technical Societies

American Society of Mechanical Engineers; American Society of Lubrication Engineers

Publications

"Particle Dynamics in Cooling Tower Plumes," Ph.D. Dissertation, University of Tennessee, August 1974.

"Treatment of Blowdown from Evaporative Cooling Systems Serving Electric Generating Plants," Siting Unit, Carolina Power and Light Co., Raleigh, N.C., 15 September 1973.

"Plume Rise and Condensation from Natural Draft Cooling Towers," Engineering Science and Mechanics Dept., University of Tennessee, June 1972.

"A Brief Survey of Plume Rise from Evaporative Cooling Towers of Large Nuclear Electric Stations," Engineering Science and Mechanics Dept., University of Tennessee, February 1972.

"Simulated Tests of GMC Truck and Coach Compressor Seals," with J.D. Symons, RM No. 35-2666, General Motors Research Laboratories, Warren, Michigan, February 1971.

"Evaluation of Mechanical Face Seals for the Chevrolet Military Vehicle," with R.L. Dega and J.D. Symons, MD-228, General Motors Research Laboratories, Warren, Michigan, 29 December 1970.

"Effects of Misalignment and Shaft Vibration on the Performance of Mechanical Face Seals," MD-223, General Motors Research Laboratories, Warren, Michigan, September 1970.

"Carbon-Graphite as a Water Pump Seal Head Material," MD-219, General Motors Research Laboratories, Warren, Michigan, July 1970.

"Design Considerations of Mechanical Face Seals," MD-207, General Motors Research Laboratories, Warren, Michigan, March 1970.

"Surface Finish Measurements and Examination of Ceramic Seal Seats, Automotive Rotary Shaft Seal," RM No. 35-2589, General Motors Research Laboratories, Warren, Michigan, December 1969.

"Vertical Takeoff or Landing Aircraft, XV-4B Inlet Development Test Report," with P.K. Shumpert and A.E. Harris, ER-9243, Lockheed-Georgia Company, Marietta, 1967.

"Effects of Injection and Suction on Pressure Distribution in Porous Wall Channels," Masters Thesis, Georgia Institute of Technology, Atlanta, 1966.

GABOR A. SOMORJAI

Consultant and Supervisor
of Catalyst Characteristic
Studies

Education

Ph.D., Chemistry, 1960, University of California at Berkeley

B.S., Chemical Engineering, 1956, University of Technical Sciences, Budapest

Experience

1964 University of California, Berkeley. Department of Chemistry, Professor of Chemistry and Principal Investigator, Inorganic Materials Research Division, Lawrence Berkeley Laboratory.
1972 Professor; 1967, Associate Professor; 1964, Assistant Professor

1960 - 64 International Business Machines Corporation, Yorktown Heights, New York. Research Staff.

Research Interests

Principal research interests are in the field of surface science. These include: (1) studies of the structure of clean metal, insulator and molecular crystal surfaces, and the structure of adsorbed molecules by low energy electron diffraction. Research in this area is presently concentrated on platinum and iron surfaces as well as on organic crystal surfaces such as naphtalene and amino acids; (2) studies of catalytic reactions on single crystal surfaces. Currently, the mechanism of catalysis of hydrocarbon reactions by platinum and the catalysis of surface reactions involving nitrogen on iron surfaces are being investigated; (3) Auger electron spectroscopy and ultraviolet photoelectron spectroscopy studies of surface composition and valency. Recently, the surface composition of binary alloys such as Ag-Au, Au-Ir, Au-Pt were examined to determine their surface phase diagrams; and (4) molecular beam scattering studies of energy transfer at surfaces. Recent studies include the $H_2 + D_2$ and $CH_4 + D_2$ surface reactions and study of the energy transfer which occurs during hydrogen atom recombination on platinum crystal surfaces.

Other Duties

Editor of the "Progress in Solid State Chemistry"; Chairman of the Division of Colloid and Surface Chemistry of the American Chemical Society; Member of the Solid State and Materials Sciences Committee of the National Research Council.

Fellowship Honors

Kokes Award, Johns Hopkins University, Baltimore, 1976.
Chairman, Division of Colloid and Surface Chemistry of the American Chemical Society, 1974.
Chairman of the Irvine Solid State Physics Conference on the "Chemical Processes at Surfaces" (with J.R. Schrieffer), 1974.
Chairman of the Berkeley Synposium on "The Frontiers of Surface Chemistry" (with W. Steele), 1974.

Chairman of the Dallas ACS Symposium on "The Structure of Solid Surfaces and of Adsorbed Molecules," 1973.
Chairman of the Physical Electronics Conference, 1973.
Unilever Visiting Professor, Chemistry Department, University of Bristol, Bristol, England, 1972.
Chairman of the Chicago ACS Symposium on the Chemical Physics of Surfaces, 1970.
Discussion Leader — Welch Foundation Conference on "Solid State Chemistry," 1970.

PAUL STONEHART Consultant

Education

Ph.D., Physical Chemistry, 1962, University of Cambridge (King's College)

Experience

1973 - 77 Stonehart Associates, Inc., Madison, Conn. Founded company to perform consultation in management of research, selective contract research and development in surface science applied to catalysis, electrochemistry, electrocatalysis, the preparation and characterization of small particles, and associated instrumentation for control and analysis. During the past ten years he has published over fifty papers, reviews and chapters in international journals and books in the fields of electrochemistry, catalysis and materials science. He is a member of the American Chemical Society, the Electrochemical Society, the Chemical Society of London, and in 1974 was elected a Fellow of The Royal Institute of Chemistry (London).

1969 - 73 Pratt & Whitney Aircraft Division, East Hartford, Conn.
1972 - 73 Heat of Advanced Fuel Cell Research Laboratories. Directed the fundamental work associated with both gas-phase catalysis and electrocatalysis. These areas involved fuel processing of light hydrocarbons (steam reforming and shift conversion) to provide hydrogen rich fuels, materials aspects of catalyst formation and deactivation, structures of porous reaction layers, electrocatalysis, and analytical techniques and instrumentation.

1964 - 69 American Cyanamid Company, Stamford, Conn. Refined theories developed earlier concerning the interactions between kinetic and diffusional processes occurring at solid electrode surfaces. In addition, he was concerned with solid surface diffusion and rearrangement processes on metal catalysts and thin film phenomena for studying properties of materials.

1962 - 64 Stanford University, Stanford, Calif. Department of Chemical Engineering. Worked on techniques for examining surface phenomena at solid electrodes with high rate electrode reactions.

Publications

"The Relation of Surface Structure to the Electrocatalytic Activity of Tungsten Carbide," with P. N. Ross, J. Catalysis, 1977.

"Support Considerations in Electrocatalysis. I. Effect of Hydrogen Atom 'Spillover' on Hydrogen Reactions in Acid at Pt/WO_3, Pt/MoO_3 and Pt/Carbon Black Surfaces," with P. N. Ross and J. MacDonald, J. C. S. Faraday Trans. I, 1976.

"Effect of Electrolyte Environment and Pt Crystallite Size on Hydrogen Adsorption — V," with K. Kinoshita and D. R. Ferrier, Electrochimica Acta, 1976.

"An Electrochemical Tool for Studying Composite Formation from PtFe and Contacting Porous Materials," with K. A. Klinedinst and W. M. Vogel, J. Materials Science, 1976.

"A Re-Interpretation of Breiter's Data in the Influence of Chemisorbed Carbon Monoxide on the Oxidation of Molecular Hydrogen at Smooth Platinum in Sulphuric Acid," J. Electroanal. Chem., 1976.

"Electrocatalysis at Transition Metal Oxide and Carbide Surfaces," Plenary Address, Proc. of Electrocatalysis on Non-Metallic Surfaces, National Bureau of Standards, 1976.

"Rheological Characterization and Thermal Degradation of PtFe — Part 2 — The Interaction Between PtFe and Carbon Black," with K. A. Klinedinst and W. M. Vogel, J. Materials Science, 1976.

"Differential Capacitance Measurements on Porous Electrodes by a Low Frequency ac Impedance Technique," with J. McHardy and J. M. Baris, J. Appl. Electrochem., 1976.

"The Preparation and Characterization of Highly Dispersed Electrocatalytic Materials," with K. Kinoshita, Chapter in Modern Aspects of Electrochemistry, Vol. 12, B. E. Conway and J. O'M. Bockris, Editors, Plenum Press, New York.

"The Use of Porous Electrodes to Obtain Kinetic Rate Constants for Rapid Reactions and Adsorption Isotherms of Adsorbed Poisons," with P. N. Ross, Electrochimica Acta, 1976.

"Crystallite Growth of Pt Dispersed on Graphitized Carbon Black: II Effect of Liquid Environment," with J. A. S. Bett and K. Kinoshita, J. Catalysis, Vol. 41, 1976, pp. 124-133.

"The Interaction Between PtFe and Porous Metals and Metal Blacks — Rheological Characterization and Thermal Degradation of the Polymer," with K. A. Klinedinst and W. M. Vogel, J. of Materials Science, Vol. 11, 1976, pp. 209-214.

"Electrochemistry of Transition Metal Oxide Bronzes and Related Compounds," Chapter in International Review of Science, Physical Chemistry Series Two, Vol. 6, with J. McHardy, Editors, J. O'M. Bockris and A. D. Buckingham, Butterworths, London, 1976, pp. 171-229.

"Effects of Gas- and Liquid-Phase Environments on the Sintering Behavior of Pt Catalysts," with K. Kinoshita and J. A. S. Bett, 4th International Conf. on Sintering and Related Phenomena, (Sintering and Catalysis) University of Notre Dame, G. C. Kuczynski, Editor, (Materials Science Research Vol. 10) Plenum Press, New York, 1975, pp. 117-132.

"Characterization of Ultra-High Surface Area Tungsten Carbide," with D. E. Fornwalt and E. J. Felten, Micron, Vol. 6, 1975, pp. 147-152.

"Surface Composition of Catalytically Active Tungsten Carbide (WC)," J. Electroanal. Chem., with P. N. Ross and J. MacDonald, Vol. 63, 1975, pp. 450-455.

"Surface Characteristics of Catalytically Active Tungsten Carbide (WC)," with P. N. Ross, J. Catalysis, Vol. 39, 1975, pp. 298-301.

"The Commonality of Surface Processes in Electrocatalysis and Gas-Phase Heterogeneous Catalysis," with P. N. Ross, Catalysis Reviews, Vol. 12, 1975, pp. 1-35.

"Electrocatalysis of Binary Alloys II. Oxidation of Molecular Hydrogen on Supported Pt-Ru Alloys," with P.N. Ross, K. Kinoshita and A.J. Scarpellino, J. Electroanal. Chem., Vol. 63, 1975, pp. 97-110.

"Hydrogen Adsorption on Pt in Hot Concentrated H_3PO_4," with D. Ferrier, K. Kinoshita and J. McHardy, J. Electroanal. Chem., Vol. 61, 1975, pp. 233-237.

"Role of Platinum Surface Morphology on Hydrogen Adsorption Isotherms — IV," with K. Kinoshita, Electroanal. Chem., Vol. 20, 1975, pp. 101-107.

"Reaction Pathways and Poisons II. The Rate Controlling Step for Electrochemical Oxidation of Hydrogen on Pt in Acid and Poisoning of the Reaction by CO," with W. Vogel, J. Lundquist and P.N. Ross, Electrochimica Acta, Vol. 20, 1975, pp. 79-93.

"Electrocatalysis of Binary Alloys I, Oxidation of Molecular Hydrogen on Supported Pt-Rh Alloys," with P.N. Ross, K. Kinoshita and A.J. Scarpellino, J. Electroanal. Chem., Vol. 59, 1975, pp. 177-189.

"Selective Poisoning of H_2-D_2 Equilibration on Transition Metals by Carbon Monoxide," with P.N. Ross, J. Catalysis, Vol. 35, 1974, pp. 391-400.

"Oxygen Solubility and Diffusivity in Hot Concentrated H_3PO_4," with K. Klinedinst, J. A. Bett and J. MacDonald, J. Electroanal. Chem., Vol. 57, 1974, pp. 281-299.

"Crystallite Growth of Platinum Dispersed on Graphitized Carbon Black," with J.A. Bett and K. Kinoshita, J. Catalysis, Vol. 35, 1974, pp. 307-316.

"The Effect of Platinum Morphology and Crystallite Size on Electrocatalytic Reactions, with J.A. Bett and K. Kinoshita, Proc. Symp. on Electrocatalysis, San Francisco, 1974, The Electrochemical Soc., Princeton, New Jersey, p. 275-284.

"The Valence Band Structure of Highly Dispersed Platinum," with P.N. Ross and K. Kinoshita, J. Catalysis, Vol. 32, 1974, pp. 163-165.

"Correlations Between Electrochemical Activity and Heterogeneous Catalysis for Hydrogen Dissociation on Platinum," with P.N. Ross, J. Res. Inst. for Cat. Hokkaido Univ., Vol. 22, 1974, pp. 22-41.

"Potential Cycling Effects on Platinum Electrocatalyst Surfaces," with K. Kinoshita and J. Lundquist, J. Electroanal. Chem., Vol. 48, 1973, pp. 157-166.

"Platinum Crystallite Size Effects on the Electrocatalytic Oxidation and Deposition of Adsorbed Hydrogen — III," with J. Lundquist, Electrochimica Acta, Vol. 18, 1973, pp. 907-911.

"Hydrogen Adsorption on High Surface Area Platinum Crystallites," with K. Kinoshita and J. Lundquist, J. Catalysis, Vol. 31, 1973, pp. 325-334.

"Platinum Crystallite Size Effects on Oxide Formation and Reduction Parameters — II,, with J. T. Lundquist, Electrochimica Acta, Vol. 18, 1973, pp. 349-354.

"A Comparison of Gas Phase and Electrochemical Measurements for Chemisorbed Carbon Monoxide and Hydrogen on Platinum Crystallites," with J. Bett, K. Kinoshita and K. Routsis, J. Catalysis, Vol. 29, 1973, pp. 160-168.

"Platinum Crystallite Size Considerations for Electrocatalytic Oxygen Reduction – I,, with J. Bett, J. Lundquist and E. Washington, Electrochimica Acta, Vol. 18, 1973, pp. 343-348.

"Adsorption Kinetics for Carbon Monoxide on Platinum in Hot Phosphoric Acid," with G. Kohlmayr, Electrochimica Acta, Vol. 18, 1973, pp. 211-223.

"Interactions Between Adsorbed Carbon Monoxide and Water on Platinum," Electrochimica Acta, Vol. 18, 1973, pp. 63-68.

"Sintering and Recrystallization of Small Metal Particles. The Loss of Surface Area by Platinum Black Fuel Cell Electrocatalysts," with P. A. Zucks, Electrochimica Acta, Vol. 17, 1972, pp. 2333-2351.

"Effect of Poisons on Kinetic Parameters for Platinum Electrocatalyst Sites," with G. Kohlmayr, Electrochimica Acta, Vol. 17, 1972, pp. 369-382.

"Surface Interactions Affecting Quasi-Equilibrium Adsorption," Electrochimica Acta, Vol. 15, 1970, pp. 1853-1864.

"Potentiodynamic Examination of Electrode Kinetics for Electroactive Adsorbed Species. Applications to the Reduction of Noble Metal Surface Oxides," with H. A. Kozlowska and B. E. Conway, Proc. Roy. Soc., A, Vol. 310, 1969, pp. 541-563.

"Potentiodynamic Examination of Surface Processes and Kinetics for the $Ag_2O/AgO/OH^-$ System," with F. P. Portante, Electrochimica Acta, Vol. 13, 1968, pp. 1805-1814.

"Potentiodynamic Determination of Electrode Kinetics for Chemisorbed Reactants. The $Ag/Ag_2O/OH^-$ System," Electrochimica Acta, Vol. 13, 1968, pp. 1789-1803.

"Quantitative Electrochemical Formation of Phosphotungstate Heteropolyanion Blues," with J. G. Koren and J. S. Brinen, Analytica Chimica Acta, 1968, pp. 65-75.

"Adsorption and Diffusional Parameters for Analysis of Carbon Monoxide in Aqueous Solutions," J. Electroanal. Chem., Vol. 15, 1967, pp. 239-244.

"Reaction Pathways and Poisons. Diffusional and Poisoning Parameters for Carbon Monoxide Anodic Oxidation in Aqueous Solutions," Electrochimica Acta, Vol. 12, 1967, pp. 1185-1198.

"Diffusion Coefficients of Tungsten Heteropolyacids," Analytica Chimica Acta, Vol. 37, 1957, pp. 350-358.

"Electrode Reactions of Tungstate Ions in the Presence of Carbon Monoxide," Analytica Chimica Acta, Vol. 37, 1967, pp. 127-134.

"Electrode Reactions of Carbon Monoxide at Noble Metal Electrodes in Aqueous Solutions," Fifth International Symposium in Research and Development of Non-Mechanical Power Sources, Brighton, Pergamon, 1967, pp. 509-529.

"Ionization of H_2 on Translating Platinum Electrodes," with G. R. Bopp and D. M. Mason, Electrochemical Technology, Vol. 4, 1966, pp. 416-423.

"Electrode Reactions of Hydrazine in Aqueous Solutions," with S. Szpak and T. Katan, Electrochimica Acta, Vol. 10, 1965, pp. 563-583.

CHARLES E. CRAVEN Electronics Research Engineer, Sr.

Education

B.S.E.E., 1971, University of Alabama in Huntsville

Experience

1965-Present LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center.
1969-77 Laser Doppler Velocimetry Program. Leader, Electrical Design Group. Specify instrumentation, conduct experiments and analyze data related to laser Doppler systems. Responsible for data recording and detector electronics for laser Doppler field measurements programs including long-range wind measurements and 3-D wind measurements. Developed hardware for providing on-line velocity readout from laser Doppler systems. Involved in wind tunnel measurements program utilizing a laser Doppler velocimeter to probe wake vortices behind a wall mounted wing stub.
1965-69 Fluid Mechanics Section. Determined systems criteria for hot wire anemometry measurement of low mass, high velocity compressible flow. Maintained and operated equipment associated with flow measurement. Assisted in reducing data common to 3-D flowfield measurements.

1959 - 65 ARO, Inc., Arnold Engineering and Development Center, Tullahoma, Tenn. Propulsion and Wind Tunnel Branch. Installed, maintained and operated analog to digital and data accumulator system, closed circuit television systems, force and moment balance systems, temperature and pressure measurement systems. Provided instrumentation support as electronic technician for transonic and supersonic wind tunnel testing of scale model space vehicles. Fabricated and designed electronic circuits.

1955 - 69 U.S. Air Force Communications Technician. Maintained UHF transmitters and receivers. Repaired and maintained FM multiplex UHF telephone relay equipment.

Publications

"Testing and Analysis of a Laser Doppler Velocimeter Technique for Remotely Measuring Wind over a Carrier Deck," with D.J. Wilson, J.L. Jetton and T.R. Lawrence, LMSC-HREC TR D496515, September 1975.

"Conduct Overall Test Operations and Evaluate Two Doppler Systems to Detect, Track and Measure Velocities in Aircraft Wake Vortices — Final Report," with M.C. Krause, D.J. Wilson, B.B. Edwards, E.W. Coffey and J.L. Jetton, LMSC-HREC TR D390470, December 1974.

"Field Operations Manual for Laser Doppler Velocimeter at John F. Kennedy International Airport," with M.C. Krause, B.B. Edwards, J.L. Jetton and R.E. Howle, LMSC-HREC TM D390388, September 1974.

"Development and Testing of Laser Doppler System Components for Wake Vortex Monitoring – Final Report," with M.C. Krause, E.W. Coffey, B.B. Edwards, D.J. Wilson and J.L. Jetton, LMSC-HREC TR D390159, August 1974.

"A Study of Laser Doppler Atmospheric Wind Interrogation Systems," (co-author), LMSC-HREC TR D390688, October 1973.

"A Laser Velocimeter for Remote Wind Sensing," Rev. Sci. Instr., Vol. 44, No. 3, 1972, pp. 512-518.

"Three-Dimensional Gas Velocity Measurements with a Laser Doppler Velocimeter System," with Charles E. Fuller, Proceedings of the Electro-Optics Conference, New York City, September 1971.

"Optical Measurement of Multi-Plume Interaction," LMSC-HREC D162004, January 1970.

"Experimental Investigation of Base Flow Field at High Altitude for a Four-Engine Clustered Nozzle Configuration," with Edwin B. Brewer, NASA TN D-5164 (1969).

WILLIAM E. JONES, JR. Research Engineer, Sr.

Education

M. S., Aerospace Engineering, 1966, University of Alabama

B. S., Aeronautical Engineering, 1961, University of Alabama

Experience

1963 - Present LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center. Engineering Sciences Section, Structures & Mechanics Group.
1965 - 77 Project engineer for developing finite difference modeling techniques to refine the Space Shuttle Solid Rocket Booster structural analysis. Defined procedures to be used to design elevated temperature structures considering fatigue, creep rupture and creep buckling. Responsible for the structural analysis of the Improved HAWK wing using finite element computer programs. Responsible for the structural design and analysis of the AMOOS configurations. Project engineer for several metallic and non-metallic Space Shuttle wind tunnel models, thermal, pressure and force models. Responsible for structural analysis of a ring/stringer stiffened cylinder subjected to thermal and pressure loadings using finite element techniques. Responsible for the structural integrity of the Apollo 14 flight HFC demonstration. Developed computer programs and performed an analysis to calculate the structural damping coefficients for each normal mode of vibration for a clustered space vehicle. Performed structural stiffness analysis, using finite element techniques, for severa components of a clustered space vehicle. A variety of other duties have include static analyses and weight optimization studies for a multiple rocket launcher, supersonic ground-to-air missile wing, and for typical Saturn interstage ring/stringer stiffened configurations. Also responsible for the structural integrity of several aerodynamic wind tunnel models.
1963 - 65 Aeromechanics Department, Structural Design & Analysis Section. Conducted statistical analysis of random vibration data to determine the vibration environment for several zones of the Saturn vehicle. Performed load and strength analysis and weight optimization studies on proposed retrorocket systems for the Saturn S-IVB vehicle.

1961 - 62 LOCKHEED-GEORGIA COMPANY, Marietta, Georgia. C-141 Stress Group, Associate Aircraft Engineer. Performed stress and internal load analysis and weight optimization of fuselage structure of C-141 jet transport aircraf

Publications

"Study Leading to the Refinement of the SRB Structural Analysis Program," with R. L. Eidson, LMSC-HREC TR D497054, November 1976.

"Methods for Structural Design at Elevated Temperatures," with A. M. Ellison, LMSC-HREC TR D306579, April 1973.

"Loopwheel Demonstration Vehicle Cost Analysis – Final Report," with W. Trautwein, LMSC-HREC TR D390778, April 1972.

"Structural Analysis of the TPS Center Section, Test Article I – Vols. I/II-III," (co-author), LMSC-HREC D225790, April 1972.

"Structural Analysis of the Center Tank Section of the TPS Prototype I – Test Article 1 – Vols. I/II," LMSC-HREC D225664, February 1972.

"Design of an Inert Fluid Injection System, Appendix G – Detailed Structural Analysis," LMSC-HREC D162662, November 1970.

"Modal Damping Predictions Using Substructure Testing," (co-author) SAE Paper 720810, Presented SAE National Aerospace Engineering and Manufacturing Meeting, San Diego, October 1972.

"Prediction of Structural Damping for Future Saturn Flights., LMSC-HREC D149346, December 1969.

ROBERT A. WYMAN Senior Scientist

Education

Business Law, 1953-54, Pierce College, Canoga Park, California

Tool Design Course, 1941-42, University of California at Los Angeles

Completed Three LMSC Management Series Courses, 1955-58

Experience

1963 LOCKHEED MISSILES & SPACE COMPANY, Inc., Huntsville Research & Engineering Center. Supervisor, Development Laboratory. Overall responsibility for design, fabrication and inspection of wind tunnel models and other related hardware. Responsible for fabrication and testing of electrical and mechanical devices through prototype and pilot production stages. Directed fabrication of word generators, analog-to-digital converters, pulse rebalanced accelerometer electronics, dc-to-ac inverters, dc-to-dc converters, fluidic amplifiers, precision fluid gyro components, precision wind tunnel apparatus, attitude motion simulator, aerodynamic models, aerodynamic probes and many similar items of hardware. Responsible for development of advanced electronic packaging, chemical milling, fluid devices, soldering techniques, plotting and encapsulating formulations and processes.

1954-63 LOCKHEED MISSILES & SPACE COMPANY, Sunnyvale, Calif.
1961-63 Senior Scientist, Microsystems Electronics, Electronics Research. Responsible for research and development of materials and processes for coatings and connections required in microminiature electronic equipment.
1959-61 Group Head, Advanced Electronic Techniques Laboratory, Process Engineering, Electronics Research, Electronics Research and Development. In charge of laboratory fabricating, testing, evaluating and design consulting on electronic prototypes.
1958-59 Group Engineer, Packaging Design Section, Radar and Data Link Department. Responsible for the mechanical design and electronic packaging of Polaris Beacons, Polaris destruct systems, satellite timers and programmers, and the mechanical design and documentation of ACRE (automatic checkout and readiness equipment).
1954-58 Research Engineer, Test Laboratories, Leader of Advanced Electronic Techniques Group. Specialized in electronic packaging.

1952-54 PCA Electronics and Neutronics, Inc., Santa Monica, Calif. Production Manager. In charge of manufacturing all electronics components such as pulse transformers, distributed constant delay lines, and subminiature assemblies.

1950-52 Hughes Aircraft Co., Culver City, Calif. In charge of assembly and techniques laboratory in miniaturization section of Advanced Electronics Laboratory. Responsible for the first processes in etched circuits, printed circuits, silk-screen techniques, and dip soldering techniques. In charge of drafting and mechanical design.

1948-50 Bendix Aviation Corp., N. Hollywood, Calif. Design Engineer, Design Drafting Group Leader. Worked on depth recorders, underwater telephones, top and bottom sounders, and various other sonar equipment.

Robert A. Wyman Page 2

Experience

1947-48 Lear, Inc., Santa Monica, Calif. Designer. Worked on design of portable aircraft radio, wire recorders, and magnetic pickup heads.

1944-46 Hoffman Radio Corp., Los Angeles, Calif. Design Draftsman. Performed design drafting on commercial radios, frequency meters, Government recording equipment and radar.

1943-44 Air Associates, Inc., Los Angeles, Calif. Design Draftsman. Worked on design of UHF Airport Landing Equipment and electric gas gauge equipment.

1942-43 Howard Aircraft Co., St. Charles, Ill. Tool Designer. Assistant Chief Tool Designer and Group Leader, Lofting Department.

1941-42 Affiliated Engineering Co., Detroit, Mich., tool designer; Allen-Kaudered Engineering Co., Detroit, Mich., tool designer; Douglas Aircraft Co., Long Beach, Calif,, tool designer.

Societies

Member, Society of Aerospace Material and Process Engineers

REFERENCES AND APPENDIXES

REFERENCES

1. Thirsk, H. R., and J. A. Harrison, "The Fundamentals of Metal Deposition," Electroanalytical Chemistry, Vol. 5, Editor, Allen J. Bard, Marcel Dekker, Inc., New York, 1971, pp. 68-72.
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15. Abbas, A. A., and W. M. Crooks, "Catalytic Epoxidation of Ethylene," Birmingham University Chemical Engineer, Vol. 24, No. 2, 1973, pp. 40-43.

16. Stonehart, P., K. Kenoshita and J. A. S. Bett, "The Effect of Platinum Morphology and Crystallite Size on Electrocatalytic Reactions," Proceed. Symposium on Electrocatalysis, San Francisco, 13-15 May 1974, Editor, M. W. Breiter, The Electrochemical Society, New Jersey, 1974.

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18. Beeck, O., and A. W. Ritchie, "The Effect of Crystal Parameter on Hydrogenation and Dehydrogenation," Discussions of the Faraday Society, Vol. 8, 1950, pp. 159-166.

19. Boudart, M., A. W. Aldog, L. D. Ptak, and J. E. Benson, "On the Selectivity of Platinum Catalysts," J. Catalysis, Vol. 11, 1968, pp. 35-45.

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Appendix A
ECONOMIC VALUE OF CATALYSTS

About \$200 billion per year (about 20% of the gross national product) is dependent to some degree on commercial catalysts processes (Ref. 53). For example, in 1974 some 6 billion pounds of butadiene were produced with a value of more than one billion dollars. Nearly 80% of the products of chemical industry are derived catalytically. The consumption of catalysts is currently about \$600 million per year, and current expenditures in the U.S. for research and development related to catalysts is about \$700 million per year (Ref. 53). In addition to their value in strictly manufacturing processes, catalysts are a vital factor in the supply of energy. A major challenge is that of upgrading various hydrocarbons from sources such as coal, sand tar, shale oil, and petroleum residua to fuels that allow clean and efficient combustion. Processes such as gasification or liquefaction of coal with simultaneous removal of sulfur and nitrogen and/or the development of effective methods to remove oxides of sulfur and nitrogen and small particles from the flue gases are envisioned. A considerable economic incentive exists for a catalyst that would allow a rapid breaking of the strong sulfur-carbon and nitrogen-carbon bonds with less consumption of hydrogen (Ref. 54).

Electrocatalysts — agents that can accelerate the rates of electrode reactions — are important in still another energy technology — fuel cells. The extensive research and development activity in this field is indicated by the approximately 175 patents relating to fuel cells that were granted in the United States in the two year period 1965 and 1966. A sizable number of these patents relate to electrode catalysts (Ref. 55).

Catalysts for decreasing auto pollution are also the topic of much current research (Refs. 56 and 57).

Finally, a point not to be overlooked is that discovery of a new catalyst can stimulate the establishment of entirely new industries. The discovery in 1913 of an iron catalyst which permitted the production of ammonia gas from nitrogen and hydrogen led to the formation of the synthetic fertilizer industry (Ref. 58).

Appendix B
STRUCTURE-SENSITIVE AND STRUCTURE-INSENSITIVE REACTIONS

Reactions that are catalyzed are either structure-insensitive (SI) or structure-sensitive (SS), i.e., and SI reaction depends only on the total surface area of catalyst available whereas an SS reaction depends both on the total surface area and the nature of that surface. In the SI case the specific catalytic activity (activity per unit surface area of catalyst particle) does not depend on particle size whereas the specific catalytic activity of an SS catalyst does depend on particle size. Figure B-1 illustrates the general size dependencies.

1: Structure-Insensitive Reaction
2 and 3: Structure-Sensitive Reactions
4 and 5: The Sense of the Structure Sensitivity is Itself a Function of Particle Size

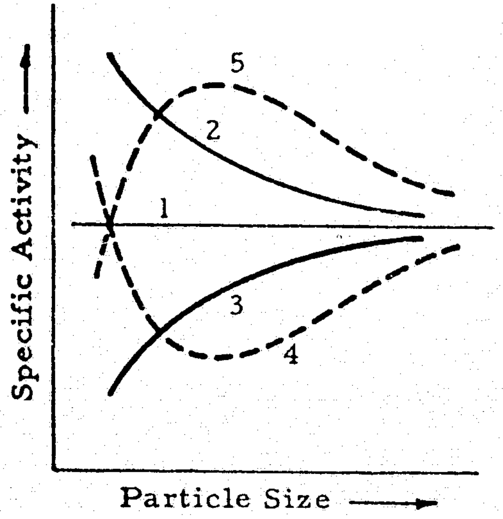

Fig. B-1 - Possible Forms of Specific Particle Size Effect (Ref. 59

made:

- A likely explanation for the observed differences between the space- and ground-grown crystals is that the rate of electrochemical displacement of silver ions from a 5% aqueous solution by copper tends to be diffusion controlled in space and kinetically controlled in one- and higher-*g* because of augmentation of mass transport by convection.
- Downward and upward growing crystal streamers are probably the result of gravity-driven convection, the flow patterns of which can be delineated. Lateral growth along an air/liquid interface may be the result of surface tension driven convection, the pattern of which also can be delineated.
- The data indicate that electrolysis in space or low-*g* environments can produce either dendritic crystals with more perfect microcrystalline structures or massive, single crystals with fewer defects than those grown on ground or at higher-*g* levels.
- Vapor transport growth of GeSe and GeTe appears to be affected by convection in a manner similar to that hypothesized for the electrochemical deposition of silver crystals.

The conclusions of the present study need, of course, to be tested by techniques more controllable than electrochemical displacement. Particularly needed are experiments in which the deposition potential can be controlled. More extensive details and discussions of the TV106 demonstration and a consideration of the technological implications will be found in ref. [9].

Acknowledgments

The authors wish to express their appreciation to Astronaut G. Carr for his enthusiastic participation in this project. A great debt of appreciation is also due Tommy C. Bannister, of the NASA Marshall Space Flight Center, who was program manager of the Skylab science demonstrations. His dedication and help in circumventing the obstacles imposed by the severe experimental constraints are in large measure responsible for the successes achieved.

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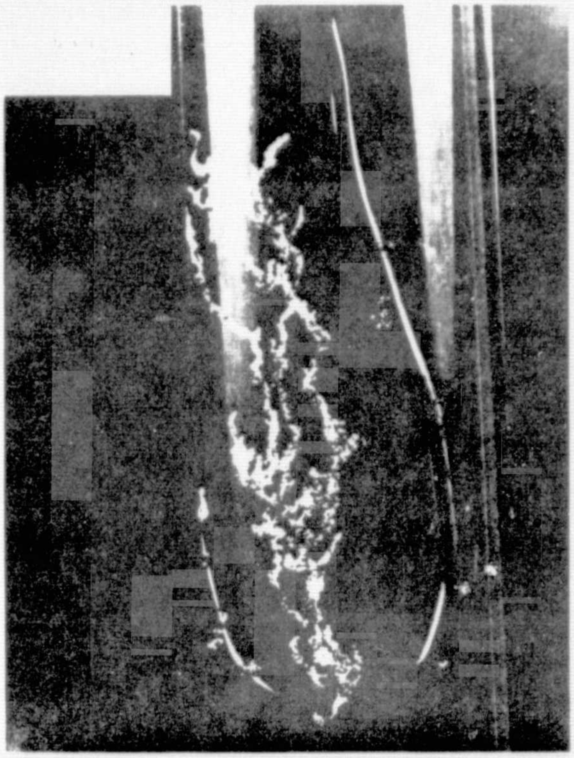

Fig. 8. Upward growing streamers on ground (50% solution concentration).

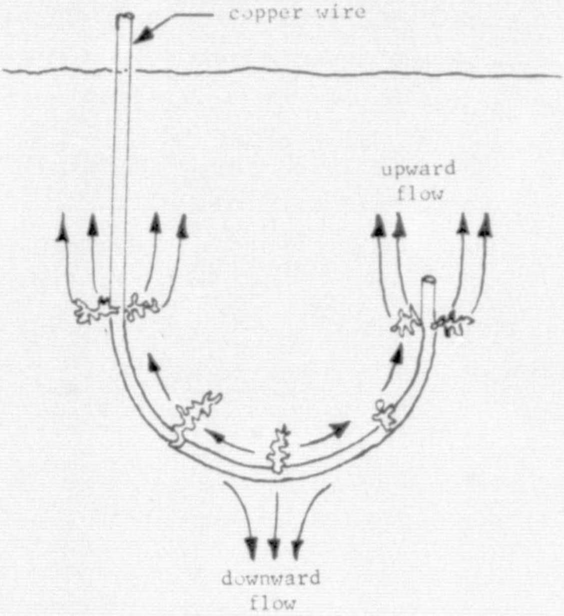

Fig. 9. Schematic of convection currents seen with shadowgraph during crystal growth from 5% aqueous solution.

phology of electrodeposited zinc changed from a smooth adherent deposit to dendritic needles when the electrolysis cell was orientated in such a manner that natural convection was minimized [7].

The observations of upward, downward, and lateral growing crystal streamers in the ground tests indicate that the crystal growth direction follows the fluid flow streamer which provides the most opportunuity for growth. In the case of the upward growing streamer, rising silver ion depleted solution would cause undepleted solution to flow toward a growing crystal tip. In the case of a downward growing dendrite, it appears that the crystal streamer follows the copper enriched, but silver ion undepleted, downward flowing fluid streamer. In the case of lateral growth along the air/liquid interface, surface tension driven convection may be involved.

Alteration of concentration profiles by convection appears to be responsible for the observed shift from relatively slow to rapid edge growth as gravity level is increased. Convection would decrease the diffusion boundary layer and thereby increase the silver ion concentration near the electrode surface. The shrinkage of the diffusion boundary layer would thereby increase electrode potential and current density. In one experimental study [8] it was found that at low current densities, layer crystal growth starts in the interior of the crystal faces, while at higher current densities growth starts at edges and corners.

The noted increase in gas generation during electrodeposition in high concentration solutions and at higher g levels suggests that convection thinning of the hydrogen ion diffusion boundary layer allows the hydrogen/hydrogen ion couple to operate.

The noted increase in twinning as convective or g levels are increased can probably be explained on the basis of increased mechanical stresses.

The noted similarities between the crystal morphologies of electrochemically deposited silver crystals and vapor deposited GeSe and GeTe crystals indicate that similar convection considerations apply to both cases.

4. Conclusions

On the basis of the data obtained from space and ground experiments, the following conclusions are

Fig. 6. Crystals grown from 5% solution on earth at 1g (illustrating rapid growth from edges and corners and also curving of crystals).

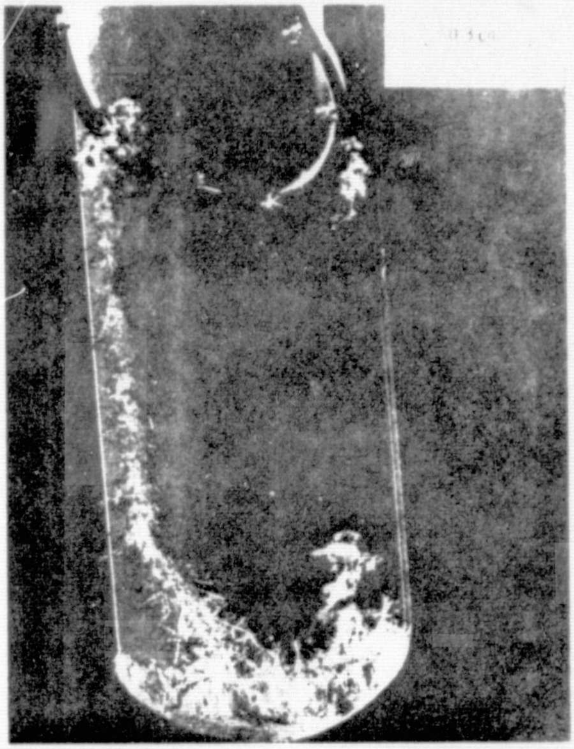

Fig. 7. Downward growing dendrites on ground (0.4% solution concentration).

relevant to the main theme of this paper, that ribbons, long thin strands, and, in a few cases, thin foils or films almost invariably developed if electrodeposited silver crystals were left standing in contact with their mother liquor for 1 or 2 months.

The noted differences in coherency and crystal structure are probably the result of convective conditions prevailing during deposition. Fig. 9 shows a schematic of an observed shadowgraph projection of an electrodeposition conducted on the ground. The downward flowing streamer consists of dense, copper nitrate reinforced solution, while the upward flow consists of lighter depleted silver nitrate solution [4]. In low-*g* the velocity of the streamers would be considerably reduced, while in a high-*g* centrifuge field it would be considerably increased. In low-*g*, therefore, it appears likely that the rate of growth of a silver crystal would tend to be diffusion controlled, while in 1- or higher-*g* it would tend to be kinetically controlled; i.e., controlled by the rate of the atomic mechanics of discharging an ion and incorporating the atom into the crystal lattice. It is fairly well established that diffusion-controlled crystal growth rates tend to produce dendrites, while kinetically controlled rates tend to produce faceted crystals. Thus, the reason for the more powdery crystals obtained in the space experiment than in ground and centrifuge experiments is readily apparent. It may be noted, however, that variations in the anodic dissolution of copper may have had a minor effect. Usually dissolution is a more rapid process than deposition. The overall rate of an electrochemical deposition, therefore, is usually controlled by the deposition rate. The postulation of a diffusion-controlled deposition for the flight crystals is also in keeping with the results of earlier studies [5,6] in which electrolytically deposited metal powders were produced in the transport-controlled regime of the current–potential curve and coherent deposits in the kinetically controlled regime. Also, in an experimental study of convection in electrolysis cells it was observed that the crystal mor-

Fig. 4. Silver crystals grown in space from 5% solution (illustrating sharp edges and corners).

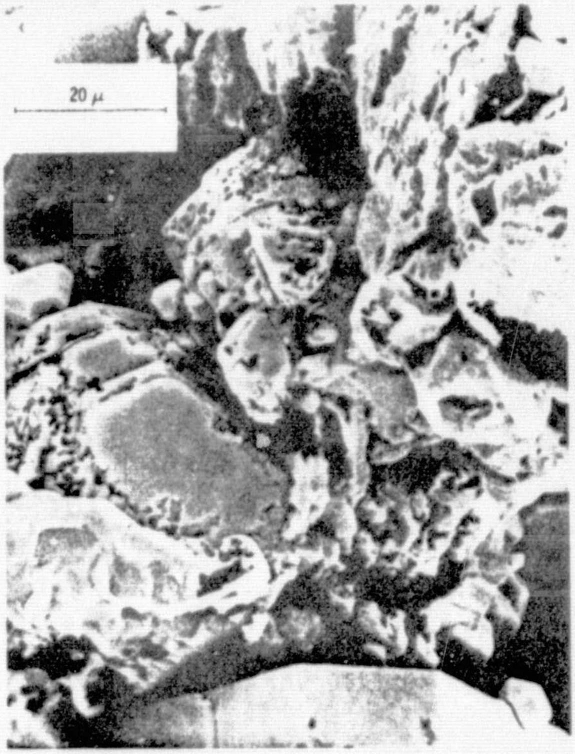

Fig. 5. Crystal grown from 5% solution at 5*g* (illustrating imperfect habit and evidence of gas formation).

tals are summarized as follows:

– The low-*g* crystals are more powdery or less coherent than the higher-*g* crystals. Furthermore, the higher the *g* level, the more coherent the crystals.

– The low-*g* crystals show more perfect crystalline forms than do the higher-*g* crystals. Also, the higher the *g* level, the more imperfect the resultant structures (figs. 3, 4 and 5).

– The higher-*g* crystals show evidence of rapid growth from edges and corners (fig. 6).

– The 1*g* crystals show examples of curvatures caused by fluid flow which are not observed in the low-*g* crystals (fig. 6).

– Steps and ledges appear more prevalent in the higher-*g* crystals than in the low-*g* crystals.

– Twinning appears more prevalent as gravity level increases.

Ground and centrifuge tests have yielded additional relevant information which is summarized as follows:

– After a number of hours, dilute solutions develop dendrite streamers that grow downward; i.e., in the same direction as the gravity vector. Concentrated solutions produce streamers of chunky, imperfect crystals that grow upward; i.e., in a direction opposite to the gravity vector (figs. 7 and 8).

– Centrifuging low concentration solutions increases coherency and size of the dendrite streamer.

– Crystal streamers in high concentration solutions spread out over a free liquid–gas surface when they encounter the same.

– More gas is apparently generated as concentration on gravity level increases.

In addition, trends in the crystalline morphologies observed in earth- and space-grown silver crystals almost exactly parallel those observed in earth- and space-grown germanium selenide (GeSe) and telluride (GeTe) crystals. The latter crystals were grown by a vapor transport technique as the Skylab experiment M556 [3].

It is also interesting to note, although not directly

Table 1
Chemical analysis of spent solutions

Sample	Ag(μg/l)	Cu(μg/l)
Flight	2.0	9300
Ground Test 1	12.0	8900
Ground Test 2	10,000	6400

Trace quantities Fe, Mg, Si, Al

was not what might have been achieved given more favorable circumstances. It is of interest, therefore, to inquire what errors or lack of experimental control might have influenced the interpretations and conclusions presented in this report.

One of the chief concerns of the study was the question of whether continued growth after re-entry would mask the crystal growth that occurred in space. A procedure having the astronaut remove the silver crystals while still in flight was judged too time consuming, complicated and risky to pass a review. Ground tests indicated that most of the growth occurred in approximately 3 days. The TV106 experiment consisted of 15 days of crystal growth in space and 3 days on the ground after re-entry. When the crystals were received in Huntsville, Alabama, they were a loose, coarse powder. At what point the crystals had been dislodged from their growth sites is not known; during re-entry, however, would appear as a likely time. In any case, the scanning electron micrographs of the flight crystals which show significantly different structures allow the conclusion that continued earth growth after re-entry did not mask the space growth.

Another concern was that perhaps impurities were introduced into the flight samples which were not present in the ground samples or vice versa. A review of the experimental conditions for the flight and ground samples shows that ample opportunity existed for such an occurrence; i.e., different people prepared the flight and the ground samples, the water used to prepare the flight and ground samples came from different sources, different people performed the flight and ground experiments, and the containers used in the ground and flight experiments were different. Also, the flight sample received an apparently appreciable dose of radiation. However, the fact that self-consistent trends could be established using not only data from the flight crystals but also from ground and centrifuge studies (in some of which another grade silver nitrate was used) indicates that lack of control over impurities was not an appreciable error. The observed self-consistent trends would also rule out vibration of *g*-jitter [2] effects as significant variables.

Finally, errors such as variations in volume or solution used for any particular test and day-to-day temperature variations were judged insignificant because many tests were done under a variety of volume and temperature conditions and, again, self-consistent trends traceable only to convection effects could be established.

3. Results and discussion

Both the Skylab and earth-grown crystals show a variety of habits. Certain tendencies in habits dependency on gravity level, however, can be discerned. The differences between the low-*g* and higher-*g* crys-

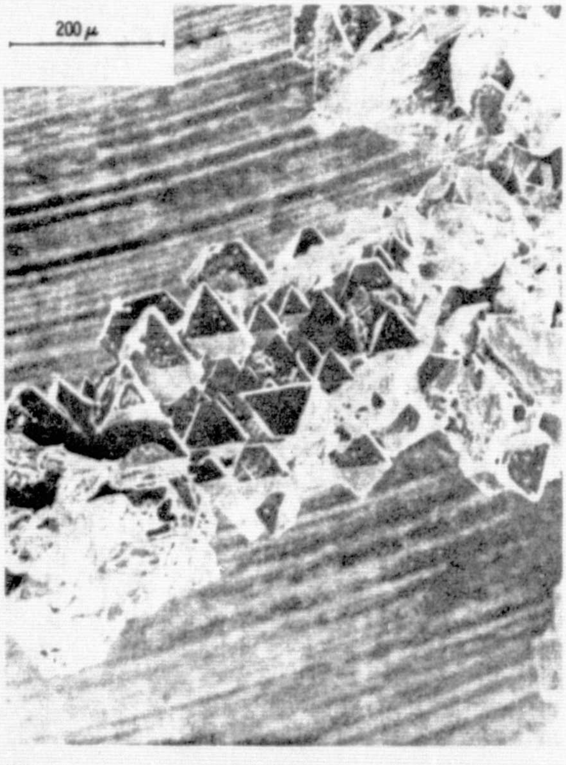

Fig. 3. Silver crystals grown in space from 5% solution (illustrating more perfect microcrystalline form).

Date of receipt by investigators of experiment package and removal of silver crystals from solution:	February 11, 1974
Solution preparer:	Person A
Radiation dose:	Significant, as judged by color change of initially clear Lexan

The procedure for conducting the flight experiment consisted of the following instructions:

(1) Coil copper wire around pen (minimize finger contact with wire) to fit within the solution volume of container, leaving a wire tip for inserting wire in solution without the fingers contacting solution (coil diameter should be smaller than container to assure minimum coil/container contact).

(2) Using pocket knife, lightly cut wire insulation several times to provide nucleation sites for crystal growth. (Areas of cut to be small; do not abrade wire, nick insulation.)

(3) Centrifuge container to settle solution. WARNING – Avoid skin contact with solution (5% silver nitrate).

(4) Wrap tissues around cap and remove cap very *slowly* to allow pressure equalization and insert wire into solution.

(5) Replace cap and restrain container for later photographing.

(6) Photograph crystals in vial after approximately 6, 24, and 76 hours. Camera setup: Nikon (per photo PAD), 55 mm – K1 adapter (F2.8, 1/30 sec) manual focus. Lighting – Hi intensity light with vial orthogonal to axis of light to minimize reflection.

The experimental conditions prevailing for the ground preparation of silver crystals for comparison to the flight grown crystals were as follows:

Containers:	Glass vials
Copper wire and silver nitrate:	Same as used for flight test
Water:	Distilled water of unspecified purity
Temperature:	Room (~22 to 24°C)
Composition of silver nitrate solutions:	5% by weight (~10 ml used in each of these preparations)
Pressure:	1 atm
Date of experiment: (Huntsville, Alabama)	January 24, 1974
Date of solution preparation: (Huntsville, Alabama)	January 15, 1974
Date of removal of silver crystals from solutions:	February 11, 1974
Solution preparer:	Person B

When the flight silver crystals were received February 11, 1974, they and the ground samples were removed from solution, washed with distilled water four times, and dried in a dessicator overnight. Approximately 0.3 g silver crystals were obtained from the flight test. A chemical analysis by emission spectroscopy gave the results shown in table 1.

Original solutions before insertion of copper wire contained approximately 3×10^7 μg/l Ag and 0 μg/l Cu. If all of the silver had been replaced and no other reactions occurred, the copper concentration would have been about 6×10^6 μg/l. The data show that obviously other reactions had occurred. This is not the only evidence for additional reactions. Small amounts of both blue and dark red crystals of unidentified materials are obtained in any ground electrochemical displacement of silver ions by copper, in addition to silver crystals. Such blue and red crystals were obtained also in the flight experiment. Further analyses of these colored crystals, however, were not undertaken.

Further ground tests involving concentration variations and centrifuging were accomplished utilizing silver nitrate of reagent grade (Baker analyzed 3426, Lot. 31522) but somewhat decomposed, as evidenced by the dark color. The copper used in these tests was generally the same as used in the flight test.

The apparatus used for centrifuging utilized a dc magnetic torque motor, low-noise electrical slip ring, dual-pen recorder, programmable power supply, and a period sensing network. A free swinging cannister which contained the sample and a counterweight were suspended from the centrifuge arms by a pivot to ensure that the acceleration force was always along the long axis of the sample tube; i.e., in the same direction as gravity when the tube stood upright.

Shadowgraph equipment consisted of some improvised flat plate cells and a high intensity microscope light. White paper served as a screen.

Control or error analysis

Because of the conditions under which the flight experiment was developed and performed, the degree of control of the experimental solutions and procedures

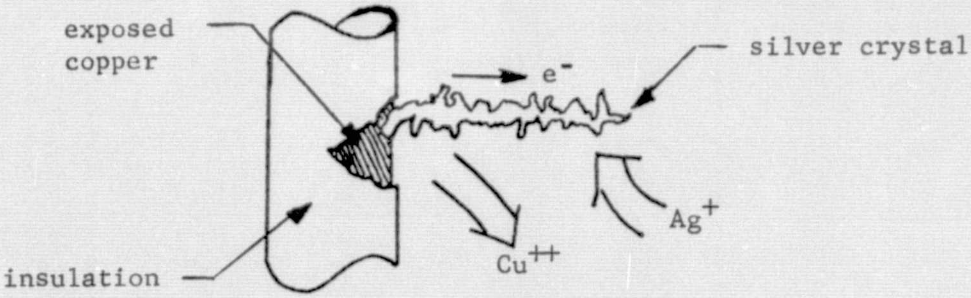

Fig. 1. Mechanism of electrochemical displacement of silver ions by copper.

standard electrochemical potential calculated for the reaction is +0.4536V. The electrochemical process can be viewed as an electrolysis in which the copper wire serves as an anode and deposited silver as the cathode as shown in fig. 1.

Commander Carr photographed the crystal growth after 6, 24, and 75 h; but unfortunately, some unknown mishap occurred and no pictures developed on the exposed film. The crystals immersed in spent solution, however, were returned to earth for examination. Fig. 2 shows the crystals (center tube) as received. The two side vials show crystals grown on earth for the same length of time as the flight crystals. It is apparent that jars during handling and re-entry dislodged the flight crystals from their growth sites. The returned flight crystals and crystals grown under a variety of conditions on earth, including gravity levels of 2 and 5*g*, were examined by scanning electron microscope. Further details of the experiments are given in the next section.

Fig. 2. Silver crystals electrodeposited in space and on earth. Center tube (8.25 × 1.6 cm) contains flight-grown crystals; side flanking vials contain crystals grown on earth for the same length of time.

2. Experimental details of Skylab experiments TV106 – electrodeposition of silver crystals

The following table summarizes the materials and conditions employed in performance of the flight experiment:

Container:	10 ml Lexan centrifuge tube with polyethylene screw top and an O-ring gasket of Buna N rubber containing carbon particles.
Silver nitrate:	Merck C.P. (Product 12C82, lot. 71731)
Copper wire:	Belden insulated wire (LE8051 22 Awg Sgl Beldsol, polyurethane and nylon insulation) Belden Corporation, Chicago, Ill. 60644
Water:	Distilled water of unspecified purity
Cabin temperature at time of experiments:	~22°C (72°F)
Cabin pressure:	~5 psi
Composition of silver nitrate solutions:	5% by weight (9.5 ml of solution made by adding 0.5 g $AgNO_3$ to 9.5 ml of water)
Date of experiment:	January 24, 1974
Date of solution preparation and packaging (in Houston, Texas):	November 1, 1973
Date of re-entry:	February 8, 1974

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Appendix C

ELECTROCHEMICAL DEPOSITION OF SILVER CRYSTALS ABOARD SKYLAB IV

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and

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Received 29 September 1975; revised manuscript received 3 February 1976

Silver crystals were grown aboard Skylab IV by an electrochemical reaction and subsequently returned to earth for comparison with crystals grown at 1g and 5g. Both the Skylab and earth-grown crystals show a variety of structures. The space-grown crystals, however, generally possess more perfect microcrystalline forms and are less coherent than earth- or centrifuge-grown crystals. The crystal habits of space and ground electrodeposited silver crystals, furthermore, are very similar to those of germanium selenide (GeSe) and telluride (GeTe) crystals grown in space and on the ground by a vapor transport technique. A likely explanation for the observed crystal habits is that the convective currents prevailing during a deposition play a determining role. In experiments conducted on the ground with solutions of varying concentrations, additional crystal forms were observed. These include downward growing dendrite streamers; upward growing chunky crystal streamers; growth along an air/liquid interface; and ribbon, film, and fiber crystal habits. Convection, again, appears to be an important factor in producing the structures described in the first three preceding cases.

1. Introduction

During the Skylab IV mission a number of science demonstration experiments were conducted aboard the spacecraft. The oppotunity for conducting these experiments arose when it became apparent that the astronauts were becoming so efficient in performing their primary duties that they would have some extra time for additional experiments. The science demonstration experiments were very simple in nature and operation because severe constraints were imposed by primary mission objectives. Some of the constraints included weight and volume limits, zero power, safety considerations, equipment compatible with spacecraft systems and operations, and limited astronaut training. Because of these constraints, the purpose of the experiments was primarily demonstration. Being, however, among the relatively few experiments in materials science that have been performed thus far in space environments, the demonstration experiments have provided useful and, in a number of cases, unexpected data as well as striking visual demonstrations of various physical phenomena in low *g* [1].

The science demonstration reported herein was called TV106 – Deposition of Silver Crystals. It was performed on January 24, 1974, by Astronaut G. Carr. The purpose of this experiment was to demonstrate crystal growth by electrochemical reaction in low *g* (1*g* equals gravity acceleration at earth surface, or 980 cm/sec^2) and to compare the resultant crystals with those grown on earth. The experiment consisted of inserting a scored, insulated copper wire into a 5% (by weight) aqueous solution of silver nitrate and allowing the crystal growth to proceed for the remainder of the mission. Silver crystals deposit when a copper wire is placed in a silver nitrate solution as indicated by the following overall electrochemical reaction:

$$\mathrm{Cu} + 2\mathrm{Ag}^+ \rightarrow \mathrm{Cu}^{++} + 2\mathrm{Ag}\,,$$

i.e., copper displaces silver ions from solutions. The

Appendix D

CONSTANT, CONTROLLED ELECTRODE POTENTIAL EXPERIMENTAL APPARATUS AND INSTRUMENTATION

The experimental apparatus and instrumentation required to conduct the electrodeposition experiments are discussed. The electrodeposition experiments are to be conducted using the controlled potential or potentiostatic coulometry method. Controlled potential electrolysis involves measuring the difference of potential between the electrode of interest and a reference electrode. Concurrently, the voltage applied across the working electrode and the other current carrying electrodes is adjusted so that the potential of the working electrode remains virtually constant. To implement the controlled potential electrolysis technique on the Space Processing Sounding Rocket Program will require that the following components be designed and fabricated which are suitable for the sounding rocket environment: (1) an electrolysis cell; (2) an electrolysis cell control module; (3) an optical data acquisition system; and (4) an experiment control package. The conceptual approach being considered in planning the electrochemical crystal growth experiment encompasses the basic intent of the space processing sounding rocket project. The planned experiments are simple while the equipment to be developed will provide a strong basis for evolutionary development as the program progresses and new experiment requirements arise. Initial equipment costs will be minimized. Also, due to the many potential electrolysis studies, the equipment will be suitable for repeated use by multiple investigators. Under the plan presented in this proposal, the experiments will involve the reduction of silver and palladium ions from aqueous solution and subsequent growth of crystals. The cell electrodes and electrolyte will be changed to facilitate further experiments. The cell control system will be adjusted to provide the desired potential control.

Details of the proposed experiment components and of the integrated experiment package are discussed in the following paragraphs.

D.1 ELECTROLYSIS CELL MODULE

The electrolysis cell module will contain three discrete cells, each cell having the basic configuration shown in Fig. D-1. The body of the cell will be constructed of an inert material suitable to withstand the sounding rocket environment. (Lexan was considered for planning purposes.) The electrolysis cavity will be formed by a rectangular opening machined into the cell body. Transparent end plates fabricated from clear inert material will complete the cell enclosure. Sealing between the end plates and the cell body will be accomplished using O-ring seals. Bolts threaded into the cell body will provide O-ring compression and will facilitate removal of the end plates for easy access to the electrodes.

Two electrodes and a reference electrode will be installed in the cell body. The egress of the leads of the electrodes from the cell body will be sealed using miniature compression fittings. Both the electrodes and reference electrode will be removable to facilitate multiple use of the cell hardware.

For the first experiment the electrodes (both auxiliary and working) will be fabricated from very thin silver plate (or foil). Each electrode will be approximately 1 sq in. (6.452 sq cm) in area. The electrodes will be held in place with a small clamp ring. The reference electrode will be formed by a platinum wire sealed in an inert shield (glass or inert plastic). Platinum foil (or wire mesh) will be utilized to form the rectangular electrodes for the second experiment. The platinum electrodes will be interchangeable with the silver electrodes and will not necessitate cell body modification. The reference electrode will remain the same.

The electrolyte temperature will be monitored during flight by means of a thermister installed in each cell. A thermister will be utilized in this application since it does not require the use of an amplifier or reference junctions to obtain a signal suitable for telemetering.

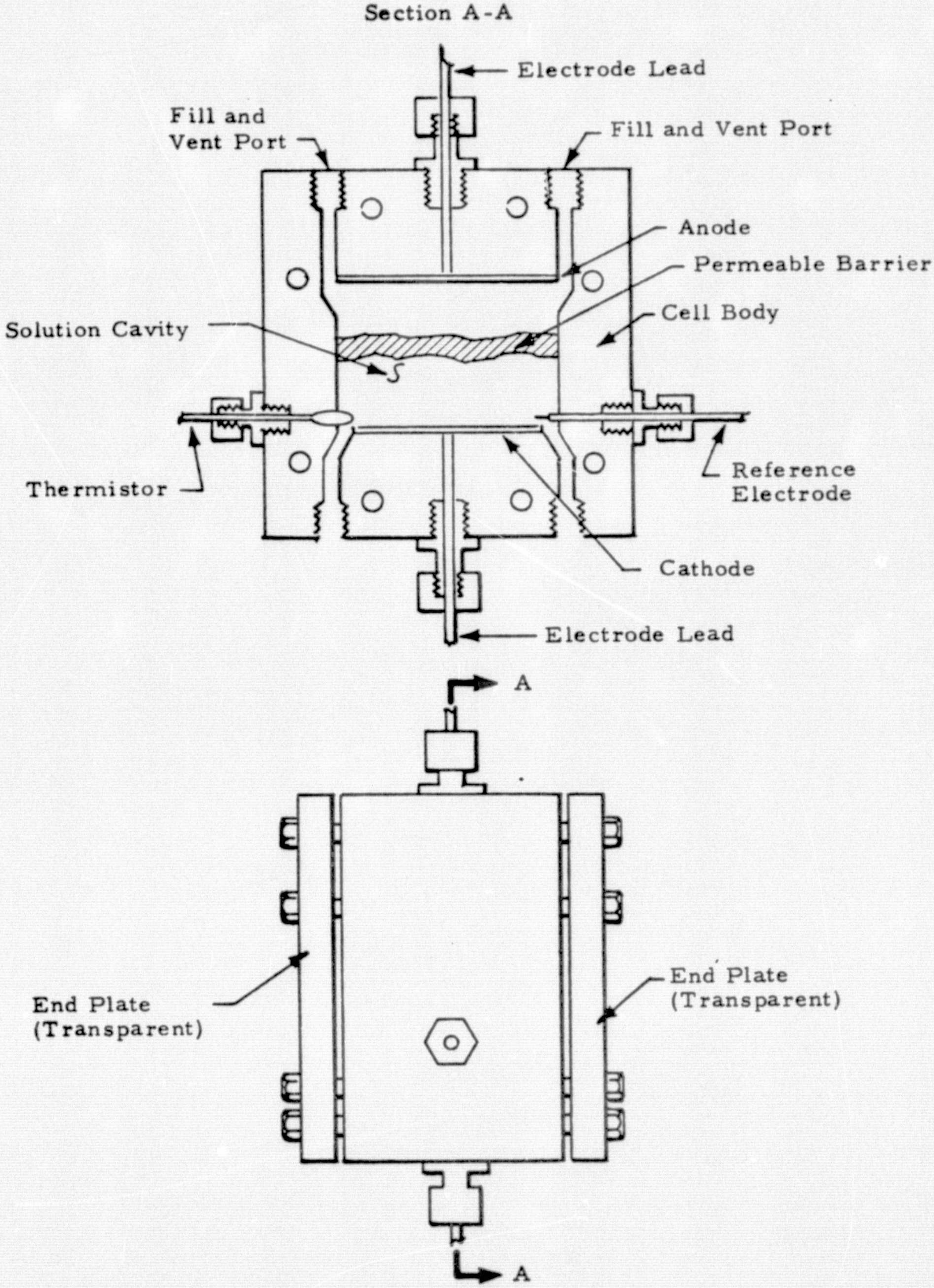


Fig. D-1 - Detail of Construction of Electrolysis Cell for Use in Crystal Growth Experiment (Three Cells to be Utilized)

Provisions for electrolyte filling and removal has been included in each cell in the form of vent and fill ports. Each port will have a positive shutoff fitting to help remove any "trapped" air during the filling process.

As stated previously, the electrolysis cell module of the experiment will contain three discrete cells. The cells will be mounted in a "side-by-side" array to facilitate the simultaneous recording of optical data from each cell. The optical data system is discussed in detail in Sections D.3, and Fig. D-5. A fourth cell, to be fabricated first, will serve as the prototype unit and will be utilized to generate terrestrial baseline electrodeposition data for later comparison with inflight results.

D.2 ELECTROLYSIS CELL CONTROL MODULE

The electrolysis cell control module will contain the components required to: (1) maintain constant potential between the working electrode and reference electrode in each cell; (2) integrate the current flowing through each cell as a function of operating time; and (3) provide a collection point for all signals to be telemetered during the experiment operating period. The basic relationship of items (1) and (2) in the cell circuitry is shown in the simple block diagram of Fig. D-2.

A constant-potential control device (potentiostat) and a current-time integrating circuit will be required for each cell. Potentiostats for use in constant-potential coulometry laboratory studies are available commercially (Brinkman, Princeton Applied Research Corporation, and others). These units are not suitably configured, however, for the sounding rocket program application. Many successful potentiostat circuit designs are also available in the literature (for instance, Refs. D-1, D-2 and D-3). The devices fabricated from these circuits designs were also not suitable for the present application. Therefore, a potentiostat device configured for the sounding rocket environment must be fabricated. The resulting device will be suitable for a multitude of constant potential coulometry experiments beyond those outlined in this proposal.

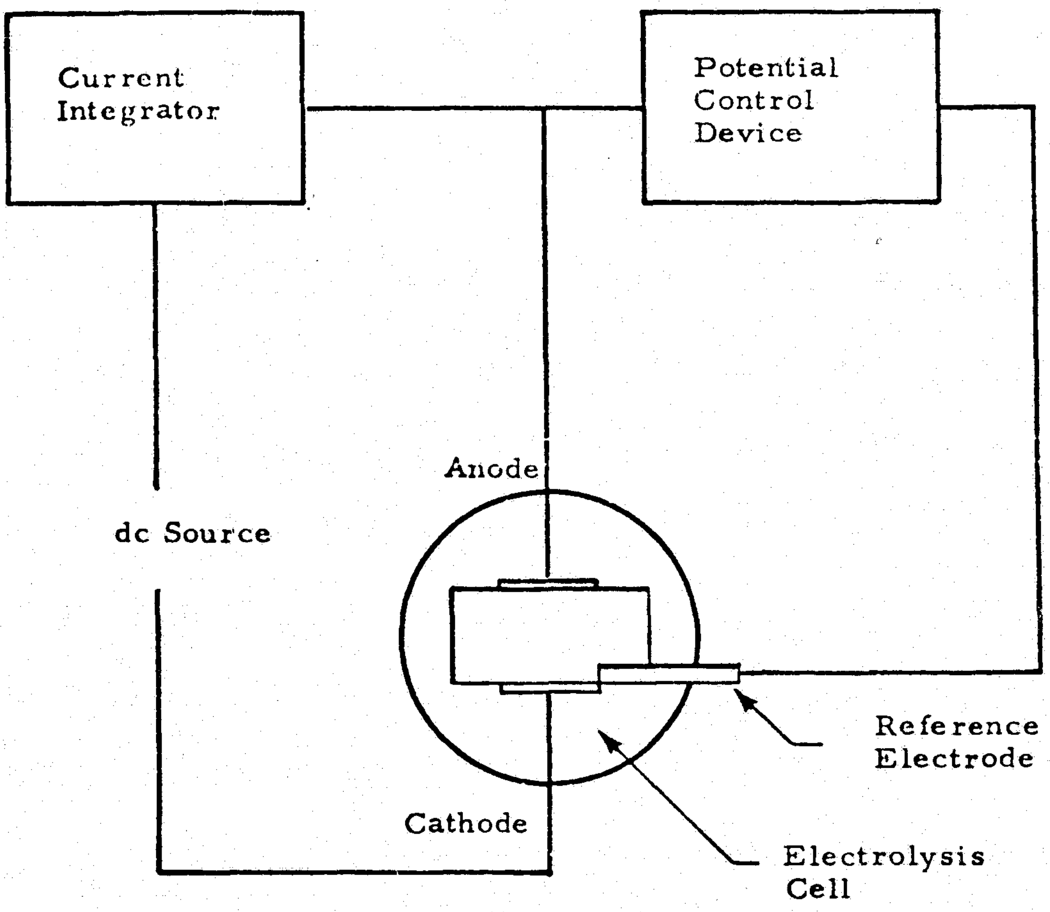


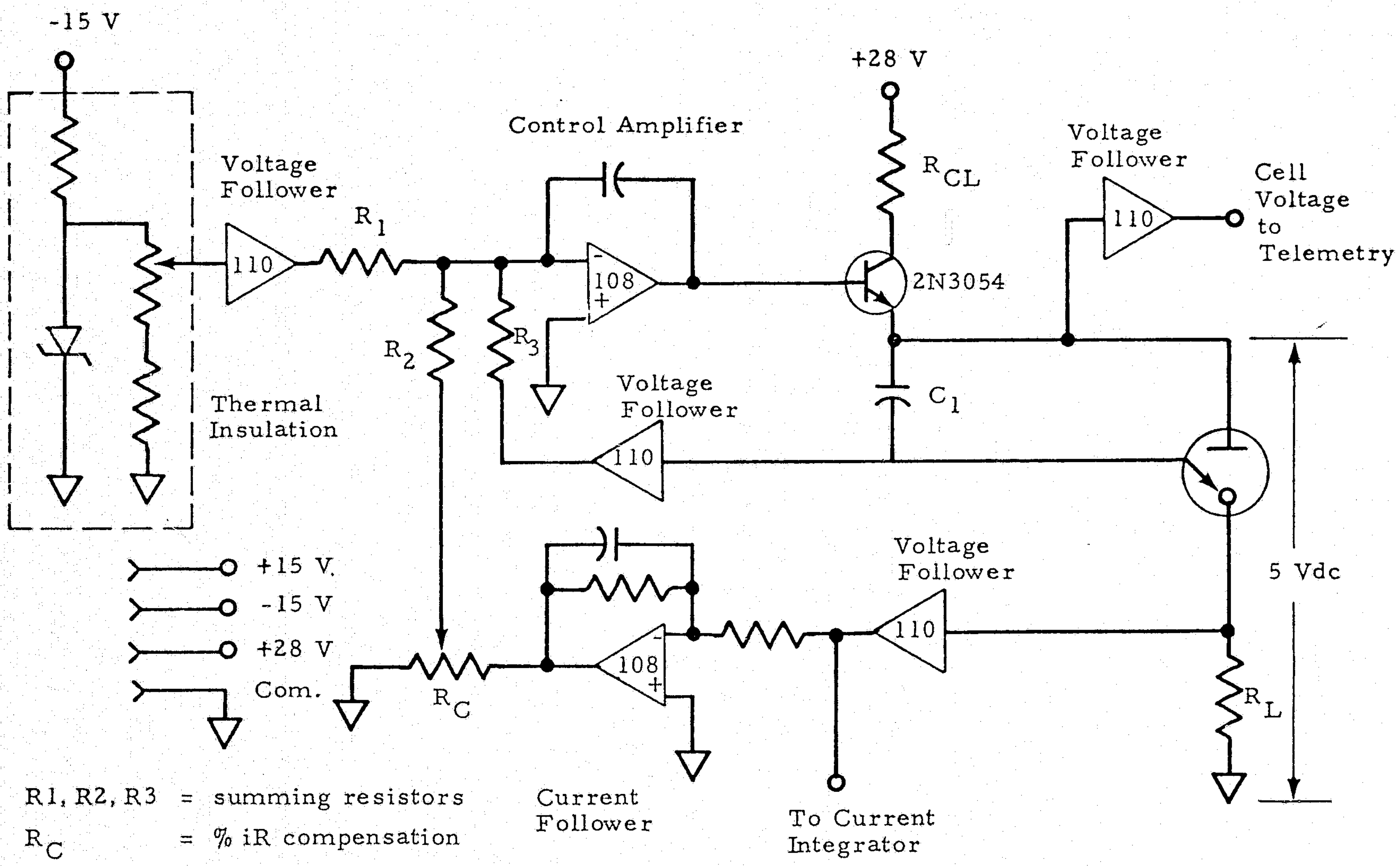
Fig. D-2 - Block Diagram Showing Relationship of the Current Integrator and Reference Electrode Potential Control Device to the Electrolysis Cell

The circuit diagram for the potentiostat to be used to control cell operation is shown in Fig. D-3. The potentiostat system consists of: (1) a voltage regulator circuit which regulates the potential drop across the cell to match a preset value; and (2) a cell "voltage drop" compensation circuit to compensate for changes in the resistance of the cell due to electrolyte modification.

Included in the potentiostat circuitry are outputs of various signals required to monitor the progress of the electrolysis process. These signals will be "conditioned" and then relayed to ground by the vehicle telemetry system. The circuitry required to "condition" the electrolysis process monitoring signals is shown in Fig. D-4. As indicated, the scaled output signals will conform to the 0 to 5 Vdc range required by the launch vehicle telemetry system.

D.3 OPTICAL DATA ACQUISITION SYSTEM

The optical data acquisition system to be utilized in the electrochemical crystal growth experiments is shown in schematic form in Fig. D-5. The basic components involved are: (1) a zirconium arc lamp; (2) a cylindrical concave mirror; (3) two large rectangular plano-mirrors; (4) one small square plano-mirror; (5) a plano-convex lens; and (6) a movie camera (28 Vdc, 25 W). The light source and the optical components are configured (as shown in Fig. D-5) to provide a folded path light system to illuminate the electrolysis cell volume and record the events occurring in the cells on movie film. The movie camera to be used is the standard NASA data acquisition camera (DAC) used on the Apollo flights (SEB 33100100, 28 Vdc). It is required that the camera be supplied as Government-furnished equipment for use during this experiment. The optical data acquisition system and electrolysis cell module will be mounted on a common support structure. Side plates will close the support structure to any outside light sources.



R1, R2, R3 = summing resistors

R_C = % iR compensation

R_L = cell load resistor

R_{CL} = current limit resistor

C_1 = stabilizing capacitor

Fig. D-3 - Circuit Diagram for Potentiostat Control System for the Electrolysis Cell

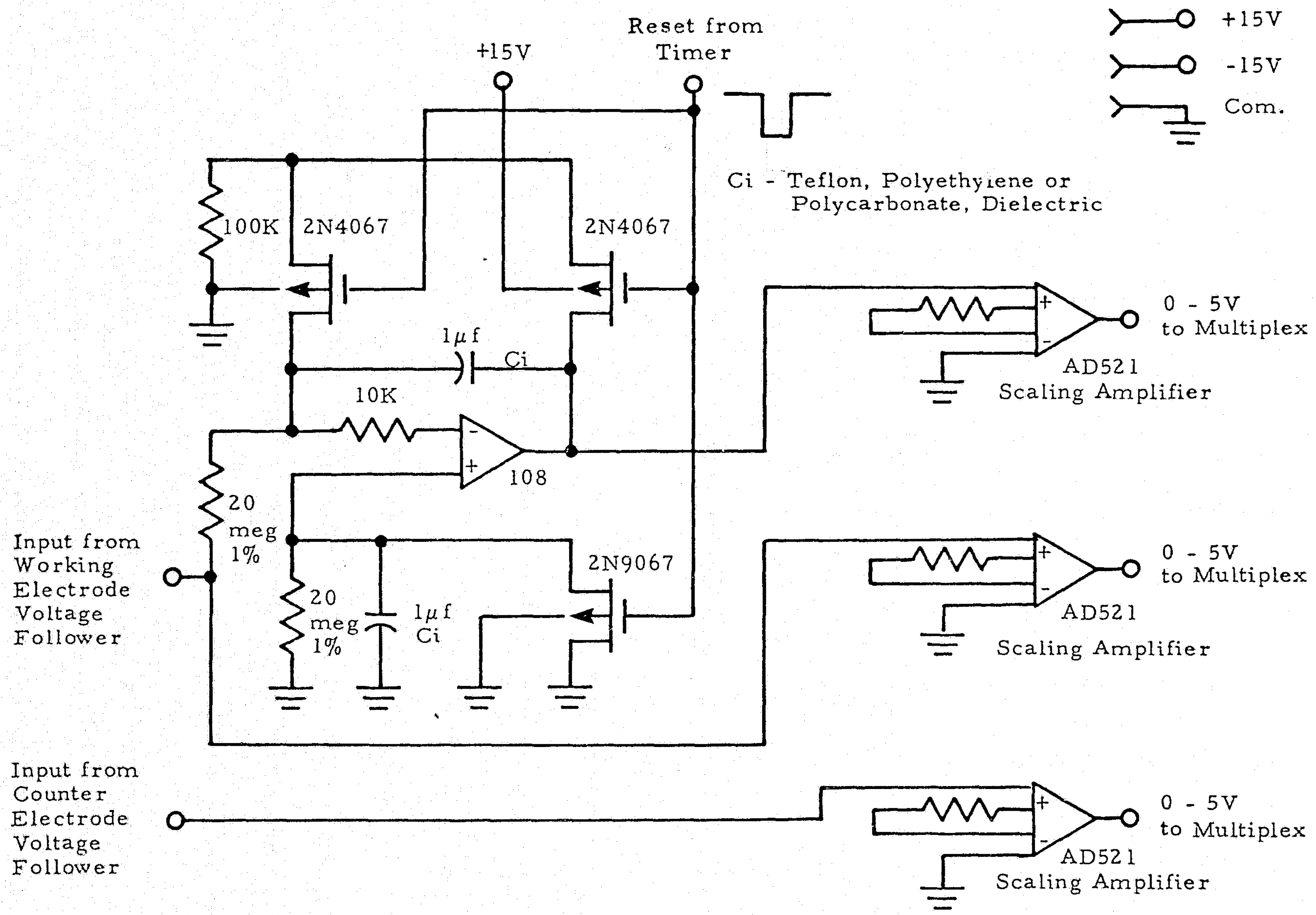


Fig. D-4 - Circuit Diagram for Current Integrating and Telemetry Signal Conditioning

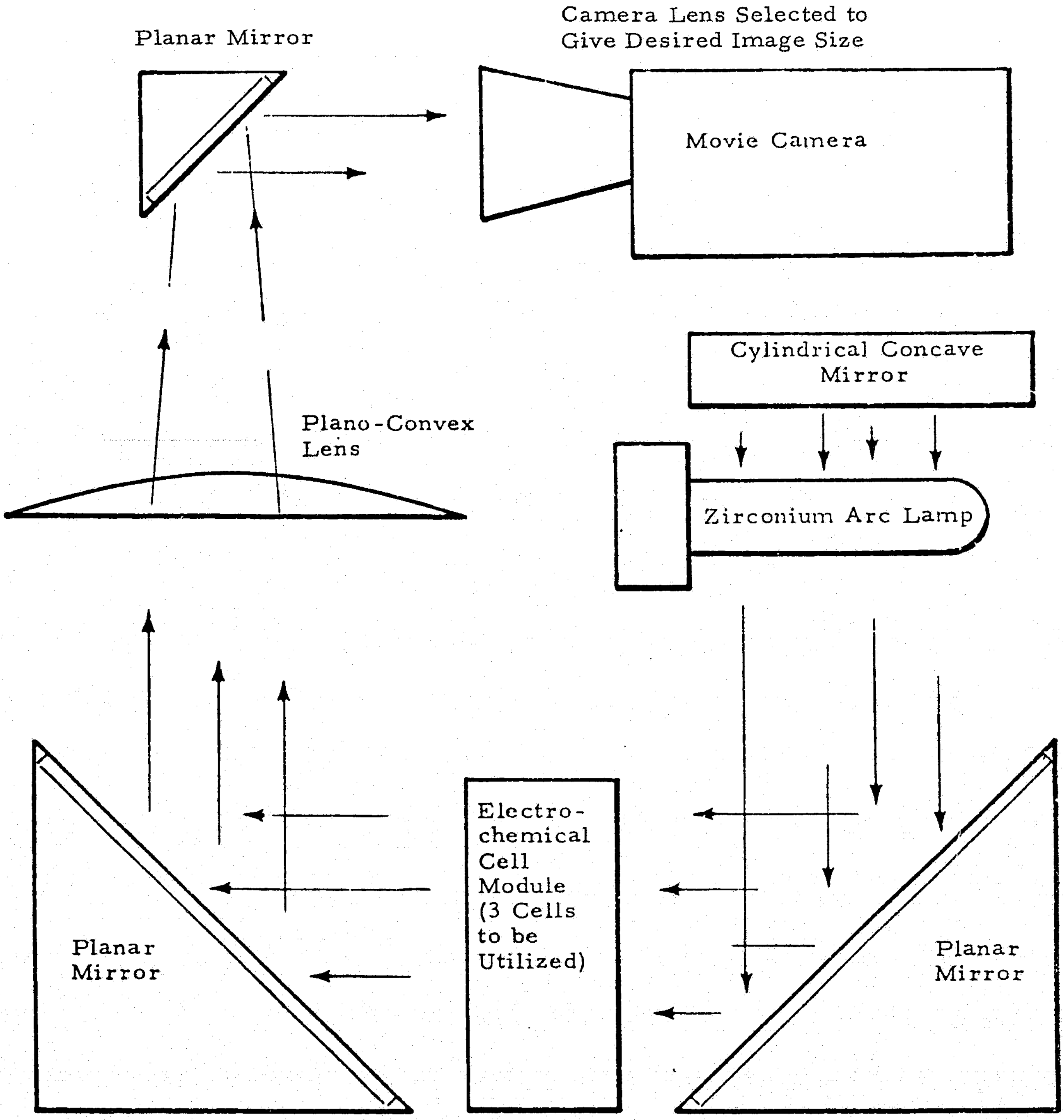


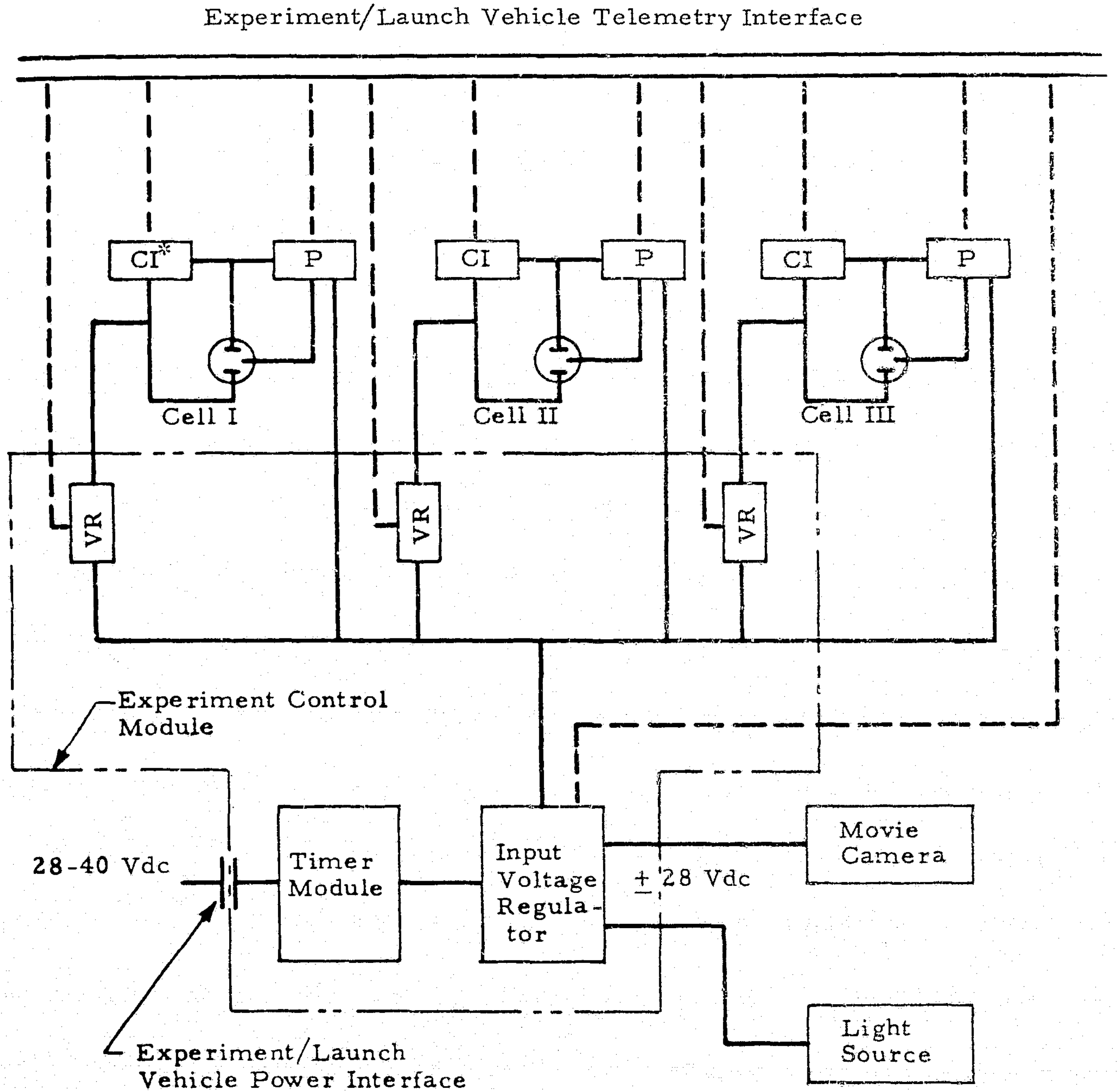
Fig. D-5 - Schematic Showing Relationship of the Components that Comprise the Optical Data Collection System

The optical data acquisition system will be activated at the same time that power is applied to the electrolysis cells to provide a correlation between optical data and telemetered information. The resulting movie film will be retrieved and developed at the termination of the flight sequence.

D.4 EXPERIMENT CONTROL MODULE

The experiment control module provides: (1) the interface with the launch vehicle power source; (2) correct sequencing of experiment functions (activation, termination, etc.); and (3) functional paths for any ground calibration and continuity checks that may be required. Figure D-6 presents a block diagram showing the relationship of the experiment control module to other experiment components.

Interface with the launch vehicle and sequencing of the experiment functions will be accomplished using the same basic system that was utilized on the Convection Measurement Package designed and fabricated in part by Lockheed-Huntsville under Contract NAS8-27015. The source voltage from the launch vehicle will be regulated to ±28 Vdc using an input regulator (NASA Dwg. No. SSL-233776). Upon launch of the sounding rocket the regulated ±28 Vdc will be supplied to a dc-to-dc converter (Abbott CC15D1-0) and to the drive motor and operating contacts of a normally open 100 second delay timer (HI-G 1722-A-1803). The converted ±15 Vdc will be utilized to provide power to the controlled potential function for cell operation and as the initial power source for the electrolyte temperature measuring system. Upon closure of the 100 second timer contacts, the ±28 Vdc voltage will be applied to activate the cells, the optical data acquisition system, and the first of two 180 second delay timers (HI-G 1722-A-1803, the first normally open, the second normally closed). Closing of the first 180 second timer contacts activates the second 180 second timer. Completion of the second 180 second delay period causes the contacts of the second timer to open, terminating all experiment functions.



*Note: CI = Current Integrator; P = Potentiostat; VR = Voltage Regulator

Fig. D-6 - Block Diagram Showing Relationship of Experiment Control Module to Other Experiment Components

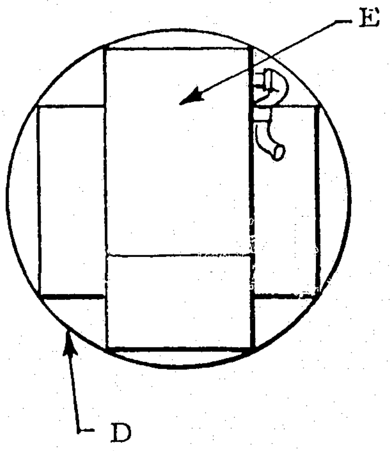
In addition to the interface and sequencing components just described, the experiment control module will also contain a source voltage regulator (zener diode type) for each of the electrolysis cells. The source voltage will not be the same for each cell. The voltage regulator component specification will therefore be fixed during the cell design task when cell potential requirements become firm.

Provision for calibration and continuity circuits is the final function to be provided by the experiment control module. An interface with the umbilical will be provided through which any calibration and continuity verification signals can be transmitted. The exact requirements for these functions will be identified as an integral part of the experiment design.

D.5 EXPERIMENT PACKAGE ASSEMBLY

The assembled Electrochemical Crystal Growth Experiment package is shown in Fig. D-7. The general experiment specifications are also noted on this figure. The experiment package will be assembled as follows: each of the primary modules (Items A, B and C, Fig. D-7) will be assembled independently and subjected to the appropriate post assembly checks and calibration. The electrolysis cell module (containing three cells) and the optical data acquisition components will be assembled into the center support structure to form Item A. Multiple pin connectors will be mounted on each side of Item A to provide the electrical interface with Items B and C (experiment power and control module and the electrolysis cell control and experiment telemetry interface modules, respectively). Assembly of the Items B and C modules is accomplished by first completing the electrical connections (plugging the modules into the multiple pin connectors) and then bolting the modules to base plate. Installation of cable F (power to optical data system) would complete the assembly of the experiment package.

The assembled experiment package would then be subjected to functional and continuity checks to ensure functional success of the experiment. Delivery of the electrochemical crystal growth experiment would then be accomplished in the schedule required to meet the sounding rocket flight sequence.



A – Electrolysis cell and folded optical system module

B – Experiment power and control module

C – Electrolysis cell control and experiment telemetry interface module

D – Base plate

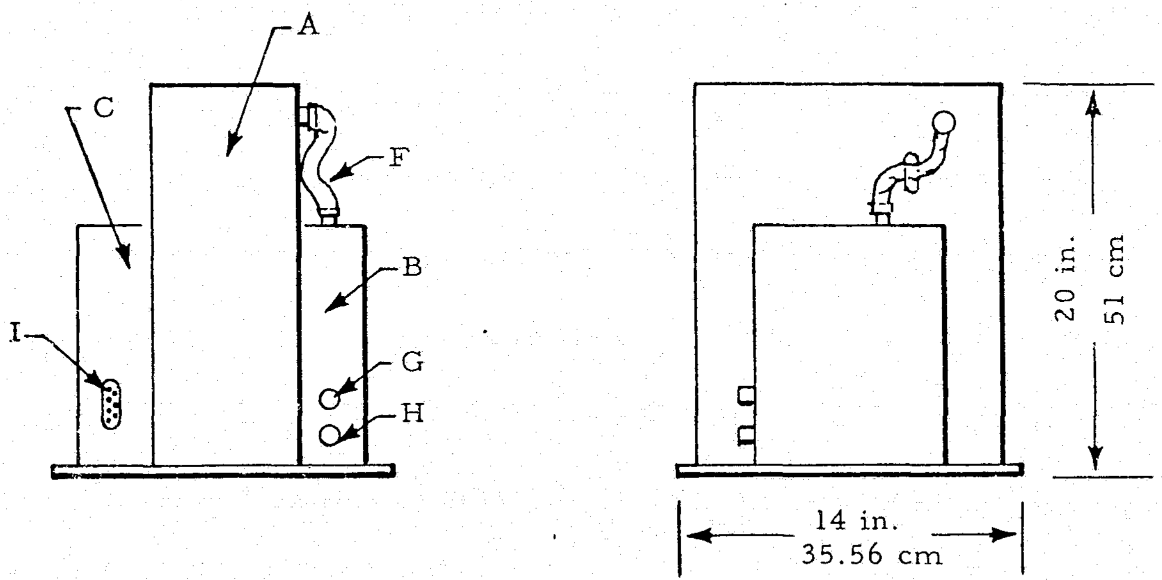
E – Movie camera access hatch

F – Optical system power and control cable

G – Interface plug for continuity and calibration

H – Interface plug for vehicle power

I – Interface connector for telemetry



Experiment Specifications

Volume: 50452 cm^3 (approx.)

Weight: 25 lb (approx) (11.36 kg)

Electrical Power Required: < 80 W

Telemetry Channels Required: 15 (approx)

Fig. D-7 - Conceptual Drawing of Assembled Electrochemical Crystal Growth Experiment for Space Processing Sounding Rocket Program

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