 D4-44

# ADVANCED CZOCHRALSKI INGOT GROWTH

KAYEX CORP.

R.L. Lane

IN TODAY'S MARKET, A VERY HIGH PERCENTAGE OF THE SOLAR CELLS PRODUCED CONTINUE TO BE MADE BY THE CZOCHRALSKI METHOD. TO SOME EXTENT, THE REASON FOR THIS IS INERTIA. CZOCHRALSKI PRODUCTION FACILITIES EXIST WITH KNOWN COSTS AND PREDICTABLE PRODUCT QUALITY. IMPROVEMENTS IN THE PROCESS AS A RESULT OF THE SEMICONDUCTOR INDUSTRY WERE MOST CERTAINLY USED TO ADVANTAGE FOR SOLAR CELL MANUFACTURE.

ON THE OTHER HAND, THE NEWER TECHNOLOGIES - CAST MATERIAL, AMORPHOUS, RIBBONS, ETC. OFFER VERY ATTRACTIVE COST SCENARIOS AND PROJECTIONS. WHY ARE NOT COMPANIES JUMPING INTO THESE NEW TECHNOLOGIES?

IT IS APPARENT THAT, ALTHOUGH GOOD PROGRESS HAS BEEN MADE ON THE MANY ALTERNATIVE METHODS, THE PROGRESS IN ADVANCED CZ HAS ALSO BEEN SIGNIFICANT, AND STILL PRESENTS THE SOLAR SHEET MANUFACTURER WITH A SATISFACTORY RETURN ON INVESTMENT.

INDEED, THE BASIC ADVANCED CZ METHOD I WILL DISCUSS IN THE NEXT FEW MINUTES IS ALREADY IN USE COMMERCIALLY FOR SOLAR CELL PRODUCTION. WITHOUT THE DOE FUNDING THROUGH JPL, THAT MIGHT NOT BE THE CASE. THE JPL-FUNDED WORK IN THE PAST HAS YIELDED SOME VERY POSITIVE RESULTS. EQUIPMENT AND PROCESSES ARE NOW AVAILABLE TO THE MARKET WHICH SATISFY CERTAIN NEEDS OF THE PV MANUFACTURER, WHICH WOULD NOT HAVE OTHERWISE BEEN FILLED.

THE VARIOUS ADVANCED CZ CONTRACTS SUPPORTED THROUGH JPL WERE NOT OF MUCH INTEREST FOR SEMICONDUCTOR APPLICATIONS. THE WORK WOULD NOT HAVE BEEN PERFORMED, EITHER BY THE SEMICONDUCTOR HOUSES OR THE EQUIPMENT MANUFACTURERS.

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WE ARE NOW ENTERING A PERIOD OF DEVELOPMENT WHERE THE SEMICONDUCTOR INDUSTRY WILL MOST DEFINITELY DIVERGE EVEN MORE FROM THE DIRECTION OF THE PHOTOVOLTAIC INDUSTRY. THE SEMICONDUCTOR HOUSES, FOR EXAMPLE, ARE STUDYING INTENSELY SUCH AREAS AS CRYSTAL DEFECT STRUCTURE, OXYGEN CONTROL, AND INTRINSIC GETTERING, AND MAGNETIC CRYSTAL GROWTH FOR STRIATION CONTROL. IT IS DIFFICULT TO FIND MUCH RELATIONSHIP OF THESE STUDIES TO PHOTOVOLTAICS.

THE ANSWERS TO PROBLEMS IN PRODUCING PV SILICON INGOTS WILL COME FROM DEVELOPMENTAL WORK WHICH DIRECTLY ADDRESS THOSE PROBLEMS.

IN THIS BRIEF PRESENTATION, I WILL ATTEMPT TO DESCRIBE THE PROGRESS THAT HAS BEEN MADE OVER THE LAST FEW YEARS, THE PRESENT STATUS, AND THE PROBLEM AREAS THAT NEED FURTHER RESEARCH AND DEVELOPMENT.

## Cost Components

| COMPONENT | ONE CRYSTAL PER CRUCIBLE | 150 KG PER CRUCIBLE |
|---|---|---|
| EQUIPMENT | 18% | 40 |
| FACTORY | 3 | 4 |
| LABOR | 12 | 9 |
| UTILITIES | 5 | 12 |
| MATERIALS (62%) | | (35%) |
| CRUCIBLES | 48 | 14 |
| GRAPHITE | 8 | 12 |
| MISC. | 6 | 9 |
| | 100% | 100% |
| APPROXIMATE ADD-ON COST | $80/KG | $20/KG |

RLL APRIL 21, 1982

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THE SAMICS/IPEG METHODOLOGY DEVELOPED AT JPL (SOLAR ARRAY MANUFACTURING INDUSTRY COSTING STANDARD/INTERIM PRICE ESTIMATION GUIDELINES) DEFINES FIVE COST ELEMENTS:

EQUIPMENT
FACTORY FLOOR SPACE
DIRECT LABOR
UTILITIES
MATERIALS.

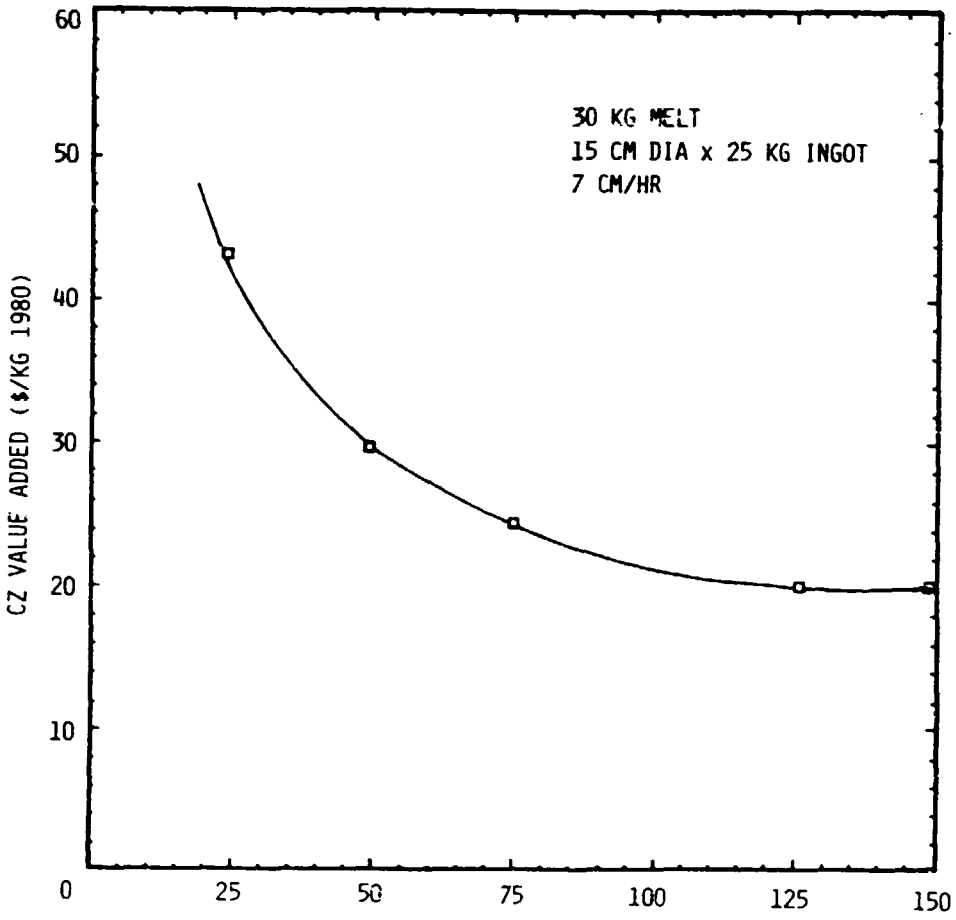
IN THE IPEG PRICE EQUATION, THE PRICE IS PROPORTIONAL TO THE SUM OF THESE ELEMENTS (MULTIPLIED X OVERHEAD) AND, OF COURSE, THE PRICE IS INVERSELY PROPORTIONAL TO THE SPEED AT WHICH THE FACILITY PRODUCES MATERIAL (THE THROUGHPUT). THE INDICATED COST ELEMENTS WERE BELIEVED TO BE THE MOST IMPORTANT.

IT WAS FOUND, BY THE JPL IPEG PRICE CALCULATION, THAT THE STANDARD CZ PROCESS CURRENTLY USED IN THE SEMICONDUCTOR INDUSTRY IS VERY HEAVY ON MATERIAL COST (NOT INCLUDING SILICON). INDEED, 62% IS MATERIAL AND MOST OF THAT IS CRUCIBLE COST ALONE.

IT WAS FURTHER CALCULATED THAT THE CRUCIBLE COST COULD BE REDUCED SIGNIFICANTLY IF ONE COULD GROW LARGE QUANTITIES, UP TO 150 KG, FROM A SINGLE CRUCIBLE. IT IS TO BE EXPECTED, SINCE THE TOTAL MUST BE 100%, THAT A REDUCTION IN THE CRUCIBLE PORTION, WOULD CAUSE OTHER COST ELEMENTS TO INCREASE.

THE NUMBERS INDICATE, HOWEVER, A DRAMATIC INCREASE IN THE COST PORTION ALLOTTED TO EQUIPMENT. ALTHOUGH THE EQUIPMENT REQUIRED TO PRODUCE THE 150 KG IS SUBSTANTIALLY MORE THAN THAT REQUIRED IN THE FIRST COLUMN, IT SHOULD BE NOTED THAT THE ADD-ON COST PER KILOGRAM IN THE SECOND COLUMN IS REDUCED BY A FACTOR OF 4.

# Cz Value Added vs Amount Produced From One Crucible




THE SENSITIVITY OF RUN SIZE UP TO 150 KILOGRAMS IS SHOWN IN THIS SLIDE. IT SHOWS A SUBSTANTIAL REDUCTION IN COST BY THE GROWTH OF TWO OR THREE INGOTS FROM ONE CRUCIBLE. THE COST BENEFIT BECOMES MARGINAL IN THE 100-150 KILOGRAM RANGES.

ALTHOUGH THE CRUCIBLE COST PER KILOGRAM CONTINUES TO DECREASE FOR LARGER RUNS, SOME COSTS INCREASE, SUCH AS EQUIPMENT; THUS, THE TOTAL COST LEVELS OUT.

OTHER COST ELEMENTS, SUCH AS UTILITIES AND LABOR, ARE INDEPENDENT OF RUN LENGTH, AND, THUS, INFLUENCE THE CURVE TO LEVEL OUT.

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JPL AWARDED CONTRACTS TO FOUR COMPANIES IN 1977 AND DIRECTED THOSE COMPANIES TO DEVELOP WAYS OF REDUCING THE COST OF INGOT-GROWN SILICON. SPECIFIC GOALS WERE TO:

(1) GROW LARGE QUANTITIES OF SILICON FROM A SINGLE CRUCIBLE (UP TO 100 KG)

(2) INCREASE GROWTH RATE TO ACHIEVE HIGHER THROUGHPUT

(3) MAINTAIN THE HIGH QUALITY OF CZ-GROWN MATERIAL.

ALTHOUGH THE OBJECTIVES WERE THE SAME, THE APPROACHES DIFFERED.

# Approaches

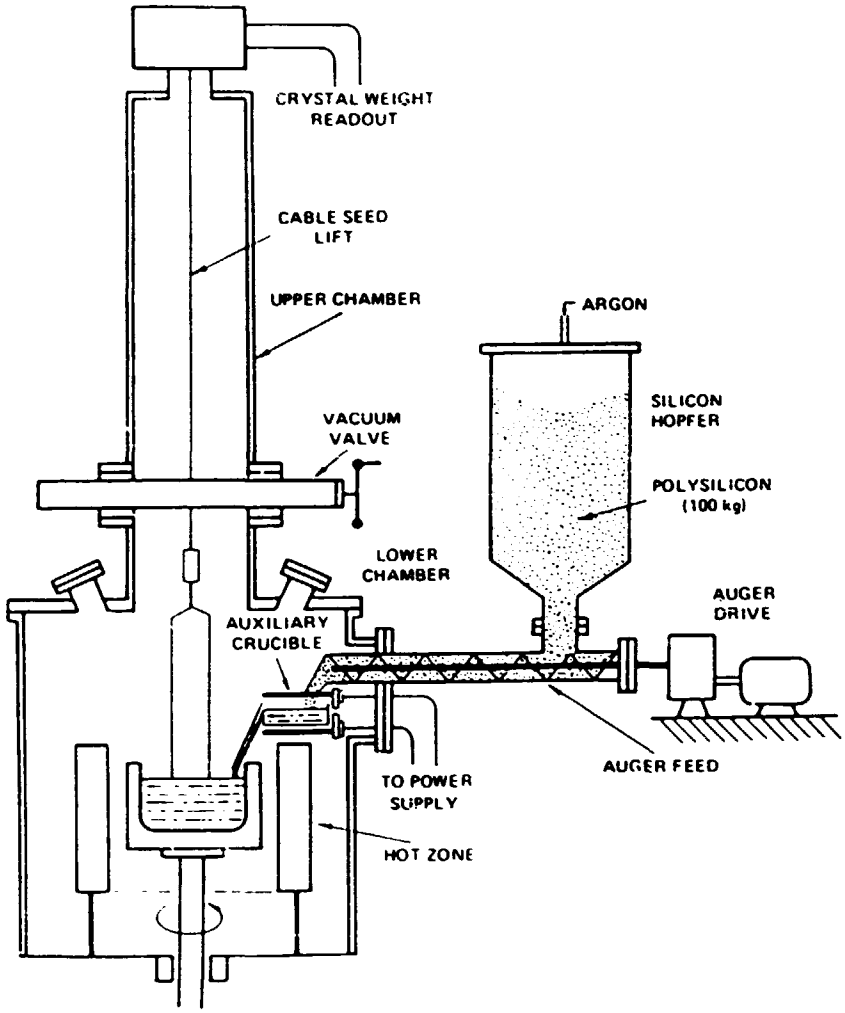
- SILTEC - CONTINUOUS LIQUID FEED FROM SECONDARY MELTING CHAMBER THROUGH A TRANSFER TUBE.
- TEXAS INSTRUMENTS - CONTINUOUS LIQUID FEED. AUGER FEED OF LUMP SILICON TO PREMELTER.
- VARIAN - INTERMITTENT GROWTH/RECHARGE CYCLES. AUGER FEED OF LUMP SILICON FROM HOPPER.
- KAYEX/HAMCO - INTERMITTENT GROWTH/RECHARGE CYCLES USING EITHER POLY-ROD FEED OR DISCHARGE FROM A HOPPER.

SILTEC - DEVELOPED A METHOD OF CONTINUOUS LIQUID FEED FROM A SECONDARY MELTING CHAMBER THROUGH A HEATED TRANSFER TUBE.

TEXAS INSTRUMENTS - ALSO CHOSE TO FEED THE GROWTH CRUCIBLE WITH LIQUID SILICON CONTINUOUSLY DURING GROWTH, BY FEEDING A PREMELTER WITH CHUNK SILICON.

VARIAN AND KAYEX/HAMCO CHOSE TO ADHERE MORE CLOSELY TO THE USUAL GROWTH METHODS, THUS REQUIRING INTERMITTENT RECHARGE AND MELTING CYCLES. THESE TWO CONTRACTORS ONLY DIFFERED IN THEIR MECHANICAL MEANS TO INSERT THE SILICON INTO THE HOT CRUCIBLE.

# Continuous Cz Silicon Furnace (Texas Instruments)




IN THE T.I. SYSTEM, THE AUGER FEEDER INTRODUCED SILICON INTO A SMALL PREMELTER. THE LIQUID SILICON THEN FLOWED INTO THE CRUCIBLE, THEREBY MAINTAINING A CONSTANT MELT LEVEL DURING GROWTH.

IF THE LIQUID TEMPERATURE WERE REASONABLY CONTROLLED, IT WAS EXPECTED THAT THERE WOULD BE NO HARMFUL THERMAL PERTURBATION OF THE MELT. INGOT SIZE WOULD ONLY BE LIMITED BY THE PULL LENGTH OF THE EQUIPMENT.

IT WAS FOUND THAT THE OPERATION OF THE PREMELTER WAS A VERY DIFFICULT TASK, PRIMARILY BECAUSE (1) THE REFRACTORY QUARTZ LINER OF THE PREMELTER DEVITRIFIED RAPIDLY, AND (2) THE UNIFORM FLOW OF SILICON WAS NEARLY IMPOSSIBLE DUE TO THE HIGH SURFACE TENSION OF THE LIQUID. CONSIDERABLE EFFORT WAS MADE ON PREMELTER DESIGN.

ALSO, T.I. REPORTED THAT THE AUGER FEED SYSTEM WORKED POORLY FOR CHUNK SILICON, AS IT TENDED TO CRUSH THE SILICON. CONCERN WAS EXPRESSED THAT THE ABRASIVE NATURE OF SILICON WAS PROBABLY CAUSING CONTAMINATION OF THE SILICON FROM THE STAINLESS STEEL AUGER.

THE T.I. WORK, HOWEVER, WAS SIGNIFICANT, IN THAT IT ILLUSTRATED THE EXTREME DIFFICULTY IN HANDLING BOTH SOLID AND LIQUID SILICON IN A CONTROLLED MANNER WITHOUT CONTAMINATION.

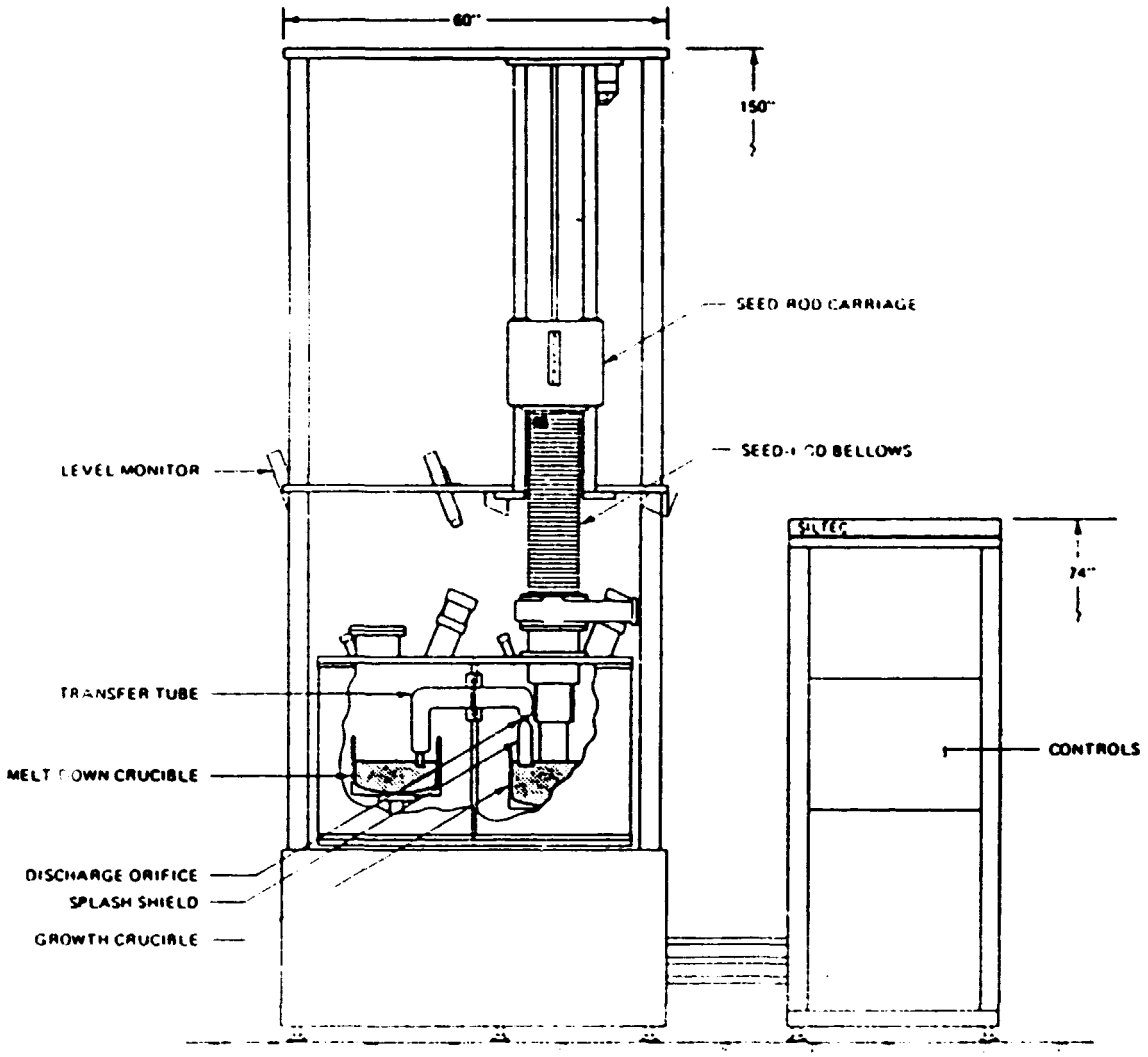
THE VARIAN SYSTEM WAS QUITE SIMILAR TO THE T.I. EXCEPT THAT RECHARGING WAS PERFORMED INTERMITTENTLY BETWEEN INGOT GROWTH CYCLES. THE AUGER SYSTEM WAS ADAPTED TO FEED LUMP SILICON DIRECTLY INTO THE CRUCIBLE.

VARIAN HAD GOOD SUCCESS WITH THIS RECHARGING SYSTEM AND REPORTED SEVERAL FAIRLY LARGE RUNS COMPLETED; UP TO ABOUT 60-70 KILOGRAMS.

PERHAPS ONE OF THE MOST VALUABLE RESULTS FROM THE VARIAN WORK WAS AN EXPERIMENT PERFORMED VERY EARLY IN THE JPL PROGRAM. A 100 KG RUN SIMULATION WAS PERFORMED, BY THE GROWTH OF FIVE 20 KG INGOTS SEQUENTIALLY FROM THE SAME CRUCIBLE AND SAME CHARGE OF SILICON. AFTER EACH INGOT WAS GROWN, IT WAS REMELTED INTO THE CRUCIBLE AND THE CYCLE WAS REPEATED.

THIS EXPERIMENT PROVED THAT THE CRUCIBLE WOULD SURVIVE FOR LONG PERIODS OF TIME THROUGH SEVERE THERMAL CYCLING. IT WAS AN ENCOURAGING RESULT FOR ALL CONTRACTORS.

## Siltec Continuous Liquid-Feed Furnace



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THE SILTEC EFFORT WAS VERY SIGNIFICANT IN THAT IT POINTED OUT A WAY TO OVERCOME OR AVOID THE EFFECT OF THE HIGH SURFACE TENSION ON THE POURING OF SILICON. A SIPHON PRINCIPLE WAS USED. ONCE THE TRANSFER TUBE WAS FILLED WITH LIQUID, UNIFORM AND CONTINUOUS FLOW COULD BE ACHIEVED BY SIMPLY RAISING THE LEVEL OF THE SUPPLY CRUCIBLE.

SIMULTANEOUS CRYSTAL GROWTH AND CONTINUOUS LIQUID FEED REPLENISHMENT WERE DEMONSTRATED. ALSO, THE POSSIBILITY OF GROWING AN EXTREMELY LARGE INGOT FROM A RELATIVELY SMALL CRUCIBLE WAS DEMONSTRATED BY THE GROWTH OF AN INGOT OF OVER 60 KG.

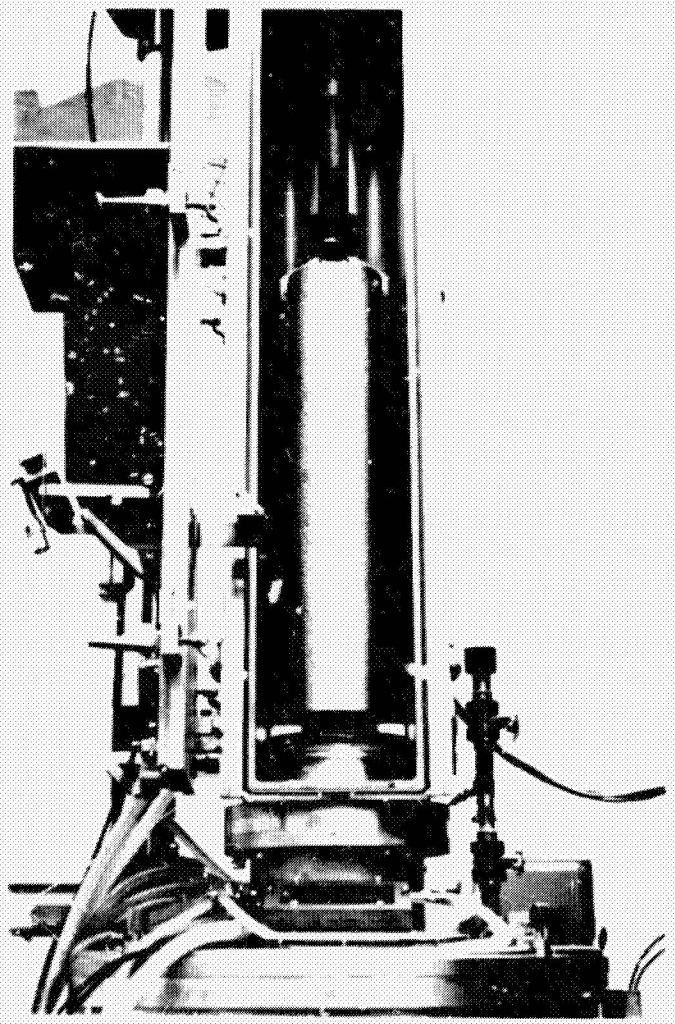
ALTHOUGH THE SILTEC METHOD IS NOT WELL ENOUGH DEVELOPED FOR COMMERCIAL APPLICATION, ITS POTENTIAL FOR LOW COST INGOT GROWTH PROBABLY SURPASSES THE OTHER APPROACHES, PRIMARILY BECAUSE ITS THROUGHPUT SHOULD BE THE HIGHEST.

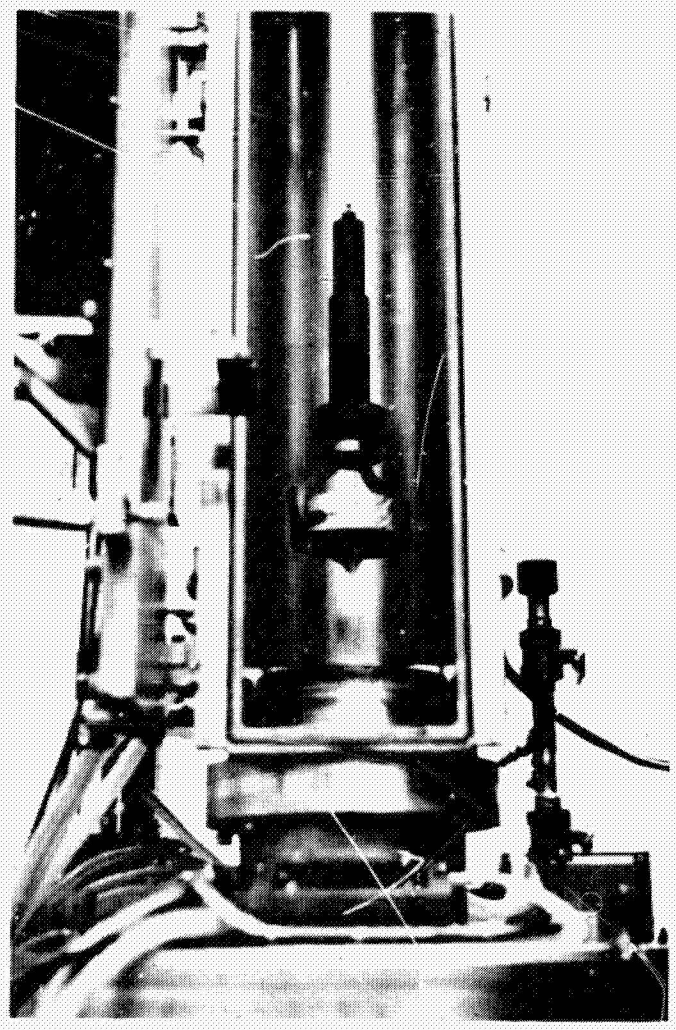
THE DEMONSTRATION OF CONTINUOUS LIQUID FEED WAS AN EXCELLENT TECHNICAL ACCOMPLISHMENT AND MAY VERY WELL HAVE A SIGNIFICANT FUTURE APPLICATION.

KAYEX CORPORATION'S HAMCO DIVISION, A MANUFACTURER OF CRYSTAL GROWERS AND OTHER SILICON PROCESSING EQUIPMENT, HAD PERHAPS THE MOST SIMPLE-MINDED APPROACH OF ALL. RATHER THAN HAVING COMPLICATED RECHARGING OR PREMELTING EQUIPMENT, IT WOULD SIMPLY PROVIDE FOR STORAGE OF A QUANTITY OF SILICA WITHIN THE VACUUM TIGHT GROWTH CHAMBER. THE SILICON FEED STOCK COULD BE EITHER LUMP OR ROD FORM. THE CHAMBER COULD BE RELOADED WITH SILICON FEED STOCK AT THE TIME OF CRYSTAL REMOVAL, THEREBY ELIMINATING A PUMPDOWN/PURGE CYCLE.

ALTHOUGH IT WAS OBVIOUS THAT RECHARGING WITH LUMP SILICON PRECLUDED CONTINUOUS GROWTH AND ALSO LIMITED ULTIMATE THROUGHPUT, THE METHOD DID OFFER A SHORTER-TERM APPROACH TO COMMERCIALIZATION.

IF THE RECHARGE METHOD COULD BE MADE SAFE AND RELIABLE, IT WOULD HAVE A SIGNIFICANT IMPACT ON COST.





IT BECAME APPARENT THAT THE USE OF POLY RODS AS RECHARGE FEEDSTOCK HAD SOME SERIOUS DISADVANTAGES:

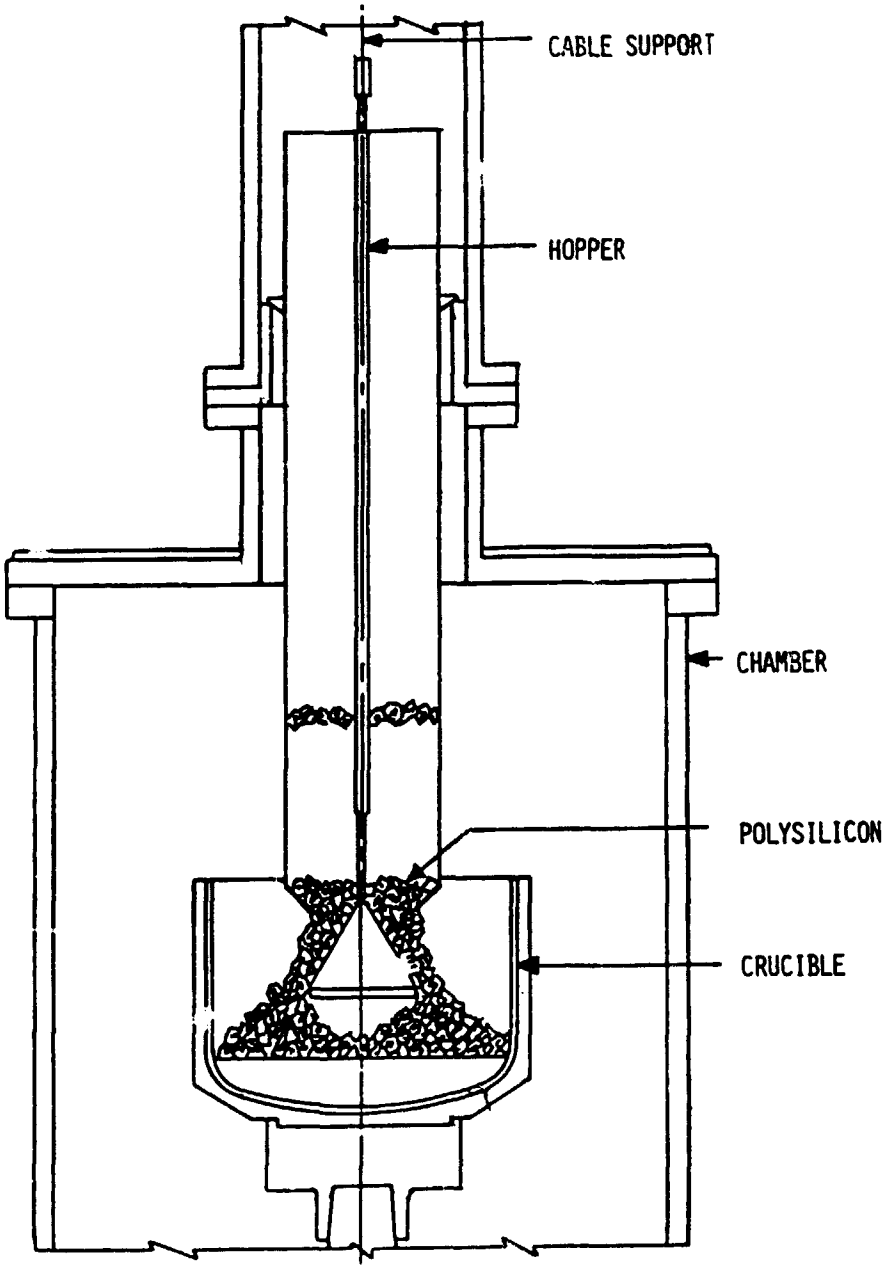
(1) RODS COULD NOT always BE OBTAINED CRACK-FREE; NEITHER COULD THEY BE DEPENDED UPON TO SURVIVE THE THERMAL SHOCK DURING RECHARGING.

(2) SUITABLE RODS WERE MORE EXPENSIVE THAN CHUNK SILICON.

(3) THE MELTING RATE WAS rather SLOW, AND REQUIRED EXCESSIVE HEATING OF THE CRUCIBLE AND MELT

(4) OPERATOR ATTENTION WAS REQUIRED, AND, OF COURSE, RODS COULD NOT BE COMPLETELY MELTED.

## Poly Chunk Recharge System




A SELF-DUMPING HOPPER WAS THEREFORE DESIGNED AND BUILT WHICH HAD ABOUT A 16 KILOGRAM CAPACITY. IT WAS A SIMPLE DESIGN AND QUICKLY BECAME THE PREFERRED METHOD. AS THE HOPPER IS LOWERED, IT COMES TO REST ON A RING AT THE ISOLATION VALVE. THE PLUG SUPPORTING THE CHARGE AT THE BOTTOM OF THE HOPPER CONTINUES TO LOWER AS THE SUPPORTING CABLE IS LOWERED, EFFECTING COMPLETE DISCHARGE OF THE SILICON.

THE HOPPER CAN BE LOWERED AND DISCHARGED IN ABOUT 5 MINUTES. SEVERAL HOPPER-FULLS CAN BE DUMPED DURING A MELTING CYCLE. NO FAILURES HAVE BEEN EXPERIENCED WITH EITHER THE HOPPER OR THE CRUCIBLE DURING MANY RECHARGE CYCLES OVER THE PAST SEVERAL YEARS.

MELT-BACK IS FASTER THAN ROD, NO TIME IS WASTED BY MULTIPLE DUMPS, AND NO CONTAMINATION HAS BEEN DETECTED FROM THE HOPPER. CHUNK SIZE IS LIMITED TO A MAXIMUM DIMENSION OF ABOUT 1 INCH.

## Advanced Cz Objectives

| | INITIAL | LATEST |
|---|---|---|
| RUN SIZE (ONE CRUCIBLE) | 100 KG | 150 KG |
| DIAMETER | 10 CM | 15 CM |
| GROWTH RATE | 10 CM/HR | 10 CM/HR |
| MELT SIZE | 25 KG | 45 KG |
| INGOT SIZE | 5 x 20 KG | 4 x 37.5 KG |
| MELT RATE | - | 25 KG/HR |
| AUTOMATION | ANALOG | MICROPROCESSOR |
| AFTER GROWTH YIELD | 90% | 90% |
| AM-1 EFFICIENCY | 14% | 14% |

AS PROGRESS WAS MADE IN ADVANCED CZ, THE INITIAL OBJECTIVES WERE MODIFIED, AS SHOWN IN THIS SLIDE. 100 KILOGRAM RUN SIZE WAS INCREASED TO 150 KG; DIAMETER INCREASED FROM 10 TO 15 CM. GROWTH RATE GOALS WERE KEPT THE SAME FOR THE HIGHER DIAMETERS. MELT SIZE INCREASED, AND NEW GOALS WERE DEFINED TO IMPROVE THROUGHPUT AND LOWER COSTS, THAT IS, MORE RAPID MELTDOWN, AND MICROPROCESSOR CONTROL TO REDUCE LABOR. YIELDS AND SOLAR EFFICIENCY GOALS HAVE REMAINED CONSTANT FROM THE BEGINNING AT 90% YIELD AND 14% AM-1, RESPECTIVELY.

# Continuous Cz Growth Summary

| DATE | RUN NO. | TOTAL PULLED (KG) | NO. OF INGOTS | DIAMETER (CM) | AVG. PULL RATE (CM/HR) | RUN TIME (HR) | THROUGH-PUT (KG/HR) | MONO-CRYSTAL (%) | AM-1 EFFICIENCY, % RUN AVG | AM-1 EFFICIENCY, % MONO-CRYSTAL | AM-1 EFFICIENCY, % POLY CRYSTAL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 4/78 | 9 | 27 | 3 | 11 | 8.7 | 39 | 0.70 | 85 | 11.5 | 11.6 | 11.4 |
| 6/78 | 11 | 43 | 4 | 11 | 9.1 | 44 | 0.97 | 88 | 11.8 | 11.9 | 11.2 |
| 10/78 | 19 | 57 | 6 | 13 | 8.9 | 64 | 0.89 | 56 | 11.8 | - | - |
| 1/79 | 30 | 99 | 6 | 13 | 8.7 | 79 | 1.25 | 27 | 11.2 | 13.3 | 9.8 |
| 6/79 | 47 | 60 | 5 | 13 | 6.8 | 52 | 1.17 | 88 | 13.0 | 13.0 | - |
| 7/79 | 49 | 108 | 9 | 13 | 7.0 | 86 | 1.26 | 85 | 13.8 | 13.8 | - |
| 10/79 | 55 | 101 | 10 | 13 | 7.2 | 91 | 1.11 | 75 | 12.0 | 13.0 | 9.7 |
| 10/79 | 2 | 100 | 9 | 13 | 7.7 | 109 | 0.92 | 64 | 12.3 | 12.7 | 10.6 |
| 12/79 | 60 | 100 | 8 | 13 | 7.6 | 85 | 1.18 | 61 | 12.0 | 13.0 | 11.0 |
| 1/80 | 62 | 103 | 9 | 13 | 7.9 | 97 | 1.06 | 89 | 12.9 | 13.2 | 11.2 |
| 2/80 | 70 | 152 | 6 | 15 | 6.9 | 99 | 1.53 | 44 | - | - | - |
| 6/81 | 10 | 146 | 5 | 15 | 5.6 | 100 | 1.46 | 52 | 13.7 | 15.3 | 12.3 |

THIS TABLE SUMMARIZES THE RESULTS FROM THE MOST SIGNIFICANT RUNS PERFORMED ON THE PROGRAM.

- THE COLUMN LABELED "TOTAL PULLED" IS THE SUM OF ALL INGOT WEIGHTS FOR A GIVEN RUN. THE GRADUAL INCREASE IN RUN SIZE SINCE THE BEGINNING OF THE PROGRAM IS APPARENT.

- "NO. OF INGOTS" - IN MANY RUNS, WE ATTEMPTED TO PRODUCE AS HIGH A YIELD OF MONOCRYSTAL AS POSSIBLE; THUS, INGOTS WERE OFTEN PREMATURELY ABORTED AND SOME RUNS PRODUCED AS MANY AS 9 OR 10 INGOTS. MORE RECENTLY, WE HAVE TRIED TO PRODUCE THE DESIRED NO. OF INGOTS.

- "DIAMETER" - EARLY RUNS WERE 10 CM. THE DIAMETER WAS INCREASED TO 14 CM WITH NO SIGNIFICANT PROBLEMS.

- "AVERAGE PULL RATE" - THE GOAL OF ALL THE WORK IS 10 CM/HR, REGARDLESS OF DIAMETER. THE PULL RATE (OR GROWTH RATE) IS LIMITED AT PRESENT BY THE TENDENCY OF THE CRYSTAL TO GO OUT OF SHAPE. LARGER CRYSTALS MUST BE GROWN SLOWER, ALTHOUGH THE SOLIDIFICATION RATE (KG/HR) IS STILL LARGER.

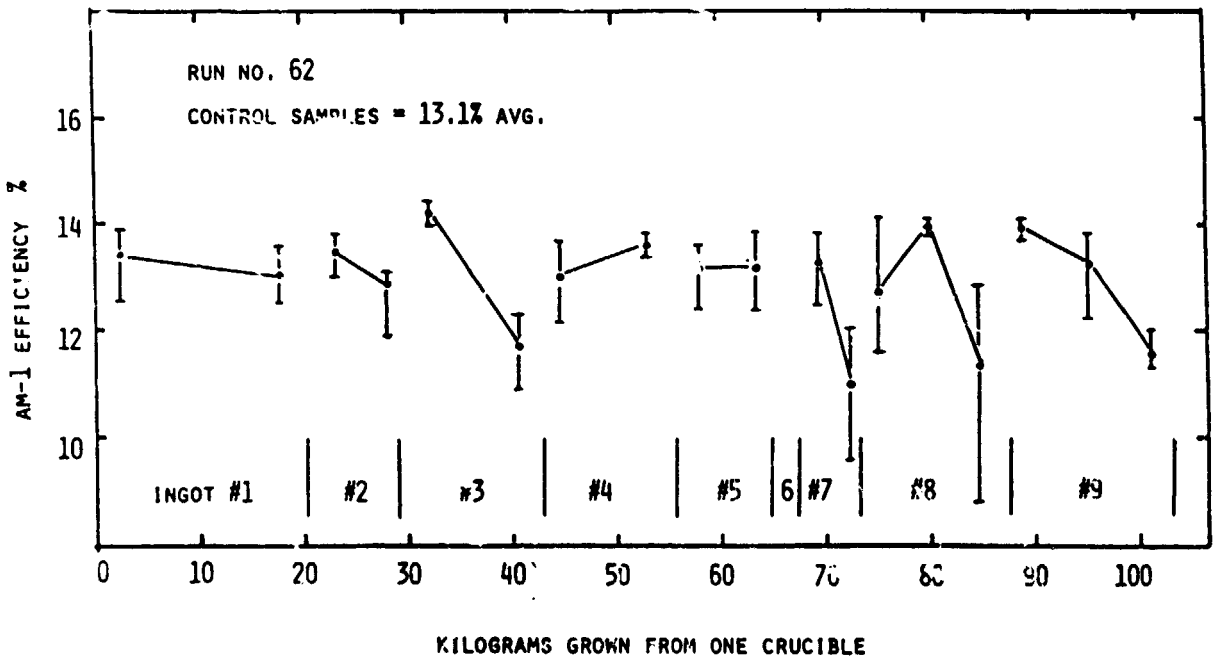
- "RUN TIME" IS TOTAL RUN TIME FROM START UP TO SHUT OFF.

- "THROUGHPUT" IS OBTAINED BY DIVIDING THE TOTAL PULLED BY THE RUN TIME IN HOURS. IT INCREASES, AS SHOWN, WITH DIAMETER. THE LATEST GOAL IS 2.5 KG/HR, WHICH MAY BE OUR MOST DIFFICULT PROBLEM.

- "PERCENT MONOCRYSTAL" - REPRESENTS THE PORTION OF THE PULLED MATERIAL THAT IS MONOCRYSTALLINE IN STRUCTURE BY VISUAL OBSERVATION. 50-80 KG OF MONOCRYSTAL CAN BE GROWN FROM A CRUCIBLE PRESENTLY.

- "AM-1 EFFICIENCY" IS FOR TEST CELLS PREPARED AND MEASURED BY APPLIED SOLAR.

## Solar Efficiency vs kg Grown



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# Run No. 62 (100 kg)

- SAMPLES WERE CUT FROM THE TOP AND BOTTOM OF EACH OF THE SMALLER INGOTS AND THE TOP, CENTER AND BOTTOM OF THE LARGER INGOTS.

- FOUR 2 X 2 CM CELLS WERE MADE FROM EACH SAMPLE.

- THE AVERAGE OF THE FOUR CELLS AND THE SPREAD WAS PLOTTED.

- EFFICIENCY IS PLOTTED ON THE ORDINATE, CUMULATIVE AMOUNT GROWN ON THE ABSCISSA.

- THE POINTS FROM THE SAME INGOT ARE CONNECTED FOR CLARITY.

THE CONCLUSIONS:

- POLYCRYSTALLINE MATERIAL WAS LESS EFFICIENT THAN THE SINGLE CRYSTAL.

- EFFICIENCY VALUES FROM THE SAME SAMPLE VARIED AS MUCH AS 1 OR 2 PERCENTAGE POINTS - PROBABLY DUE TO CELL MANUFACTURE.

- EFFICIENCY OF SINGLE CRYSTAL MATERIAL WAS AS GOOD AS, OR BETTER THAN, THE CONTROL SAMPLES.

- EFFICIENCIES REMAINED CONSTANT RIGHT OUT TO 100 KG.

- AVERAGE OF ALL SAMPLES WAS 12.9%.

C-2

# Run No. 10 (150 kg)

- SAMPLES WERE CUT FROM THE TOP, CENTER, AND BOTTOM OF ALL INGOTS.

- EXCEPT FOR THE FIRST INGOT, THE BOTTOMS OF ALL CRYSTALS WERE POLY, THE TOPS WERE ALL SINGLE.

- EFFICIENCY OF MONOCRYSTAL WAS SURPRISINGLY CONSTANT AND ALL ABOVE 15%, AVERAGING 15.3%.

- EFFICIENCY OF POLY WAS IN THE 11-13% RANGE AND ALSO CONSTANT TO 150 KG, AVERAGING 12.3%.

- THE INCREASE IN EFFICIENCY COMPARED TO THE PREVIOUS RUN IS BELIEVED TO BE A DIFFERENCE IN THE CELL ~~JOB~~ PAR TECHNIQUE, AS THE CONTROL CELLS WERE ALSO HIGHER EFFICIENCIES AND THE CONTROL CELLS MEASURED VERY CLOSE TO THE SINGLE CRYSTAL MATERIAL.

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## Control System: Microprocessor Controls

ONE OF THE GOALS OF THE PROGRAM WAS TO PRODUCE CRYSTAL GROWTH EQUIPMENT WITH MICROPROCESSOR AUTOMATION FOR IMPROVED YIELDS WITH LESS LABOR.

THE PROTOTYPE EQUIPMENT PRODUCED ON THIS WORK WAS RETROFITTED WITH A HAMCO AUTOMATIC GROWER LOGIC SYSTEM. IN A PARALLEL EFFORT, IMPROVED SENSORS WERE DEVELOPED ON THE CONTRACT FOR:

(1) MELT SEEDING TEMPERATURE
(2) DIAMETER AND SHOULDER SENSOR
(3) MELTBACK SENSOR.

ALTHOUGH THE SYSTEM IS STILL NOT AN OPERATOR "HANDS OFF" ONE, THE OPERATOR INPUT HAS BEEN REDUCED, AND WE BELIEVE WE HAVE MADE GOOD PROGRESS TOWARD IMPROVED AUTOMATION.

THE MELT TEMPERATURE AND DIAMETER/SHOULDER SENSORS ARE INTERFACED WITH THE MICROPROCESSOR AND ARE USED ON ALL RUNS. THE MELT LEVEL SENSOR IS SET UP TO MONITOR MELT LEVEL, BUT IS NOT ACTUALLY USED IN CLOSED LOOP CONTROL YET.

PLENARY SESSION: R.L. LANE

## Problems and Concerns

| PROBLEM AREA | APPROACH |
|---|---|
| ● YIELD OF MONOCRYSTAL, LOWER EFFICIENCY OF POLY MATERIAL | ● STUDY STRUCTURE LOSS MECHANISMS, PRIMARILY MELT CONTAMINATION<br>- CRUCIBLE DISSOLUTION<br>- CRUCIBLE DEVITRIFICATION<br>- GAS AMBIENT PURITY & FLOW |
| ● THROUGHPUT AS RELATED TO RATE LIMITING FACTORS<br>- MELTING RATE<br>- CORKSCREWING<br>- STABILIZATION OF MELT TEMPERATURE | ● IMPROVE HOT ZONE DESIGN<br>- FURTHER WORK WITH RADIATION SHIELDING<br>- TEMPERATURE PROFILING OF MELT<br>- IMPROVED TUNING OF MICROPROCESSOR TO SPEED UP STABILIZATION, SEEDING AND NECKING |

IN ATTEMPTING TO EXPRESS PROBLEMS IN SOME MEANINGFUL WAY, I FINALLY CAME UP WITH TWO GENERALIZED PROBLEM AREAS, WHICH ARE LIMITING OUR ABILITY TO ACHIEVE THE PREDETERMINED GOALS; THESE ARE:

(1) YIELD OF MONOCRYSTALLINE MATERIAL, AND

(2) THROUGHPUT.

YIELD - IF THE MATERIAL COULD BE ALL PRODUCED IN MONOCRYSTALLINE FORM, THEN IT IS APPARENT THAT SOLAR EFFICIENCIES WOULD BE HIGHER.

STRUCTURE LOSS IS BELIEVED TO BE CAUSED BY CONTAMINATION OF THE MELT, EITHER BY PARTICLES FROM THE CRUCIBLE OR BY CARBON CONTAMINATION FROM THE CARBON MONOXIDE FURNACE GAS ATMOSPHERE.

THROUGHPUT CAN BE INCREASED BY INCREASING THE SPEED OF THE NON-GROWTH OPERATIONS, AS WELL AS THE GROWTH RATE.

- PRESENTLY, THE MELTING RATE DOES NOT MEET OUR GOALS. BECAUSE A CRYSTAL GROWER IS DESIGNED FOR HIGH THERMAL GRADIENTS AND LARGE HEAT LOSSES ABOVE THE MELT, THE MELTING RATE IS LIMITED AND MUCH HEAT IS LOST. PRELIMINARY EXPERIMENTS SHOW THAT TEMPORARY RADIATION SHIELDING OVER THE CRUCIBLE DURING MELTING WOULD SPEED UP THE MELTING RATE WHILE, AT THE SAME TIME, REDUCE POWER CONSUMPTION.

## PLENARY SESSION: R.L. LANE

- THE GROWTH RATE IS NOT LIMITED BY STRUCTURE LOSS CONSIDERATIONS; RATHER, IT IS LIMITED BY THE TENDENCY OF CRYSTALS TO LOSE CYLINDRICAL SHAPE - WHICH WE CALL CORKSCREWING. A NUMBER OF RUNS WITH A CONE-SHAPED RADIATION SHIELD HAVE GIVEN ENCOURAGING RESULTS; HOWEVER, THE PROBLEM HAS NOT BEEN ELIMINATED. IT MAY BE THAT STEEPER THERMAL GRADIENTS WILL BE REQUIRED TO PREVENT CORKSCREWING AND, THUS, HOT ZONE REDESIGN MAY BE REQUIRED. WE BELIEVE THAT QUANTITATIVE MEASUREMENTS OF MELT TEMPERATURE GRADIENTS AS A FUNCTION OF VARIABLE GROWTH PARAMETERS WOULD LEAD THE WAY TOWARD IMPROVED THERMAL CONDITIONS FOR FASTER GROWTH AND STRAIGHTER CRYSTALS.

- ALTHOUGH THE MICROPROCESSOR IS CAPABLE OF STABILIZING THE MELT REPRODUCIBLY, IT ACTUALLY TAKES MORE TIME PRESENTLY THAN AN EXPERIENCED OPERATOR. MORE SOFTWARE DEVELOPMENT WILL BE REQUIRED TO IMPROVE THIS SITUATION.

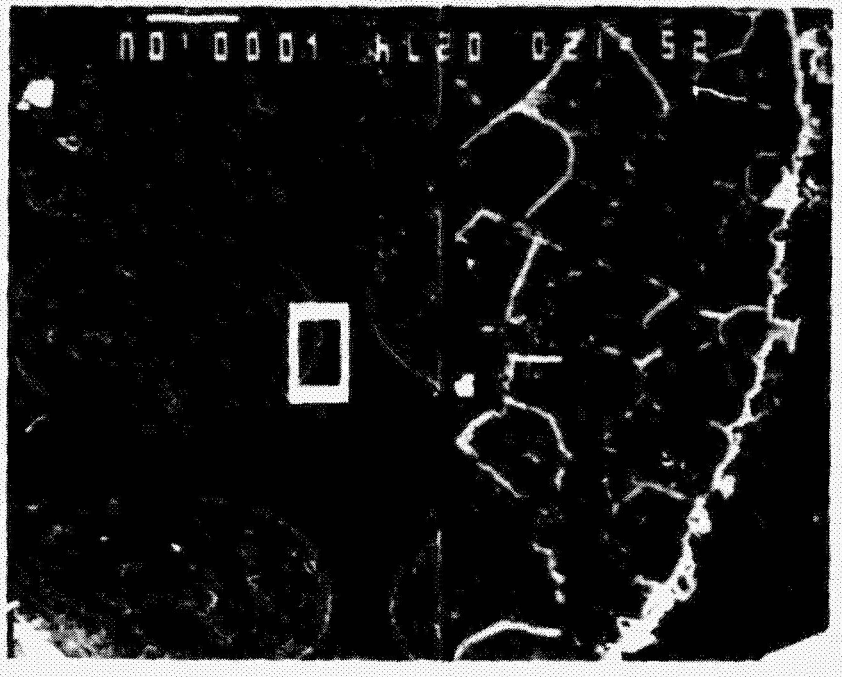
# Devitrification

THIS SLIDE SHOWS THE INSIDE SURFACE OF A TYPICAL CRUCIBLE AFTER ABOUT 100 HOURS OF EXPOSURE TO MOLTEN SILICON. IT IS COVERED WITH "ROSETTES" - APPROXIMATELY 1 MM DIAMETER AREAS OF CRYSTALLIZED $SiO_2$. IT IS CRYSTOBALITE, THE STABLE FORM OF QUARTZ AT THAT TEMPERATURE.

THE ROSETTES TEND TO MULTIPLY, PROBABLY BY SOME FORM OF NUCLEATION, EVENTUALLY COVERING THE COMPLETE INNER SURFACE OF THE CRUCIBLE. IF THEIR RATE OF NUCLEATION AND GROWTH IS EXCEEDET BY their DISSOLUTION rate, COMPLETE COVERAGE MAY NOT OCCUR. IN FACT, IN THE GLASSY AREAS (BLUE), THERE APPEARS TO BE SOME [redacted]OLOGICAL REMAINS OF [redacted], NOW TOTALLY DISSOLVED.

ALSO, ONE CAN SEE DARK SPOTS. THESE ARE SIMPLY VOIDS IN THE CRUCIBLE (BUBBLES) FORMED DURING ITS MANUFACTURE, WHICH HAVE BECOME FILLED WITH SILICON.

DEVITRIFICATION AND EXPOSED BUBBLES ARE OF CONSIDERABLE CONCERN BECAUSE THEY ARE POTENTIAL SOURCES OF $S_iO_2$ PARTICULATE MATTER, WHICH, IF IT ENTERS THE CRYSTAL GROWTH INTERFACE, WILL CAUSE STRUCTURE LOSS.



A CLOSER LOOK AT THESE DEVITRIFICATION ROSETTES WITH THE SCANNING ELECTRON MICROSCOPE IS SHOWN IN THIS SLIDE. THERE ARE A NUMBER OF OBSERVATIONS THAT CAN BE MADE:

(1) THEY ARE FRACTURED AND FLAKING OFF, INDICATING THEIR DIFFERENCE IN STRUCTURE FROM THE GLASSY SURROUNDING AREAS.

(2) THE ROSETTES ARE ROUND, INDICATING THAT THEY "GROW" OUT FROM A CENTER.

(3) THE CENTER NUCLEUS HAS MANY SMALL BUBBLES SURROUNDING A RELATIVELY CLEAN AREA.

(4) A MAGNIFIED AREA AT THE EDGE SHOWS THAT THE EDGE APPEARS TO BE LIFTED FROM THE BULK MATERIAL, AND THE ROSETTE APPEARS TO HAVE GROWN INTO THE BULK AS WELL AS LATERALLY.

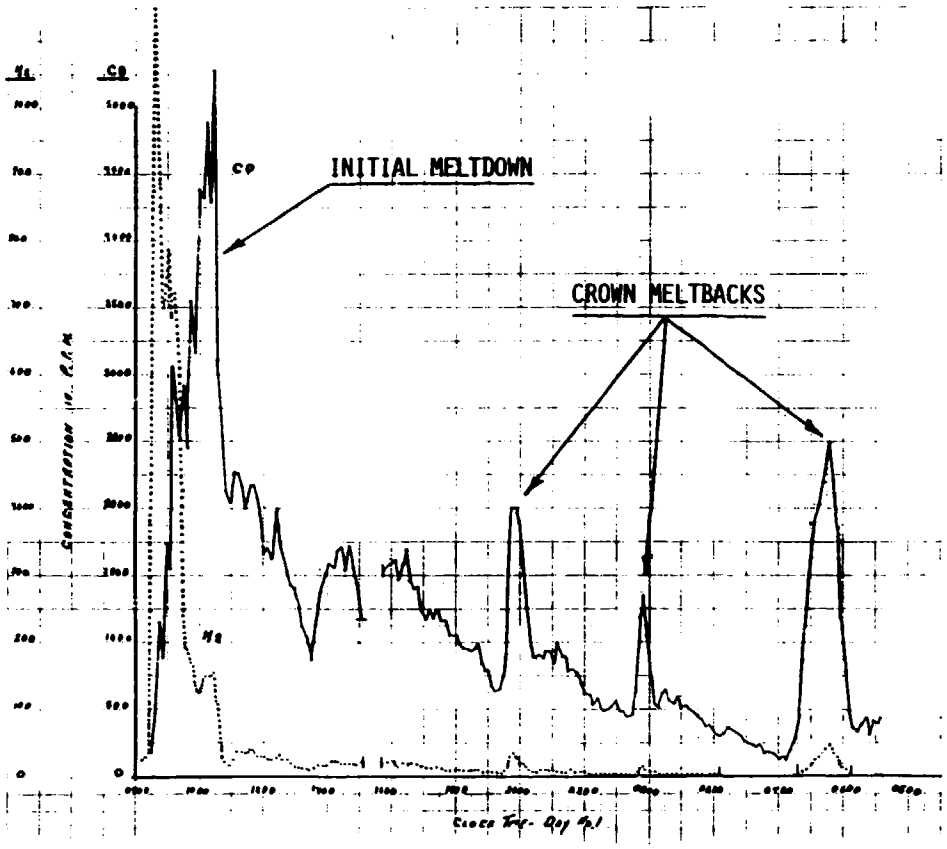
(5) ENERGY DISPERSIVE X-RAY ANALYSIS SHOWS ONLY SILICON WITH NO DETECTABLE IMPURITIES IN (A) THE BULK $SiO_2$ AND (B) THE INTERNAL PORTIONS OF THE ROSETTES. HOWEVER, RIGHT AT THE EDGE, SURPRISINGLY HIGH CONCENTRATIONS OF METAL ARE DETECTED.

EXPRESSED AS ATOMIC PERCENT:

| | |
|---|---|
| Si | 89.92 |
| S | 1.78 |
| Cl | 5.01 |
| K | 2.15 |
| Ca | 1.14 |

WE BELIEVE THAT THIS DEVITRIFICATION IS ASSOCIATED WITH LOSS OF MONOCRYSTALLINITY. WE ALSO BELIEVE THAT IT IS ACCELERATED BY IMPURITIES IN CRUCIBLE, MELT, ON FURNACE.

## CO and $H_2$ vs Run Time




IN AN ATTEMPT TO UNDERSTAND THE MECHANISM OF STRUCTURE LOSS, WE HAVE CONSTRUCTED A DEVICE WHICH SAMPLES AND ANALYZES THE GROWER EXHAUST GAS FOR CARBON MONOXIDE, HYDROGEN, AND WATER. IF CARBON IS CONTAMINATING THE MELT, IT WILL BE CONCENTRATED IN THE RESIDUAL MELT AND COULD LEAD SUBSEQUENTLY TO SILICON CARBIDE PRECIPITATION, AS MORE AND MORE CRYSTALS ARE GROWN.

CARBON MONOXIDE HAS BEEN FOUND IN SURPRISINGLY HIGH CONCENTRATIONS, AND IS A FUNCTION OF TEMPERATURE.

DESCRIBE PLOT -

- SCALES, ORDINATE & ABSCISSA
- CO AND $H_2$
- MELTBACKS VS. RECHARGE
- TREND DOWNWARD WITH TIME

APPROXIMATELY 5000 PPM ARE SEEN DURING MELTDOWN.

$H_2$ EVOLUTION AND WATER (NOT SHOWN) REACT LIKE TYPICAL OUTGASSING.

# Advanced Cz Status

GOALS ACHIEVED

150 KG FROM ONE CRUCIBLE (5 x 30 KG INGOTS)
15 CM DIAMETER x 37.5 KG GROWTH DEMONSTRATED
MICROPROCESSOR CONTROLS WITH IMPROVED SENSORS
PROTOTYPE EQUIPMENT TRANSFERRABLE TO INDUSTRY
AFTER-GROWTH YIELD - 90% OF MELT PULLED
14% AM-1 IN MONOCRYSTAL

| GOALS NOT ACHIEVED | DEMONSTRATED |
|---|---|
| THROUGHPUT - 2.5 KG/HR | 1.5 KG/HR FOR 150 KG RUN |
| YIELD OF MONOCRYSTAL - 90% | 50% MONO 150 KG |
| RECHARGE MELTING RATE - 25 KG/HR | 14 KG/HR |

TO SUMMARIZE THE STATUS, WE HAVE MADE A LARGE NUMBER OF MULTIPLE INGOT RUNS WITH RUN SIZE UP TO 150 KILOGRAMS. THE RECHARGE PROCEDURE IS RELIABLE FROM A PRODUCTION STANDPOINT.

- 30 KG INGOTS HAVE BEEN PRODUCED IN 150 KG RUNS, AND THE FEASIBILITY OF EVEN LARGER INGOTS HAS BEEN ESTABLISHED ON THE JPL FACILITY.

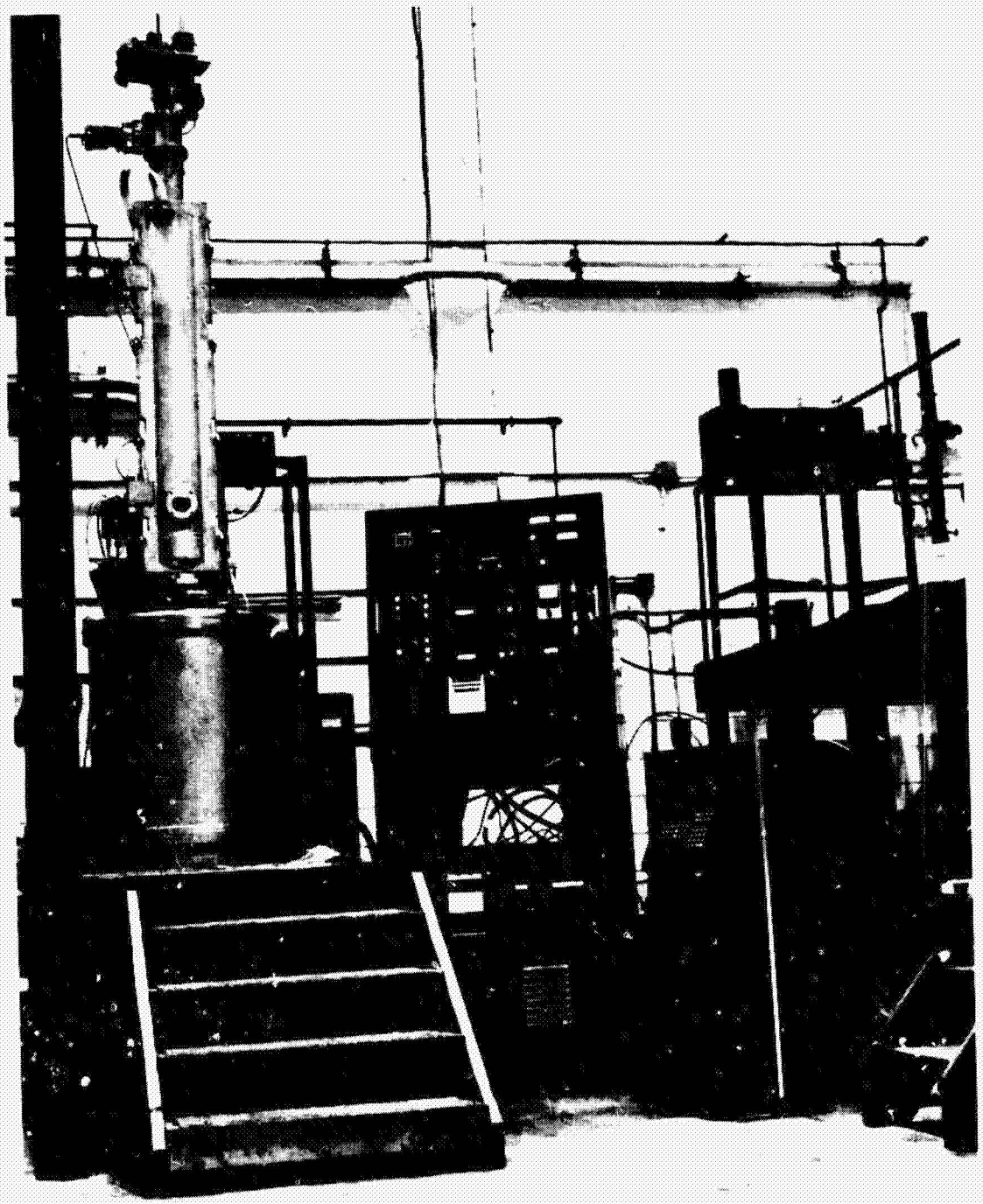
- THE MICROPROCESSOR SYSTEM REQUIRES MORE SOFTWARE DEVELOPMENT, BUT HAS THE POTENTIAL TO LOWER LABOR COST AND IMPROVE PERFORMANCE.

- KAYEX RECENTLY HAS INTRODUCED THE HAMCO CG6000 CRYSTAL GROWER WITH A CHARGE CAPACITY IN THE 50-60 KILOGRAM RANGE, WHOSE DESIGN IS BASED UPON THE JPL PROTOTYPE. THUS, THE LATEST TECHNOLOGY IS NOW AVAILABLE TO THE INDUSTRY.

- CERTAIN IMPORTANT GOALS WERE NOT COMPLETELY ACHIEVED.

- SELECTIVE R & D PROGRAMS, AS SUGGESTED, WOULD ADDRESS THESE GOALS:
  THROUGHPUT - RADIATION SHIELDING, TEMPERATURE PROFILING.
  YIELD - CRUCIBLE AND GAS ANALYSES.
  RECHARGE MELTING RATE - TEMPORARY RADIATION SHIELDING DURING MELTING.

# CG6000



THIS IS A PHOTOGRAPH OF THE FIRST CG6000 WHEN IT WAS BEING ASSEMBLED. THIS MACHINE WAS PURCHASED SPECIFICALLY FOR SOLAR INGOT PRODUCTION USING THE JPL-DEVELOPED PERIODIC RECHARGE METHOD. IT IS PLANNED TO PRODUCE 60 TO 80 KILOGRAMS FROM EACH CRUCIBLE. ALTHOUGH THIS IS NOT THE QUANTITY DEEMED TO BE THE MOST COST EFFECTIVE, IT IS CERTAINLY A MAJOR STEP TOWARD LOWER COST SOLAR GRADL MATERIAL.

I WOULD LIKE TO SUMMARIZE BY EXPLAINING OUR POSITION WITH RESPECT TO GROWTH EQUIPMENT.

- FIRST, THE CZ PROCESS, WITHOUT QUESTION, IS CAPABLE OF PRODUCING CONSISTENTLY THE HIGHEST EFFICIENCY PHOTOVOLTAIC CELLS.
- HIGH EFFICIENCY IS A VERY IMPORTANT INGREDIENT TO LOW COST, BACAUSE LESS OF ALL OTHER MATERIALS ARE REQUIRED TO PRODUCE A KILOWATT OF POWER - LESS GLASS, LESS STRUCTURE, LESS SILICON, EVEN LESS REAL ESTATE.
- SECONDLY, IT IS FAST. EVEN AT 1.4 KG/HR, IT IS EQUIVALENT TO OVER 1 $M^2$/HR FOR ONE PULLER, WHICH IS ABOUT 100 WATTS OF GENERATING CAPACITY PER HOUR. IT TAKES 6 TO 7 OF THE FASTEST RIBBON PULLERS TO KEEP UP WITH ONE CZOCHRALSKI PULLER IN TERMS OF SQUARE METERS PER HOUR.
- AS A THIRD POINT NOT MENTIONED PREVIOUSLY, I WOULD LIKE TO SUGGEST CZOCHRALSKI AS AN ALTERNATIVE TO CASTING. LARGE DIAMETER INGOTS (IN THE 12" RANGE) HAVE BEEN GROWN IN THE KAYEX TECHNOLOGY CENTER AT A RATE OF 6 KG/HR. CERTAINLY, SEVERAL OF THESE INGOTS COULD BE PRODUCED FROM THE SAME CRUCIBLE. THERMAL SHOCK CRACKING AND CRUCIBLE OR MOLD PROBLEMS WOULD BE ELIMINATED. THE INGOTS WOULD BE CROPPED, SECTIONED AND SLICED JUST LIKE CAST INGOTS.

YOU MAY ASK, "WHY DOESN'T INDUSTRY GO AHEAD AND INVEST DOLLARS IN THESE IDEAS IF THEY ARE SO GOOD?"

SMALL EQUIPMENT MANUFACTURERS LIKE KAYEX ARE DRIVEN BY THE CUSTOMERS AND THE MARKETPLACE. 90% OF OUR PROJECTED CRYSTAL GROWER BUSINESS FOR THE FORESEEABLE FUTURE WILL COME FROM THE SEMICONDUCTOR INDUSTRY OUR CORPORATE R AND D WILL BE DIRECTED TOWARD THAT MARKET - WE WILL BE CONCERNING OURSELVES AND OUR DESIGNS WITH, FOR EXAMPLE:

- OXYGEN CONTROL AND PRECIPITATION
- MICROSCOPIC AND MACROSCOPIC UNIFORMITY
- THERMAL GRADIENTS IN THE GROWING CRYSTAL AND IN THE MELT
- MAGNETIC CZ GROWTH
- AUTOMATION

TO THE EXTENT THESE DEVELOPMENT PROJECTS ARE USEFUL TO PHOTOVOLTAICS, WE WILL APPLY THEIR RESULTS; HOWEVER, IT WILL BE OF LIMITED USE.

IT TAKES 2-3 YEARS TO BRING A CONCEPT FROM THE LABORATORY TO THE MARKETPLACE. WE ARE SEEING SOME OF THE RESULTS OF OUR PREVIOUS WORK BEING USED NOW.

THE MOMENTUM OF DEVELOPMENT OF PHOTOVOLTAIC CZ MATERIALS AND EQUIPMENT MUST BE KEPT UP IF REAL COMMERCIAL PROGRESS IS TO BE MADE IN FUTURE YEARS.