DRD NO. SE-9
DRL NO. 60

DOE/JPL 954888 78/4
DISTRIBUTION CATEGORY UC-63

# CONTINUOUS CZOCHRALSKI GROWTH

## SILICON SHEET GROWTH DEVELOPMENT OF THE LARGE AREA SILICON SHEET TASK OF THE LOW COST SILICON SOLAR ARRAY PROJECT

FIRST ANNUAL PROGRESS REPORT
OCTOBER 1, 1977 - SEPTEMBER 30, 1978
PROGRAM MANAGER: DR. R. L. LANE

(NASA-CR-158096) CONTINUOUS CZOCHRALSKI GROWTH: SILICON SHEET GROWTH DEVELOPMENT OF THE LARGE AREA SILICON SHEET TASK OF THE LOW COST SILICON SOLAR ARRAY PROJECT Annual Progress Report, 1 (Kayex Corp., Rochester, G3/44

N79-16369

Unclas
43629



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"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."

## PREFACE

This work was performed by the Kayex Corporation Hamco Division Research and Engineering Department, R. L. Lane, Director. Others who participated in this research are D. Fanale, C. Saulsbury, R. Moore, F. Merz, and M. Johnson.

Dr. A. R. Kachare is the JPL Project Monitor.

## ABSTRACT

The primary objective of this contract is to develop equipment and methods for the economic production of single crystal ingot material by the continuous Czochralski (CZ) process. Continuous CZ is defined for the purpose of this work as the growth of at least 100 kilograms of ingot from only one melt container.

The approach to the project is to utilize a Hamco production CG2000 crystal grower suitably modified to grow large crystals and to permit periodic replenishment of the liquid. Key modifications were a vacuum tight isolation valve to separate the furnace chamber from the pull chamber for recharging, an automatic recharge mechanism, and an enlarged pull chamber to hold a supply of poly material. Other suitable modifications to permit the growth of crystals up to about 35 kilograms each included an optical diameter control system to allow crystals up to 12.5 cm to be grown, and a pull mechanism capable of supporting the large crystal to be grown. Furnace hot zone modifications were designed and built to accommodate 14-inch diameter crucibles having a melt capacity of about 40 kilograms.

During the reporting period (October, 1977 - September, 1978), the modified grower was made fully functional and several recharge runs were performed. The largest run lasted 44 hours and over 42 kg of ingot was produced.

Samples from two recharge runs were sent to JPL for solar cell fabrication and analysis. Little, if any, degradation in efficiency was observed as a result of pulling multiple crystals from one crucible. Solar efficiencies observed were between 9.3 and 10.4% AMO (13.0 and 14.6% AMI) compared to 10.5% (14.7% AMI) for optimum CZ material control samples.

The two primary reasons for the termination of recharge runs were (1) the throughput of high quality single crystal material being produced became so low

that continued running was deemed to be unfruitful in terms of learning more about the process, and (2) unexpected shut downs occurred due to operator error, equipment failure, or utility interruption.

The reason for the reduction in crystalline quality of produced material with time is not fully understood, although foreign particles entering the melt are considered the most probable reason. Silicon monoxide particles from furnace interior walls, quartz particles from the crucible, or silicon carbide particles precipitating from the melt are being investigated. More study is needed to solve this problem.

Using the SAMICS/IPEG format, economic analysis of continuous CZ suggests that 1986 DoE cost goals can only be met by the growth of large diameter, large mass crystals.

Emphasis will be placed in the future on determining the causes of structure loss and on the production of larger crystals in order to achieve the project goals.

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## 1.0 INTRODUCTION

The production of several crystals (up to 100 kg total) from one container by the Czochralski (CZ) method requires equipment and process modifications not commonly used in the semiconductor device industry. In general, these modifications are required for the purpose of permitting some method of silicon replenishment without cooling the residual melt and container or contaminating the silicon.

Although a number of methods of accomplishing continuous CZ growth can be devised, it is very important that the chosen method be suitable for a production environment and that it is attractive in terms of safety, reliability and cost. In a production-type process, variables should be well-controlled and equipment should not require operators with great skill or technical ability.

It is the purpose of this project to develop equipment and process to achieve low-cost continuous CZ production of silicon single crystal ingot by repeated refilling (recharging) of the vitreous silica crucible and subsequent crystal growth cycles.

The equipment used in this work was a Hamco model CG-2000 Czochralski crystal growing furnace, extensively modified for the purpose of growing several crystals from the same crucible.

Evaluation of the equipment designs, verification of the economic model (or revision) and study of the process parameters were carved out by performing a number of recharge runs.

## 2.0 PROGRESS

### 2.1.0 GROWTH FACILITY DESIGN, CONSTRUCTION, AND MODIFICATION

Since the vitreous silica crucible is always destroyed by fracture during the cooling-off cycle due to the thermal expansion difference between silicon and silica, it was necessary to provide a means to refill the crucible while maintaining it at high temperature. Furthermore, it was necessary to continuously maintain an inert atmosphere surrounding the molten silicon and the graphite furnace parts, to prevent oxidation. A pressure range of about 25 mbar minimizes the deposits of silicon monoxide which tend to collect on the cooler surfaces of the furnace. The monoxide, formed by the reaction between the molten silicon and the silica crucible, sometimes falls from surfaces to land in the melt causing loss of zero-dislocation crystal structure, twinning, or polycrystalline growth. Therefore, the following criteria were established for the design of the furnace modifications:

1. The recharge operation must permit the crucible to remain at or above the melting point of silicon.
2. The hot zone must be always protected by vacuum or low argon pressure.
3. Polysilicon insertion and crystal removal must be performed in a safe, reliable way without contamination of the silicon or the hot zone environment.

During the reporting period, a new CG-2000 production crystal grower was completed with special modifications to the chambers to make it suitable for recharging. The pull chamber is enlarged to permit polysilicon to be stored within the furnace in a ready condition for recharging. (The initial effort utilized polysilicon rods for recharging. A subsequent design will be suitable for powdered or nugget silicon).

Other initial modifications to the chambers included a vacuum-tight isolation valve, larger viewports, a larger pull length capacity and a larger growth chamber suitable for 14-inch diameter crucibles. Subsequently, additional modifications to the crystal growth facility were made, including a recharge mechanism, a larger capacity cable-pull mechanism, an automatic diameter co trol optical system suitable for 12.5 cm diameter crystals, and a dopan[illegible] ishing fixture.

[illegible]heral equipment to accommodate recharging included a crystal-poly transfer device for loading raw material and retrieving grown crystals, and a special vacuum pumping manifold to control furnace pressure during the crystal retrieval process.

Recently, an etching facility was designed and built to clean poly feed stock rods or ingots for recycling.

Figure 1 is a photograph of the JPL continuous CZ facility after installation in the room specially prepared and designated for the project.

Figures 2 and 3 are schematic illustrations of the growth chambers with modifications showing both the crystal growth and the recharge modes of operation.

Polysilicon (semiconductor grade) is readily available in rod form, and the initial recharge procedure which was used on the program was to slowly

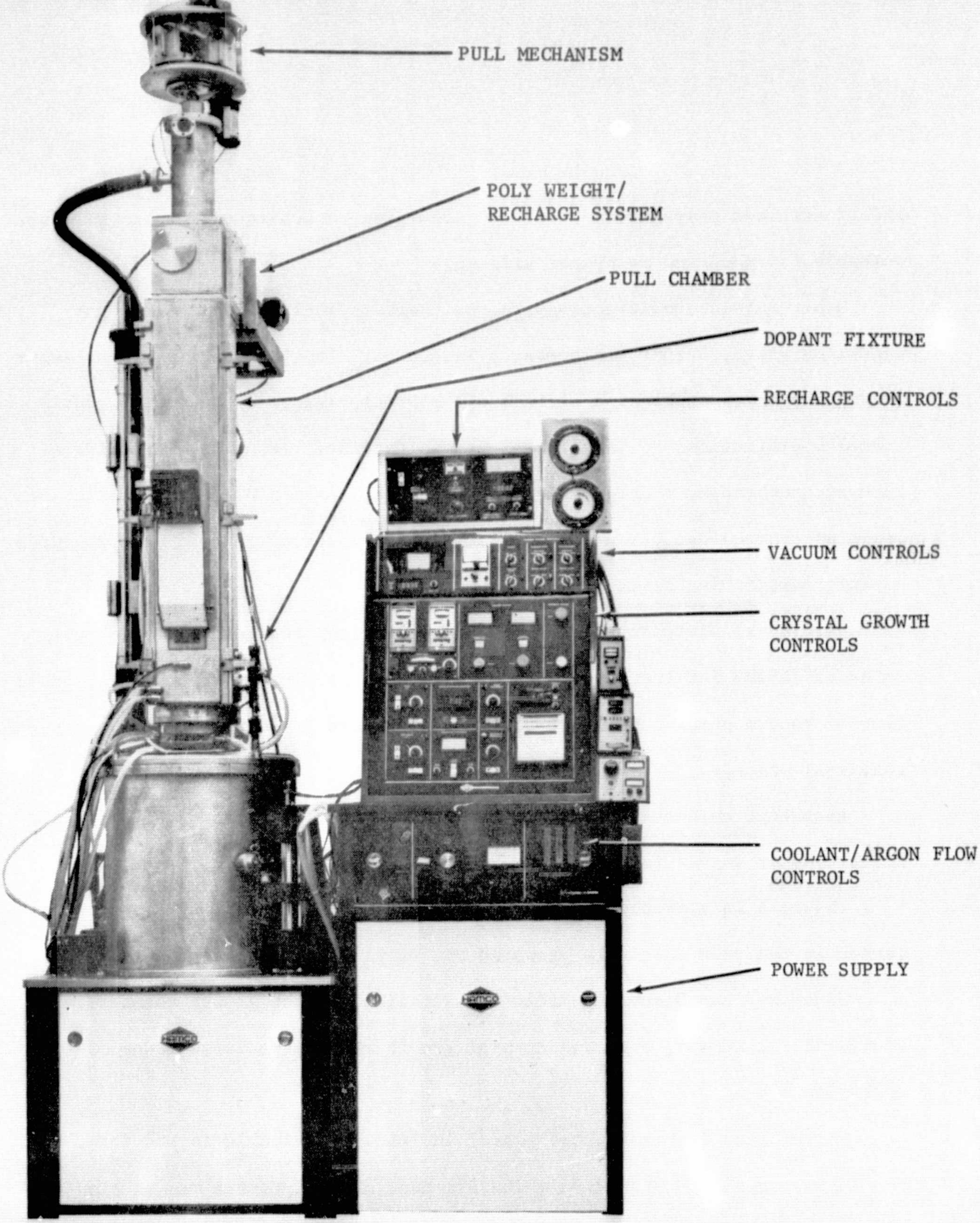


FIGURE 1

JPL CONTINUOUS CZ GROWTH FACILITY

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OF POOR QUALITY

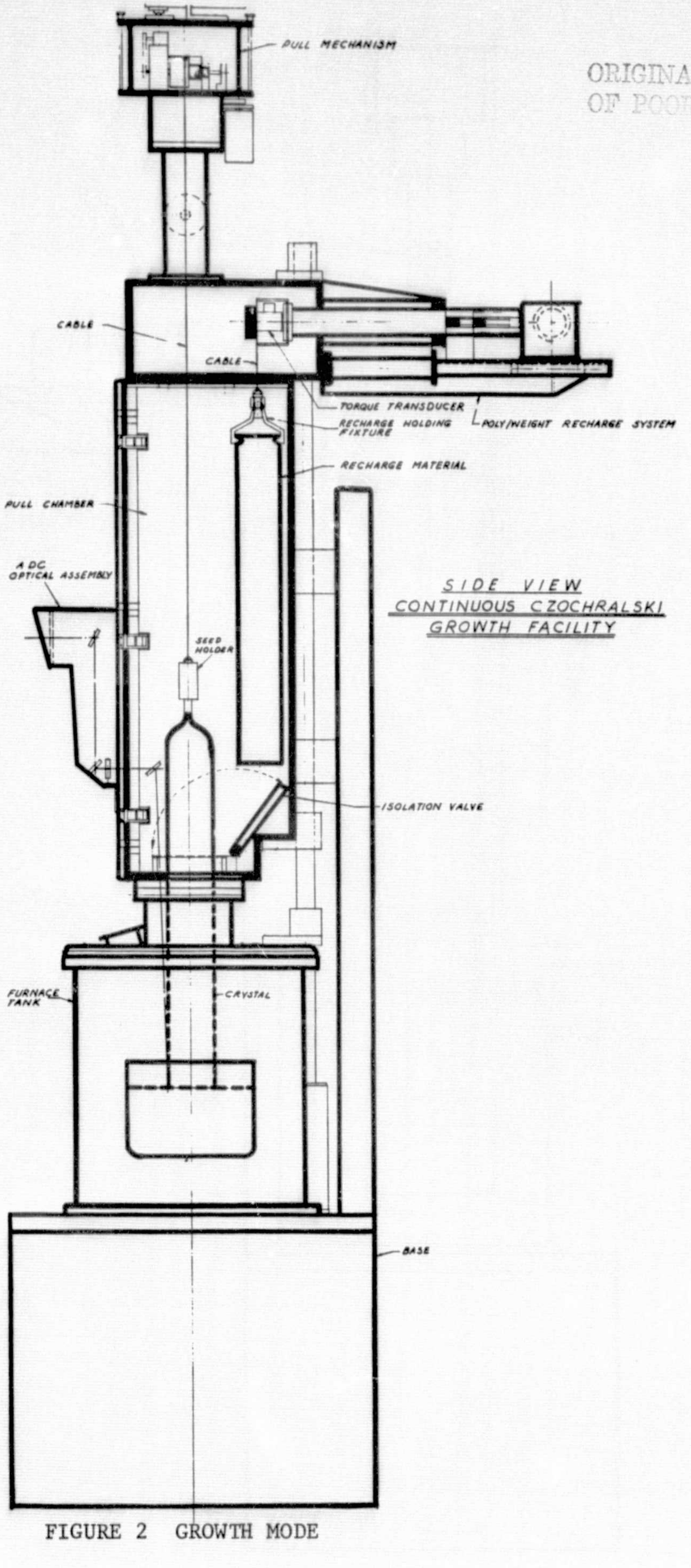



FIGURE 2 GROWTH MODE

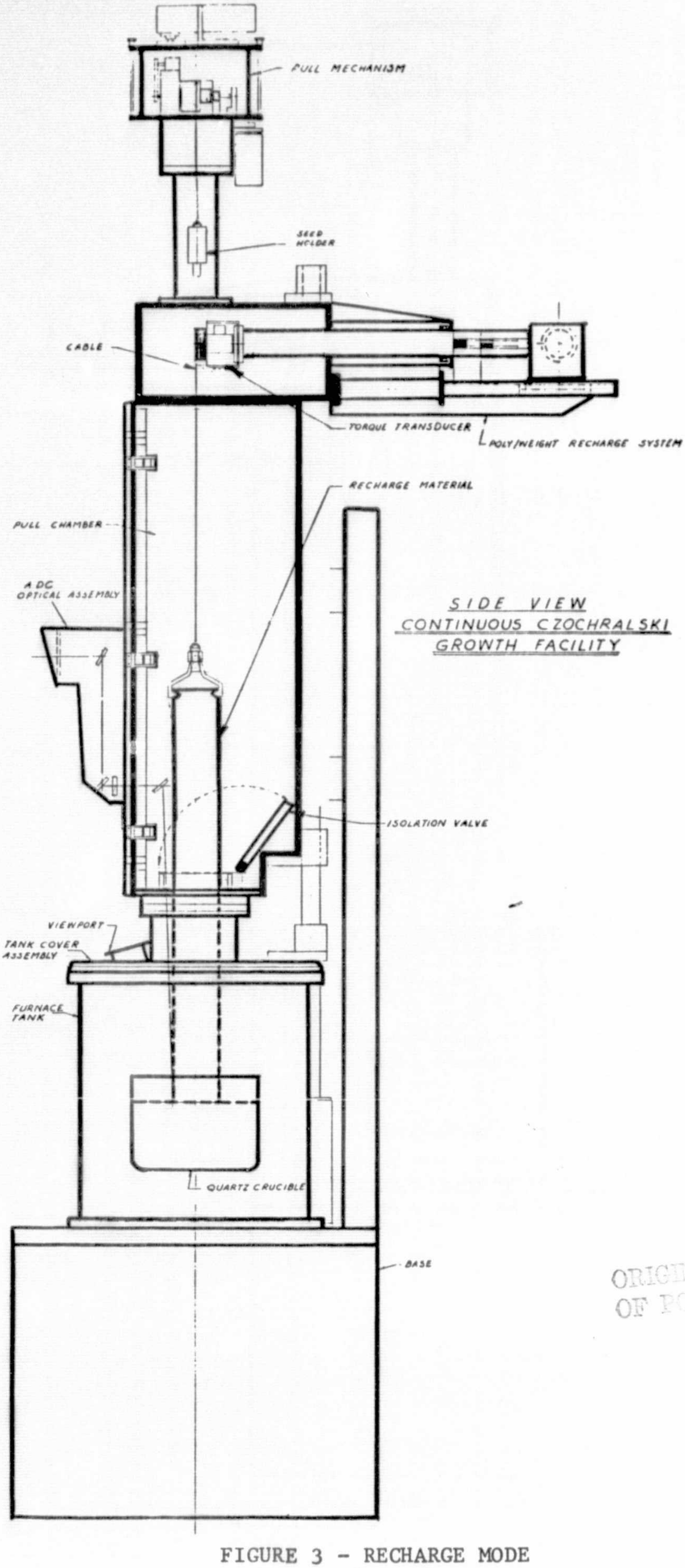





FIGURE 3 - RECHARGE MODE

lower the poly rod feed stock into the hot crucible, melting it off until the desired amount of liquid was obtained in the crucible.

Poly rod feed stock can be stored in the rear of the pull chamber. When a crystal is complete, the valve is closed and the pull chamber is brought up to atmospheric pressure by introducing argon. After removal of the crystal, the pull chamber is closed and returned to low argon pressure by pumping down and purging. After the valve is opened, the poly rod is moved forward, lowered into the hot zone and the crucible is filled. (At this time the seed is positioned above the recharge mechanism). After recharging, the remaining poly is retracted and the seed is lowered into the furnace to start a new crystal.

After the second crystal is grown and removed from the furnace, a new poly rod can be placed on the recharge cable, if required.

This concept of recharging has been demonstrated to be feasible by a number of successful multiple recharge runs. The basic furnace chamber has performed adequately with the exception of vacuum leak problems around the larger viewports, primarily related to stress cracking at welds. The design has been modified now to eliminate this problem.

Following is a summary of the design criteria for the modifications which were added to the JPL continuous CZ facility.

### 2.1.1 ISOLATION VALVE

A "flapper"-type isolation valve was designed for simplicity, reliability, and ease of cleaning. It is water cooled, relying on an O ring seal. A manually operated external lever actuates the valve. Initial tests of the valve were satisfactory and the performance of the valve over many runs has been excellent. The water-cooled O ring has not been degraded at all by pulling hot crystals through the valve.

2.1.2 AUTOMATIC DIAMETER CONTROL OPTICAL SYSTEM (Figure 4)

The design of this unit was made similar in principle to a standard CG-2000; however, because of the vacuum isolation valve throat, certain modifications had to be made. First, a larger focal length lens was required. The image of the growth interface was obtained having exactly a 2:1 reduction for convenience in determining crystal diameter. A larger viewport in the chamber door was added for visual confirmation that crystals clear the valve before closure of the valve and removal of the crystal.

2.1.3 POLY WEIGHT/RECHARGE SYSTEM

The design work on this device constituted a major portion of the effort. After consideration of a number of alternatives, the concept of a torque sensing transducer fixed to the end of a horizontal shaft (Figure 5) was the preferred route. The shaft rotates for raising or lowering the poly and translates horizontally to move the poly over the crucible. A commercially available torque sensing strain-gauge transducer was purchased and included in the design.

The POLY WEIGHT/RECHARGE DEVICE is a removable, independent unit positioned between the pull chamber and the pull mechanism support. This minimized rework cost to the pull chamber, increased the recharge capacity of the furnace and simplified the modification procedure.

The control circuitry for the Poly Weight/Recharge Device provides for (1) continuous weighing of the poly so that accurate measure of the amount remaining (and therefore the amount melted off) is always available, and (2) automatic shut-down of the lowering motor when the desired amount of silicon has been melted. A tachometer feedback DC-PM motor raises and lowers the poly while the lateral movement is produced by a hydraulic cylinder operating off of the main hydraulic pump.

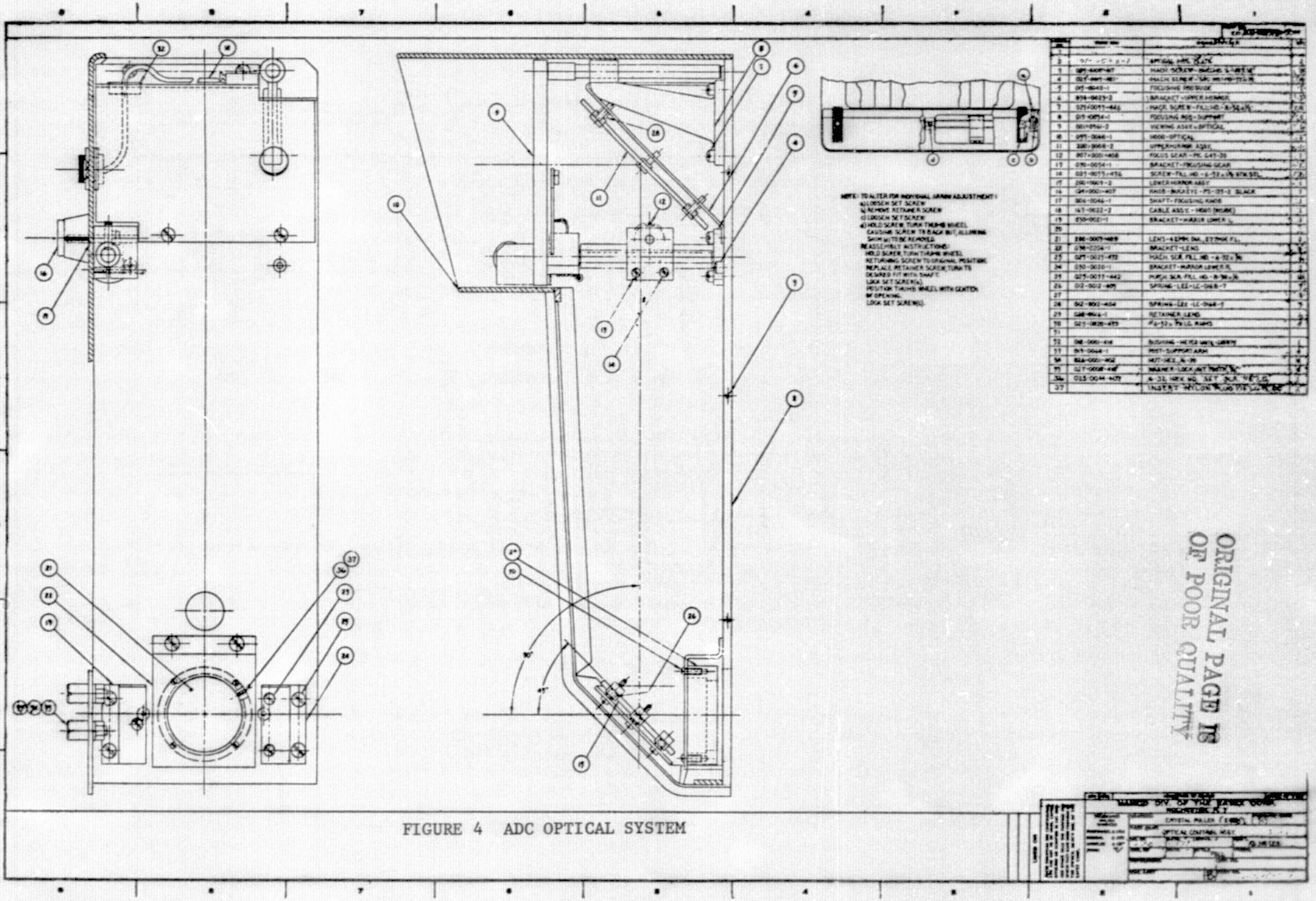


FIGURE 4 ADC OPTICAL SYSTEM

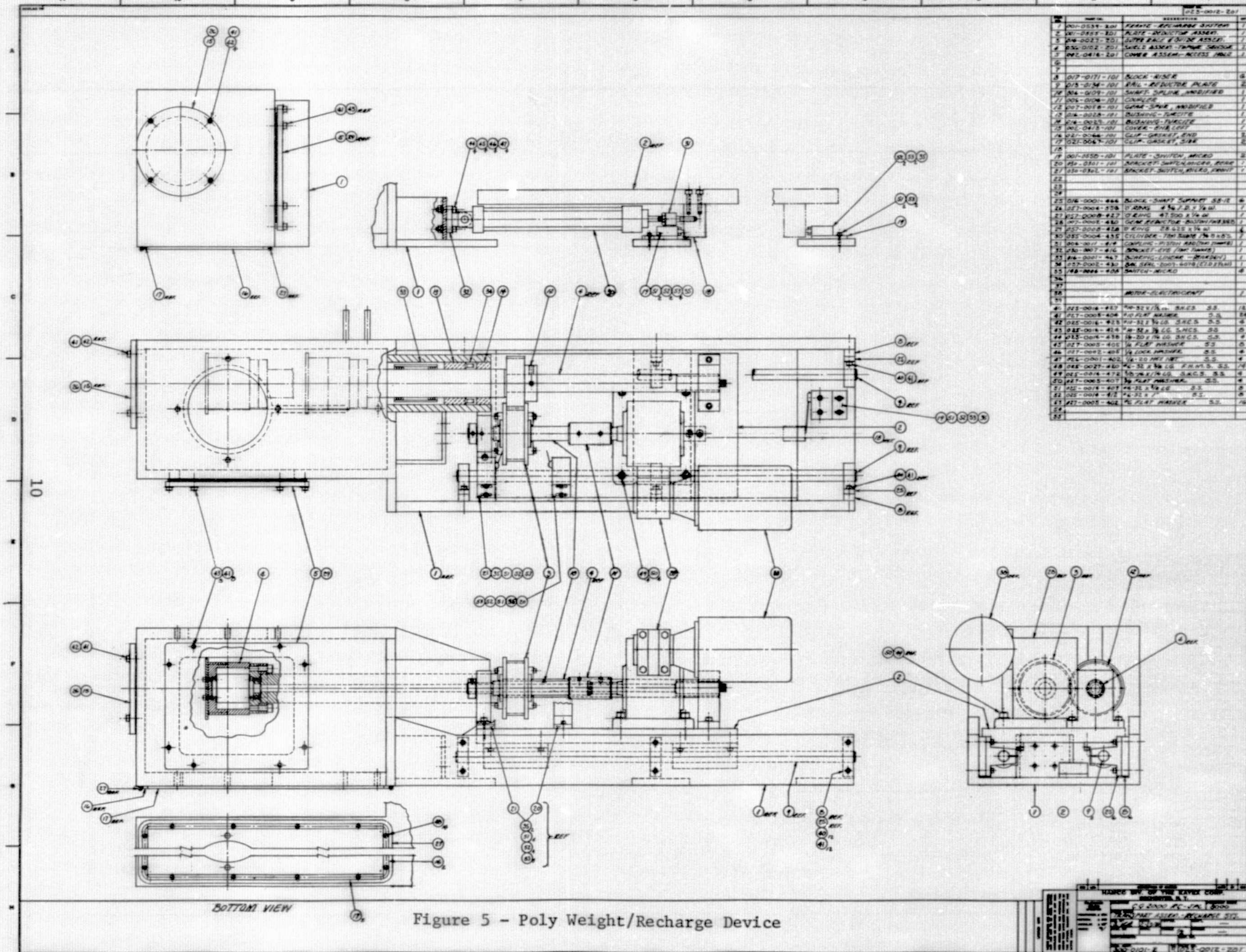



Figure 5 - Poly Weight/Recharge Device

Considerable difficulty was experienced in fabrication and testing of the recharge device. The major problems related to achieving a leak-tight system. Additionally, the weight sensing circuitry failed and had to be returned to the vendor for repair.

A poly attachment device (Figure 6) fabricated of molybdenum was found to perform adequately for recharging with poly rods up to 5 inches in diameter. Two notches are required to be cut in the rod to accept the teeth of the clamp. Poly can be melted off to a point where only about one kilogram remains unmelted.

### 2.1.4 DOPANT FIXTURE

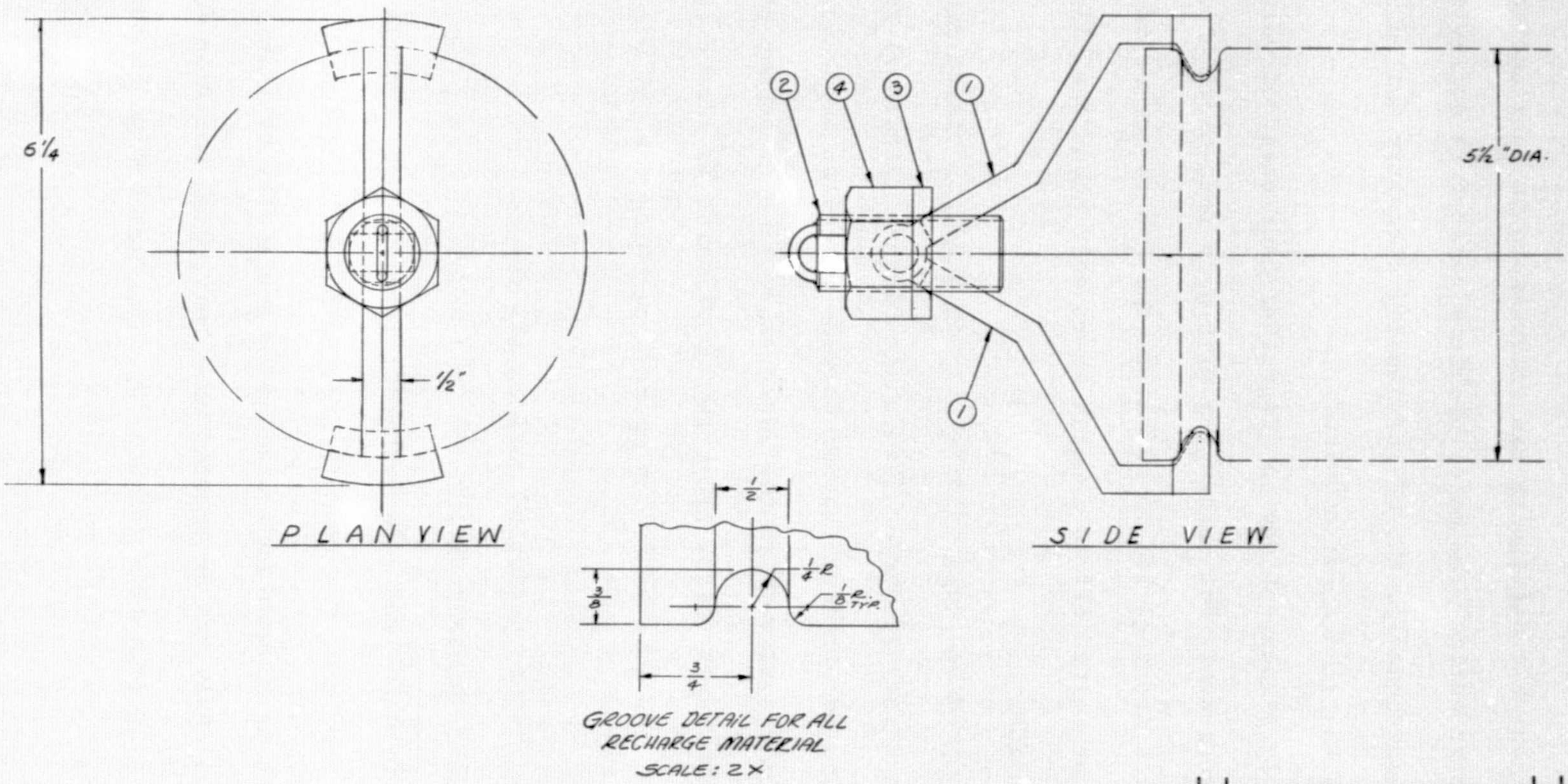
When predoped poly material is not used, provision must be made to add dopant with each recharge. The dopant fixture (Figure 7) consists of two vacuum valves with an air-lock chamber between which can be evacuated using the secondary pumping system. With the lower valve closed, dopant pellets can be added through the upper valve. After the upper valve is closed and the air-lock evacuated, the lower valve is opened, allowing the pellets to drop into the crucible.

This fixture performed well on all runs.

### 2.1.5 CRYSTAL/POLY TRANSFER DEVICE

A convenient and safe method of removing hot crystals and inserting poly rods was required. The transfer device was built (Figures 8 & 9) for this purpose. The device is mounted on a standard commercially available manual lift truck. The crystal/poly can be rotated to a horizontal position for placing on the cooling or storage rack (Figure 10). The rack design is shown in Figure 11.

| ITEM NO. | PART NO. | DESCRIPTION | QTY |
|---|---|---|---|
| 1 | 021-0065-201 | HOOK - R.C. HOLDING FIXTURE | 2 |
| 2 | 024-0079-201 | ROD - R.C. HOLDING FIXTURE | 1 |
| 3 | 027-[illegible]-101 | WASHER - R.C. HOLDING FIXTURE | 1 |
| 4 | 026-0001-438 | NUT - 1"-8 STD. S.S. | 1 |




HAMCO DIV. OF THE KAYEX CORP.
ROCHESTER, N. Y.

MATL. 316 S.S.
SCALE FULL
NEXT ASSY. 300-0101-2
SIZE C — PART NO. 361-0054-201

POLY ATTACHMENT DEVICE

FIGURE 6

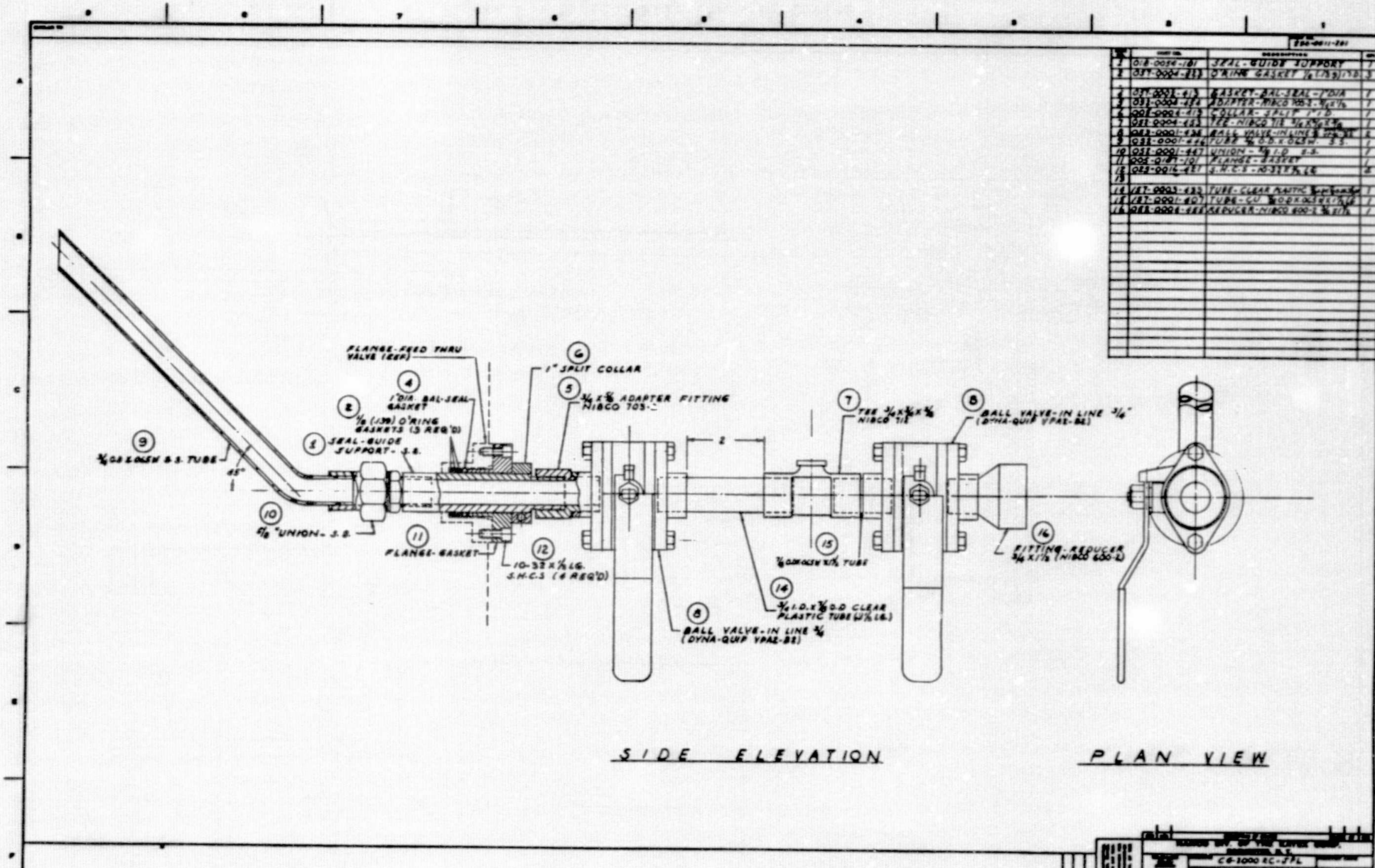



Figure 7 - Dopant Fixture

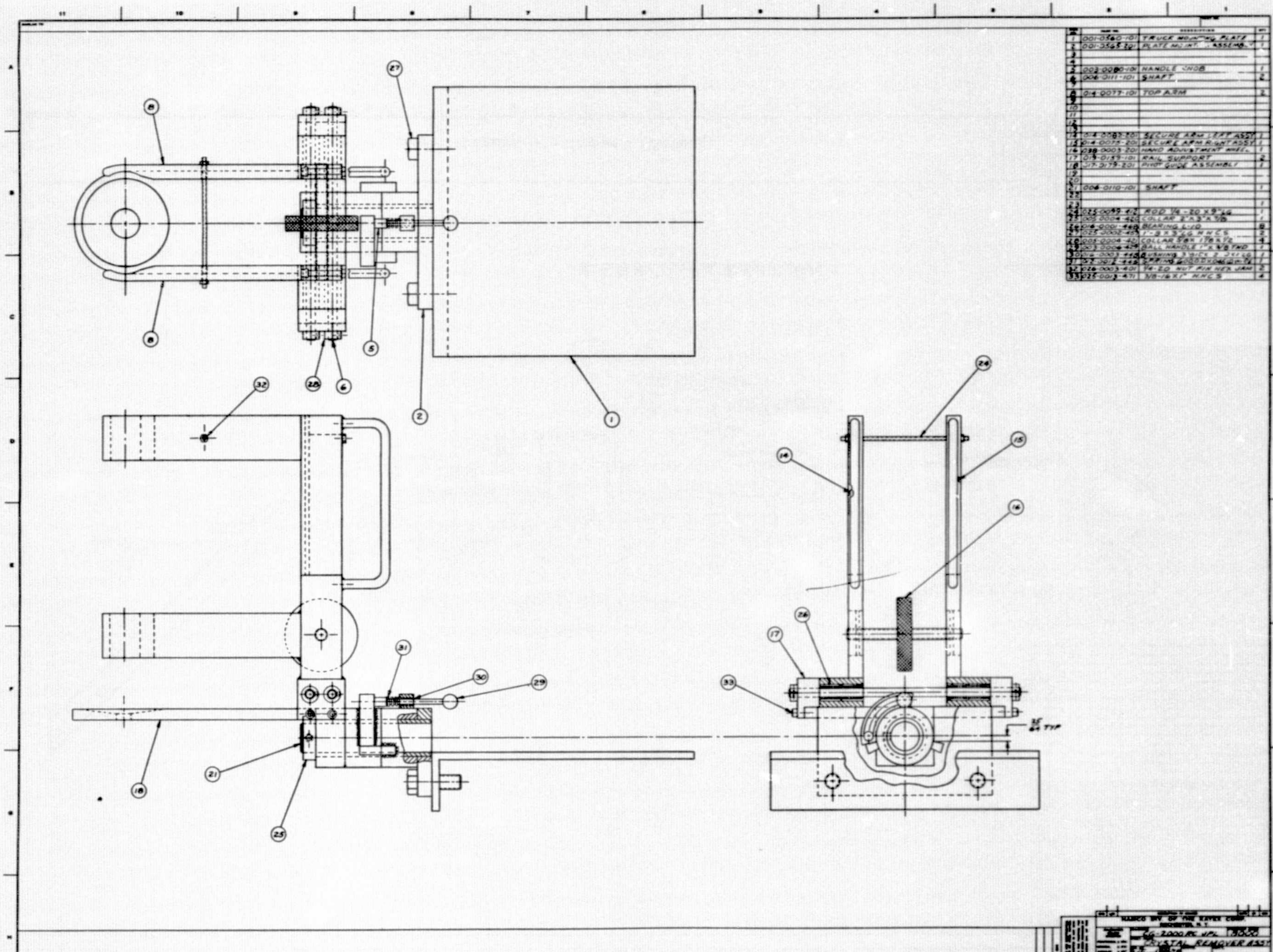



Figure 8 - Crystal/Poly Transfer Device

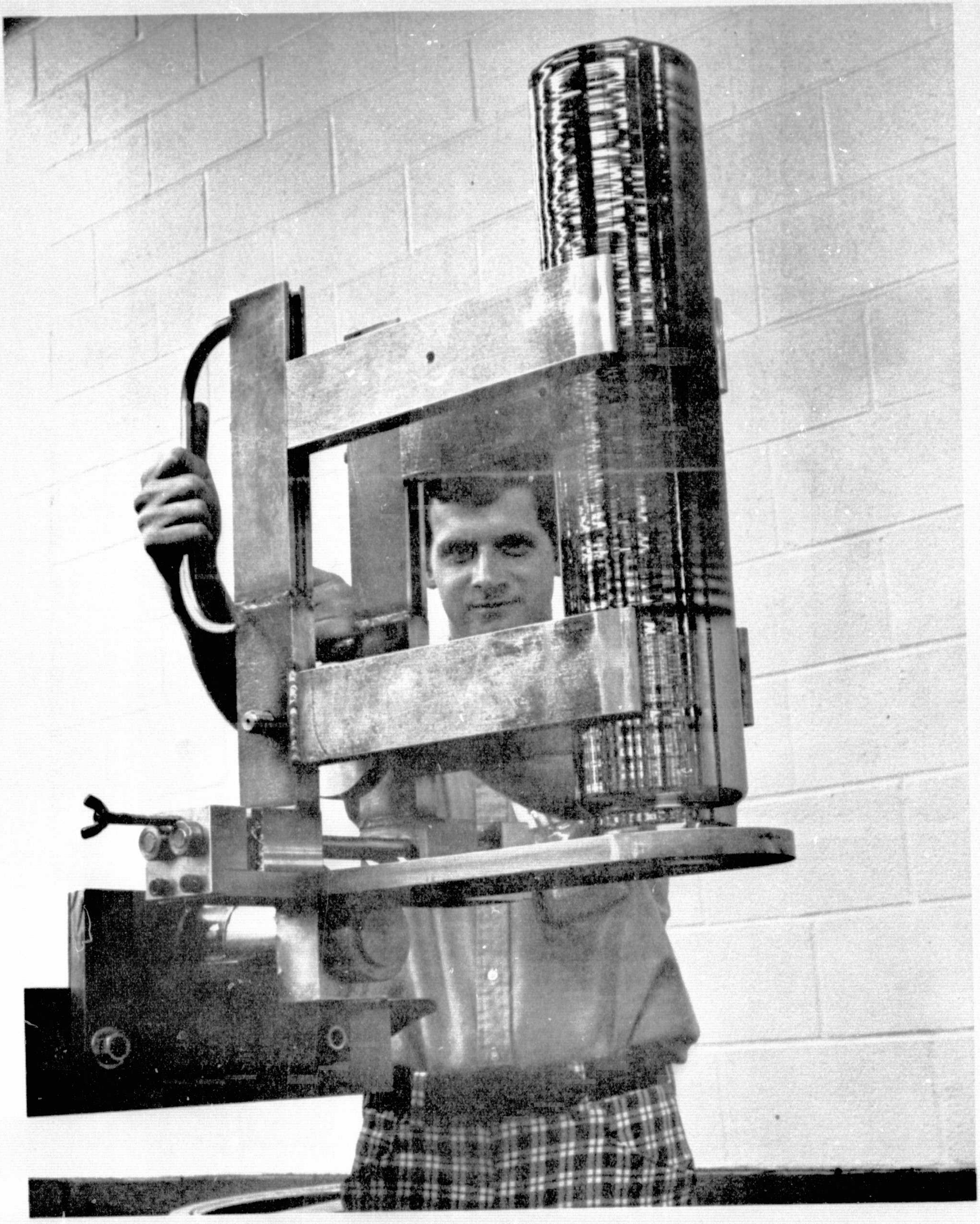


FIGURE 9 CRYSTAL/POLY TRANSFER DEVICE

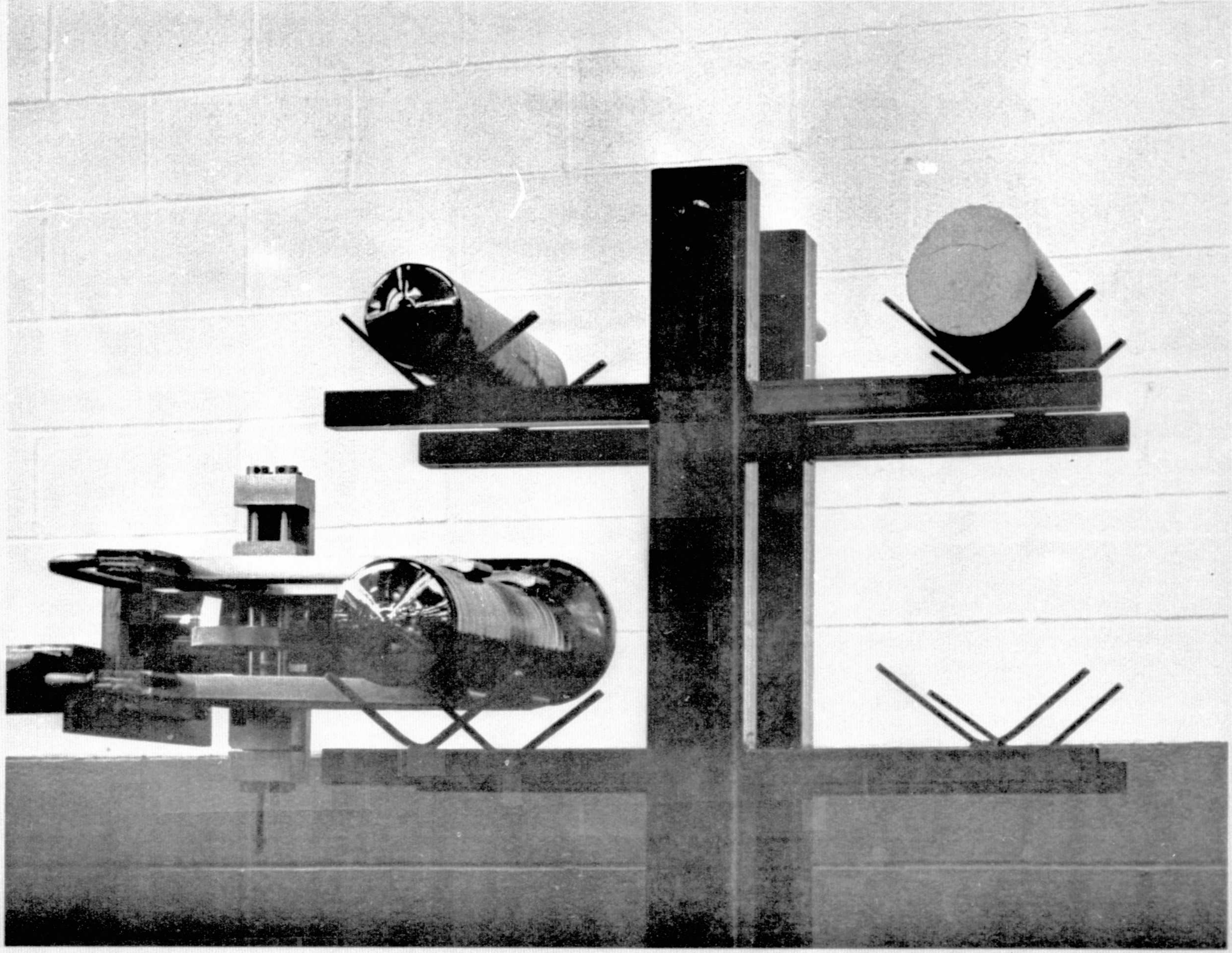


FIGURE 10  PLACING HOT CRYSTAL ON COOLING RACK

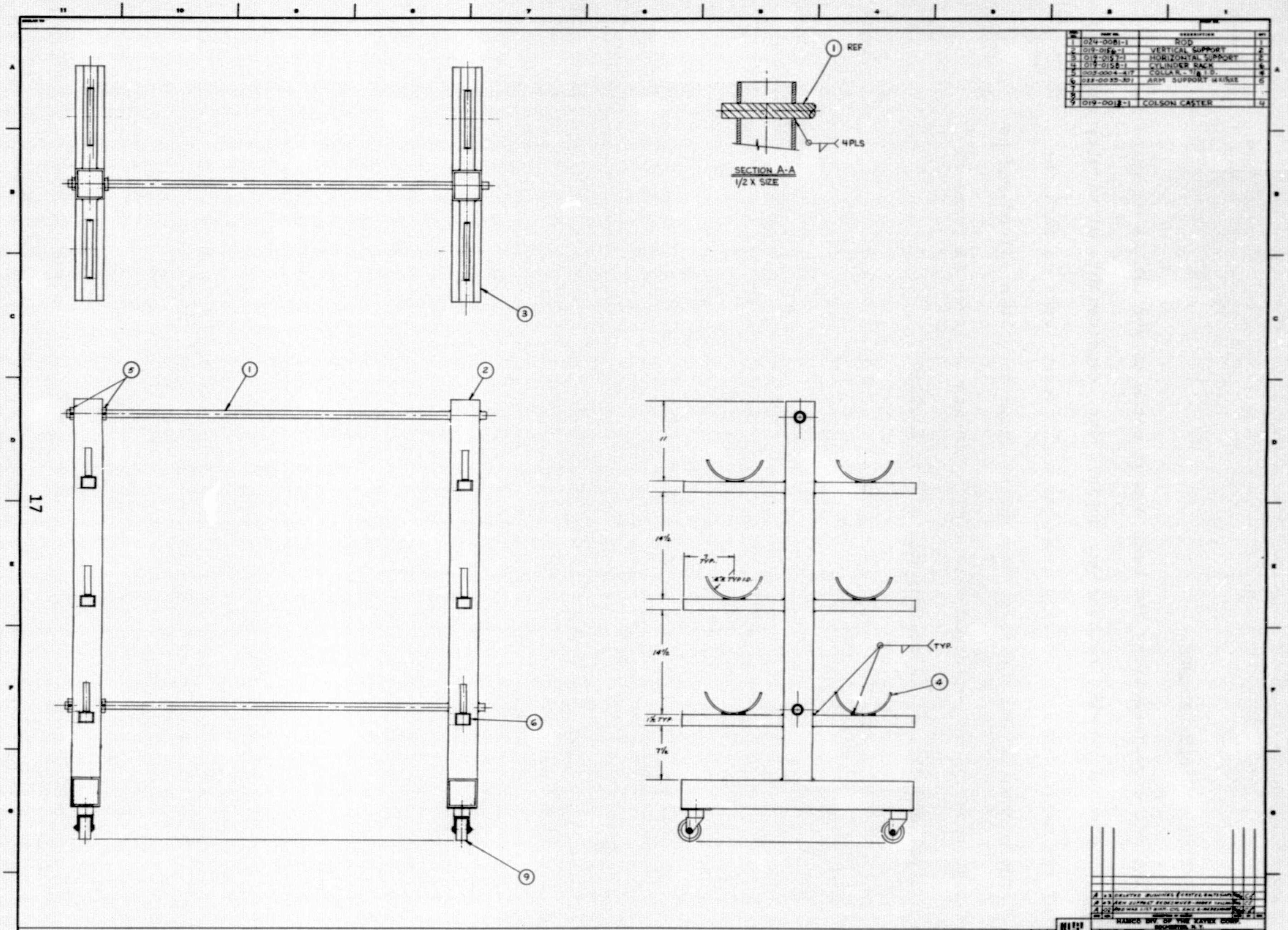



Figure 11- Crystal/Poly Storage Rack

## 2.2.0 GROWTH FACILITY PERFORMANCE

The JPL growth facility has been demonstrated to perform as expected in terms of crystal growth, recharging, doping, etc. Considerable difficulty and delay was experienced in solving problems with vacuum leaks in both the recharge mechanism and the large viewports. These have been solved and now recharge runs can be performed with a high confidence level in terms of mechanical and electrical performance of the facility.

## 2.3.0 PROCESS DEVELOPMENT

The equipment being used in this project has a design capacity of 16 kilograms pulled from a 20 kilogram melt, utilizing a 12-inch diameter by 9-inch high vitreous silica crucible. Since the goal of the project is to pull 100 kg from one crucible, this would mean 6.25 crystals would be required to achieve the goal.

The economic analysis indicated that cost reduction would result by pulling large crystals; therefore, the effort was directed at larger melts (24-25 kg) in a 12-inch crucible and subsequently 35-40 kg melts in a 14-inch crucible. The goal for the work was subsequently defined to be five 20 kg crystals from a 12-inch crucible and three 33 kg crystals from the 14-inch crucible.

Since neither the 12-inch or 14-inch crucible will hold that amount of silicon in unmelted chunk form (chunk poly has a packing density of only about 50% of theoretical silicon density), it was proposed that the first melt size be increased by "topping up" or hot-filling prior to starting the first crystal growth cycle. This would be performed by adding silicon by the same method proposed for recharging.

## 2.3.1 HOT FILL EXPERIMENTS

The first task was to establish the feasibility of hot filling using a polycrystalline silicon rod as feed material.

Five-inch diameter by 24-inch long rods were obtained (approximately 20 kg each). They were notched on one end to accept the poly attachment clamp.

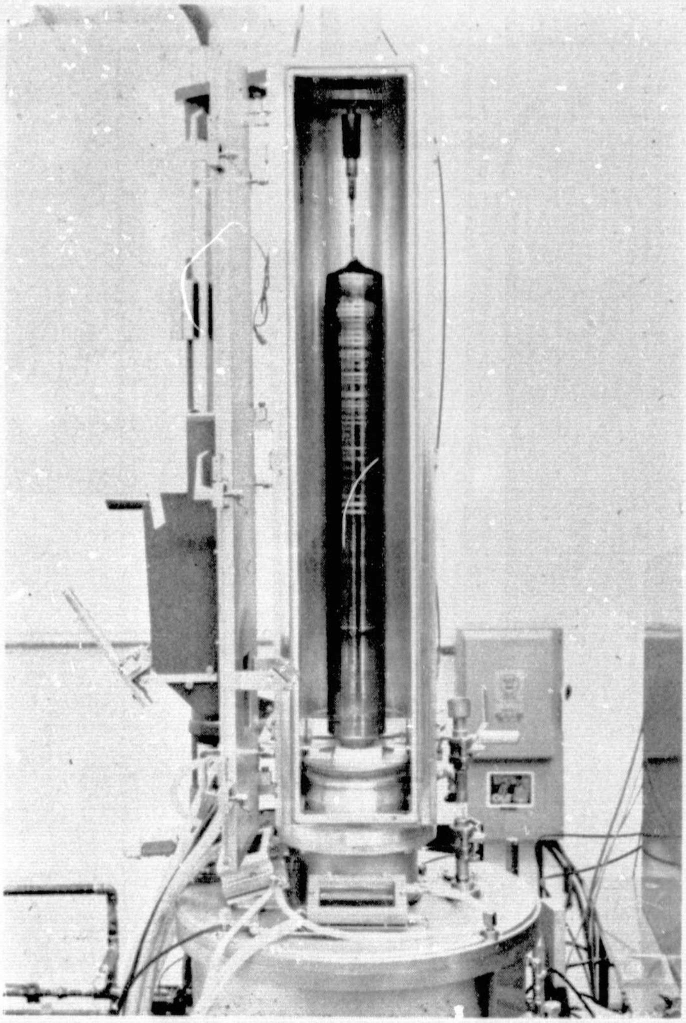
The crucible was loaded with 18 kg of chunk poly and melted. When melting was complete, the poly rod was lowered into the crucible until it established contact with the melt.

Seven kg of silicon were melted from the rod to achieve a total melt size of 25 kg. From this melt, a 22 kg high quality crystal was grown (Figure 12). Anticipated problems in achieving zero dislocation structure with such a full crucible did not occur.

The hot filling experiment showed that this method of filling a crucible to a high level is feasible and that 100 kg could be achieved with five 20 kg crystals.

Power consumption was monitored throughout this run (see Figure 13), and the data showed that the assumptions used in the economic model were valid.

The melt-off rate for the poly rod was 14 kg/hr, i.e. the seven kilograms required 30 minutes to melt, once the rod had achieved melt-back temperature.



A 22 kg crystal grown by hot filling technique

FIGURE 12

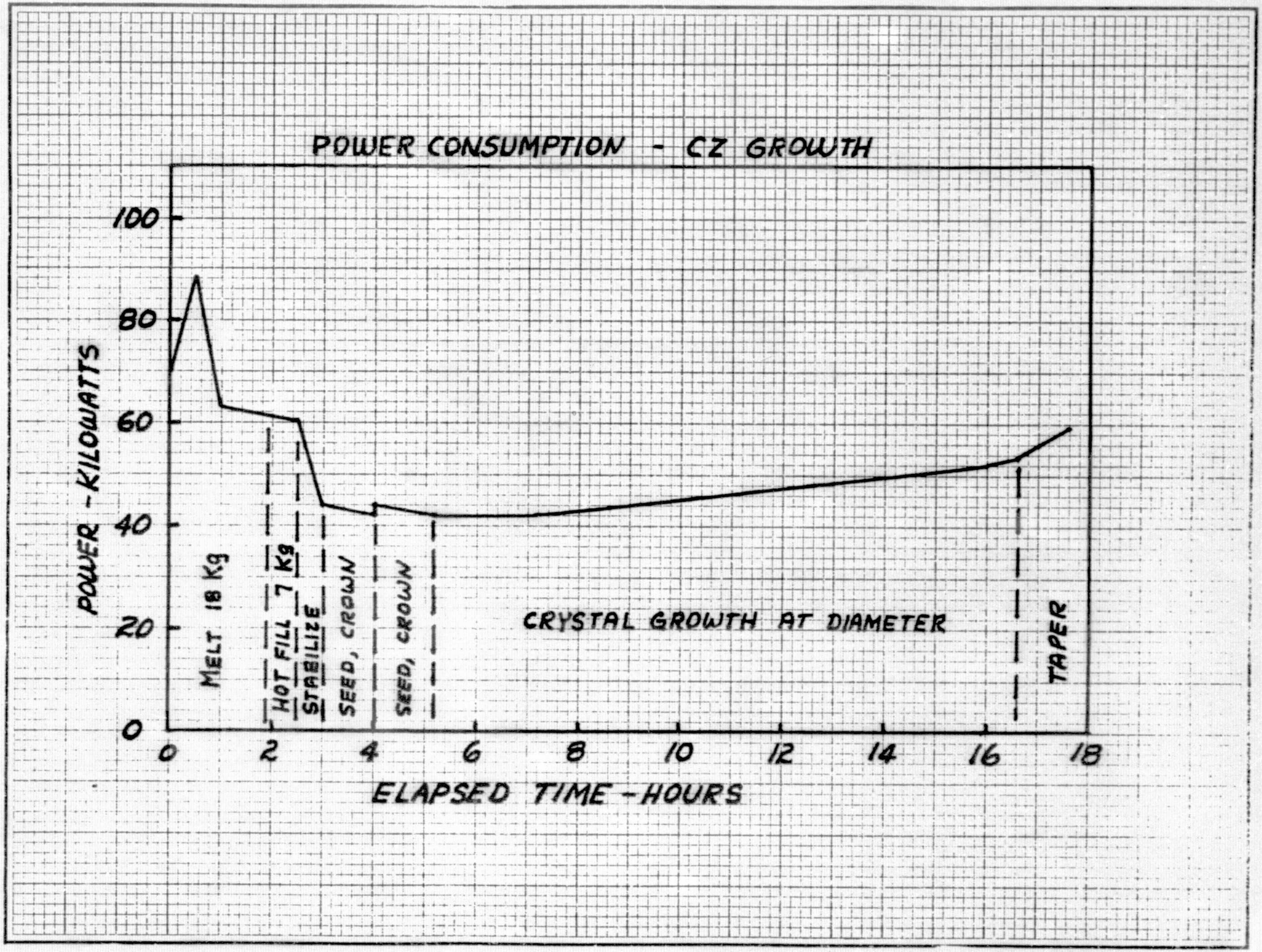

POWER CONSUMPTION - CZ GROWTH
POWER - KILOWATTS
0
20
40
60
80
100
ELAPSED TIME - HOURS
0
2
4
6
8
10
12
14
16
18
MELT 18 Kg
HOT FILL 7 Kg
STABILIZE
SEED, CROWN
SEED, CROWN
CRYSTAL GROWTH AT DIAMETER
TAPER

Figure 13

### 2.3.2 RECHARGE RUNS

Eight recharge runs were attempted during the reporting period. Most of these runs were not intended to be long runs and consisted of one or two recharges. On later runs, preparations were made to run as long as possible.

Table 1 lists several significant runs in the program. Run No. 5 was the hot-fill run previously described. Run No. 9 was a two-melt run producing 27 kg of single crystal ingot.

To date the longest run (#11) lasted 44 hours and resulted in 42.5 kilograms of ingot, most of which (88%) was high quality, zero dislocation material. This run was terminated because of excessive build-up of silicon monoxide on one viewport of the grower, causing structure loss problems, as silicon monoxide fell into the melt. The cause of the build-up of monoxide was determined later to be a pin-hole leak in a weld which allowed water vapor to enter the chamber.

The less successful runs were terminated for the following reasons:

1. The graphite upper crucible support failed in two instances, allowing the crucible to bulge. The runs were terminated because it was feared that the crucible wall would fail.
2. A polysilicon rod fractured and fell into the melt. This is believed to have been caused by a too rapid heat-up of the rod during recharging.
3. The ability to maintain zero dislocation crystal structure was lost. This has been the most frequent reason for terminating a run. At this point, the causes of structure loss are not known for certain, although foreign particles in the melt are the most probable cause. This is discussed later in this report.
4. A plant compressed air failure caused the vacuum system valves to close. The argon pressure in the furnace increased to atmosphere and excessive

monoxide developed. This mode of failure has been eliminated with a compressed air back-up system.

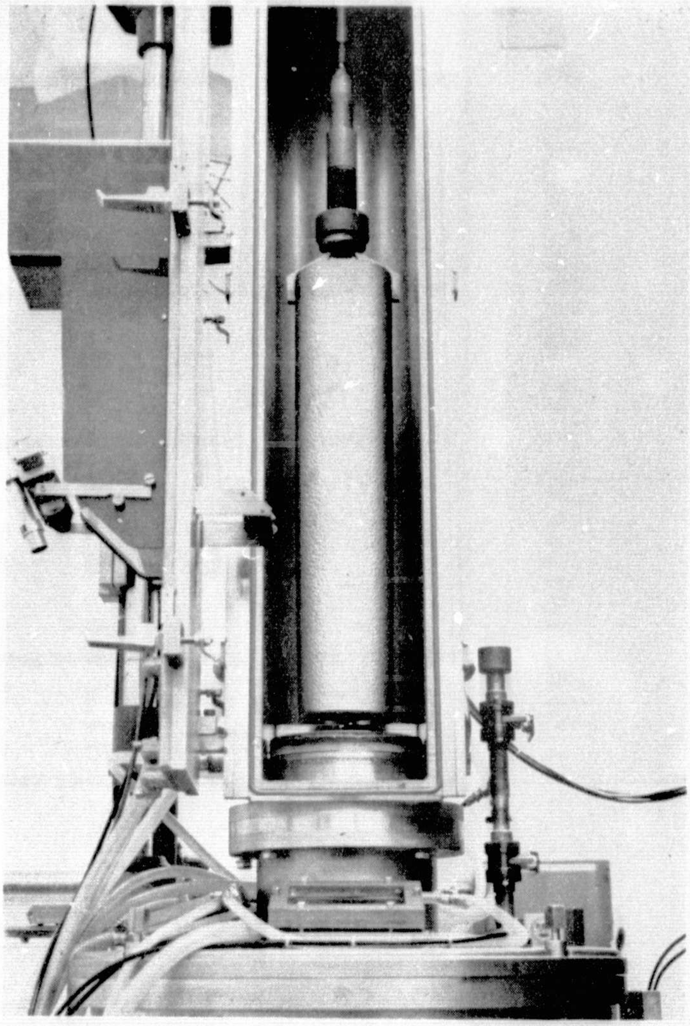
There have been no crucible failures in the program, although devitrification has been moderate to severe in some cases. This is discussed below.

Initially, all recharging was done with poly rods. Figures 14 and 15 illustrate a typical rod before and after recharging. Later, as it was necessary to recycle the silicon, we recharged with ingots from previous runs.

# CONTINUOUS CZ GROWTH SUMMARY

| RUN NO. | TOTAL MELTED (kg) | TOTAL PULLED (kg) | NO. CRYSTALS | DIAM. (cm) | AVG. PULL SPEED (cm/hr) | RUN TIME (hr) | THROUGH-PUT (kg/hr) | PULLED YIELD (%) | ZERO DISLOC. (%) |
|---|---|---|---|---|---|---|---|---|---|
| 5 | 25.0 | 22.0 | 1 | 11.0 | 9.1 | 18 | 0.82 | 88 | 100 |
| 9 | 31.2 | 27.1 | 3 | 11.2 | 8.7 | 39 | 0.70 | 87 | 85 |
| 11 | 48.7 | 42.5 | 4 | 10.7 | 9.1 | 44 | 0.97 | 87 | 88 |

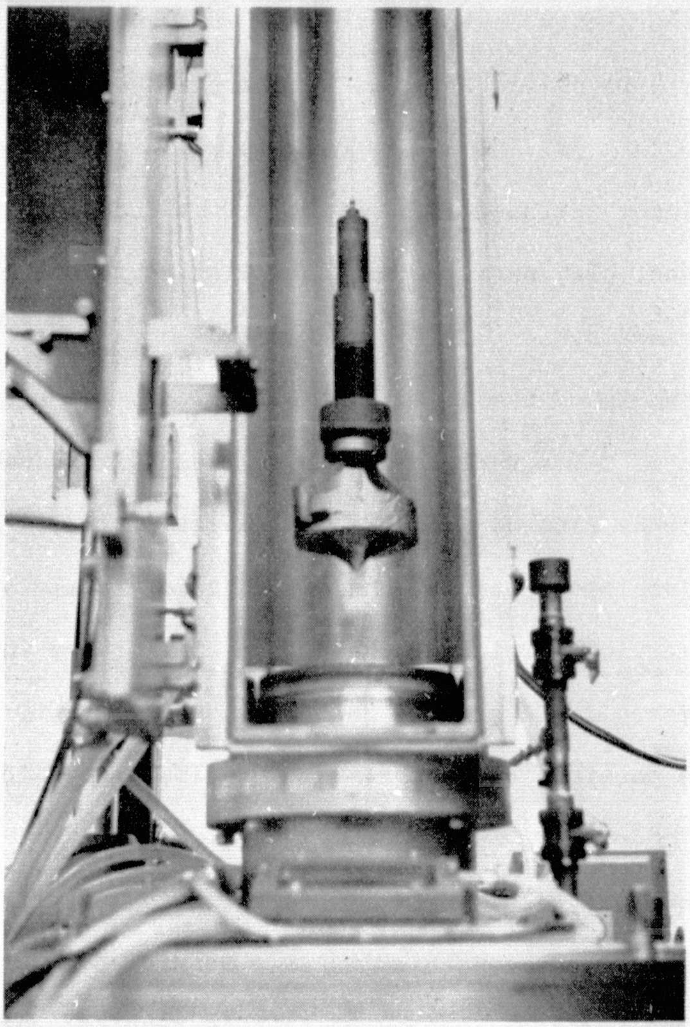
Table 1





Polysilicon rod ready for recharging

FIGURE 14



Polysilicon rod after recharging

FIGURE 15

2.3.3 CRYSTAL QUALITY

It is considered highly desirable to pull single crystal ingots having a zero dislocation (0-D) characteristic. With a clean system, 0-D crystals are relatively easy to grow. "Clean" means we use a high purity crucible free of defects on the inner surface, high purity polysilicon, high purity graphite parts in the hot zone, high purity argon, and that there are no leaks in the furnace chambers or seals.

It is well established that a foreign particle in the silicon melt will cause at least loss of 0-D structure and most likely will cause twinning or polycrystalline growth. Silicon monoxide and quartz particles have been observed to cause loss of structure, the effect being easily observed by the operator. Silicon carbide particles behave in a similar way in EFG growth. The possibility exists, in CZ growth, that SiOx, $SiO_2$, or SiC could enter the melt. Thus, all are possible causes of structure loss and should be investigated.

(a) Silicon Monoxide - This material is formed by vaporization from the melt and subsequent condensation as a submicron powder on cold surfaces in the furnace. The oxygen that is required for its formation comes from the crucible or from other sources such as air or water leaks, or impure argon. Although considerably more care must be taken on recharge runs to have a leak-tight system, it is believed that normal SiO deposition as a result of reaction between liquid silicon and the crucible can be tolerated. Gas turbulence in the furnace must be minimized to prevent dislodging particles from the inner walls and, to some extent, gas flow can be controlled in the furnace, directing the deposits to areas that are away from the growth region.

(b) Quartz ($SiO_2$) - Occasionally, quartz particles are seen floating on the melt. These are probably due to chips from the crucible produced during the loading or melt-down. Quartz is distinguishable from monoxide because it does not evaporate, and the operator waits until the particle lodges against the wall of the crucible before starting a crystal.

Assuming proper care is exercised in loading the crucible initially and that the crucibles are high quality, then visible quartz particles appearing on the melt initially should be very rare.

During long runs, however, microscopic particles of quartz may be released from the crucible, which could float to the surface and destroy the crystal structure. Defects in the inner wall of the crucible such as unfused areas, pinholes, blisters or gas bubbles near the surface are potentially sources of quartz particles.

(c) Silicon Carbide (SiC) - Little is known about silicon carbide in CZ melts. Carbon contamination in CZ furnaces occurs, but the mechanism of its production is not clear, and the level of contamination is below saturation in semiconductor grade wafers.

If there is a CO or $CO_2$ phase present in the furnace atmosphere due to oxygen from leaks or from the crucible, then gaseous transport to the melt would appear to be feasible. In long recharge runs, saturation could occur, leading to precipitation of SiC particles at the growth interface.

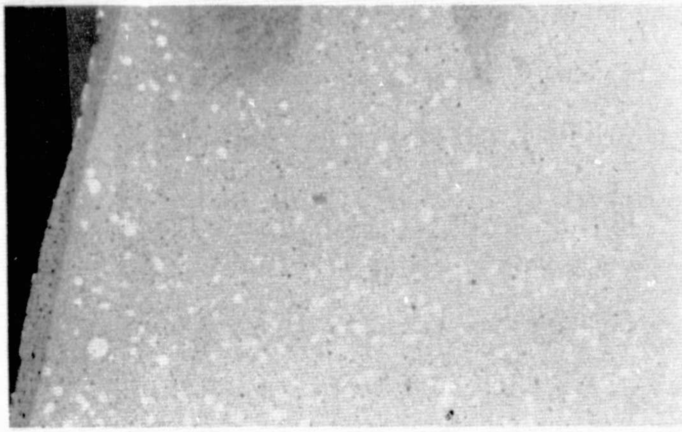
The desired crystal quality for this program has been largely achieved up to 42.5 kg (Run 11), but attempts at increasing the throughput have failed due to structure loss. Investigation of the mechanism of structure loss will be carried out in the future.

## 2.3.4 CRUCIBLE PERFORMANCE

The role of the crucible in structure loss is not well understood. In a typical non-recharge run of 12 to 18 hours duration (Figure 16), the inner surface of the crucible usually shows a slight amount of devitrification in the form of small circular spots about 1 mm in diameter randomly distributed. The surface, however, is generally smooth and free of pits or open bubbles.

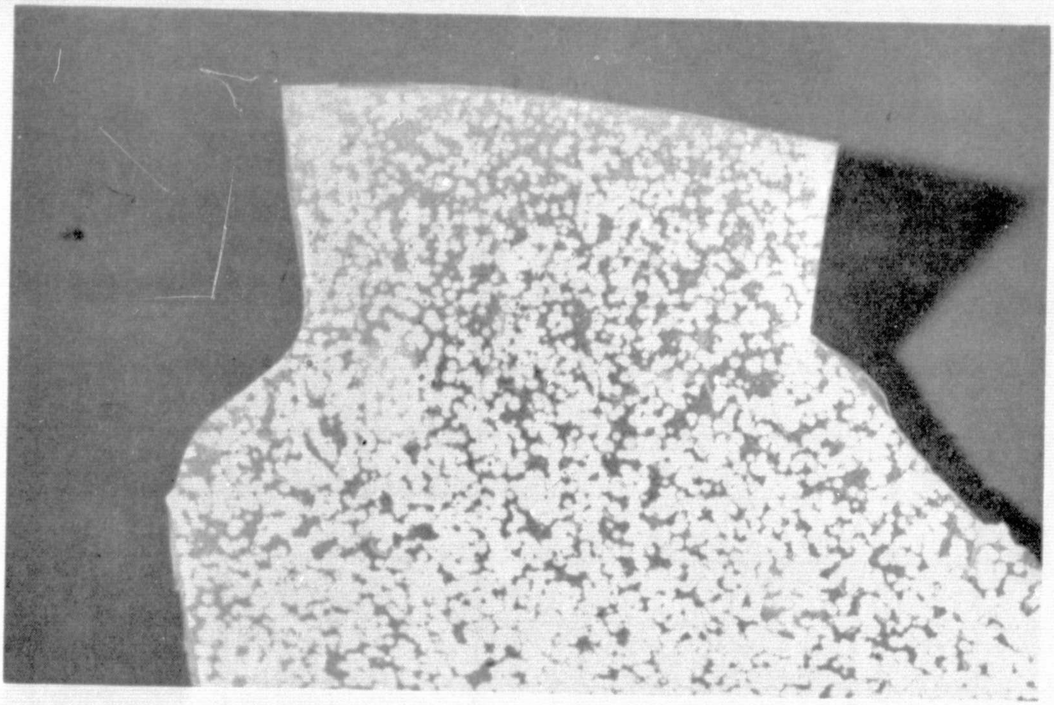
Severe devitrification (Figure 17) has been observed occasionally after long periods in contact with the melt. The circular devitrified areas grow and become more numerous. Bubbles originally beneath the surface are exposed. The illustration is not typical, however, of all long recharge runs. In this case, the condition of the crucible was far worse than usually observed.

The visual appearance of crucibles from various commercial vendors varies considerably. Differences in the degree of fusion, the inside surface quality, and the bubble structure are apparent. Chemical purity of all crucibles is good, yet some differences are apparent. The analyses in Table 2 were supplied by the vendors.

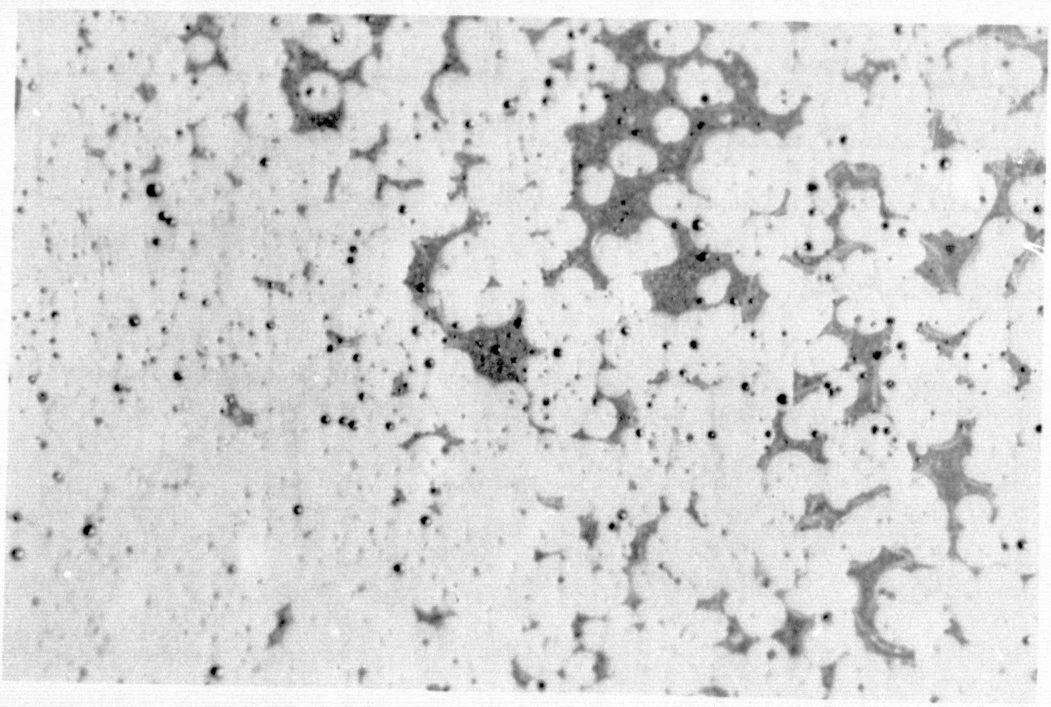


Crucible devitrification after 18 hours
(Inside surface, actual size) ⇤ ⇥ 1 cm

FIGURE 16



17a Actual size ⇤ ⇥ 1 cm



17b Magnified 3.5X ← 1 cm →



Unusually severe crucible devitrification

FIGURE 17

CRUCIBLE IMPURITIES

| Impurity | Amersil | Quartz Products | General Electric | Corning |
|---|---|---|---|---|
| Al | 12 | 20 | 28 | 5 |
| B | <1 | NR | <1 | 0.5 |
| Ca | 4 | 3 | 3 | 1 |
| Ca | < 1 | NR | <1 | 0.05 |
| Fe | 3 | 1 | 4 | 5 |
| Li | 1 | 1 | 0.5 | NR |
| Mg | 1 | 1 | 1 | 0.1 |
| K | 2 | 3 | 3 | 0.1 |
| Na | 3 | 1 | 3 | 2 |
| Ti | < 1 | 2 | 2 | 10 |
| Cr | NR | NR | 3 | NR |
| Zn | NR | NR | 2 | NR |
| Mn | NR | NR | 2 | NR |

NR: Not Reported

Maximum impurities in Fused Quartz (vitreous $SiO_2$) crucibles provided by vendors. Expressed as parts per million by weight.

Table 2

### 2.3.5 SOLAR CELL EVALUATION

Recharge runs No. 9 and 11 were selected for solar cell measurements. Samples were taken from the top and bottom of all crystals and were sent to JPL for evaluation. The cell fabrication and testing was performed at OCLI.

Table 3 presents the resulting data from these runs. Each value represents the average value of four cells prepared from four quadrants of one wafer cut from the ingot. All data were taken under AMO illumination. (AMI values are calculated from AMO values.)

Resistivities were measured by the four point probe method.

The control sample was optimum high quality CZ material provided by OCLI and had an average efficiency of 14.6% AMI. Efficiencies for run No. 9 varied from 14.1 down to 12.6, whereas run No. 11 had a range of 14.6 to 13.0.

There is a generally small trend downward in efficiency throughout the run; however, in both runs, the sample of lowest efficiency was not from the last material grown. This is surprising for the last material grown in both runs was dislocated.

In general, the efficiency measurements are so close that the spread of data could account for the unexplained values. For example, the control sample average was 14.7 whereas the eight samples which made up the average ranged from 14.0 to 15.1% AMI.

SOLAR CELL LIGHT CURRENT-VOLTAGE CHARACTERISTICS
DATA TAKEN AT AMO (135.3 mw at 28°C)

| Run No. | Ingot No. | Sample Description | Resistivity ohm-cm | $I_{sc}$ (mr) | $V_{oc}$ (mv) | FF | $P_{max}$ (mw) | $n_{avg}$ AMO | $n_{avg}$ AMI |
|---|---|---|---|---|---|---|---|---|---|
| Control | | High Quality Optimum CZ | -- | 134.8 | 567.2 | .74 | 56.9 | 10.5 | 14.7 |
| 9 | 2 | Top of ingot, OD | 5.75 | 137.5 | 542.4 | .74 | 54.9 | 10.1 | 14.1 |
| 9 | 2 | Middle of ingot, OD | 6.00 | 133.3 | 536.6 | .71 | 51.0 | 9.5 | 13.3 |
| 9 | 2 | Bottom of ingot, OD | 5.6 | 135.0 | 543.8 | .73 | 53.3 | 9.9 | 13.9 |
| 9 | 2 | Bottom, dislocated | 5.6 | 135.2 | 543.2 | .73 | 53.7 | 9.9 | 13.9 |
| 9 | 3 | Top of ingot, OD | 5.2 | 134.0 | 537.6 | .67 | 48.4 | 9.0 | 12.6 |
| 9 | 3 | Top of ingot, dislocated | 5.2 | 132.5 | 537.4 | .69 | 49.2 | 9.1 | 12.7 |
| 9 | 3 | Bottom - polycrystalline | 4.4 | 132.5 | 540.7 | .71 | 50.9 | 9.4 | 13.2 |
| 11 | 1 | Top of ingot, OD | 2.99 | 134.3 | 564.4 | .74 | 56.3 | 10.4 | 14.6 |
| 11 | 2 | Top of ingot, OD | 2.74 | 133.9 | 546.8 | .72 | 52.5 | 9.7 | 13.6 |
| 11 | 2 | Bottom of ingot, OD | 2.57 | 135.8 | 558.3 | .72 | 54.4 | 10.1 | 14.1 |
| 11 | 3 | Top of ingot, OD | 3.69 | 137.2 | 552.3 | .72 | 54.9 | 10.2 | 14.3 |
| 11 | 4 | Top of ingot, OD | 3.52 | 135.2 | 549.7 | .71 | 52.9 | 9.3 | 13.0 |
| 11 | 4 | Bottom of ingot, OD | 3.62 | 132.4 | 546.6 | .70 | 50.6 | 9.3 | 13.0 |
| 11 | 4 | Bottom - dislocated | 3.62 | 131.4 | 550.7 | .72 | 52.0 | 9.6 | 13.4 |

Table 3

## 2.4.0 ECONOMIC ANALYSIS

The SAMICS format was used to calculate the add-on cost of continuous CZ growth for the two modes of operation to be studied during this contract.

The following is a comparison of the CZ No. 1 method (12-inch diameter crucible, five 20 kg crystals) with the lower cost method, CZ No. 2, which assumes four 33 kg crystals to be grown from one 14-inch diameter crucible.

Table 4 gives the assumed conditions for CZ No. 1 and CZ No. 2. Table 5 contains time cycles for the two methods.

The criterion for choosing the conditions for CZ Nos. 1 and 2 was to keep the total run time approximately constant for both methods, i.e., approximately 75 hours. CZ No. 2 produces considerably more crystal in the same run time because the crystal diameter is larger while the growth rate remains the same at 10 cm/hr. The resulting significant increase in throughput is the largest single factor in reducing the CZ add-on cost.

Input data for the SAMICS/IPEG cost calculation are detailed in Table 6. The add-on cost for CZ No. 1 is \$19.40/kg and for CZ No. 2 is \$15.07/kg. All costs are expressed in 1975 dollars. The calculations illustrate the economy of increasing the throughput by an increase in crystal diameter.

The cost per meter$^2$ indicated assumes that one kg of ingot will produce one square meter of silicon sheet.

## CZ GROWTH METHODS

| CONDITIONS | CONTINUOUS NO. 1 | CONTINUOUS NO. 2 |
|---|---|---|
| CRUCIBLE SIZE (in.) | 12 x 9 h | 14 x 11-1/2 h |
| CRYSTAL DIAMETER (cm) | 10 | 12.5 |
| GROWTH RATE (cm/hr) | 10 | 10 |
| TOTAL POLY MELTED (kg) | 105 | 143.3 |
| TOTAL CRYSTAL PULLED (kg) | 100 | 134 |
| PULLED YIELD | 95 | 94 |
| USABLE AFTER GRINDING (kg) | 87.5 | 117 |
| GROUND CRYSTAL YIELD (%) | 83 | 81.5 |
| NO. CRYSTALS/CRUCIBLE | 5 | 4 |

TABLE 4

PROCESS CYCLE TIME

| OPERATION | CZ NO. 1 (MIN.) | | CZ NO. 2 (MIN.) | |
|---|---|---|---|---|
| 1. Preparation | | 180 | | 240 |
| Load Poly | 45 | | 45 | |
| Close Furnace | 5 | | 5 | |
| Pump Down | 10 | | 10 | |
| Melt | 120 | | 180 | |
| 2. Hot Fill/Growth Cycle | | 765 | | 895 |
| Position Poly | 5 | | 5 | |
| Lower Poly | 5 | | 5 | |
| Melt Poly | 60 | | 120 | |
| Retract Poly* | 5* | | 5* | |
| Lower Seed* | 15* | | 15* | |
| Stabilize Temperature | 40 | | 40 | |
| Seed Growth | 30 | | 30 | |
| Crown Growth | 60 | | 60 | |
| Straight Growth | 525 | | 575 | |
| Taper End | 40 | | 60 | |
| 3. Recharge/Growth Cycles | | 3460 (4 cycles) | | 3210 (3 cycles) |
| Cool Crystal | 30 | | 45 | |
| Remove Crystal | 10 | | 10 | |
| Position Poly | 5 | | 5 | |
| Lower Poly | 5 | | 5 | |
| Melt Poly | 120 | | 220 | |
| Retract Poly* | 5* | | 5* | |
| Lower Seed* | 15* | | 15* | |
| Stabilize Temperature | 40 | | 60 | |
| Seed Growth | 30 | | 30 | |
| Crown Growth | 60 | | 60 | |
| Straight Growth | 525 | | 575 | |
| Taper End | 40 | | 60 | |
| 4. Shut Down Cycle | | 120 | | 160 |
| Cool Furnace | 60 | | 80 | |
| Remove Crystal** | 10** | | 10** | |
| Clean, Set Up | 60 | | 80 | |
| Total Time (Min.) | | 4525 | | 4505 |
| (Hr.) | | 75.4 | | 75.1 |

*Completed during stabilization of melt temperature.
**Completed during furnace cooling time.

TABLE 5

## SAMICS/IPEG INPUT DATA AND COST CALCULATION FOR CZ NO. 1 AND CZ NO. 2

| Conditions: (Per Cycle) | CONTINUOUS CZ NO. 1 | CONTINUOUS CZ NO. 2 |
|---|---|---|
| Total Si Melted (kg) | 105 | 143.3 |
| Crystal Weight (kg) | 20 | 33.4 |
| No. of Crystals/Crucible | 5 | 4 |
| Diameter of Crystal (cm) | 10 | 12.5 |
| Growth Rate (cm/hr) | 10 | 10 |
| Cycle Time (hrs) | 75.4 | 75.1 |
| Crucible Size | 12" x 9" high | 14" x 11-1/2" high |
| Yield (%) | 83 | 81.5 |
| **Input Data (1975 $)** | | |
| Capital Equipment Cost (EQPT) | $124,700 | $124,700 |
| Manufacturing Floor Space (SQFT) | 100 | 100 |
| Annual Direct Labor Salaries | | |
| Prod. Operator (1.33 prsn/yrs) | $ 11,572 | $ 11,572 |
| Elect. Tech. (1.33 prsn/yrs) | $ 1,018 | $ 1,018 |
| Inspector (.1 prsn/yrs) | $ 763 | $ 763 |
| Total (DLAB) | $ 13,353 | $ 13,353 |
| **Direct Used Material and Supplies** | | |
| 85% Usage per year | | |
| Cycles/yr; hrs/cycle | 98.7; 75.4 | 99.1; 75.1 |
| Poly - kg/yr (charged) | 10,363 kg | 14,201 kg |
| Seed ($4.16/cycle) | $ 410 | $ 412 |
| Dopant (not costed) | --- | --- |
| Argon (100 $ft^3$/cycle - hr @ $0.014/$ft^3$) | $ 9,563 | $ 9,563 |
| Crucibles - $166/12"; $208/14" | $ 16,384 | $ 20,613 |
| Miscellaneous (including graphite $2.25/cycle-hr (12"); $2.50/cycle-hr (14")) | $ 16,753 | $ 18,615 |
| Material Total (MATS) | $ 43,110 | $ 49,203 |
| **Utilities (Process)** | | |
| Electricity (C1032B) (50 kw)($.025/kw) (cycle time-3 hr)(#cycles) | $ 8,932 | $ 8,932 |
| Cooling Water (C11280) (50 kw)($.00528/kw)(cycle time-2 hr)(#cycles) | $ 1,912 | $ 1,912 |
| Utilities Total (UTIL) | $ 10,844 | $ 10,844 |

TABLE 6

| | | CONTINUOUS CZ NO. 1 | CONTINUOUS CZ NO. 2 |
|---|---|---|---|
| **IPEG PRICE** | | | |
| $C_1$ | *EQPT = $0.489/yr-dollar *EQPT | $ 60,978 | $ 60,978 |
| $C_2$ | *SQFT = $96.9/yr-ft *SQFT | 9,690 | 9,690 |
| $C_3$ | *DLAB = $2.133/yr-dollar *DLAB | 28,482 | 28,482 |
| $C_4$ | *MATS = $1.255/yr-dollar *MATS | 54,103 | 61,750 |
| $C_5$ | *UTIL = $1.255/yr-dollar *UTIL | 13,609 | 13,609 |
| | TOTAL | $166,862 | $174,509 |
| | QUAN (total charge x % yield)(kg) | 8,602 kg | 11,574 kg |
| | Throughput | 1.12 kg/hr | 1.56 kg/hr |
| | ADD-ON COST ($/kg or $/$m^2$) (assume 1 kg → 1 $m^2$) | $19.40 | $15.07 |

TABLE 6

## 3.0 PLANS

Continued efforts toward increasing the throughput of recharge runs consistent with the production of high quality ingots will be the main thrust of the work.

Investigation of the causes of structure loss by analysis of residual silicon, grown ingots and crucibles will be performed.

A lump silicon recharge device will be designed. See Program Plan, Figure 18.

## 4.0 COSTS AND LABOR SCHEDULE

The following costs and man-hours have been expended on the first year of the project.

| | |
|---|---|
| Man-hours | 5833 |
| Cost | $219,719 |

Figures 19 and 20 compare projected with actual costs and man-hours.

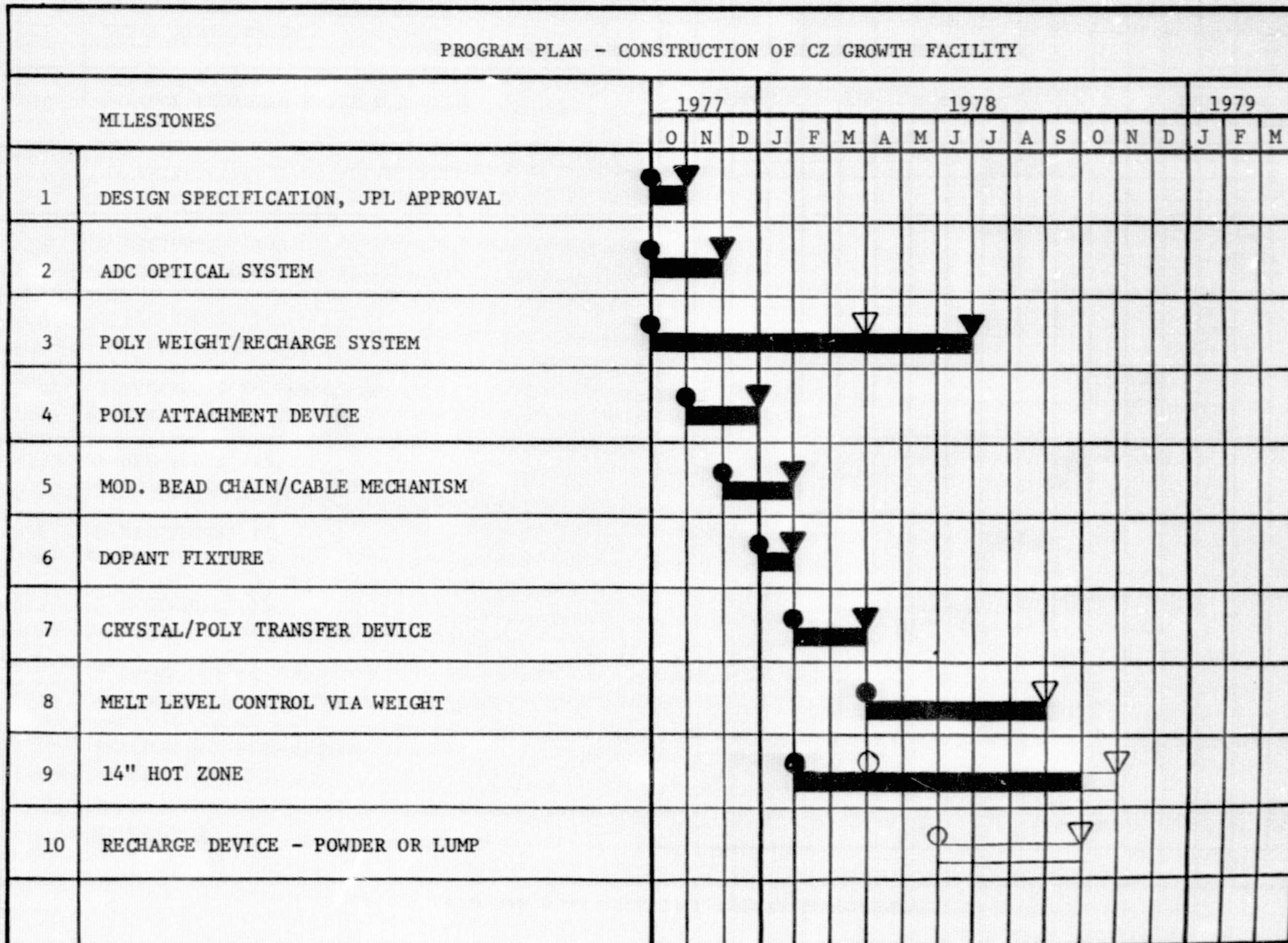

PROGRAM PLAN - CONSTRUCTION OF CZ GROWTH FACILITY
MILESTONES
1977
1978
1979
O N D J F M A M J J A S O N D J F M
1 DESIGN SPECIFICATION, JPL APPROVAL
2 ADC OPTICAL SYSTEM
3 POLY WEIGHT/RECHARGE SYSTEM
4 POLY ATTACHMENT DEVICE
5 MOD. BEAD CHAIN/CABLE MECHANISM
6 DOPANT FIXTURE
7 CRYSTAL/POLY TRANSFER DEVICE
8 MELT LEVEL CONTROL VIA WEIGHT
9 14" HOT ZONE
10 RECHARGE DEVICE - POWDER OR LUMP

Figure 18a

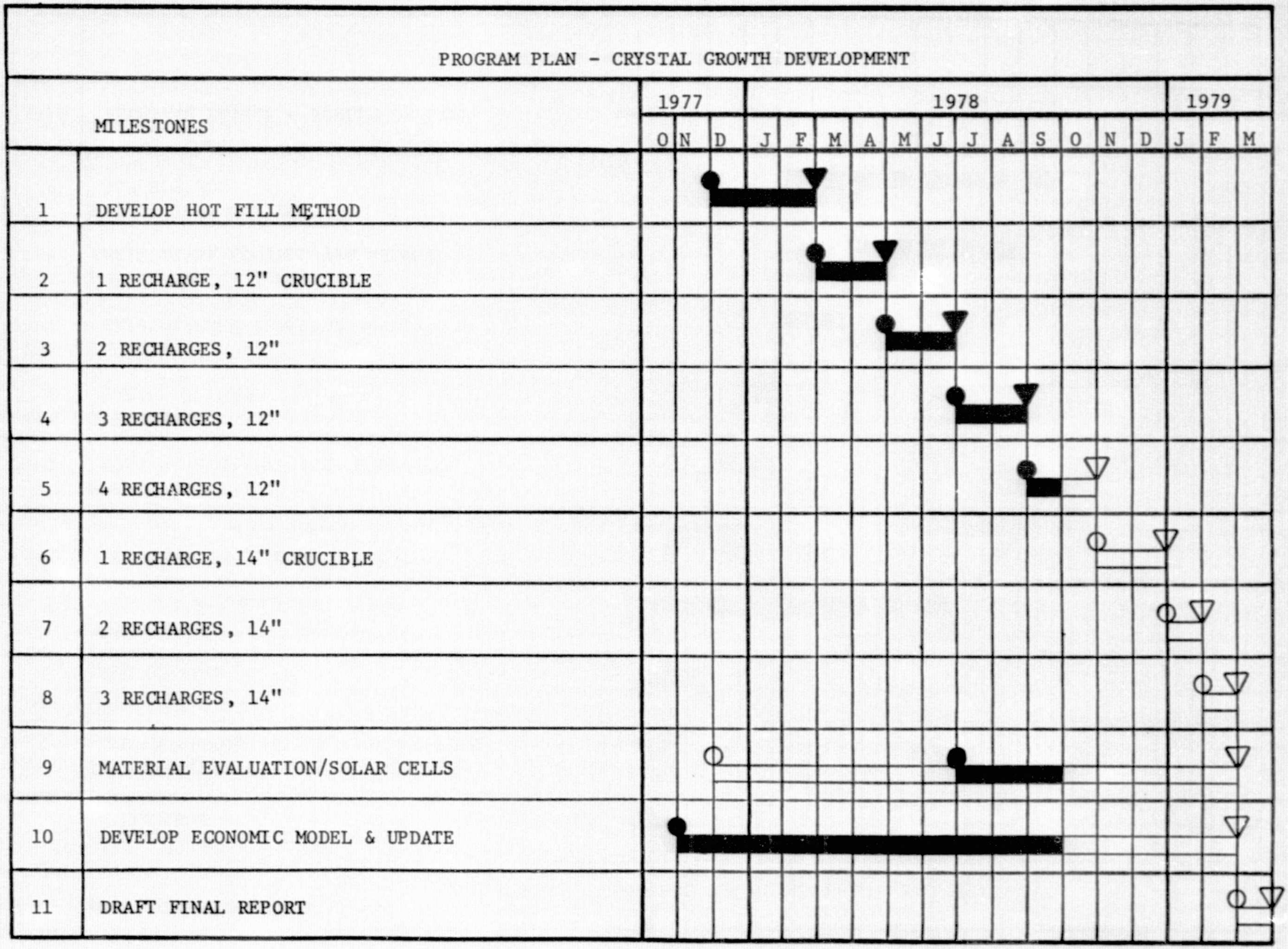

PROGRAM PLAN - CRYSTAL GROWTH DEVELOPMENT
MILESTONES
1977
1978
1979
O N D J F M A M J J A S O N D J F M
1 DEVELOP HOT FILL METHOD
2 1 RECHARGE, 12" CRUCIBLE
3 2 RECHARGES, 12"
4 3 RECHARGES, 12"
5 4 RECHARGES, 12"
6 1 RECHARGE, 14" CRUCIBLE
7 2 RECHARGES, 14"
8 3 RECHARGES, 14"
9 MATERIAL EVALUATION/SOLAR CELLS
10 DEVELOP ECONOMIC MODEL & UPDATE
11 DRAFT FINAL REPORT

Figure 18b

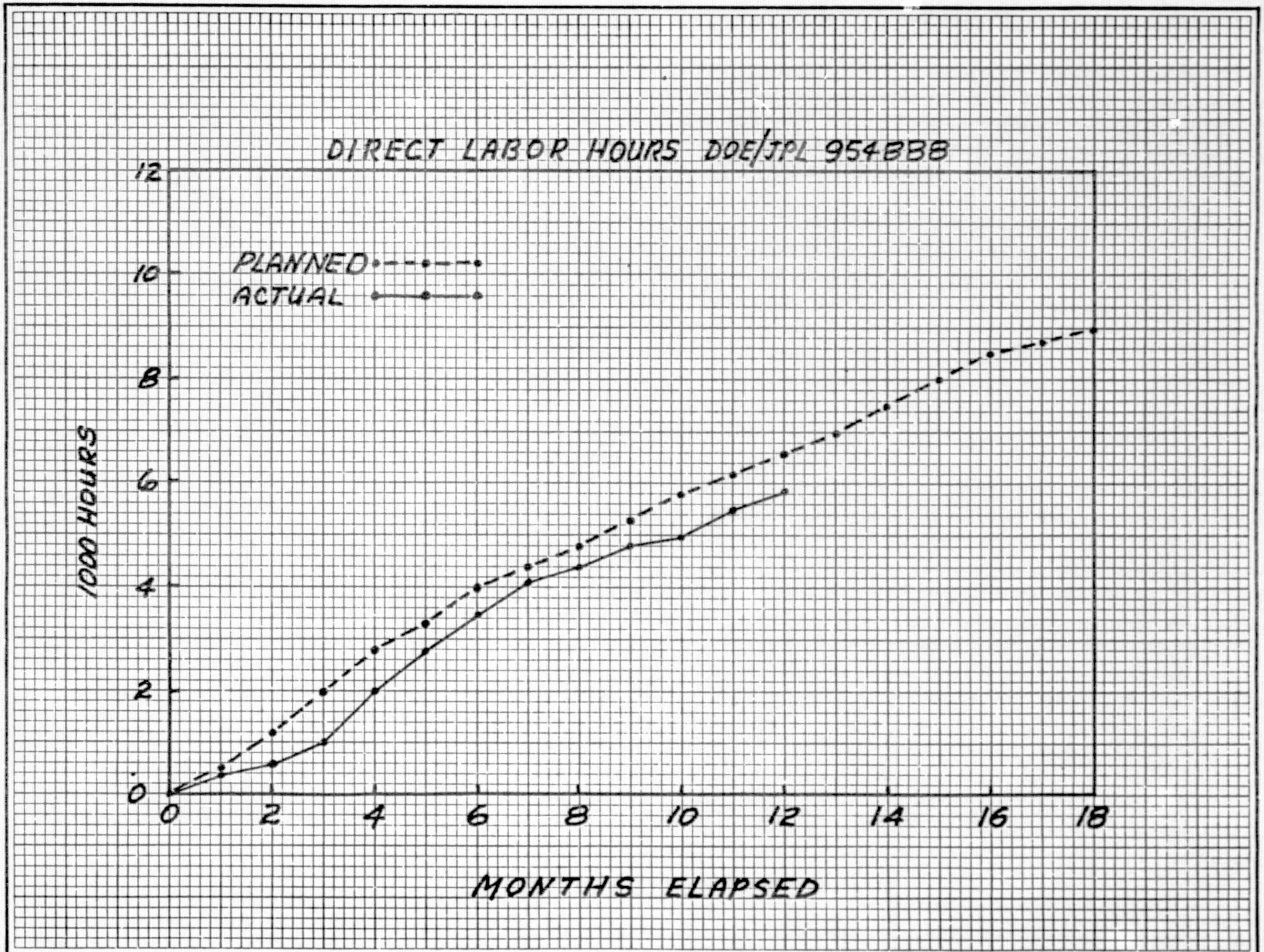

DIRECT LABOR HOURS DOE/JPL 954BBB
PLANNED
ACTUAL
1000 HOURS
0
2
4
6
8
10
12
14
16
18
MONTHS ELAPSED

Figure 19

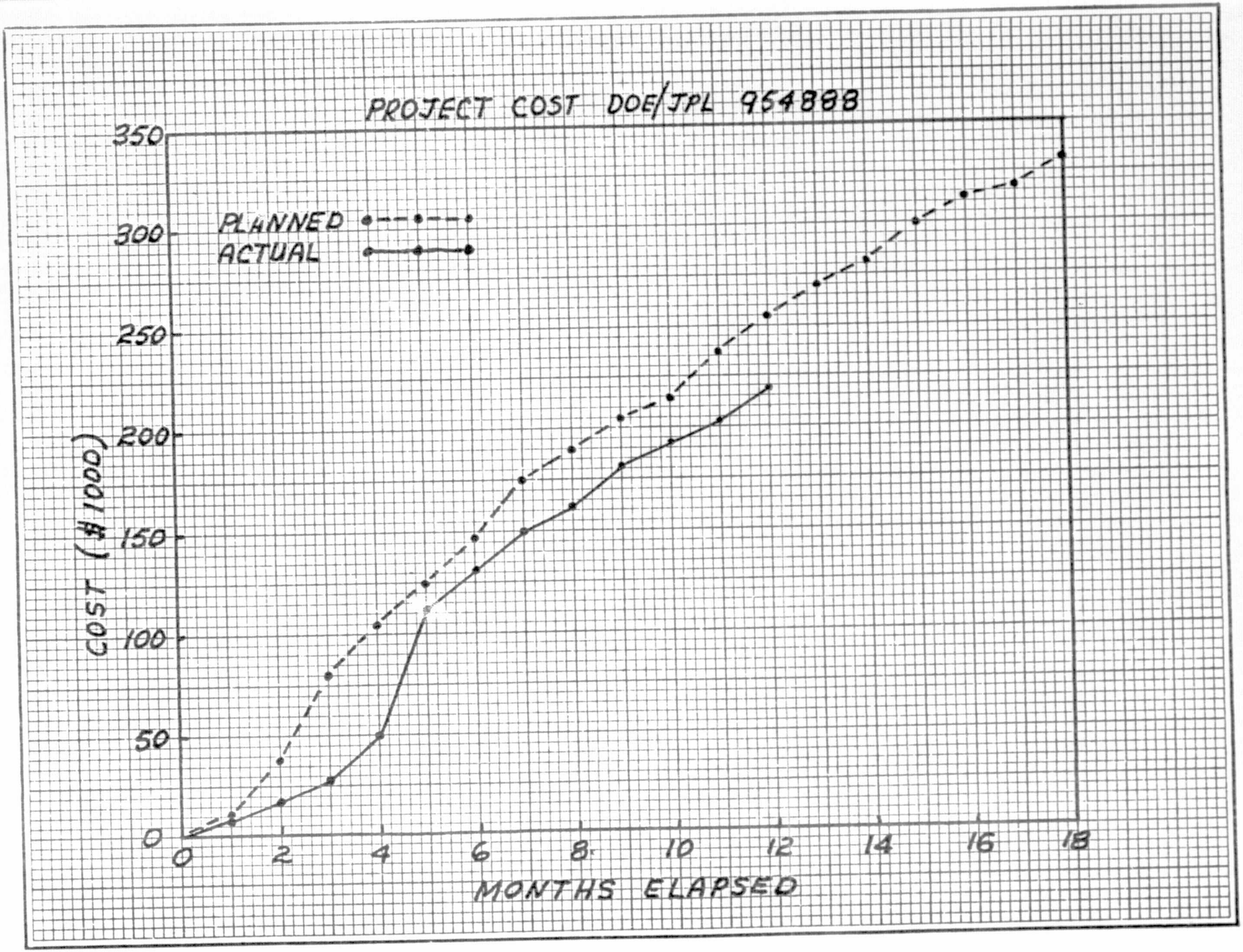

PROJECT COST DOE/JPL 954888
PLANNED
ACTUAL
COST ($1000)
0
50
100
150
200
250
300
350
0
2
4
6
8
10
12
14
16
18
MONTHS ELAPSED

Figure 20