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DOE/JPL/955733--T1
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SECOND QUARTERLY PROGRESS REPORT
JANUARY 1 - MARCH 31, 1981
CPFF CONTRACT NO. 955733

DEVELOPMENT OF ADVANCED CZOCHRALSKI GROWTH PROCESS TO PRODUCE LOW-COST 150-KG SILICON INGOTS FROM A SINGLE CRUCIBLE FOR TECHNOLOGY READINESS

PROGRAM MANAGER: R. L. LANE

KAYEX CORPORATION
1000 MILLSTEAD WAY
ROCHESTER, NEW YORK 14624

"The JPL Low Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE."

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I INTRODUCTION

This program for "Advanced Czochralski Growth Process To Produce Low-Cost 150 kg Silicon Ingots from a Single Crucible for Technology Readiness" has several goals:

A. Provide a modified CG2000 crystal grower capable of pulling a minimum of five crystals, each of approximately 30 kg in weight, 150 mm diameter from a single crucible with periodic melt-replenishment.

B. Crystals to have: resistivity of 1 to 3 ohm-cm, p-type; dislocation density below 10^4 per cm^2; orientation (100); after growth yield of greater than 90%.

C. Growth throughput of greater than 2.5 kg per hour of machine operation using a radiation shield.

D. Prototype equipment suitable for use as a production facility.

E. The overall cost goal is $.70 (1980 $) per peak watt by 1986.

To accomplish these goals, the modified CG2000 grower and development program includes:

A. Increased automation with a microprocessor based control system which reduces operator attention and avoids operator errors.

B. Sensors development which, during the program, will increase the capability of the automatic controls system.

C. Process development which will: define the process control variables for accelerated growth rate using a radiation shield; analyze variations in the effects of silicon feed material and meltback rate of greater than 25 kg per hour; analyze the effects of these changes on the economic model; investigate and evaluate the effects of process variations on the "quality" of silicon produced by performing purity analysis of the silicon, solar cell fabrication/analysis, and furnace atmosphere analysis.

D. Provide technology transfer of the developed systems.

To accomplish the above goals, the program has been divided into five general task categories:

A. Construction and Test - to provide a modified CG2000 grower for process development and sensor/automated controls integration.

B. Process Development - for accelerated growth, accelerated recharge and yield/cost improvement.

C. Controls and Automation - for sensor development and microprocessor controls integration to the Mod CG2000.

D. Analytical Study - for purity analyses and solar cell fabrication.

E. Documentation - for reporting, economic analysis, and process specification.

These categories overlap in time phasing (see program plan).

II SUMMARY

The modified CG2000 crystal grower construction, installation, and machine check-out was completed.

The process development check-out proceeded, with several dry runs and one growth run. Several machine calibrations and functional problems were discovered and corrected. Several exhaust gas analysis system alternatives were evaluated and an integrated system approved and ordered.

A contract presentation was made at the Project Integration Meeting at JPL, including cost-projections using contract projected throughput and machine parameters.

Several growth runs on a development CG2000 RC grower have shown that complete neck, crown, and body automated growth can be achieved with only one operator input. Work continued for melt level, melt temperature, and diameter sensor development.

The overall program is very close to the expected program schedule.

III PROGRESS/PLANS

A. Construction and Test

As part of the 1982 Technology Readiness goals, the design modifications necessary for this contract were performed under JPL Contract No. 954888. The purchase and construction task of this project has proceeded in conjunction with the design completion.

Previous to this quarter, all design for the Mod CG2000 had been completed. During this quarter, all necessary components and subassemblies were available to complete the construction of the grower. (See Program Plan - Purchase and Construction).

The grower was located in its own room to ensure an uninterrupted utilities supply. All the corresponding utilities - argon, water, electrical , vacuum pumps - were installed.

Some modifications were necessary to the water, vacuum, and argon systems because of the dedicated room requirements. However, Hamco testing personnel, with the assistance of the Kayex Technology Center, completed the installation and grower functional checkout within two weeks of the expected time frame.

The photograph in Figure 1 shows the completed grower.

The Mod CG2000 was turned over to process development personnel to begin the preliminary hot-zone and functional process checkout.

The construction and machine debug task was completed during this reporting period. Any further design modification deemed necessary by process development or support of machine problems will be reported under process development.

B. Process Development

After support of the installation of the Mod CG2000, process "dry runs" commenced.

Three problems were apparent in the Power Controls Corporation (PCC) 150 kW power supply. Greater than expected power fluctuation (control) appeared during dry runs and the power unit is acoustically noisy at about 73 decibels. The power readout was also out of calibration. A PCC field engineer verified the minor power output fluctuations, recalibrated the power readout, and will prepare a solution to the acoustical problem.

On March 19, 1981, a growth run using a 30 kg charge of re-cycle silicon was tried. All the crystal was polycrystalline and diameter control of the crystal greater than 5.4 inches was not possible becuase a misalignment of the cable lift mechanism prevented correct imaging of the crystal/melt meniscus under vacuum. Heavy smoking and excessive oxide build-up also indicated a leak occurred in the chamber. Several other problems were noted:

1. MKS vacuum gage calibration.
2. The crystal diameter cathetometer required modification for more accurate diameter measurement.
3. The crystal length readout and the temperature channel of the recorder failed to operate.

All of these problems were rectified as well as:

1. Revision of the argon system
2. Relocation of the vacuum pumps external to the grower room
3. Installation of a seed lift cable
4. Shimming of the imaging mirror to extend the meniscus view for larger diameter crystal control
5. The entire grower was realigned under vacuum conditions.

Further evaluation runs on the grower are necessary to finish de-bug of the control system and hot-zone design before extended recharge runs are performed.

Other activities during the quarter included:

1. Receipt of crucibles, silicon, hot zone parts, dopant, etchant and other consumables
2. Design, quotation and order placement for a pyrolytic graphite coated radiation shield
3. Contract presentation at the Project Integration Meeting at JPL on February 5, 1981
4. Visit by the JPL technical project manager to Kayex on February 18, 1981.

C. Controls and Automation

1. General

The controls and automation tasks of this contract address the development of sensors for melt level, melt temperature and ingot diameter, and the integration of these sensors with the Kayex Automatic Grower Logic (AGL) computer-based control system* and the modified CG2000 grower.

During this quarter, an AGL system was made available to the project. The system was connected to a standard CG2000 RC crystal grower being used for sensor development under this contract. During the quarter, a total of twelve crystal growth runs were performed for the purpose of:

*Automatic Grower Logic is a proprietary development of the Kayex Corporation.

a. implementation and test of the automatic dip temperature setting technique;

b. definition and test of sensors for fully automatic growth of the ingot crown, shoulder and body.

During the quarter, activities also continued in the design and fabrication of the sensor mounts for the modified CG2000 grower being built for the contract.

2. Sensor Development

a. Melt Temperature

A commercial two color pyrometer (IRCON R-series) has been mounted on the standard development grower. The pyrometer views the melt at near-normal incidence through a port located at the top of the pull chamber. The control arrangement, shown schematically in Figure 2, automatically trims the heater power control loop to produce the proper melt temperature for seed dip.

This system has been used to set dip temperature for the last eight crystal growth runs on the developmental grower. The accuracy (and adequacy) of the dip temperature settings were evaluated based on the ability of the AGL computer system to automatically grow an acceptable neck following seed dip.

b. Ingot Diameter

A goal of this effort is growth of the ingot neck, crown and body with a minimum of operator judgement and input. Continued work with the Kayex-Hamco Automatic Grower Logic computer system has led to the conclusion that this goal is achievable without the complexity of a sensor system that continuously measures diameter through all stages of neck, crown and body growth.

The seed is dipped after melt temperature is stabilized as described above. Neck, crown and shoulder growth are controlled according to process specifications stored in tabular form in the AGL computer. The transition from neck to crown is initiated in response to an operator input indicating that the neck is thin enough. The operator obtains a prompting request for this input. This single decision is readily performed by an operator and eliminates the need for a complex sensor capable of responding to small changes in the growing neck diameter. Moreover, it is appropriate for the operator to make this decision since operator observation and judgement are desirable at this point in the process to determine whether the neck growth has acceptable structure.

During the quarter, candidate hardware for the shoulder and body sensors have been fabricated and evaluated. The selected sensors are IRCON type Z pyrometers with modified optics. Integrated tests with the AGL system will be performed first on the standard CG2000 RC development grower and then on the modified CG2000 during the next quarter.

c. Melt Level

A reflected laser melt level sensing system similar to that described by C. S. Duncan, et. al.* is being developed for implementation on the modified CG2000 grower. During this quarter, a preliminary test was conducted by using the laser/detector system to control the height of a water filled crucible. Water was siphoned from the crucible to simulate lowering of the silicon melt level. Water level in the grower was maintained $\pm$ 0.5 mm, while the water level in the crucible changed by 50 mm. Designs are in progress for mountings and optics to test the system on the modified CG2000 grower.

D. Analytical Study

The analytical task consists of three areas:

1. Purity Analyses of Silicon - The control of silicon purity will be achieved by chemical impurity analysis of selected samples from feedstock, grown ingots and residual melt.
2. Solar Cell Fabrication and Analysis - Selected ingot material from all 150 kg runs will be sliced into wafers and, along with several control samples, fabricated into solar cells. These cells will also be tested for solar efficiency.

* "Silicon Web Process Development Annual Report", June 30, 1980, Contract No. 954654 DOE/JPL - 954654/80/11.

3. Furnace Atmosphere Analysis - A gas chromatograph and sampling system will be used to monitor the oxygen and other possible impurities.

During this quarter, a suitable furnace atmosphere analysis system was found. However, several approaches to the problem were investigated and evaluated. This work showed that standard gas chromatograph systems (G.C.) using normal detectors would not be sensitive enough to analyze the low concentrations of elements and compounds present. The system would be operating at approximately one hundredth (1/100) of an atmosphere. A standard G.C. system with sensitivities rated at one atmosphere would therefore be too insensitive. Also, the grower is purged with argon, which would also be difficult to separate from oxygen using common detectors and G.C. techniques.

Several integrated systems capable of resolution and analysis of the low concentrations in a grower were successfully pursued.

The chosen system consists of a gas chromatograph using a suitable solid state detector, allowing the measurement of concentrations of carbon monoxide and hydrogen.

Coupled with the G.C. will be an oxygen analyzer and a hygrometer for measurement of moisture concentration.

The system was presented to the JPL technical project manager on February 18, 1981 and approved.

Approval was also gained to obtain the necessary components on a lease/buy arrangement as far as possible.

Acquisition of components, system design and construction should be complete by mid-May.

E. Documentation - Economic Analyses

A SAMICS/IPEG CZ add on cost projection was completed in February.

The cost is projected utilizing a desired throughput rate of 2.5 kg per hour at 6" diameter. Projections are made for the production of 150 kg of crystal in a total time of 60 hours as follows:

	CZ ADD-ON $/M^2	$/Peak Watt
1. Cost 1 - pulling 5 crystals each 30 kg in weight	20.12	0.1419
2. Cost 2 - pulling 3 crystals each 50 kg in weight	19.24	0.1357

It should be noted that to complete either example in a total of 60 hours necessitates pulling at a faster straight growth rate per hour for the five 30 kg example (4.49 in/hour). The reason for this is that the pulling of five crystals requires more crucible recharging operations to be performed than growing three 50 kg crystals. This reduces significantly the amount of time available for crystal growth.

A further cost projection (Cost 3) shows the cost advantage that could be gained if a straight growth rate of 4.49 in/hour could be achieved when producing three crystals each of 50 kg in weight.

	$/M^2	$/Peak Watt
3. Cost 3 - pulling 3 crystals each 50 kg in weight at 4.49 in/hour	16.54	0.1166

IV PROGRAM PLAN

The program is proceeding well, with only minor delay in the process development area due to de-bug problems. The final construction and test phase schedule update is included in Figure 3. This task is now completed. An update of the program plan is included in Figure 4.

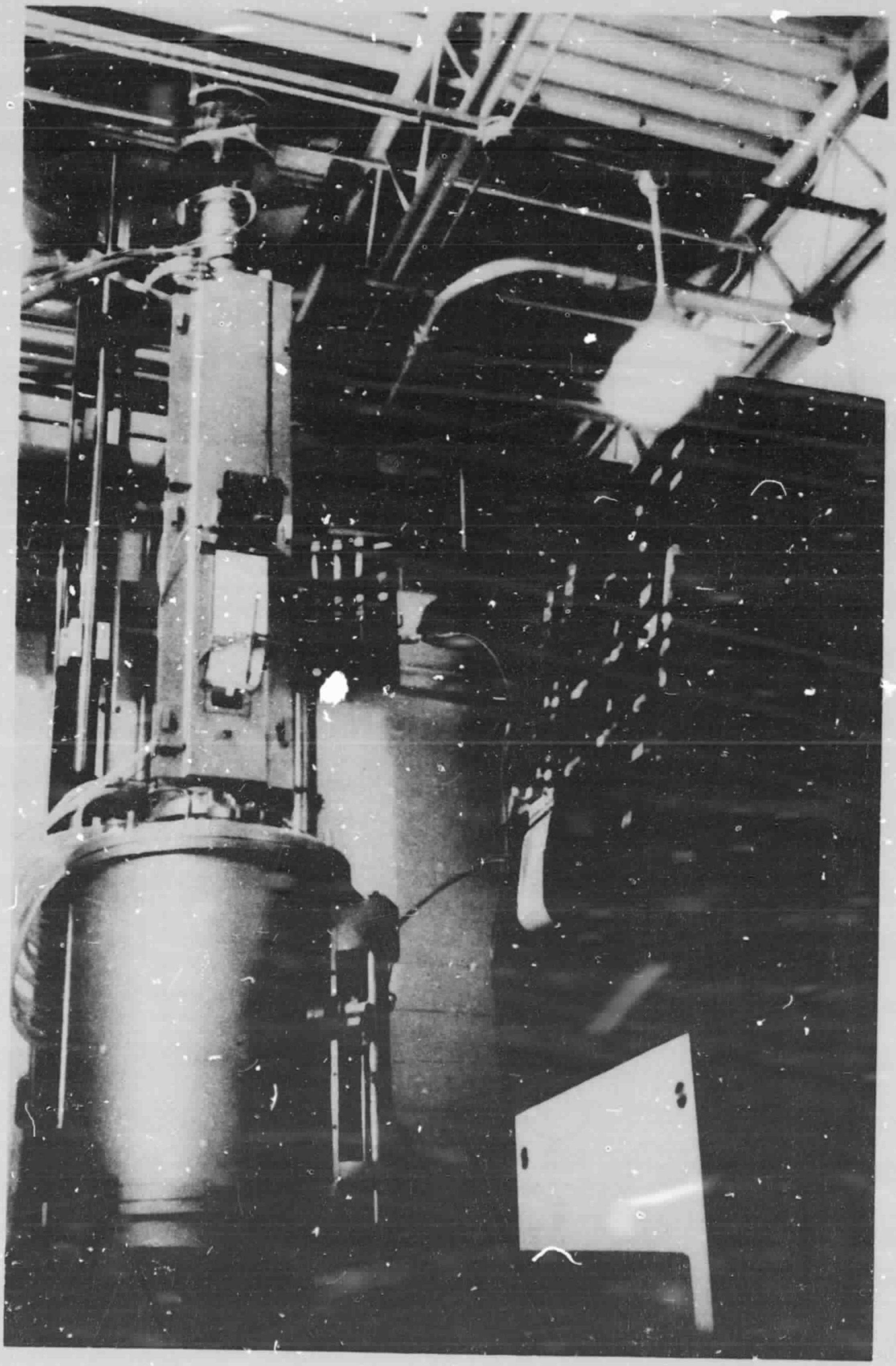

Figure 1
Photograph of Completed Mod CG2000 RC

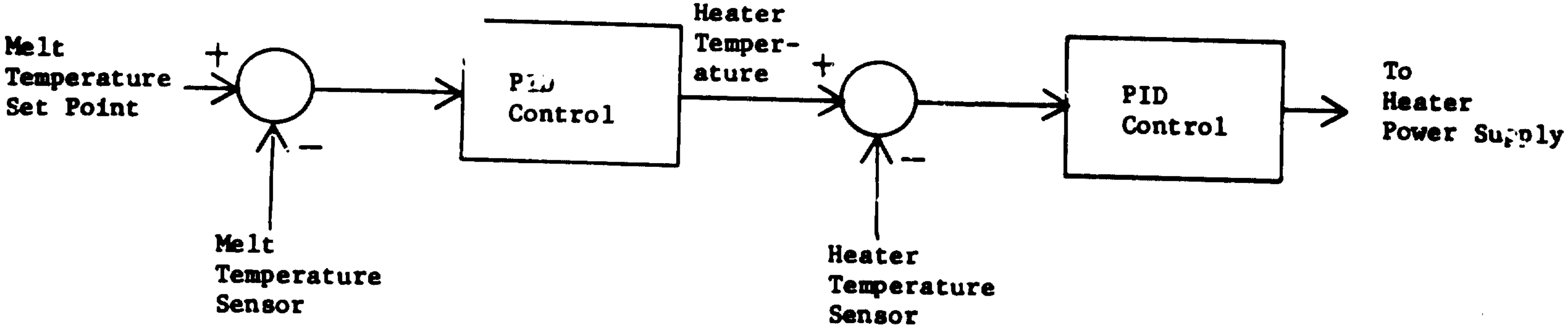

Figure 2: Cascade control configuration for setting melt temperature prior to seed dip

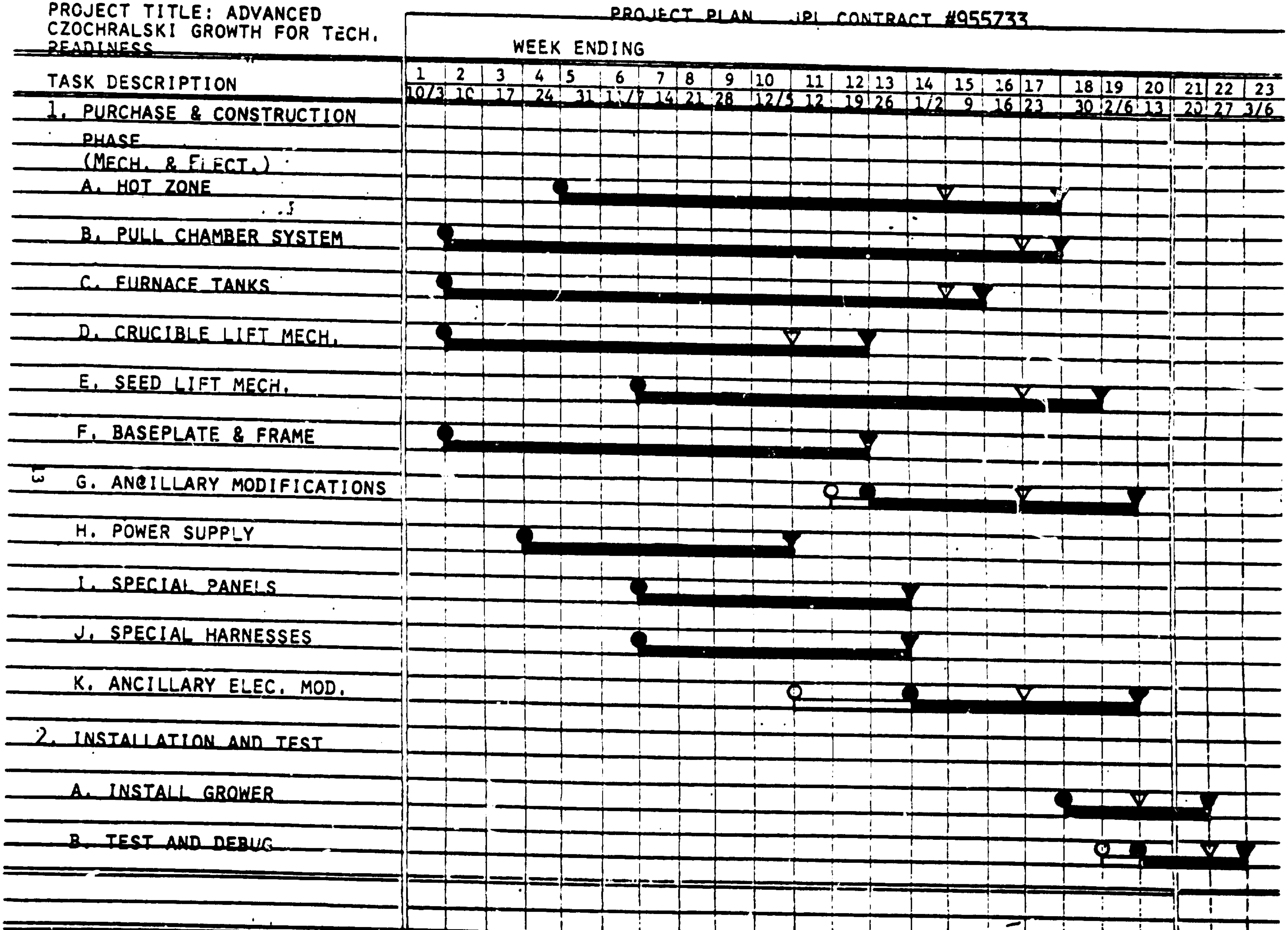
PROJECT TITLE: ADVANCED CZOCHRALSKI GROWTH FOR TECH. READINESS
PROJECT PLAN JPL CONTRACT #955733
WEEK ENDING
TASK DESCRIPTION
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23
10/3 10 17 24 31 11/7 14 21 28 12/5 12 19 26 1/2 9 16 23 30 2/6 13 20 27 3/6
1. PURCHASE & CONSTRUCTION PHASE (MECH. & ELECT.)
A. HOT ZONE
B. PULL CHAMBER SYSTEM
C. FURNACE TANKS
D. CRUCIBLE LIFT MECH.
E. SEED LIFT MECH.
F. BASEPLATE & FRAME
G. ANCILLARY MODIFICATIONS
H. POWER SUPPLY
I. SPECIAL PANELS
J. SPECIAL HARNESSES
K. ANCILLARY ELEC. MOD.
2. INSTALLATION AND TEST
A. INSTALL GROWER
B. TEST AND DEBUG

Figure 3

Project Title:
Advanced Czochralski Growth
for Technology Readiness

Program Plan Revision No. 1

CONTRACT #953733