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(NASA-CR-169186) DEVELOPMENT OF ADVANCED
CZOCHELSKI GROWTH PROCESS TO PRODUCE LOW
COST 150 kg SILICON INGOTS FROM A SINGLE
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FIFTH QUARTERLY PROGRESS REPORT
OCTOBER 1 - DECEMBER 31, 1981

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 Flat Plate Solar Array (FSA) 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 in 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 these goals, the program has been divided into five general 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 control integration to the Mod CG2000.
- D. Analytical Study - for purity analyses and solar cell fabrication.
- E. Documentation - for reporting, economic analysis, and process specification.

II SUMMARY

Process development continued with redirection from a Technical Direction Memorandum. The emphasis for these last months of the contract will include continued investigation of (1) throughput rate using a cone-shaped radiation/purge shield and (2) crucible-melt interaction with the Furnace Gas Analysis System. Availability of crucibles with minimum "bubble" content and/or synthetic quartz will also be investigated.

Six growth runs used the Kayex-Hamco Automatic Grower Logic (AGILE) computer based system for growth from larger melts in the Mod CG2000. Also, the implementation of the melt pyrometer sensor allowed for dip-temperature monitoring and usage by the operator/AGILE system. Use of the AGILE during recharge operations was successfully evaluated. The tendency of crystals to lose cylindrical shape (spiraling) still continues to be a problem.

The hygrometer was added to the Furnace Gas Analysis System and used on several growth runs. The gas chromatograph, including the integrator, was also used for more accurate carbon monoxide concentration measurements. Efforts continued for completing the automation of the total Gas Analysis System. The oxygen analyzer was added in late December, but preliminary tests indicate it will not measure properly at the reduced pressure of the crystal grower atmosphere.

An economic analysis, based on revised achievable straight growth rate, is presented. The throughput is 1.92 kg/hour. When achieved, the program goal of 2.5 kg/hour will result in an additional 15.3% reduction in CZ add-on cost.

III PROGRESS

A. Construction and Test

The Mod CG2000 was completed in March, 1981. When necessary, modifications to the grower are reported in other sections of the report.

B. Process Development

Preface:

As a result of the Final Design and Progress Review, along with a Technical Direction Memorandum received in September, 1981, increased emphasis will be placed on two areas for the remainder of the contract:

- a. The throughput rate will be addressed with a heat shield (cone) for both melt recharge and crystal growth rates.
- b. Crucible-melt interaction will continue to be monitored by the Furnace Gas Analyzer System reported in Section D. Because of concern for size, migration, and quantity of bubbles in the fused quartz, availability of crucibles will be investigated using synthetic quartz and also for crucibles with "minimim" bubble size and numbers.

1. Accelerated Growth

The method chosen for increasing crystal growth and recharge rates is the use of a radiation shield/purge cone. The materials considered for cone fabrication were molybdenum, graphite, and pyrolytic coated graphite. Since the purge cone will be used in conjunction with the recharge hopper, it was decided to proceed with a molybdenum cone. The possible bumping of the silicon chunks on the cone might cause some particles of graphite from other type cones to fall into the silicon melt. Purchase orders were placed for both the molybdenum sheet (.060" thickness) and graphite supports. The molybdenum cone and support assemblies are expected to be available in January, 1982.

2. Crucible Melt Interaction:

A prime suspect for the cause of persistent structure loss during multi-crystal runs is crucible deterioration. Although the exact mechanism is unknown, some sort of a picture has emerged from technical discussion with those involved with crucible manufacture. There is evidence that the typical "rosette" pattern on the inner crucible wall, where it has been in contact with molten silicon, is composed of an island of cristobalite surrounded by a ring of SiO_{2-x} . These rosettes grow and coalesce as time and the extent of crucible dissolution proceed. The problem in a multi-crystal run occurs when the molten silicon has dissolved away the inner glassy surface of the crucible (after, say, 40 hours) and starts to expose bubbles. The bursting of bubbles at the melt/crucible interface might then release cristobalite particles into the melt, leading directly to crystal structure loss.

To test this theory, it is necessary to obtain some crucibles with zero, or at least greatly reduced, bubble content. Several possibilities exist:

- a) Synthetic quartz - high purity, bubble-free material is currently produced in tube form by Thermal American ("Spectrosil") and Heraeus-Amersil ("Supersil"). Size and cost are the most likely limiting factors with this material.
- b) Clear fused quartz - crucibles would be produced from tube starting stock.
- c) Slip-cast silica - crucibles currently produced by Pyromatics are sintered and fired after slip-casting and this technique produces a transparent, low-bubble content crucible.
- d) Synthetic quartz lining - it might be possible to put a synthetic quartz lining into a standard arc-fused crucible.

- e) "Re-glazed" crucible - an extra fusing step is utilized to produce a thicker glassy layer on the inner crucible surface.

Crucible manufacturers (i.e. QSI, Thermal American, Heraeus-Amersil) have been asked to quote on crucibles in the size range 12" - 16" diameter fabricated from this above list of possible materials. The results of these inquiries will be reported as soon as these potential vendors respond.

3. Process Runs

During November, Run 14A (a duplicate run number 14 was entered in the log by error and has been changed to 14A) was made for continued testing and training with the Kayex-Hamco Automatic Grower Logic Control System. This run was originally scheduled to produce a 30 kg crystal from a 40 kg melt, and, if successful, i.e. zero-D or single, to proceed with recharging and growth of subsequent crystals until retention of structure was no longer possible. For this run, the latest additions to the gas analyzer system were also to be tested (see section II-D).

During the meltdown sequence, at about the time that molten silicon was first observed in the bottom of the crucible, it was apparent that one crucible support segment was skewed relative to the crucible and almost touching the upper heat shield ring. Although the situation was awkward, it was decided to complete the meltdown and try to grow a crystal rather than abort a 40 kg charge. However, by the end of meltdown, the situation had worsened with two crucible support segments pushed out and touching the top ring. It was obvious at this stage that two crucible support segments had cracked, and that vibration caused by these segments rubbing against the top ring would make crystal growth difficult. Vibration was reduced by adjusting crucible rotation rate and crystal growth was initiated after seed dip. However, crucible distortion had occurred, where the supports had pushed out, and, consequently, a thermal stability

problem arose which led to wall-freeze.

The run was continued in an attempt to recover as much silicon as possible. The resulting crystal was approximately 15 kg in weight, with diameter greater than 6 in., grown under manual control and not automatic diameter control (ADC). Because of the continuing problem of wall-freeze, the growth was very slow, but it did allow plenty of time to operate the gas analyzer and, thus, monitor the grower atmosphere.

During the remainder of the quarter (December), five crystal growth runs were made. The main objectives of these runs were to determine the effect of some hot zone changes on the continuing problem of spiraling, and to more closely evaluate the interface between microprocessor control and growth from large melts.

In preparation for run No. 15, the crucible shaft was removed and machined to clean up the top area where it had overheated and reacted with the graphite. After clean up, the shaft was re-installed and fitted with a new pedestal. The graphite insert for the heat pack was relocated to rest on the heat pack itself, effectively lowering the top ring assembly by 1/2". Run No. 15 was made without computer control.

This run produced 16.3 kg of crystal, of which approximately 11 kg were zero-D. The growth was terminated prematurely because of a control problem in the diameter controller, causing wide pull rate fluctuations. The result was a crystal distortion which resembled an exaggerated version of the spiral growth already evident in the first 10-1/2" of growth. See Table I for further run data.

The problem with the diameter controller was circumvented for run No. 16 by utilizing the microprocessor system and its implicit control logic. During meltdown, it was found necessary to interrupt the computer

in order to alter crucible position and heater power to melt off silicon chunks adhering to the crucible wall. After stabilization and dip, a neck and crown were grown successfully at the first attempt. However, the crown was grown quite quickly and the microprocessor recipe could not turn the shoulder fast enough. Consequently, the crystal diameter drifted out to approximately 6.5" before straightening up. Structure loss occurred after 2" of body growth and spiral growth was evident at 4". This run was terminated at an ingot weight of 19 kg (Table I) due to wall-freeze.

Run No. 17 was attempted under similar conditions to the previous run. The main differences were changes made to the computer recipe for the power levels during meltdown, and for the shoulder turning sequence during crystal growth. The result was a successful neck, crown, and shoulder, and a zero-D crystal grown at 5.98-6.0 inches diameter with an average pull speed of 3.08 in/hr (Table I). As in the preceding run, however, the wall-freeze problem was encountered and the run was stopped.

In order to increase the radial thermal gradient and thus reduce the tendency to wall-freeze, the melt level for run No. 18 was raised by 1/2". This change was successful in avoiding wall-freeze, but structure loss occurred after 6" of crystal growth. There was still a slight spiral appearance to the resulting crystal. Refinements to the computer recipe continued to be made.

At the end of run No. 17, it had been found after measurement that the heater was slightly distorted and not concentric about the crucible. The concentricity was improved in preparation for run No. 19 to see whether there would be any beneficial effect in regards to spiral growth.

Run No. 19 was initiated with a 20 kg charge, but it was planned to hot-fill a further 5 kg using the recharge apparatus. The object of this

exercise was to see how successfully the computer program could be interrupted to accommodate a recharge operation. In fact, there was no problem making manual adjustments to power for the hot-fill sequence and later returning to computer control, but the total meltdown time was a lengthy 3.6 hours.

After dipping the seed, a zero-D crystal was grown at the first attempt with no problems during the shouldering step. Structure loss occurred after 10.4 kg growth and final ingot weight was approximately 20 kg (Table I). There was apparently no difference in the degree of spiral evident on the crystal.

Plans for next quarter include the following:

- a) Two more growth runs will be made in 15" crucibles with charge weights of up to 40 kg.
- b) The hot zone will be changed to 16" size and run with the molybdenum radiation shield/purge cone.

C. Controls & Automation

1. Mod 2000 Controls

The Kayex-Hamco Automatic Grower Logic (AGILE) computer-based control system has been operational since August. It was used during the quarter for process development runs as reported in Section B.

2. Sensor Development

a. General

The development and test program conducted on the standard CG2000 RC grower has been concluded. The implementation of the shoulder and diameter sensors was discussed in the June, 1981 Quarterly Progress Report.

Activities this quarter centered on the implementation and testing of the melt pyrometer on the Mod CG2000 grower and the completion and bench testing of the melt level sensor electronics.

b. Melt Temperature Sensor

The melt temperature sensor implementation was changed to improve the accuracy and reproducibility as described in the September, 1981 Quarterly Progress Report. The periscope that allows the pyrometer to view the melt at 90° is illustrated in Figure 1. The periscope assembly replaces the spacer section between the Mod 2000 pull chamber and the cable lift mechanism. The optical pyrometer mounts on the flange (item 1) and views infra red radiation from the melt reflected by the gold-coated front-surface mirror (item 2). Pyrometer optics were specified to give a 0.5 inch diameter field of view at the melt surface.

The output of the pyrometer electronics module is a linear signal proportional to melt temperature. This signal is filtered to minimize the effects of variations in the melt surface temperature. Melt temperature stabilization is achieved by means of the control arrangement illustrated in Figure 2. The filtered output of the melt pyrometer is the input to a PID Dip Temperature Controller whose set point is the desired melt temperature. The output of the Dip Temperature Controller modifies the set point of the PID Heater Controller. This arrangement results in the automatic determination of the proper melt temperature for seed dip.

c. Melt Level Sensor

The melt level sensing technique being implemented is based on the system described by C.S. Duncan, et. al. in the March, 1981 Quarterly Report for DOE/JPL Contract 954654. The principle of operation is illustrated in Figure 3.

The output of the He-Ne laser enters the grower through a port and is reflected off the surface of the silicon melt to a second port. The receiver lens mounted just outside this port serves to reconverge the

laser light that has been scattered by ambient disturbances on the melt surface. An interference filter is used to eliminate light except for a narrow band centered on the laser wavelength. A linear, position-sensitive detector located at the focus of the receiver lens generates outputs which are processed by the electronics to give a signal proportional to the position of the spot on the detector being illuminated by the reflected laser light. A change in melt level results in a position change of the illuminated spot. The change in the output signal will be used as a servo input to drive the crucible height to maintain melt level constant.

The electronics have been assembled and the system has been bench tested. These tests indicate sensitivity to melt level changes of less than 0.5 mm. The detector is a Model LSC 30-D manufactured by United Detector Technology of Culver City, California. The difference of the detector's output signals is proportional to the product of the illumination intensity and its position along the detector axis. The sum of the output signals is proportional to intensity only. The circuit illustrated includes a divider whose output is then positioned independent of intensity. This output is buffered and filtered to give the final melt level output.

Designs for mechanical mounts for the laser and other components are complete and are being fabricated. The system will be mounted on the Mod CG2000 for tests with a silicon melt during the next quarter.

D. Analytical Study

One of the goals of this task is to use a Furnace Gas Analysis System to monitor furnace atmosphere during process development. The system includes a gas chromatograph with integrator and automatic sampling system for carbon

monoxide (CO) and possibly other gases. It also includes a hygrometer and oxygen analyzer for water and oxygen concentration measurements.

During this quarter, the integrator for the gas chromatograph, the hygrometer, and the oxygen analyzer were released from hold status.

The H.P. 3390 Reporting Integrator was received the second week in October, interfaced with the G.C., and preliminary testing performed.

No crystal growth runs on the Mod CG2000 were made in October and the Reporting Integrator coupled with the G.C. were used during a bakeout on October 27. Two peaks were indicated during this bakeout: one at 35 seconds retention time (R.T.), the second at approximately 2 minutes and 30 seconds retention time. A CO calibration gas sample had a peak at an R.T. similar to the second peak of the furnace sample. The 35 second R.T. peak corresponds to peaks seen on earlier bakeout and growth runs and is due to a light gas, possibly hydrogen. The gas will be verified later.

On 11/3/81, the Panametrics Hygrometer was installed into the Furnace Gas Analysis System and tested using argon flow through a cold crystal grower (Mod CG2000). The hygrometer and sensor appeared to be operating correctly, as evidenced by a decrease in the dew point of the sampled gas when the sampling valve to the grower (argon atmosphere) was opened. Conversely, when the sampling valve to the grower was closed, the dew point (moisture level) of the sampled gas increased. This would be consistent with the argon having a lower dew point than the residual gas in the lines from the sampling valve to the sensor housing.

On 11/6/81, a test was performed to compare peak characteristics of the integrator charts with those peaks produced on the two pen chart recorder. The comparison was performed using a calibration gas with 2390 ppm of carbon monoxide (CO) in argon. The effects of simultaneous operation of the integrator and the chart recorder were also investigated at this time.

The results indicate that simultaneous operation of the H.P. integrator and the two pen recorder do not create electrical interference problems that adversely affect the peak heights and corresponding areas under the curves. For example, an increase of 0.2 torr from 7.8 torr to 8.0 torr resulted in a peak height variation of 14% on the two pen recorder. Comparisons of area changes, as indicated by the Integrator when the vacuum levels of the sampling system vary, have not been attempted as yet. However, it was observed in this test that the areas under the curves varied by 0.8% to 0.9% when calibration gas samples were repeatedly run. Since this is a highly sensitive GC, it is expected the large percentage deviations in area convert to relatively small concentration deviations in parts per million.

A change in the equilibration time before sample injection was increased to one minute during this test without affecting the peak height (no external leaks).

Retention times of the carbon monoxide peaks during this test were from 2.45 minutes to 2.48 minutes as recorded by the integrator printout. Each sample and corresponding analysis was performed manually.

The procedure is as follows:

1. Purge rotary valve in load (backflush) position with calibration gas for one minute.
2. Close valve to G.C. calibration gas supply and allow sample in sample loop to equilibrate to the system vacuum (7.6 torr). Normal time - 30 seconds.
3. Manually inject gas in sample column thru G.C. columns by switching the rotary valve position using the Digital Valve Interface (DVI) manual switch.

NOTE: The DVI can be activated electrically by using a timer system or

a programmable control system, when either is used as part of the system.

Due to the equilibration time (usually 30 seconds), the actual retention time following injection of the sample was actually one minute and fifty seven seconds to one minute and fifty nine seconds. The equilibrium time must be subtracted from the recorded retention times.

NOTE: Retention times also vary as a function of the flow rate of the carrier gas. Therefore, the carrier gas flow rate must be maintained constant.

Based on the results of this test, it appears there will be no problem operating the integrator in conjunction with the two pen recorder for recording the outputs from the hygrometer and oxygen analyzer.

On November 19, the first crystal growth run (Run No. 14A) using the integrator to record sample peaks analyzed by the G.C. was performed. The hygrometer was also used to monitor moisture levels (dew points) continuously through the run.

All G.C. and integrator operations were performed manually. Random samples were taken from the crystal grower during meltdown, stabilization and portions of the crystal growth cycle. The G.C. outputs were recorded both on the integrator chart and the two pen recorder and corresponded well.

The results of this growth run are as follows:

- 1) Three calibration sample runs were performed initially to establish a reference peak area.

Average area = 1.52×10^7 for CO concentration standard of 2390 ppm.

These calibration runs were made with the hygrometer line open to the grower and the furnace power on.

- 2) The hygrometer was started before the furnace power was turned on while the grower was being purged with argon at 20 torr. The moisture content of the gases being sampled decreased until the power was turned on, then

remained fairly constant until the power level was increased to 60 KW. When the power level was increased to 60 KW, the moisture level started to increase significantly. The rate of rise to the dew point of the gases from the furnace increased further when the power level was raised to 100 KW. The dew point increase lasted for approximately thirty minutes following the power increase, where it leveled off and started a slow gradual decline. This decline in dew point levels continued throughout the remainder of the growth run.

- 3) When the first furnace sample was analyzed by the G.C., forty minutes after the furnace power was turned on, and 18 minutes after the 100 KW power was set, a sharp peak at 0.34 minutes was observed along with the characteristic CO peak at a 2.60 minute retention time. The first peak area continued to increase during the meltdown for approximately one hour until a bridge of silicon chunk fell into the melt.
- 4) Prior to the bridge dropping (9:54 AM), the CO peak started increasing sometime between 9:22 AM and 9:34 AM. The CO concentration had increased from a low of 500 ppm at 9:09 AM to 3770 ppm at 9:34 AM. The CO concentration increase seems to correspond to the start of silicon melting during the meltdown. The CO concentration continued to increase to a maximum of over 5800 ppm during the meltdown (9:59 AM). The CO concentration remained in the 5000 ppm range for at least 30 minutes during the meltdown until near the end when the power level was decreased.
- 5) As the CO concentration increased during the meltdown, the first peak that had been so high in the initial stages of the meltdown was decreasing. Finally, when the power level was decreased near the end of the meltdown, this first peak was nearly gone. The first peak continued to decrease to a barely noticeable level during the remainder of the run.
- 6) The normal CO levels during the initial stages of the crystal growth were in the 2000 ppm to 2500 ppm range and continued to decrease during

the run. The last sample analyzed was at 1:38 AM the morning of the 20th and indicated a CO concentration of 700 ppm to 800 ppm. At this time, approximately 10 kg of crystal had been grown and the crucible had traveled up 2.7 inches in the heater.

Previous to the run on the 19th, it was learned that the I.P. Integrator and Event Control Module could not be interfaced to automate the G.C. system without the addition of an accessory kit. This was ordered on the 12th of November.

The subminiature, vacuum-tight solenoid valve needed to automate the G.C. had not been received and a different valve was purchased from Hughes Industrial Products Co.

In order to identify the first peak, it was decided to order a new calibration gas standard made up of hydrogen, carbon monoxide and argon. This was done on the 2nd of December.

During the month of December, five crystal growth runs were performed with each run being monitored by the gas analyzer. In each case, the hygrometer was run on a continuous basis (recorded on the two pen recorder) and the G.C. was run on a manual basis, injecting samples randomly throughout the run and analyzing the G.C. output on the H.P. Integrator. The first three crystal growth runs, No.'s 15, 16, and 17, recorded G.C. output on the second pen of the two pen recorder in addition to the H.P. Integrator.

Analysis cycle time data was accumulated with the Integrator. A cycle time of seven to eight minutes was normal for a carrier gas flow rate of 24 cc to 25 cc/minute (regulated tank pressure \sim 13 psi).

During all of these crystal growth runs, the same pattern of CO concentration levels was evident.

- a) Little or no CO detected during the initial stages of the meltdown as the silicon and hot zone parts are heating up.

- b) A rapid increase in the CO concentration when the silicon starts to melt.
- c) Continued increase in CO concentration as more silicon melts and the furnace temperature rises.
- d) Maximum CO concentrations at the end of the meltdown if heater power is not decreased well before the completion of meltdown.
- e) Unusually high concentrations of CO if the melt is super heated following meltdown.
- f) Gradual decrease in CO concentrations as the melt temperature cools to seed dip temperatures following meltdown.
- g) Continued decreases in CO concentrations as the crucible is raised to the start height for crystal growth.
- h) Slow gradual decreases in CO concentration as the crucible is slowly raised out of the hot zone during crystal growth.
- i) CO concentrations sensitive to furnace temperature changes (power changes) during any stage of the growth cycle.

A pattern also developed for the as yet unidentified first peak detected by the G.C.:

- a) Rapid increase in the concentration of this substance in the beginning stages of the meltdown when the power is increased to the normal maximum during meltdown. This peak increases to a maximum about thirty minutes into the meltdown and then slowly decreases. It continues to decrease as the CO concentration starts increasing dramatically when the silicon starts to melt.
- b) The first peak concentration (approximately 21 seconds after sample injections) decreases throughout the remainder of the crystal growth run following its maximum levels in the early stages of the meltdown.
- c) Significant changes in temperature (power levels) during the run do seem to affect the concentration of this substance; increased power levels -

concentration up; decreased power levels - concentration down. This characteristic is much more evident in the early stages of the crystal growth run than the later stages.

It is interesting to note that, at the same time the first peak concentration is increasing to its maximum values, the moisture levels of the furnace gases are also increasing, as reflected in the dew point measurements of the hygrometer. Moreover, the moisture levels of the furnace gases stabilize and start to decrease around the same time the first peak concentrations start decreasing.

More study is needed to not only identify the first peak substance (thought to be hydrogen), but also the correlation between this first peak and dew point data collected by the hygrometer.

The Hewlett Packard accessory kit for the H.P. 3390 Integrator was received on the seventh of December and sent to the local office for installation. It was discovered in December that the Humphrey solenoid valve was not bubble tight and could not be used in conjunction with the gas sampling system for the G.C.. Automatic operation of the G.C. analysis portion of the gas analyzer cannot be accomplished without an electrically operated bubble tight solenoid valve. A 3-way Skinner valve was finally located that would do the job and ordered on 12/21/81. The valve was received on 12/28/81 and scheduled for installation, along with the modified integrator the first part of January.

The oxygen analyzer was installed in the Furnace Gas Analysis System. Initial trials indicated that the unit works fine under atmospheric conditions, but does not appear to receive sufficient flow of gases from the crystal grower when the grower is at normal operating vacuum levels (reduced pressures). Further testing is scheduled for January.

E. Documentation - Economic Analysis

The goal of this program is 2.5 kg per hour throughput at 6" diameter for 150 kg total pulled weight. Previous reports have assumed the pull speed goal

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had been achieved and the resulting three cases shown below were presented:

<u>Case</u>	<u>CZ Add-On Cost</u>	
	<u>\$/Kg</u>	<u>\$/Peak Watt</u>
1 Pulling 5 crystals, each 30 kg	21.62	0.1525
2 Pulling 4 crystals, each 37.5 kg	21.13	0.1490
3 Pulling 3 crystals, each 50 kg	20.66	0.1457

In the last quarterly report (July - September), the average straight growth rate of 2.5 in/hr achieved to that date was used and the results presented:

	<u>CZ Add-On Cost</u>	
	<u>\$/kg</u>	<u>\$/Peak Watt</u>
Pulling 5 crystals, each 30 kg	27.56	0.1944

During this quarter, the achieved rate was increased to 3.0 in/hr and the results are:

Pulling 5 crystals, each 30 kg	25.51	0.1799
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Table 2 shows the total economic analysis.

A 7.4% reduction in cost will result from the latest proven parameters over the analysis of last quarter. Also, another 15.3% reduction in cost will result from increasing the throughput from 1.92 kg/hr to the goals of 2.5 kg/hr. Some of this improvement is expected during the last three months of this project.

IV PROGRAM PLAN

The revised program plan is updated and shown in Figure 4.

V COST AND DIRECT LABOR DATA

The total incurred cost and direct labor graphs have been updated and are shown in Figures 5 and 6 respectively.

	<u>Previous Total</u>	<u>Current Month</u>	<u>Total To Date</u>
Costs	\$ 615,764	\$ 20,061	\$ 635,825
Man-Hours	5,912.6	570.7	6,483.3

TABLE 1: GROWTH RUN DATA - DECEMBER, 1981

ITEM	UNITS	PROCESS DATA										DATE	
		15	16	17	18	19	12/1/81	12/8/81	12/10/81	12/15/81	12/21/81		
1 Run #	-												
2 Date	-	12/1/81	12/8/81	12/10/81	12/15/81	12/21/81							
3 Crystal #	-	1	1	1	1	1							
4 Charge-Cold Fill	kg	25	25.5	25	25	20.1							
5 -Hot Fill	kg	-	-	-	-	5							
6 Crucible Diam.	in	15	15	15	15	15							
7 Silicon-New/Recycle	-	Rec.	Rec.	Rec.	Rec.	Rec.							
8 Seed	-	1-0-0	1-0-0	1-0-0	1-0-0	1-0-0							
9													
10 Meltdown Power	kw	110	100	108	109	110							
11 Total Meltdown Time	hr	2.08	2.5	2.58	2.08	3.67							
12 Start Neck Power	kw	68	67	66	68	68							
13 15" Body Power	kw	70	67	-	70	72							
14													
15 Crystal Diameter	in	5.84	6.56	6.0	6.1	6.05							
16 Pulled Weight	kg	16.3	19.15	10.8	17.75	19.92							
17 Residual Melt	kg	8.7	6.35	14.2	7.25	5.18							
18 Zero D-length	in	10.5	2	10	5.5	9.5							
19 -weight	kg	10.75	2.5	10.8	6.14	10.4							
20 Body Growth Time	hr	5.33	5.42	3.5	4.5	5.2							
21 Cycle Time (power on-power off)	hr	14.58	12.42	9.75	11.37	14.85							
22													
23													
24 Pulled Yield	%	65.2	75.1	42.2	71.0	79.4							
25 Avg Pull Speed	in/hr	3.0	2.9	3.08	3.1	2.75							
26 Zero D Yield	%	65.9	9.8	100	41.2	52.2							
27 Machine Throughput	kg/hr	1.14	1.54	1.08	1.56	1.34							
28													
OPERATING UNIT:		PREPARED BY:										DATE:	

TABLE 2

ECONOMIC ANALYSIS

CZ ADD-ON COST BASED ON MOD CG2000
PROCESS PARAMETERS AS OF DECEMBER, 1981CONDITIONS

Crucible Diameter (in)	15
Crystal Diameter (in)	6
Total Poly Melted (kg)	158
Total Crystal Pulled (kg)	150
Avg. Straight Growth Rate (in/hr)	3.0
Pulled Yield (%)	94.9
Yield After C.G. (% of Melt)	83.5
Individual Crystal Wt. (kg)	30
No. Crystals/Crucible	5
Cycle Time (hr)	78

PROCESS CYCLE TIMES

OPERATION

TIME (MINS)

1. PREPARATION

Load Polysilicon	20
Close Furnace	10
Pump Down	20
Meltdown	150
Subtotal	<u>200</u>

2. GROWTH CYCLE (INITIAL)

Lower Seed	*
Stabilize Melt	60
Neck Growth	20
Crown Growth	70
Straight Growth	470
Taper End	60
Subtotal	<u>680</u>

3. RECHARGE/GROWTH CYCLE (x 4)

Cool Crystal	30
Remove Crystal	10
Load Hopper, Vac. Down (x 2)	60
Lower Hopper (x 2)	10
Dump and Melt	125
Lower Seed	*
Stabilize Melt Temp.	60
Neck Growth	20
Crown Growth	70
Straight Growth	470
Taper End	60
Subtotal	<u>915</u>

x 4 3660

4. SHUT DOWN CYCLE

Cool Furnace	80
Remove Crystal	**
Clean, Set Up	60
Subtotal	<u>140</u>

Total Cycle Time 4630 mins = 78 hours

* Completed during Melt Stabilization Time.

** Completed during Furnace Cooling Time.

GROWTH RATE PARAMETERS

Grow Diameter (in)	6.2
Straight Crystal Wt. (kg)	27
Straight Growth Time (hr)	7.8
Avg. Growth Rate (kg/hr)	3.46
Wt. Per Unit Length (kg/in)	1.153
Avg. Pull Rate (in/hr)	3.0

SAMICS/IPEG INPUT DATA AND COST CALCULATION

INPUT DATA (\$1980)

1. CAPITAL EQUIPMENT COST [EQPT]	\$ <u>247,560</u>
2. FLOOR SPACE [SQFT]	<u>120</u>
3. ANNUAL DIRECT SALARIES	
Prod. Operator (0.65 man @\$13,160/yr)	8,554
Elec. Tech. (0.3 man @\$16,940/yr)	5,082
Inspector (0.1 man @\$11,550/yr)	1,155
Total [DLAB]	\$ <u>14,791</u>
4. DIRECT MATERIALS Usage Based on Machine	
Utilization of 85% = 95.5 cycles/yr	
Crucibles 15" x 12" @\$300 ea	28,650
Seeds (\$20/cycle)	1,910
Dopant (\$25/cycle)	2,388
Argon (60 ft ³ /hr @\$0.04/ft ³)	17,520
Graphite (3 sets/yr)	26,661
Materials Total [MATS]	<u>77,129</u>
5. UTILITIES	
Electricity @\$0.04/KW hr	
Meltdown @100 KW	4,138
Avg. Grow @75 KW	<u>20,055</u>
Water @c0.7/ft ³	<u>12,121</u>
Utilities Total [UTIL]	\$ <u>36,314</u>

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IPEG PRICE 5 x 30 CRYSTALS

C1 EQPT x \$0.57/yr	= \$EQPT	\$ 141,109
C2 SQFT x \$109/yr	= \$SQFT	13,080
C3 DLAB x \$2.1/yr	= \$DLAB	31,061
C4 MATS x \$1.2/yr	= \$MATS	92,555
C5 UTIL x \$1.2/yr	= \$UTIL	43,577

TOTAL ANNUAL COST	\$ 321,382
-------------------	------------

Quan (Total Charged x Yield After C.G.) kg	=	12,599
Add-On Cost \$/kg	=	25.51
Add-On Cost ¢/pk watt	=	17.99
(assuming 1 kg = 1 m ²)		
Machine Throughput kg/hr	=	1.92

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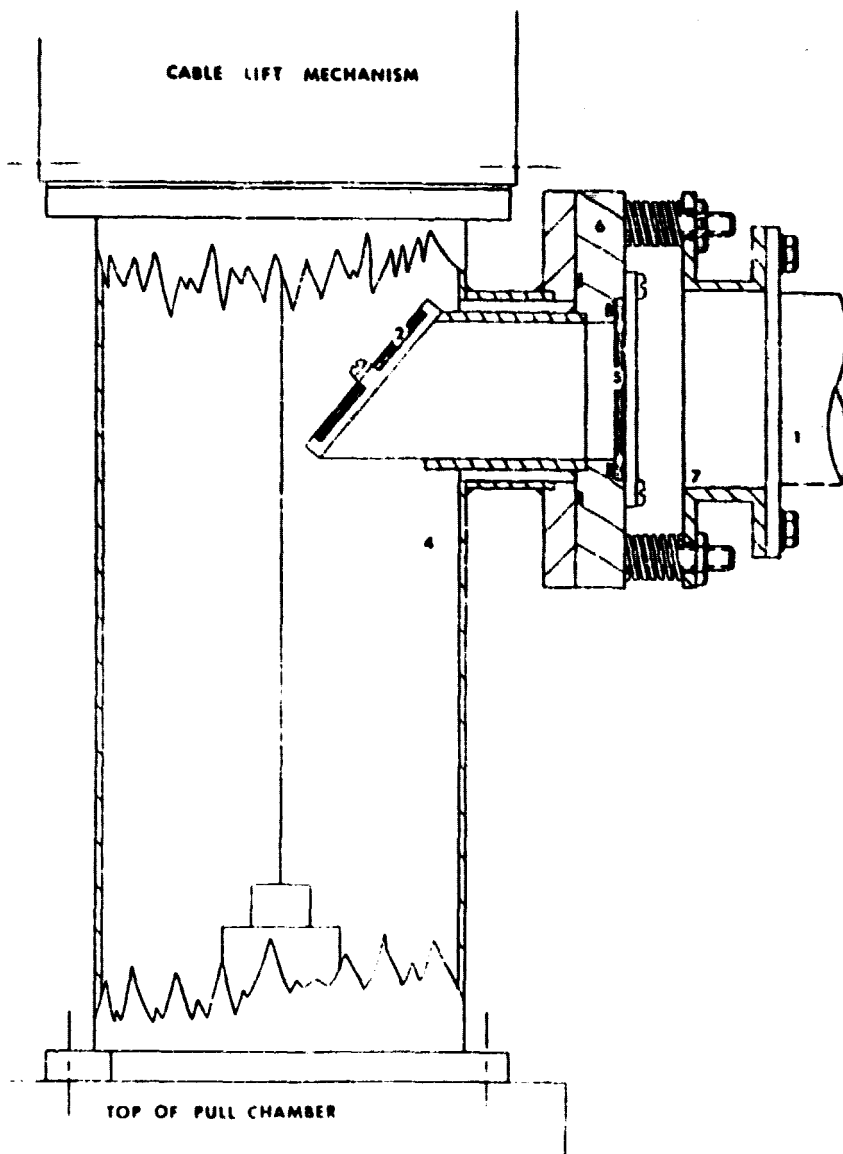


Figure 1
Melt Temperature Pyrometer

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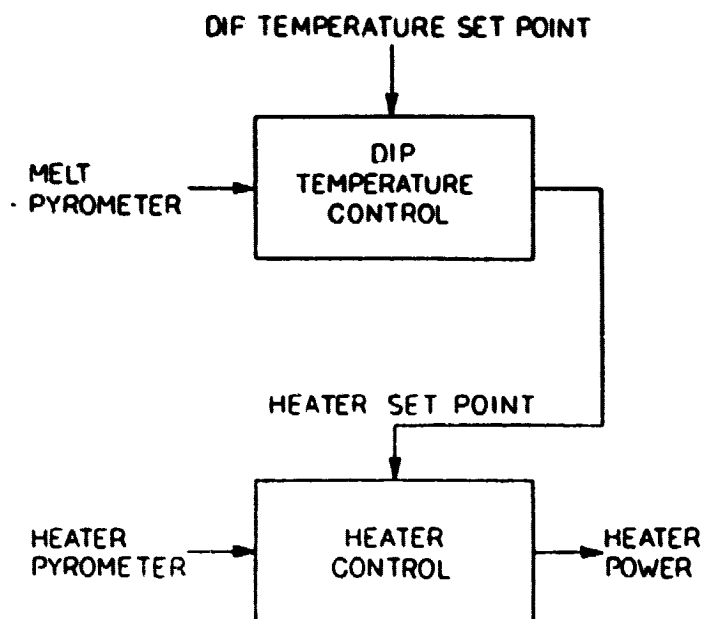
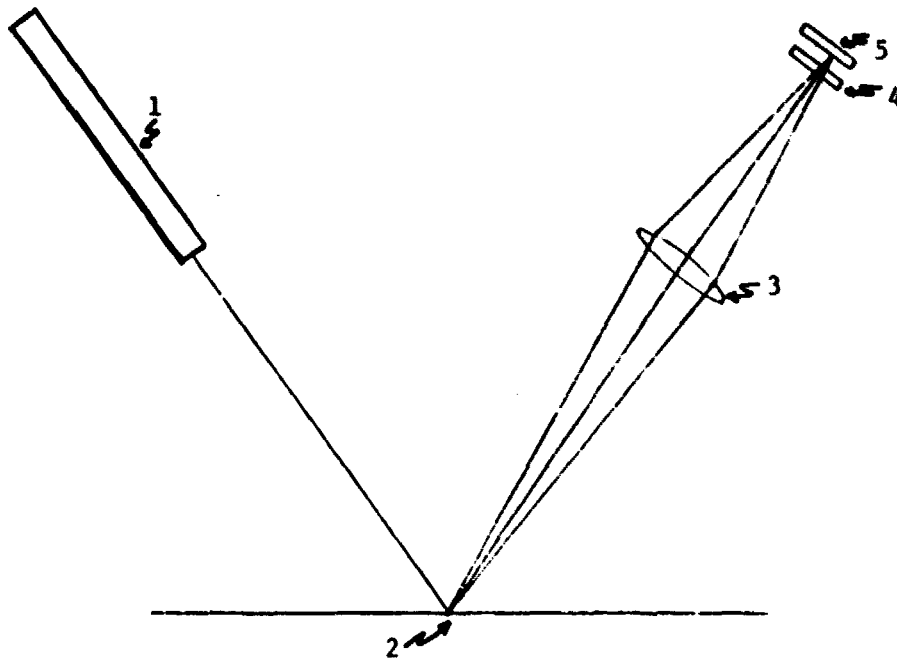


Figure 2

CONTROL CONFIGURATION DURING AUTOMATIC
DIP TEMPERATURE SETTING

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1. 2-Milliwatt HeNe Gas Laser
2. Surface of Silicon Melt
3. Receiver Lens
4. Narrow Band Interference Filter
5. Position-Sensitive Photodetector

Figure 3
Melt Level Sensor

Advanced Czochralski Growth
For Technology Readiness

Program Plan, Revision No. 2
DOE/JPL 955733
Kayex Corporation
April 21, 1981

1980 → ← 1981

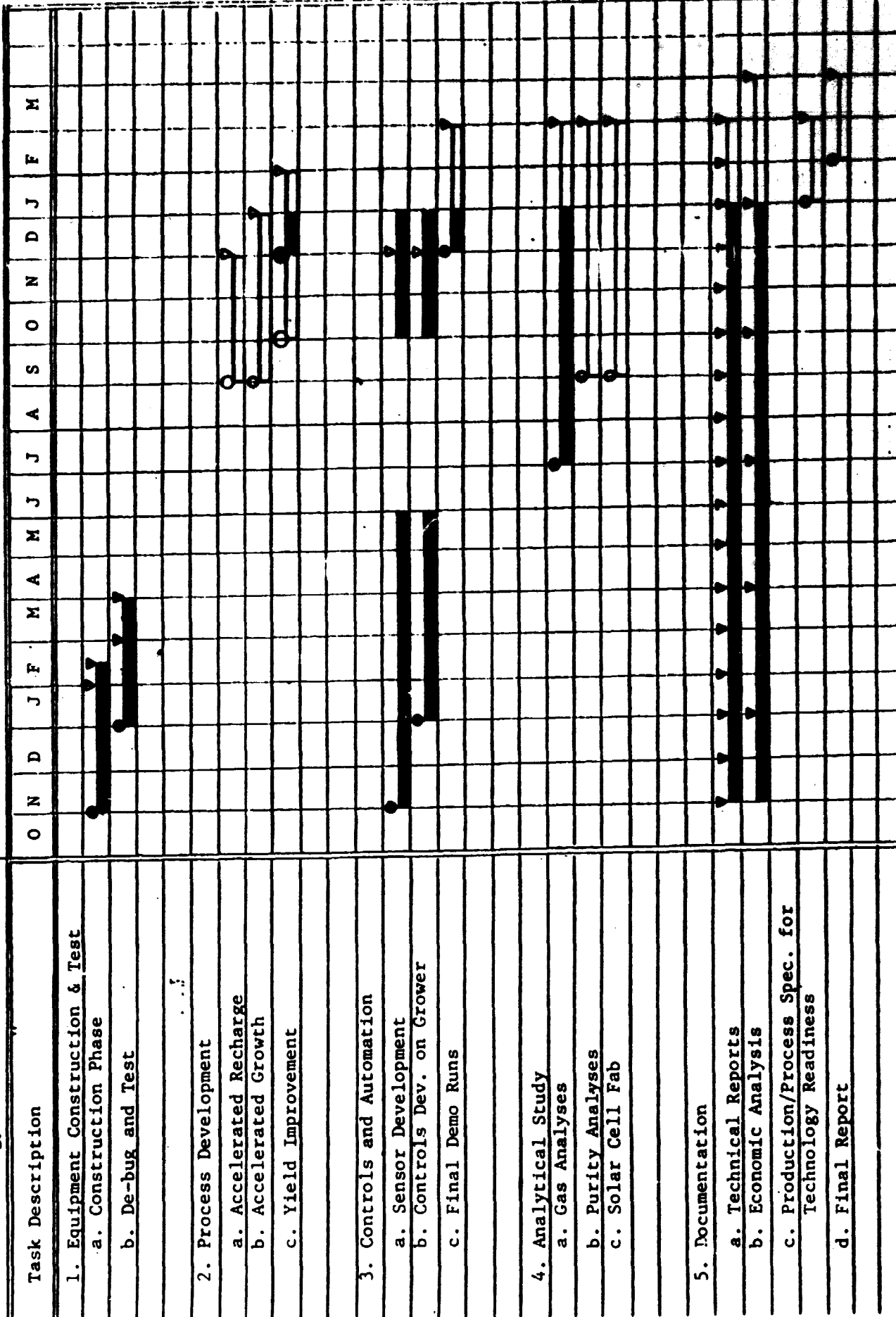


Figure 4

KAYEX CORPORATION

DOE/EPPL 955733

ADVANCED CE GROWTH

TECHNOLOGY READINESS

TOTAL INCURRED COSTS

TOTAL CONTRACT COSTS (X 1000)

PROTECTED
ACTUAL

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REV. A; MAY 1, 1981

OCT '80 NOV '80 DEC '80 JAN '81 FEB '81 MAR '81 APR '81 MAY '81 JUN '81 JUL '81 AUG '81 SEP '81 OCT '81 NOV '81 DEC '81 JAN '82 FEB '82 MAR '82

1982

1981

1980

Figure 5

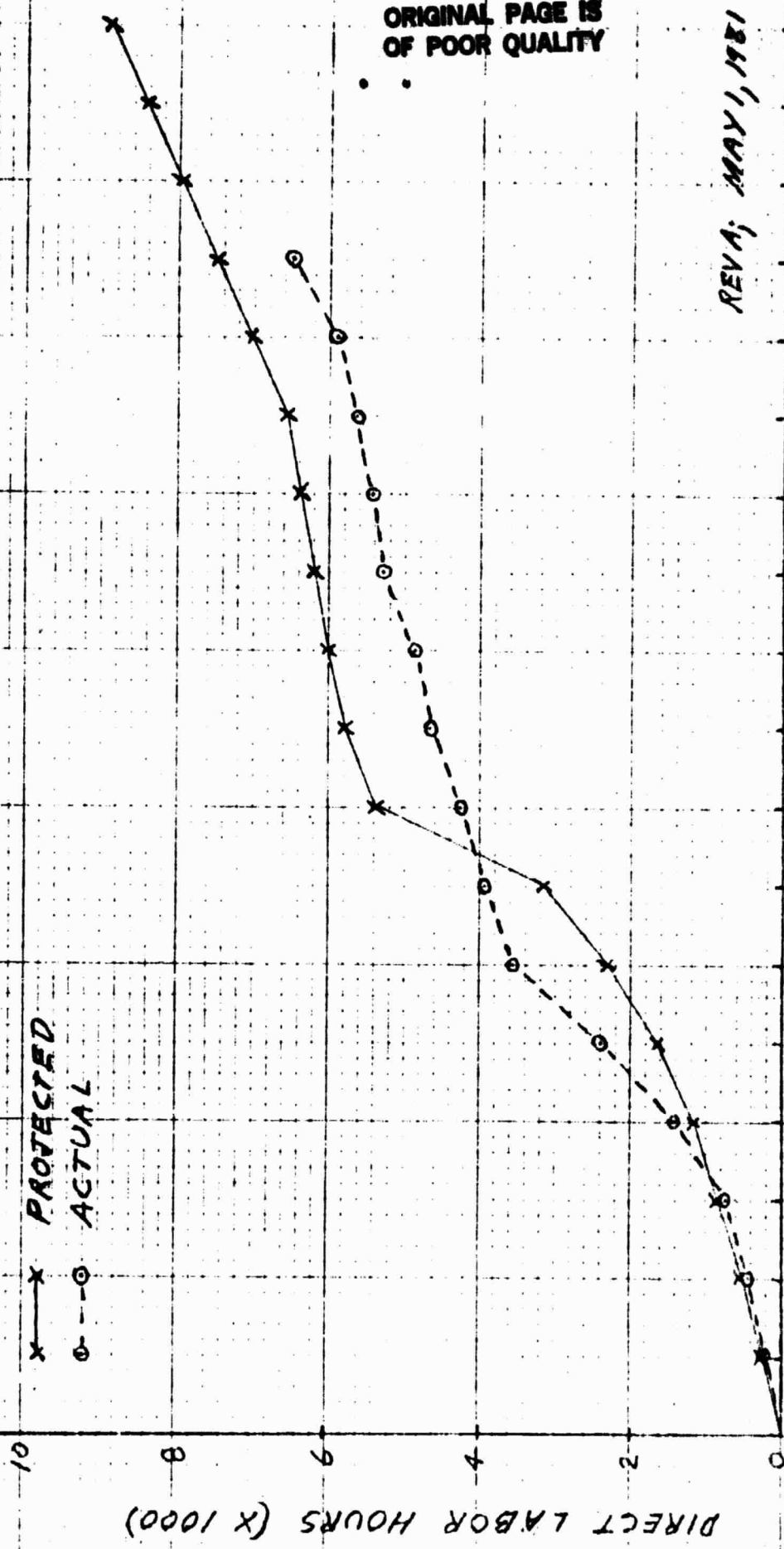
KAYEX CORPORATION

DOE/JPL 955733

ADVANCED CE GROWTH

TECHNOLOGY READINESS

DIRECT LABOR HOURS



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REVA; MAY 1, 1981

Figure 6