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**A NEW METHOD OF METALLIZATION FOR
SILICON SOLAR CELLS.**

(NASA-CR-162823) A NEW METHOD OF
METALLIZATION FOR SILICON SOLAR CELLS Final
Report, Dec. 1978 - Sep. 1979 (SOL/LOS,
Inc., Los Angeles, Calif.) 67 p
HC A04/MF A01

N80-19620

Unclas
47471

CSSL 10A G3/44

FINAL REPORT.

DECEMBER 1978 TO SEPTEMBER 1979

DECEMBER 1979.

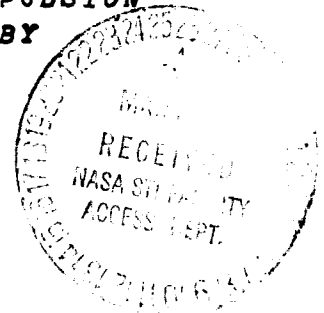
BY

DR. MILO MACHA

JPL CONTRACT No. 955318

**SOL/LOS INCORPORATED
2231 S. CARMELINA AVENUE
LOS ANGELES, CALIFORNIA 90064**

**" THE JPL LOW-COST SILICON SOLAR ARRAY PROJECT IS SPONSORED
BY THE UNITED STATES 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
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I. ABSTRACT.

THE NEW METALLIZATION PROCESS BASED ON Mo-Sn SYSTEM IS THE SUBJECT OF THIS NINE MONTHS PROGRAM.

MoO₃ IS USED AS THE SOURCE OF Mo, SINCE ITS RELATIVELY LOW MELTING POINT AND EASE OF REDUCTION TO METALLIC MOLYBDENUM.

THE TASKS PERFORMED DURING THIS STUDY INCLUDE:

1. ESTABLISHING THE REDUCTION CYCLE FOR MoO₃
2. DETERMINING THE REACTION MECHANISM FOR MoO₃-Sn MIXTURE
3. ESTABLISHING THE RATIO OF MoO₃-Sn FOR THE INK COMPOSITION
4. FORMULATION OF SCREENABLE INK
5. EVALUATION OF PHOTOVOLTAIC CELLS METALLIZED WITH THE INK
6. COMPARISON OF THE Mo-Sn METALLIZATION WITH NICKEL PLATED AND SILK SCREENED SILVER CONTACTS
7. ENVIRONMENTAL TEST OF METALLIZED CELLS
8. METALLIZATION OF N/P CELLS WITH BSF AND COMPARISON WITH CELLS METALLIZED WITH EVAPORATED Ti-Ag CONTACT
9. COST ANALYSIS OF THE PROCESS

THE FIRST PART OF THE CONTRACT, I.E. THE REACTION MECHANISM STUDY OF MoO₃ AND ITS MIXTURE WITH Sn WAS CONDUCTED IN AN EXPERIMENTAL STATION CONSISTING OF A GRAPHITE

STRIP-HEATER AND A PYREX BELLJAR, UNDER CLOSE CONTROL OF TEMPERATURE-ATMOSPHERE-TIME, WHILE ALLOWING VISUAL OBSERVATIONS OF THE REACTIONS.

THE METALLIZATION OF THE CELLS WAS DONE IN A DIFFUSION TUBE FURNACE.

IN ORDER TO OBTAIN A LOW OHMIC CONTACT TO THE CELL, THE BASIC INK COMPOSITION WAS MODIFIED WITH A SMALL ADDITION OF TITANIUM IN THE FORM OF TITANIUM RESINATE.

THE ELECTRICAL CHARACTERISTICS OF THE CELLS WERE COMPARABLE WITH THE EXISTING METALLIZATION PROCESSES.

THE FIRING CYCLE STILL HAS TO BE OPTIMIZED FOR THE PROCESS USED IN THE CONTINUOUS CONVEYOR BELT FURNACE, ESPECIALLY IN THE COOLING CYCLE AROUND 450°C, WHICH INDICATED A SIGNIFICANT EFFECT ON THE ELECTRICAL CHARACTERISTICS.

THE COST ANALYSIS OF THE PROCESS WAS BASED ON PROJECTED PRODUCTION OUTPUT OF ONE MEGAWATT PER YEAR, USING 2" DIAM. SILICON CRYSTAL WAFERS AND THE CURRENT MATERIAL COSTS. THEREFORE THE CALCULATED COST DEVIATES FROM THE PROJECTED PRICE GOAL SET UP FOR THE YEAR 1986.

IN COMPARISON WITH THE STANDARD PROCESSES USING SILVER AS THE CONTACTING METAL, THE SAVING OBTAINED BY THE USE OF THIS NEW PROCESS IS A DIRECT RESULT OF THE PRICE DIFFERENCE BETWEEN SILVER AND MOLYBDENUM OXIDE WITH TIN.

II. INTRODUCTION

METALS FOR OHMIC CONTACT TO SILICON SOLAR CELLS ARE SELECTED ON BASIS OF THEIR ELECTRICAL CONDUCTIVITY, THERMAL EXPANSION COEFFICIENT, ENVIRONMENTAL STABILITY AND COST.

TABLE I SHOWS THE PERTINENT PROPERTIES OF METALS. THIS TABLE SHOWS THAT MOLYBDENUM IS MOST DESIRABLE WITH RESPECT TO THE STATED REQUIREMENTS. IT HAS THE CLOSEST MATCH OF THERMAL EXPANSION COEFFICIENT WITH SILICON FROM ALL METALS AND IT IS AMONG TEN OF THE HIGHEST CONDUCTIVE METALS, ONLY SECOND TO COPPER AMONG THE COMMON METALS. FOR THIS REASON MOLYBDENUM IS USED IN SILICON DEVICE TECHNOLOGY AS A STRUCTURAL ELEMENT IN THE DEVICE AND CIRCUIT ASSEMBLY. IT IS USED AS A MECHANICAL SUPPORT AS WELL AS A HEAT SINK. AS A CONTACTING ELEMENT HOWEVER MOLYBDENUM IS USED VERY RARELY AND THE REASON FOR THIS IS THAT ITS HIGH MELTING AND BOILING POINT MAKE THE DEPOSITION OF THE FILM BY EVAPORATION OR SPUTTERING RATHER DIFFICULT AND FILM FORMATION BY PYROLYTIC DECOMPOSITION OF CARBONYLS OR HALIDE COMPOUNDS REQUIRES COMPLEX EQUIPMENT AND PROCESSING STEPS.

THE FORMATION OF MOLYBDENUM FILMS BY A SIMPLE AND ECONOMIC PROCESS IS THE SUBJECT OF THIS PROGRAM. IT IS BASED ON THE CONVERSION OF MOLYBDENUM TRIOXIDE (MoO_3) INTO METALLIC MOLYBDENUM IN A REDUCING ATMOSPHERE AT

TABLE I

PROPERTIES OF METALS

	ELECTRICAL RESISTIVITY (μ OHMS-CM)	COEF. OF THERMAL EXPANSION (CM/CM/ $^{\circ}$ C). 10^{-6}	MELT. P. ($^{\circ}$ C)	BOIL. P. ($^{\circ}$ C)	DENSITY (G/CM 3)
TITANIUM	176	7.1	1725 $^{\pm}$ 10		4.5
NICKEL	65.3	9.2	1455	3075	8.9
LEAD	20.6	16.3	327	1740	11.3
PLATINUM	14.9	4.9	1774	4530	21.5
TANTALUM	12.4	4.0	2996	4100	16.6
TIN	11.5	13	232	2260	7.3
PALLADIUM	10.8	6.5	1555	3980	12.0
ALUMINUM	6.3	13.7	660	1800	2.82
ZINC	6.1	19.3	419	904	7.17
TUNGSTEN	5.48	2.2	3410	5900	19.4
MOLYBDENUM	5.17	3.1	2622	4570	10.2
BERYLLIUM	5	6.4	1292 $^{\pm}$ 8	2980	1.84
RHODIUM	4.51	4.6	1966		12.4
GOLD	2.35	7.9	1065	2700	19.3
COPPER	2.03	9.8	1083	2595	8.95
SILVER	1.6	10.9	960	2000	10.5
SILICON		4.2	1420		2.33

ELEVATED TEMPERATURES. MOLYBDENUM TRIOXIDE (MoO_3) IS THE MOST STABLE OXIDATION STATE OF MOLYBDENUM AND HAS AN ADVANTAGEOUS CHARACTERISTIC IN THAT IT HAS A RELATIVELY LOW MELTING POINT OF 795°C AND IS ALSO EASILY REDUCED INTO MOLYBDENUM METAL. WHEN HEATED IN AIR IT STARTS TO SUBLIME ABOVE 550°C , MELTS INTO AN OILY LIQUID AT 795°C , AT WHICH POINT THE SUBLIMATION IS EXCEEDINGLY HEAVY. IN REDUCING ATMOSPHERE, ON THE OTHER HAND, THIS OXIDE REDUCES AT APPROXIMATELY 600°C INTO LOWER OXIDES, MOSTLY MoO_2 , WHICH IS CHARACTERIZED BY A PURPLE COLOR. MoO_2 DOES NOT SUBLIME AND CAN BE ULTIMATELY REDUCED TO MOLYBDENUM METAL ABOVE 600°C , IF KEPT FOR A LONG ENOUGH TIME. WHEN THE TEMPERATURE IS INCREASED TO 795°C OR ABOVE, THE RESIDUAL MoO_3 STILL PRESENT IN THE OXIDE MIXTURE MELTS AND IS CONVERTED INTO A DENSE MOLYBDENUM FILM.

SINCE THE MoO_3 IS COMMONLY AVAILABLE IN A FINE POWDER FORM, IT IS VERY SUITABLE TO USE IN A SUSPENSION ADJUSTED FOR SILK SCREENING PROCESSES.

THE SECOND ELEMENT IN THE PROPOSED METALLIZATION METHOD IS TIN. IT IS SELECTED BECAUSE OF ITS GOOD SOLDERABILITY AT LOW TEMPERATURES AND ALSO BECAUSE IT HAS THE HIGHEST ELECTRICAL CONDUCTIVITY AMONG THE LOW MELTING METALS.

NO CONCLUSIVE INFORMATION IS AVAILABLE ON THE CONSTITUTION OF MOLYBDENUM-TIN SYSTEM.

THERE ARE REFERENCES STATING THAT UPTO 0.13% TIN IN MOLYBDENUM FORMS A SINGLE PHASE. THIS REFERENCE CAN BE FOUND IN THE CONSTITUTION OF BINARY ALLOYS BY F.A.SHUNK, 1969 (SECOND EDITION).

THERE IS EXPERIMENTAL EVIDENCE THAT MOLYBDENUM IS READILY WET BY TIN IN THE ABSENCE OF OXIDE AND THIS REACTION IS MORE PRONOUNCED WHEN IT TAKES PLACE AT THE TIME WHEN MOLYBDENUM IS REDUCED FROM ITS OXIDE. THEREFORE THE AIM IS TO FORMULATE A MOLYBDENUM OXIDE-TIN COMPOSITION, DISPERSE IT IN A SUITABLE VEHICLE FOR SILK SCREENING AND FORM THE CONTACT ON THE SOLAR CELL SURFACE BY FIRING THIS MIXTURE IN REDUCING ATMOSPHERE.

FORMING GAS IS USED FOR SAFETY REASONS.

THE PROJECT IS PLANNED IN THREE PHASES.

1. THE DETERMINATION OF THE CYCLE FOR CONVERSION OF MoO_3 INTO Mo AND THE RATIO OF $MoO_3:Sn$.
THIS PHASE IS DONE IN AN EXPERIMENTAL STATION, ESPECIALLY CONSTRUCTED FOR THIS PURPOSE.
2. THE FORMULATION OF SCREENABLE INK AND THE EVALUATION OF THE CONTACT ON SOLAR CELLS. THIS WORK IS CONDUCTED IN A TUBE FURNACE CALIBRATED TO PARAMETERS ESTABLISHED EXPERIMENTALLY IN THE FIRST PHASE.
3. THE SPECIFICATION OF THE PROCESS.

III. TECHNICAL DISCUSSION

THE FIRST QUARTER OF THE PROGRAM WAS CONCERNED WITH ESTABLISHING OF THE TEMPERATURE-TIME-ATMOSPHERE CYCLE REQUIRED FOR THE REDUCTION OF MOLYBDENUM TRIOXIDE INTO MOLYBDENUM AND ALSO DETERMINATION OF MOLYBDENUM TRIOXIDE-TIN RATIO TO OBTAIN SOLDERABLE AND ADHERENT COATINGS. A SPECIAL STATION WAS BUILT FOR THIS WORK.

III. 1. CONSTRUCTION OF EXPERIMENTAL STATION.

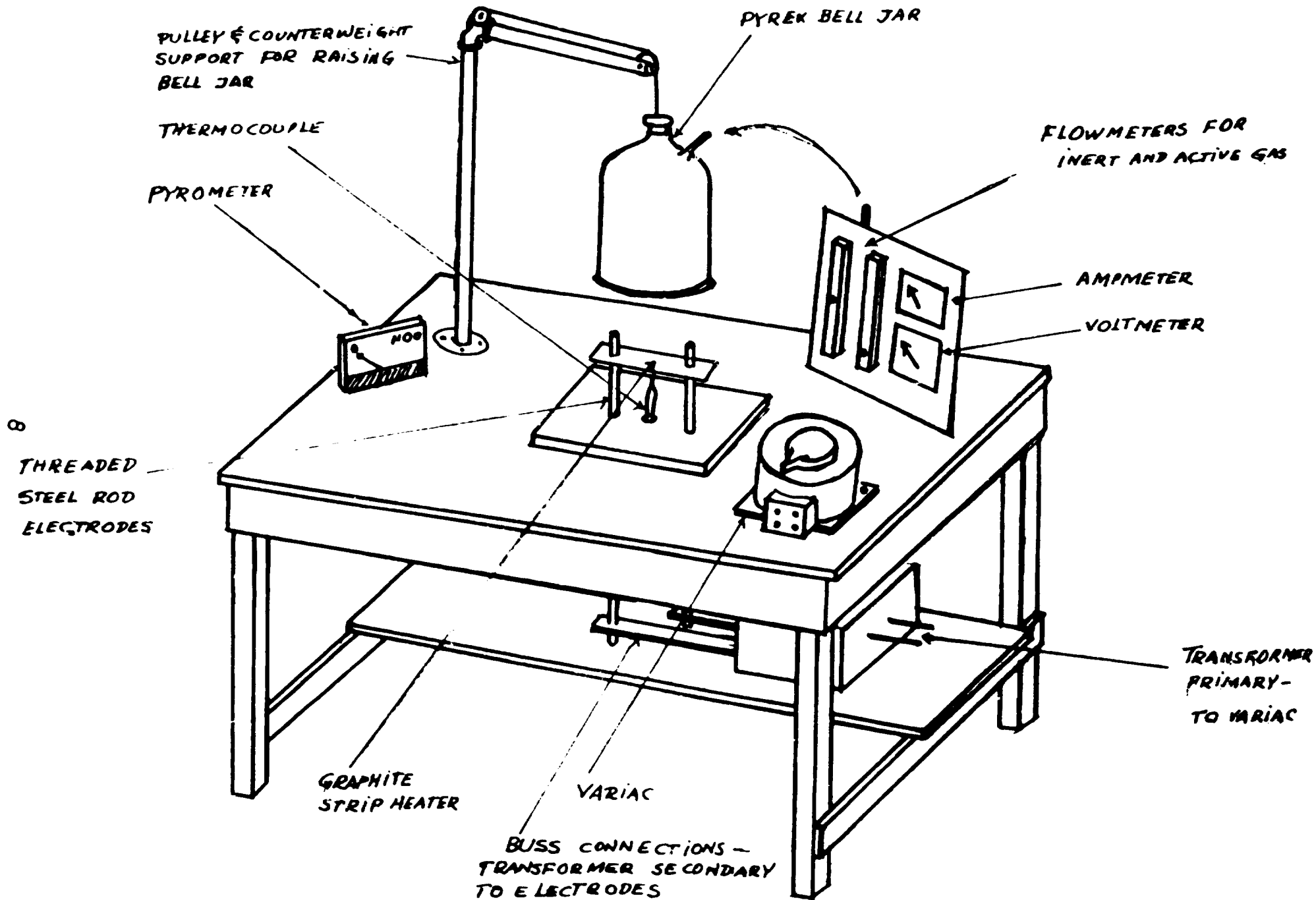
THE PURPOSE OF THIS STATION IS TO ALLOW FOR VISUAL OBSERVATION OF REACTIONS TAKING PLACE UNDER VARIOUS TEMPERATURE-TIME-ATMOSPHERE HEATING CYCLES AND TO PERFORM INDIVIDUAL EXPERIMENTS IN A SHORT TIME. THE STATION SHOWN IN THE ATTACHED SKETCH CONSISTS OF A GRAPHITE STRIP HEATER, LOCATED UNDER A PYREX BELLJAR WITH AN INLET FOR GASFLOW. THE GRAPHITE STRIP IS HEATED BY MEANS OF AC CURRENT AND DESIGNED TO GENERATE 1000°C. THE TEMPERATURE OF THE STRIP IS MONITORED BY A CHROMEL-ALUMEL THERMOCOUPLE AND DISPLAYED BY A PYROMETER. THE ATMOSPHERE IN THE BELLJAR IS CONTROLLED BY FLOWMETERS AND VALVES TO PROVIDE A DESIRABLE INERT OR REACTIVE GASEOUS ENVIRONMENT.

THE HEATER IS CONSTRUCTED FROM A HIGH PURITY POCO GRAPHITE

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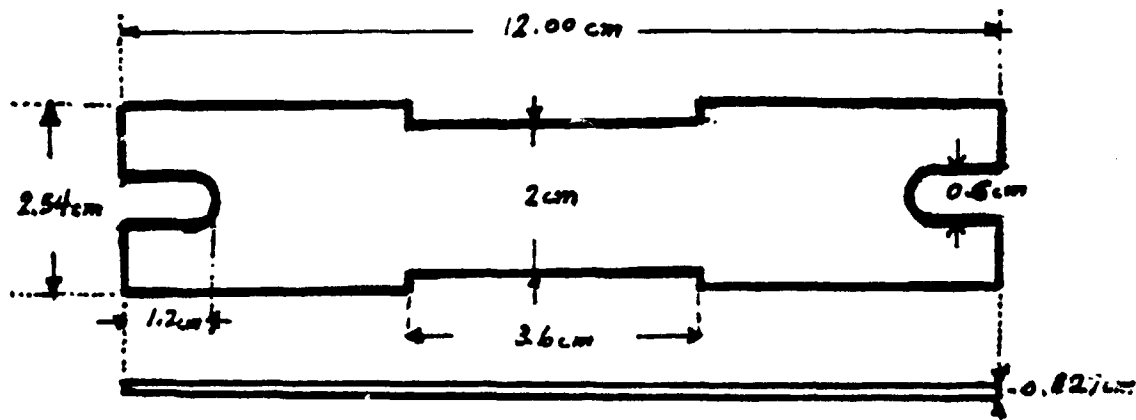


CONTROLLED ATMOSPHERE EXPERIMENTAL STRIP HEATER

THE DENSITY OF THIS MATERIAL IS 1.84g/cm^3 AND RESISTIVITY $1.27 \times 10^{-3} \Omega\text{-cm}$.

THE SUPPLIED MATERIAL IS 4" (10.16CM) WIDE, 6" (15.24CM) LONG AND .050" (0.127CM) THICK.

THE HEATING ELEMENT IS SHAPED FROM THIS PLATE AND HAS DIMENSIONS OF 1" (2.54CM) X 4.72" (12CM) WITH SLOTS ON EACH NARROW END FOR JUNCTING TO THE ELECTRODES TERMINALS (SEE DRAWING).



THE ELECTRICAL ENERGY FOR THE HEATER IS SUPPLIED BY 120VOLTS-20AMPS IN-HOUSE LINE VIA THREE TRANSFORMERS EACH ONE HAVING 208 VOLTS-10.8AMPS RATING ON THE PRIMARY AND 30VOLTS-60AMPS ON THE SECONDARY WINDING.

THE PRIMARIES OF THESE TRANSFORMERS ARE CONNECTED IN SERIES, THE SECONDARIES IN PARALLEL. THE VOLTAGE TO THE PRIMARY IS REGULATED BY A VARIAC.

THE TEMPERATURE OF THE STRIP IS MONITORED BY A CHROMEL-ALUMEL THERMOCOUPLE, GAUGE 28, PURCHASED FROM CALIFORNIA ALLOY Co., EL MONTE, CALIF. AND RECORDED ON A PYROMETER WITH A SCALE FROM 0 - 1100°C, PURCHASED FROM THE SAME COMPANY. THE EXTERNAL RESISTANCE OF THE THERMOCOUPLE IS ADJUSTED TO 10 OHMS IN ORDER TO MATCH THE RESISTANCE OF THE PYROMETER.

THE BELLJAR IS FROM PYREX GLASS, HAS THE DIAMETER OF 8"(20.32 CM) AND HAS A VOLUME OF 12 LITERS.

IT IS SUSPENDED BY A WIRE CORD AND BALANCED BY A COUNTER WEIGHT.

THE FLOWMETERS TO CONTROL THE GAS FLOW ARE MADE BY BROOKS INSTRUMENT DIVISION EMERSON ELECTRIC Co., HATFIELD, PENN. ONE, USED FOR FORMING GAS, HAS A MAXIMUM FLOW-RATE OF 4.7 STD.L/MIN AND THE MINIMUM FLOW-RATE OF 0.1 STD L/MIN. THE OTHER, USED FOR NITROGEN, HAS THE MAXIMUM FLOW-RATE OF .272 STD L/MIN AND A MINIMUM RATE OF .014 STD L/MIN.

III. 2. REDUCTION OF MoO_3 INTO Mo .

MATERIALS USED:

SILICON WAFERS P-TYPE, 1 OHM-CM AND
N-TYPE, 0.1 OHM-CM

MOLYBDENUM OXIDE REAGENT (MALLINCKRODT)

TRICHLOROETHYLENE (LOS ANGELES CHEMICAL Co.)

ETHYLCELLULOSE (DOW CHEMICAL Co.)

THE SILICON WAFERS WERE SCRIBED TO 1 X 1 CM APPROX.

AND ETCH-CLEANED IN 9:1 = HNO_3 :HF.

THE MOLYBDENUM OXIDE SUSPENSION WAS PREPARED BY DISPERSING MoO_3 IN TRICHLOROETHYLENE AND A SMALL AMOUNT OF ETHYLCELLULOSE WAS ADDED TO ADJUST TO A CONSISTENCY OF A PAINT.

NO EXACT RATIO OF INDIVIDUAL INGREDIENTS WAS ESTABLISHED AS THE PURPOSE OF THESE TESTS WAS TO DETERMINE QUALITATIVELY THE REDUCTION OF MoO_3 .

THE SILICON WAFERS WERE COATED ON ONE SIDE WITH THIS SUSPENSION.

THE SAMPLES WERE SUBJECTED TO A HEAT CYCLE TO FORM Mo . THE Mo COATINGS WERE EVALUATED FOR LATERAL RESISTANCE AND BOND STRENGTH. THE RESISTANCE WAS MEASURED BY AN OHM METER AND THE BOND STRENGTH BY A SCRATCH TEST USING AN X-ACTO KNIFE.

THE FIRST CYCLE HAD A PEAK TEMPERATURE OF 800°C AND THE HEAT-UP WAS DONE IN STEPS TO OBSERVE MATERIAL CHANGES AT DIFFERENT TEMPERATURES.

THE GRAPH SHOWS THIS CYCLE. (FIG. 1)

THE INITIAL HEATING WAS DONE IN NITROGEN FLOW OF .25L/MIN AND AT 225°C THE DARK COLOR APPEARED, CORRESPONDING TO THE CARBONIZATION OF THE ORGANIC BINDER IN THE COATING. THE FORMING GAS WAS TURNED ON AT 300°C WITH A FLOW-RATE OF 2.5 L/MIN.

AT 400°C YELLOW COLOR OF THE ORIGINAL MoO_3 REAPPEARED. AT 540°C THE COLOR BECAME GREENISH AND AT 550°C SOME SUBLIMATION WAS OBSERVED.

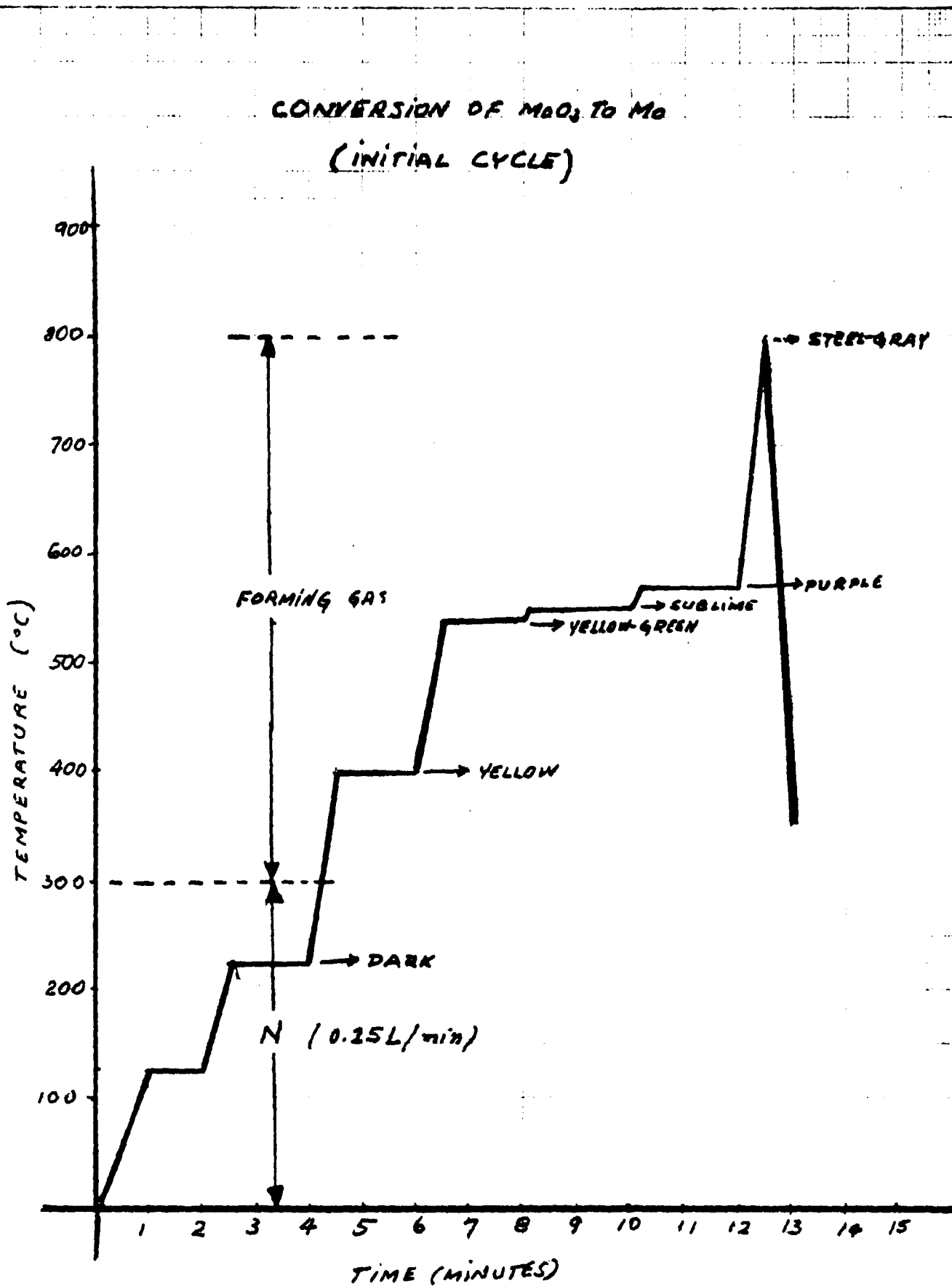


FIG. 1.

AT 570°C THE COLOR TURNED DARK PURPLE AND THIS PERSISTED TILL THE PEAK OF 800°C, AT WHICH POINT THE COLOR CHANGED TO STEEL-GRAY. THE TEMPERATURE WAS QUENCHED SUDDENLY TO ROOM TEMPERATURE.

THE FILMS HAD A RESISTANCE BETWEEN 5-10 OHMS AND A REASONABLE GOOD BOND TO SILICON, P-TYPE AS WELL AS N-TYPE, YET IT WAS POSSIBLE TO BE SCRAPPED OFF BY A X-ACTO KNIFE. THE FORMING GAS USED FOR THE REDUCTION WAS COMPOSED OF 85% NITROGEN AND 15% HYDROGEN.

EXPERIMENTS WERE DONE ALSO WITH 60% NITROGEN AND 40% HYDROGEN.

THE RESULTS IN BOTH CASES WERE IDENTICAL EXCEPT WITH THE HIGHER HYDROGEN CONTENT THE REDUCTION OF THE OXIDE WAS FASTER.

THE CONCLUSION DRAWN FROM THESE EXPERIMENTS WAS THAT THE TRANSITION TEMPERATURE FROM 550°C TO 650°C WAS CRITICAL WITH RESPECT TO THE SUBLIMATION AND LOSS OF MoO_3 .

THEREFORE THE CYCLE WAS MODIFIED TO SLOW-DOWN THE HEATING RATE WITHIN THIS RANGE AND ALSO THE PEAK TEMPERATURE WAS INCREASED TO 900°C, TO DETERMINE THE EFFECT OF HIGHER TEMPERATURE ON THE BOND STRENGTH.

THE INITIAL HEATING IN NITROGEN ATMOSPHERE WAS INCREASED TO 400°C TO ASSURE COMPLETE REMOVAL OF ORGANIC MATERIALS. A FIVE MINUTE PURGE IN FORMING GAS FOLLOWED.

THE GRAPH SHOWS THE MODIFIED CYCLE. (FIG. 2)

CONVERSION OF MoO_3 TO Mo .
(MODIFIED CYCLE)

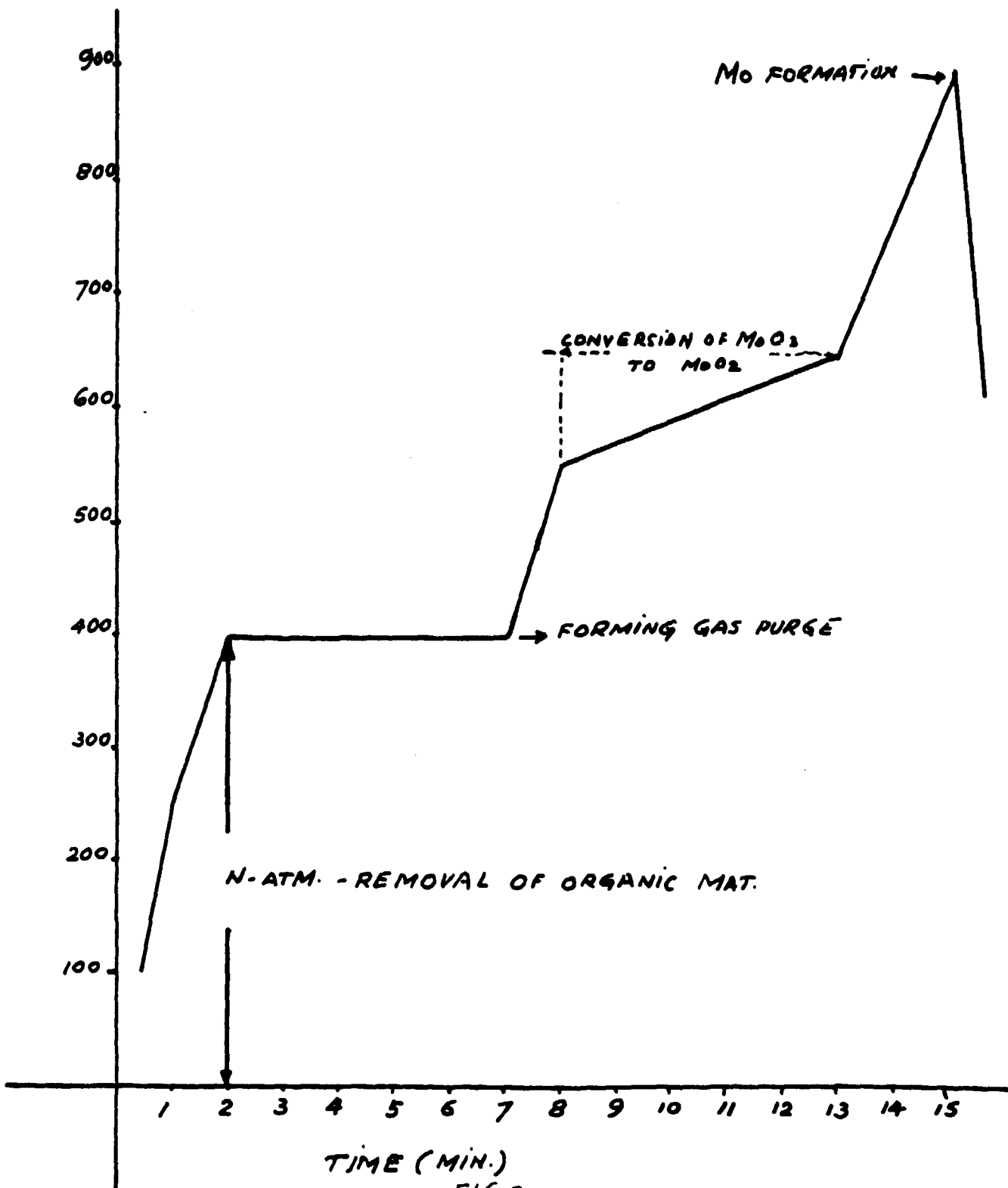


FIG. 2

THE MOLYBDENUM FILM RESULTING FROM THESE CYCLES HAD LATERAL RESISTANCE BELOW 1 OHM AND COULD NOT BE SCRAPED OFF BY THE X-ACTO KNIFE.

III. 3. DETERMINATION OF $MoO_3:Sn$ RATIO.

THE PRELIMINARY TESTS FOR THIS TASK WERE DONE BY COATING SILICON WAFERS WITH MOLYBDENUM OXIDE SUSPENSION AND LOCATING A FEW GRAINS OF 20 MESH SIZE TIN ON ONE EDGE OF THE COATING.

THE PURPOSE OF THIS TEST WAS TO DETERMINE THE WETABILITY OF THE MOLYBDENUM BY TIN.

THE SAMPLES WERE HEATED FOLLOWING THE MODIFIED CYCLE.

THE REACTION PROCEEDED BY FASHION PREVIOUSLY DESCRIBED FOR MoO_3 . AT $900^{\circ}C$ HOWEVER, WHEN THE Mo WAS FORMED, TIN SPREAD OVER THE ENTIRE SURFACE.

RESULTED COATINGS PASSED THE ADHESION TEST AND HAD A GOOD SOLDERABILITY BY LEAD-TIN SOLDER.

NEXT TESTS WERE DONE WITH MIXTURES OF $MoO_3:Sn$ (325 MESH PARTICLE SIZE) USING THE FOLLOWING RATIOS.

$\frac{MoO_3}{Sn}$	5	3	2	1	.5
	5	7	8	9	9.5

THE MIXTURE WAS SUSPENDED IN TRICHLOROETHYLENE WITH ETHYL-CELLULOSE BINDER SUCH AS WAS DONE BEFORE WITH MoO_3 ONLY. THE OBTAINED RESULTS SHOWED THAT WITH THE $MoO_3:Sn$ RATIO GREATER THAN 3:7, THE FORMED FILMS HAD A TEXTURE WITH A LACY PATTERN WITH TIN FORMING THE RIDGES.

THE BOND PASSED THE ADHESION TEST, BUT THE SOLDERABILITY WAS POOR AND REQUIRED A SCRUBBING ACTION.

FILMS FORMED FROM $\text{MoO}_3:\text{Sn}$ RATIOS LESS THAN 3:7 HAD A DENSE SOLDERABLE FINISH, BUT THE COATINGS WITH $\text{MoO}_3:\text{Sn}$ IN RATIOS OF 1:9 AND .5:9.5 HAD A WEAKER BOND.

THE BEST RESULTS WERE OBTAINED AT A RATIO OF $\text{MoO}_3:\text{Sn}=2:8$.

THE FIRING CYCLE TO ACHIEVE GOOD BONDING WITH THIS COMPOSITION WAS BETWEEN 800°C AND 900°C .

III. 4. FORMULATION OF A SCREENABLE INK FROM MoO_3 :SN MIXTURE.

MATERIALS USED:

TIN POWDER (COMINGO & ROIC) 325 MESH-99.999% PURITY

TRICHLOROETHYLENE-ANALYTICAL REAGENT, (LOS ANGELES CHEM. CO.)

ETHYL CELLULOSE-ETHOCCEL (R) STANDARD 10, PREMIUM ETHYL
CELLULOSE (DOW CHEMICAL CO.)

CARBITOL SOLVENT (DIETHYLENE GLYCOL MONOETHYL ETHER)-
TECHNICAL GRADE (ORANGE COUNTY CHEMICAL CO.)

CARBITOL ACETATE-TECHNICAL GRADE (ORANGE COUNTY CHEMICAL CO.)

MOLYBDENUM TRIOXIDE-REAGENT (MALLINCKRODT)

MoO_3 AND SN WERE MIXED IN A RATIO OF 78% SN AND 22% MoO_3 .

10 GRAMS OF THE MIXTURE WERE USED FOR THE FORMULATION
OF THE INITIAL INK COMPOSITION.

2.2 GRAMS OF MoO_3 AND 7.8 GRAMS OF SN WERE DRY MIXED IN
A QUARTZ MORTAR.

THE VEHICLE FOR THE INK CONSISTED OF 75% TRICHLOROETHYLENE,
15% ETHYL CELLULOSE, 8% CARBITOL SOLVENT AND 2% CABITOL
ACETATE, ALL AMOUNTS IN PERCENTAGES BY WEIGHT.

10 GRAMS OF THE MoO_3 :SN MIX WERE BLENDED WITH 5 GRAMS
OF THE VEHICLE AND HOMOGENIZED ON A GLASS PLATE WITH
A SPATULA.

THE SCREENING TEST WAS DONE USING A 200 MESH SCREEN.

III. 5. SET-UP AND CALIBRATION OF THE TUBE FURNACE FOR INK FIRING.

A HEAVY DUTY 3-ZONE DIFFUSION FURNACE WITH 3" I.D. QUARTZ TUBE WAS USED FOR THIS PURPOSE. THE INITIAL PEAK TEMPERATURE WAS SET AT 800°C. THE PROFILE OF THE FURNACE WAS MEASURED BY A CHROMEL-ALUMEL THERMOCOUPLE IN ORDER TO ESTABLISH THE TEMPERATURE ZONES CRITICAL FOR FIRING THE MoO_3 :SN MIXTURES.

NITROGEN AND FORMING GAS (60%N-40%H) WERE CONNECTED THROUGH A COMMON FLOWMETER TO ONE END OF THE TUBE.

THE EXPERIMENTS WERE DONE WITH N-TYPE SILICON .5 TO 1.5 OHM-CM RESISTIVITY WITH ONE SIDE POLISHED AND THE OTHER SIDE ETCHED.

AFTER APPLYING THE INK ON BOTH SIDES OF THE WAFER THE SAMPLES WERE AIR-DRIED AND ORGANIC MATERIALS BURNED OUT IN A SMALL CERAMIC MUFFLE KILN (21CM X 16CM X 23CM) ON A QUARTZ BOAT.

THE AIM OF THE FIRST RUNS WAS TO DUPLICATE THE CYCLE PREVIOUSLY ESTABLISHED ON THE GRAPHITE STRIPHEATER IN THE EXPERIMENTAL STATION.

IT HAS BEEN FOUND THAT IN ALL CASES THE REDUCTION OF MoO_3 INTO PURPLE MoO_2 TOOK PLACE WITHIN THE DETERMINED TEMPERATURE RANGE, I.E. BETWEEN 550°C AND 650°C , BUT IN CONTRAST WITH THE REACTION OBSERVED ON THE STRIPHEATER, WHERE THE METALLIC TIN FORMED AT 800°C , THE TIN FORMATION IN THE TUBE FURNACE TOOK PLACE SHORTLY AFTER THE CONVERSION OF MoO_3 INTO MoO_2 .

THIS WAS CONFIRMED BY A RUN OF MoO_3 COATING ALONE, DURING WHICH CONDUCTIVE Mo FILM WAS OBTAINED BETWEEN 550°C AND 650°C IN 5 MINUTES.

THE Mo:SN LAYER FORMED AT LOWER TEMPERATURES HAD A GOOD SOLDERABILITY AND IN SOME CASES A GOOD BOND.

IT REMAINED TO BE DETERMINED WHETHER IT WAS IMPORTANT TO CONDUCT THE HEATING CYCLE IN SUCH A WAY TO ESTABLISH IF THE TOTAL CONVERSION OF MoO_3 INTO Mo WAS NECESSARY BEFORE THE TEMPERATURE WAS RAISED TO 800°C .

III. 6. EVALUATION OF Mo:SN CONTACT.

THE CONTACT FORMED FROM A SCREENABLE MIXTURE OF 78% SN : 22% MoO_3 WAS EVALUATED FOR MECHANICAL AS WELL AS ELECTRICAL CHARACTERISTICS.

THE PATTERN WAS SCREENED THROUGH 200 MESH SILK

SCREENS ON DIFFUSED P ON N SILICON SOLAR CELL
STRUCTURES.

AFTER SCREENING, THE TEST SAMPLES WERE DRIED AND
ORGANIC MATERIAL BURNED OFF IN A CERAMIC MUFFLE
FURNACE.

THE FIRST EXPERIMENTS WERE DONE WITH A PRE-HEAT
AT 560°C FOR 5 MINUTES AND A SOAK AT 800°C PEAK
FOR 1 MINUTE.

THE ATMOSPHERE WAS 60% NITROGEN AND 40% HYDROGEN,
(FORMING GAS, PURCHASED FROM ANWELD INC.)

THE CONTACT HAD A GOOD ADHERENCE, TESTED BY THE
X-ACTO KNIFE AND HAD A GOOD SOLDERABILITY.

THE VOLTAGE-CURRENT CHARACTERISTICS DETERMINED
ON A X-Y PLOTTER SHOWED A STRAIGHT LINE BETWEEN
THE V_{OC} AND I_{SC} VALUES.

THERE WAS NO DEGRADATION IN SHORT CIRCUIT CURRENT NOR
IN OPEN CIRCUIT VOLTAGE VALUES, INDICATING THAT
THE CONTACT WAS OHMIC, BUT WITH A HIGH RESISTANCE
(ABOVE 10 OHMS).

IN ORDER TO ESTABLISH THE EFFECT OF THE PEAK TEMPE-
RATURE ON THIS HIGH SERIES RESISTANCE VALUE, OTHER
SAMPLES WERE RUN WITH THE SAME PRE-HEAT CYCLE, BUT
WITH PEAK TEMPERATURES OF 700°C AND 900°C RESPECTIVELY.

THE RESULTS SHOWED THAT THE SAMPLES FIRED AT 700°C HAD THE SAME SLOPE AS THE SAMPLES AT 800°C, WHILE THE SAMPLE FIRED AT 900°C WAS DEGRADED IN OPEN CIRCUIT VOLTAGE. (FIG. 3).

IN ORDER TO DETERMINE WHETHER THIS DEGRADATION WAS CAUSED BY THE ACTION OF THE METALLIC CONTACT OR BY THE TEMPERATURE ALONE, NON-METALLIZED CELLS WERE SUBJECTED TO THE SAME CYCLE OF 900°C PEAK. THE RESULTS OF THIS TEST SHOWED ALSO DEGRADATION IN OPEN CIRCUIT VOLTAGE, WHICH PROVED THAT THIS DEGRADATION WAS CAUSED BY THE HEAT CYCLE ALONE.

III. 7. IMPROVEMENT OF THE CONTACT SERIES RESISTANCE.

IN ORDER TO IMPROVE THE HIGH SERIES RESISTANCE Mo/Sn-Si CONTACT, REFERENCES WERE SEARCHED RELATING TO STUDIES OF Mo-Si INTERFACE AND NATURE OF THE CONTACT.

AN ARTICLE PUBLISHED BY R.C. HOOPER, J.A. CUNNINGHAM AND J.G. HARPER IN SOLID STATE ELECTRONICS, VOL. 8, PP 831-833 (1965), LISTS CONTACT RESISTANCE VALUES OF Mo AND OTHER METALS EVAPORATED ON Si OF VARIOUS RESISTIVITIES.

ACCORDING TO THIS STUDY THE CONTACT RESISTANCE OF Mo TO P-TYPE Si WITH RESISTIVITIES OF 0.002 AND 0.1 OHM-CM IS 4.4×10^{-6} OHM/CM² AND 1.1×10^{-1} OHM/CM² RESPECTIVELY.

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V-I CHARACTERISTICS OF PN CELLS.
(SCREENED Mo/Sn CONTACTS)

CELL AREA: APPROX. 2 cm²

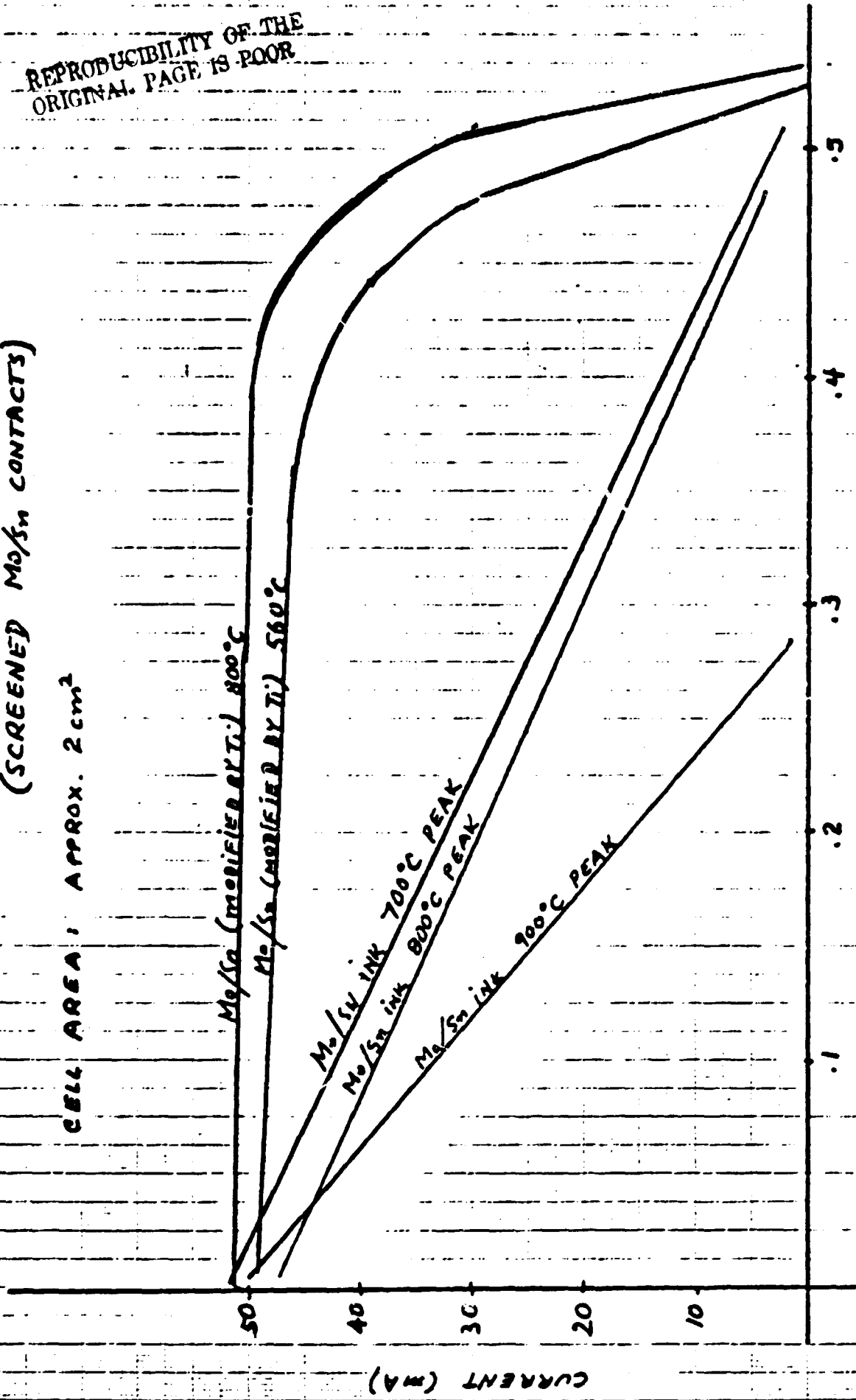


FIG. 3.

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THE CONTACT RESISTANCE OF Mo TO 0.5 OHM-CM P-TYPE Si IS 9.4×10^{-2} OHM/CM².

FOR N-TYPE Si OF 0.005 OHM-CM THE CONTACT RESISTANCE IS 7.8×10^{-5} OHM/CM², FOR 0.01 OHM-CM 6.1×10^{-1} OHM/CM², FOR 0.05 OHM-CM 2.0 OHM/CM² AND FOR 0.5 OHM-CM 26 OHM/CM².

SINCE THE STARTING SILICON CRYSTAL USED IN OUR SOLAR CELL STRUCTURE IS N-TYPE OF A RESISTIVITY RANGE BETWEEN 0.3 - 0.7 OHM-CM, THE HIGH RESISTANCE OF THE Mo CONTACT COULD BE EXPLAINED BY THE REFERENCED DATA.

ANOTHER ARTICLE PUBLISHED BY R.W. BOWER AND J.W. MAYER IN APPL. PHYS. LETT., VOL.20 - No.9 (MAY 1972) PP. 359-361, IS CONCERNED WITH THE FORMATION OF METAL SILICIDES, SPECIFICALLY Pd, Ti, Cr AND Mo.

ACCORDING TO THE AUTHORS, THE GROWTH RATE OF Pd₂Si AND TiSi₂ VARIES LINEARLY WITH THE SQUARE ROOT OF TIME, WHILE CrSi₂ AND MoSi₂ GROW LINEARLY WITH TIME.

THE FIRST CASE SUGGESTED DIFFUSION LIMITED RATE GROWTH MECHANISM, WHILE THE SECOND CASE CORRESPONDED TO REACTION LIMITED RATE GROWTH.

THE FORMATION DEPTH OF Pd, Ti SILICIDES IS THERE-

FORE EASIER TO CONTROL THAN THE DEPTH OF CR AND Mo SILICIDES.

WHILE THE PRESENCE OF AN OXIDE FILM ON SI SURFACE INHIBITS THE FORMATION OF Pd, CR AND Mo SILICIDES,

IT HAS NO DETECTABLE EFFECT FOR THE Si-Ti SYSTEM AND THE $TiSi_2$ FORMS AT RELATIVELY LOW TEMPERATURES $\sim 600^\circ C$.

BASED ON THESE REFERENCES, EXPERIMENTS WERE PERFORMED TO DETERMINE THE SIGNIFICANCE AND THE EFFECT OF TITANIUM SILICIDE ON THE VALUE OF CONTACT RESISTANCE.

AS THE SOURCE OF TITANIUM, TITANIUM RESINATE WAS USED, PRODUCED BY ENGELHARD, INDUSTRIES DIV., NEWARK, N.J.

THIS ORGANO-METALLIC COMPOUND CONTAINS 4.3% SOLIDS AS TITANIUM AND IS IN A SOLUTION FORM WHICH IS COMPATIBLE WITH THE SOLVENTS PRESENT IN THE INK. ONE EYEDROP OF THE RESINATE SOLUTION CORRESPONDING TO APPROX. 0.0013 G OF TITANIUM WAS ADDED TO 30 G OF INK.

THE RATIO OF ADDED TITANIUM TO THIS MIXTURE CORRESPONDED THEN TO APPROX. 1 PART OF Ti TO 15000-16000 PARTS OF MoO_3/Sn SOLID MIXTURE.

THE EXPERIMENTS OF THE INK FIRING DESCRIBED FOR THE

STANDARD Mo:Sn MIXTURE WERE DUPLICATED WITH THE INK MODIFIED BY THE TITANIUM.

THE RESULTS SHOWED A VERY DISTINCT EFFECT ON V-I CHARACTERISTICS.

THE SERIES RESISTANCE HAD A VALUE OF APPROX. 1 OHM AT A CYCLE CONSISTING OF A PRE-HEAT AT 560°C FOR 3 MINUTES, 620°C FOR 3 MINUTES AND A SOAK AT 800°C FOR 1 MINUTE.

SIMILAR VALUES OF THE SERIES RESISTANCE WERE ALSO OBTAINED BY HEATING AT 560°C FOR 9 MINUTES. (FIG. NO. 3)

THE BONDS ON SAMPLES FIRED AT 560°C WERE WEAKER THAN THE BONDS OBTAINED AT 800°C.

III. 8. OPTIMIZING THE FIRING CYCLE FOR THE Mo:Sn INK MODIFIED BY Ti.

THE OBJECTIVE OF THIS TASK WAS TO DETERMINE THE EFFECT OF VARIOUS FIRING CYCLES ON THE COMBINED QUALITY OF MECHANICAL ADHESION, SOLDERABILITY AND ELECTRICAL PERFORMANCE OF SOLAR CELLS METALLIZED WITH Mo:Sn INK MODIFIED BY Ti.

THREE FIRING CYCLES WERE EMPLOYED FOR EVALUATION

- A) 560°C - 20 MINUTES AND QUENCH
- B) 560°C - 3 MINUTES, 620°C - 3 MINUTES, 800°C - 1 MINUTE AND QUENCH
- C) 560°C - 10 MINUTES, 700°C - 1 MINUTE AND QUENCH.

ALL CYCLES WERE DONE IN A FORMING GAS ATMOSPHERE CONSISTING OF 60% N AND 40% H AT A FLOW RATE OF 3L/MIN.

THE ADHESION WAS DETERMINED BY SCRATCH TESTS (X-ACTO) AND PULL TESTS WITH SOLDERED LEADS. THE LEAD ATTACHMENT WAS ALSO A MEASURE OF SOLDERABILITY.

THE ELECTRICAL CHARACTERISTICS WERE EVALUATED BY THE SHAPE OF V-I CURVES.

THE QUALITY OF MECHANICAL PROPERTIES (ADHESION

AND SOLDERABILITY) CONFIRMED RESULTS OF TESTS PREVIOUSLY PERFORMED WITH Mo:Sn INK WITHOUT TI ADDED, I.E. THE BOND WAS WEAKER WITH THE FIRING CYCLE AT 560°C THAN AT CYCLES WITH 700°C AND 800°C PEAK TEMPERATURES.

THE SOLDERABILITY WAS ALSO BETTER FOR LAYERS FORMED AT HIGHER TEMPERATURES.

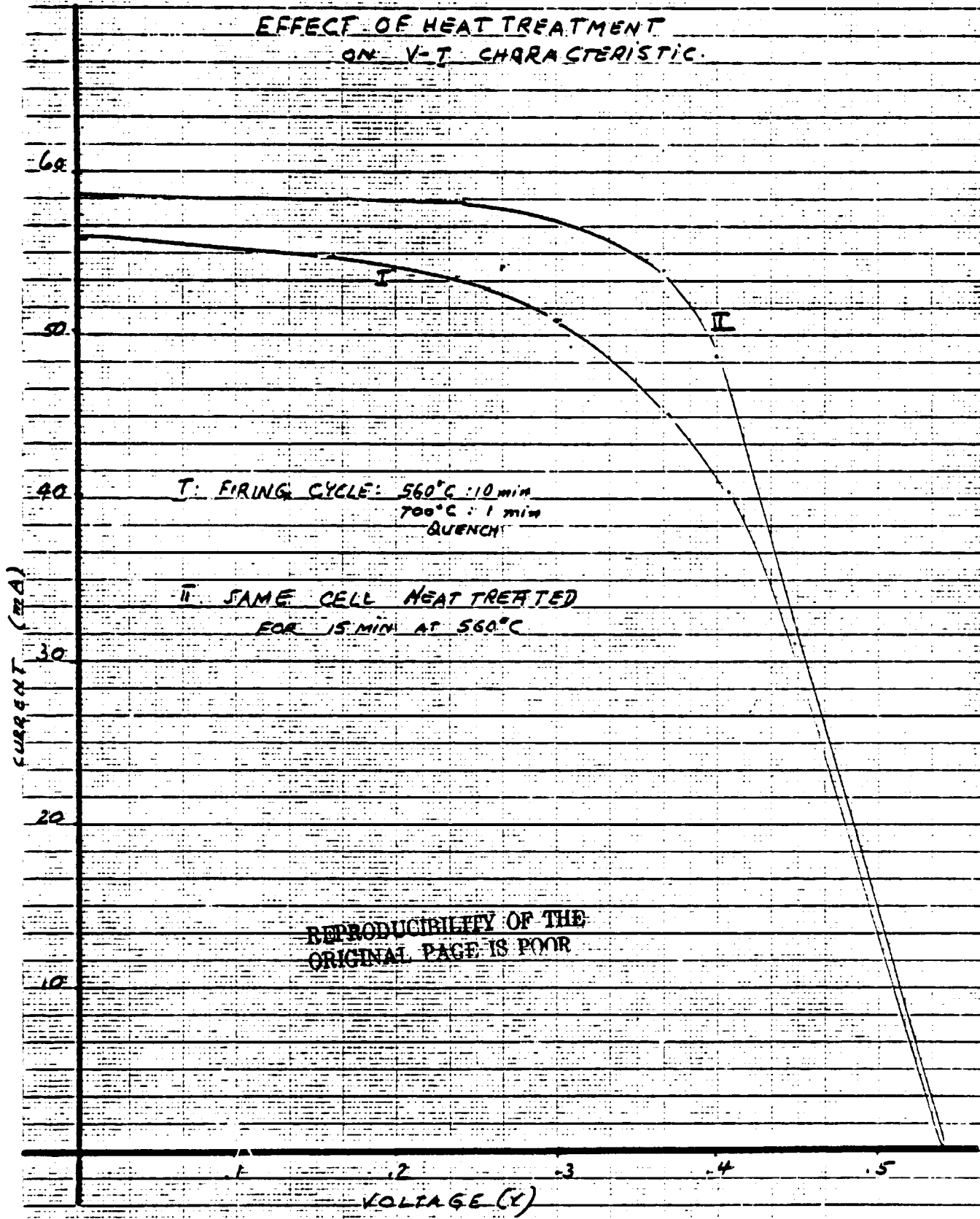
ELECTRICAL CHARACTERISTICS OF SOLAR CELLS METALLIZED AT THE STATED FIRING CYCLES WERE ON THE OTHER HAND SIGNIFICANTLY BETTER AT THE LOW TEMPERATURE FIRING CYCLE

IN ORDER TO ESTABLISH WHETHER THIS CHARACTERISTIC WAS A RESULT OF THE PEAK TEMPERATURE OF THE FIRING CYCLE ONLY OR OF ADDITIONAL REACTIONS TAKING PLACE DURING LOW TEMPERATURE ANNEALING PROCESS, THE CELLS METALLIZED AT HIGHER TEMPERATURES WERE HEAT TREATED AT LOW TEMPERATURE AFTERWARDS.

THE RESULT OBTAINED FROM THIS TEST ESTABLISHED THAT THE V-I CHARACTERISTICS IMPROVED TO THE QUALITY OF THE CELLS METALLIZED AT LOW TEMPERATURE ONLY. (FIG. 4)

FROM THESE RESULTS OTHER SETS OF EXPERIMENTS WERE PLANNED TO DETERMINE WHETHER THIS HEAT

EFFECT OF HEAT TREATMENT ON V-I CHARACTERISTIC.



I. FIRING CYCLE: 560°C : 10 min
700°C : 1 min
QUENCH

II. SAME CELL HEAT TREATED
FOR 15 MIN AT 560°C

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Fig. 4

TREATMENT CAN BE DONE AS A PART OF THE FIRING CYCLE OR MUST BE PERFORMED AS A POST HEAT TREATMENT STEP. ALSO IT WAS NECESSARY TO DETERMINE WHETHER 550°C IS THE OPTIMUM HEAT TREATMENT TEMPERATURE OR WHETHER THE TREATMENT HAD ANOTHER PEAK.

THE RESULTS OBTAINED FROM THESE TESTS POINTED OUT THAT THE HEAT TREATMENT CAN BE INCORPORATED IN THE FIRING CYCLE AND THE OPTIMUM HEAT TREATMENT IS 450°C FOR 15 MINUTES. (FIG. 5)

III. 9. COMPARISON OF Mo:Sn CONTACT WITH NICKEL PLATED AND SILVER SCREENED CONTACT.

NICKEL PLATED CELLS USED FOR THE COMPARATIVE TESTS WERE PRODUCTS OF THREE MANUFACTURERS.

THE SCREENED SILVER CELLS WERE FROM ANOTHER SOURCE. THE RESULTS OF V_{oc} , I_{sc} AND PULLSTRENGTH ARE LISTED IN TABLE II.

SINCE THE CELLS DID NOT HAVE IDENTICAL DIMENSIONS THE VALUES OF I_{sc} WERE NORMALIZED TO 1 cm^2 AREA.

ELECTRICAL CHARACTERISTICS DISPLAYED BY THE V-I CURVES IS SHOWN IN FIG. 6.

TABLE II

COMPARISON OF Mo/Sn INK WITH Ni-PLATED AND Ag-SCREENED

CONTACT.

SOURCE	TYPE	METALLIZATION	V_{oc} (V)	$\frac{I_{sc} (mA)}{cm^2}$	PULL STRENGTH (g)
MANUFACTURER "A"	P/N	Ni-PLATED	.555	26	200+
"B"	P/N	Ni-PLATED	.56	28	200+
"C"	N/P	Ni-PLATED	.55	28	200+
"D"	N/P	Ag-SCREENED	.54	25	0
EXPERIMENTAL INK	P/N	Mo/Sn-SCREENED	.56	26	200+

V-I CHARACTERISTICS OF CELLS
 LOW TEMP. METALLIZATION: I (560°C)
 HIGH TEMP. + 450°C HEAT TREATMENT.

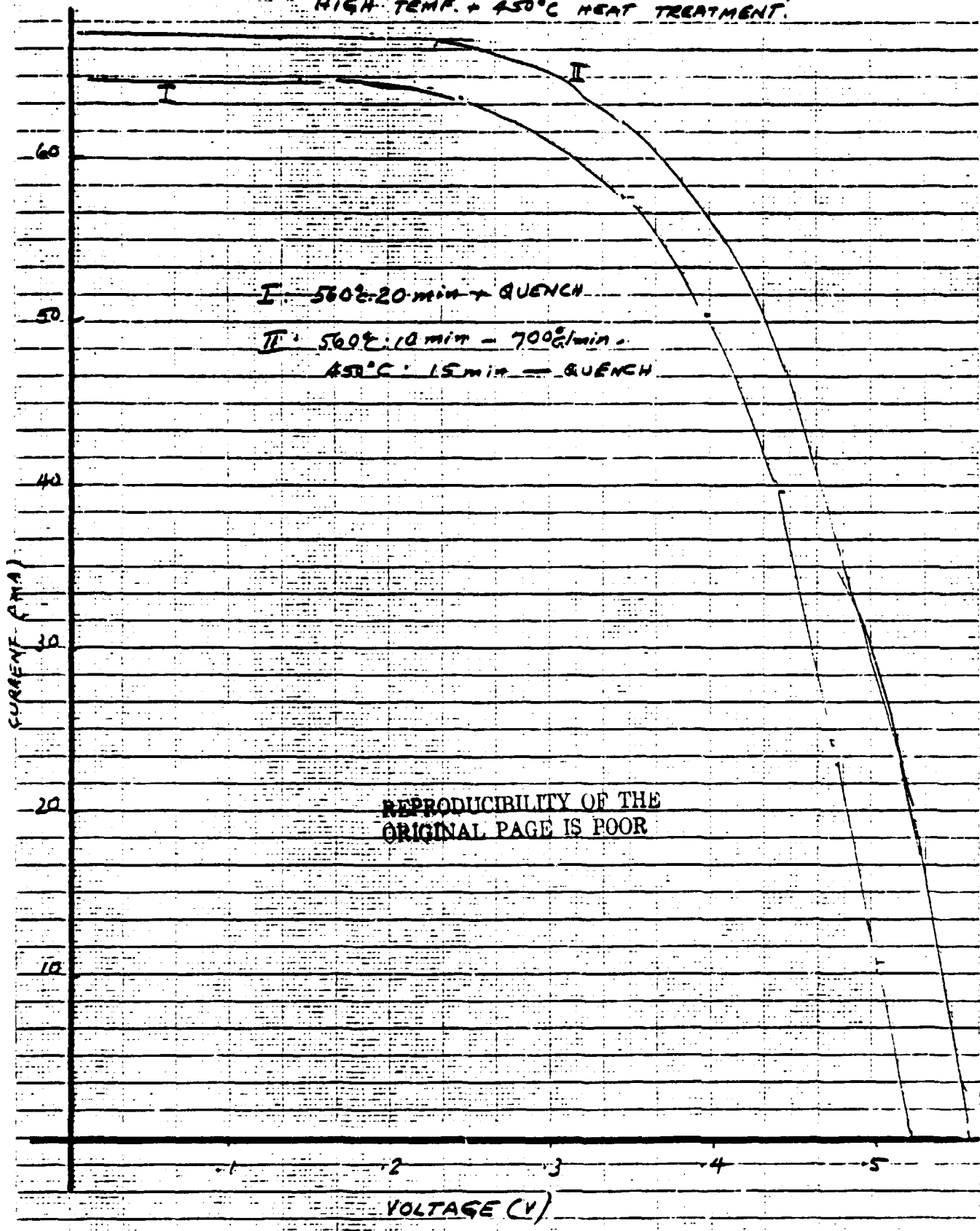


FIG. 5

THE MECHANICAL CONTACT OF THE Mo:Sn CELLS WAS EQUAL TO THE BEST NICKEL PLATED CONTACT AND SUPERIOR TO THE SCREENED SILVER CONTACT. ACTUALLY THE SILVER SCREENED CONTACT WAS INFERIOR SINCE THE PATTERN AS WELL AS THE BACK METALLIZATION COULD BE PEELED OFF WITHOUT ANY EFFORT.

IT WAS COMPARABLE WITH THE CHARACTERISTICS OF OTHER METALLIZATION. ONLY IN CASES WHEN THE TEST SAMPLES WERE SCRIBED TO SIZE, THE SHUNT RESISTANCE AND SERIES RESISTANCE SHOWED SOME DEGRADATION CAUSED BY THE MECHANICAL DAMAGE. GENERALLY THE SERIES RESISTANCE OF ALL CELLS TESTED WAS 1Ω IN AVERAGE.

V-I CHARACTERISTIC OF CELLS
 WITH VARIOUS METALLIZATIONS
 (CELLS OF DIFFERENT SIZES)

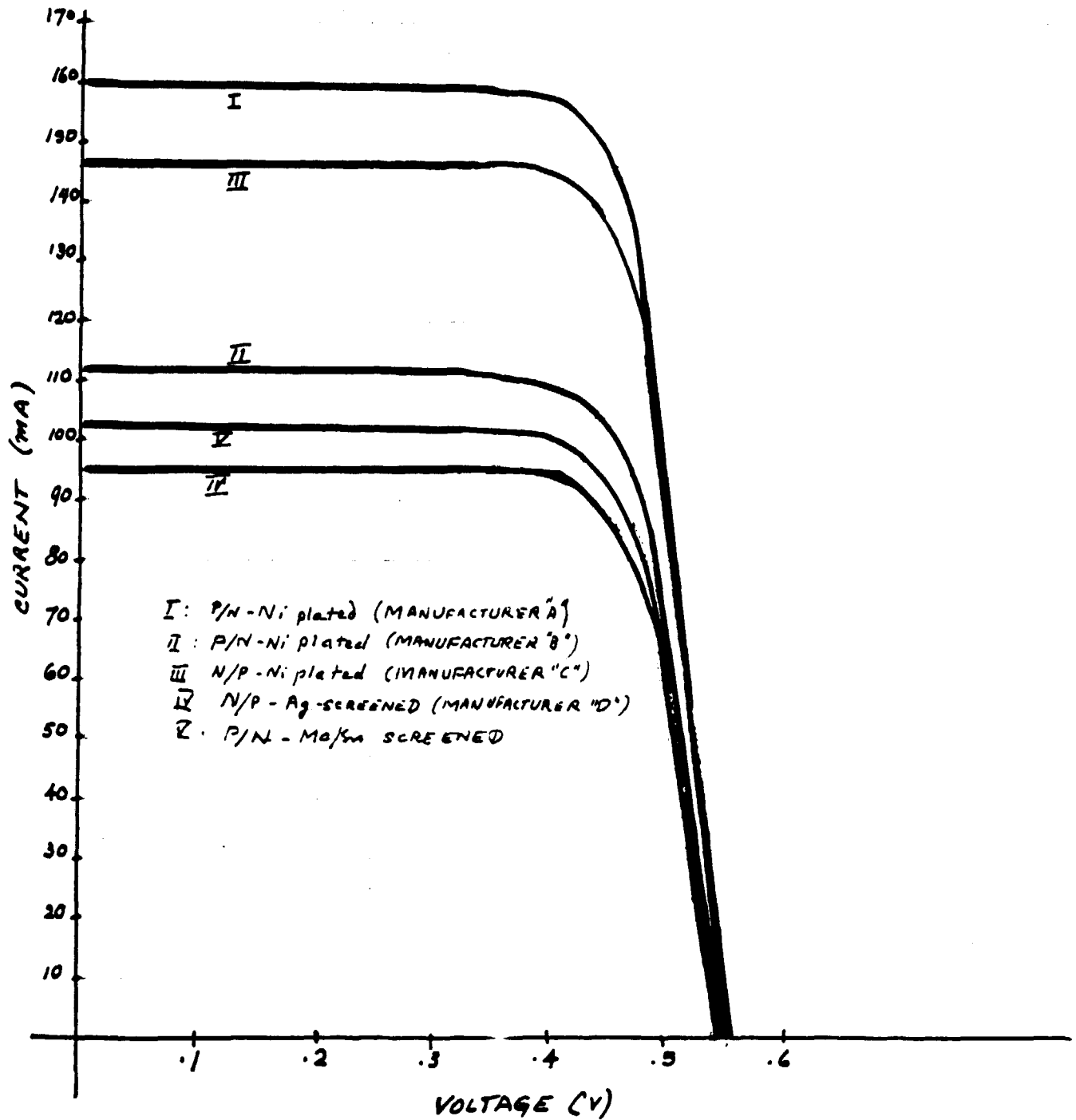


FIG. 6

III. 10. OPTIMIZATION OF INK COMPOSITION.

THE INK PREPARED PREVIOUSLY REVEALED DIFFICULTIES IN SEGREGATION OF THE TIN FROM MoO_3 DURING STANDING.

THIS SEGREGATION RESULTED IN NON-REPRODUCIBLE RESULTS, SINCE TIN WITH THE DENSITY OF 7.3 GR/CM^3 SETTLED BELOW THE MoO_3 WITH A DENSITY OF 4.5 GR/CM^3 WITHIN ONE HOUR.

THEREFOR IT WAS NECESSARY TO ADJUST THE RHEOLOGY OF THE INK IN SUCH A WAY THAT THE HOMOGENEITY OF THE MIXTURE WAS MAINTAINED FOR AT LEAST A PERIOD OF ONE WORKING DAY AND WAS RECOVERED THE NEXT DAY.

THE GOAL WAS ACHIEVED BY PARTIAL SUBSTITUTION OF TRICHLOROETHYLENE BY TERPINEOL WHICH HAS A LOWER VAPOR PRESSURE AND HIGHER BOILING POINT.

THE INK WAS PREPARED BY THE FOLLOWING PROCEDURE: Sn-MoO_3 POWDERS WERE PREMIXED DRY AND PASSED THROUGH 140 MESH SIEVES TO REMOVE ANY POSSIBLE LUMPS.

THE VEHICLE WAS PREPARED FROM ETHYL CELLULOSE BINDER DISSOLVED IN TRICHLOROETHYLENE.

TO THIS SOLUTION WAS ADDED TERPINEOL AND CARBITOL ACETATE.

THE PERCENTAGE BY WEIGHT OF THE INDIVIDUAL
INGREDIENTS WAS AS FOLLOWS:

6% ETHYL CELLULOSE DISSOLVED IN 20% TRICHLORO-
ETHYLENE, ADDED WAS 70% TERPINEOL AND 4% CARBI-
TOL ACETATE.

20% OF THIS VEHICLE AND 80% MoO_3 -SN MIXTURE
WERE BLENDED TO FORM THE INK. THIS WAS DONE BY
ADDING THE DRY MIXTURE UNDER CONSTANT STIRRING
TO THE VEHICLE.

FIVE OZ. OF THIS INK WAS SHIPPED TO JET
PROPULSION LABORATORY.

III. 11. SCREENING, FIRING AND TESTING OF 2CM X 2CM CELLS.

THE CELLS WERE P ON N STRUCTURE AND WERE SCREENED WITH A GRID PATTERN SPECIFICALLY DESIGNED FOR THIS PURPOSE.

THE FIRING CYCLE WAS 560°C : 10 MIN -
700°C : 1 MIN -
450°C : 15 MIN.

THE CHARACTERISTICS OF THE CELLS WERE TESTED ON A V-I PLOTTER AND IS SHOWN ON THE ATTACHED GRAPH. (FIG. 7)

COMPARATIVE TESTS WERE DONE WITH LOW TEMPERATURE CYCLES AT 550°C FOR 15 MIN.

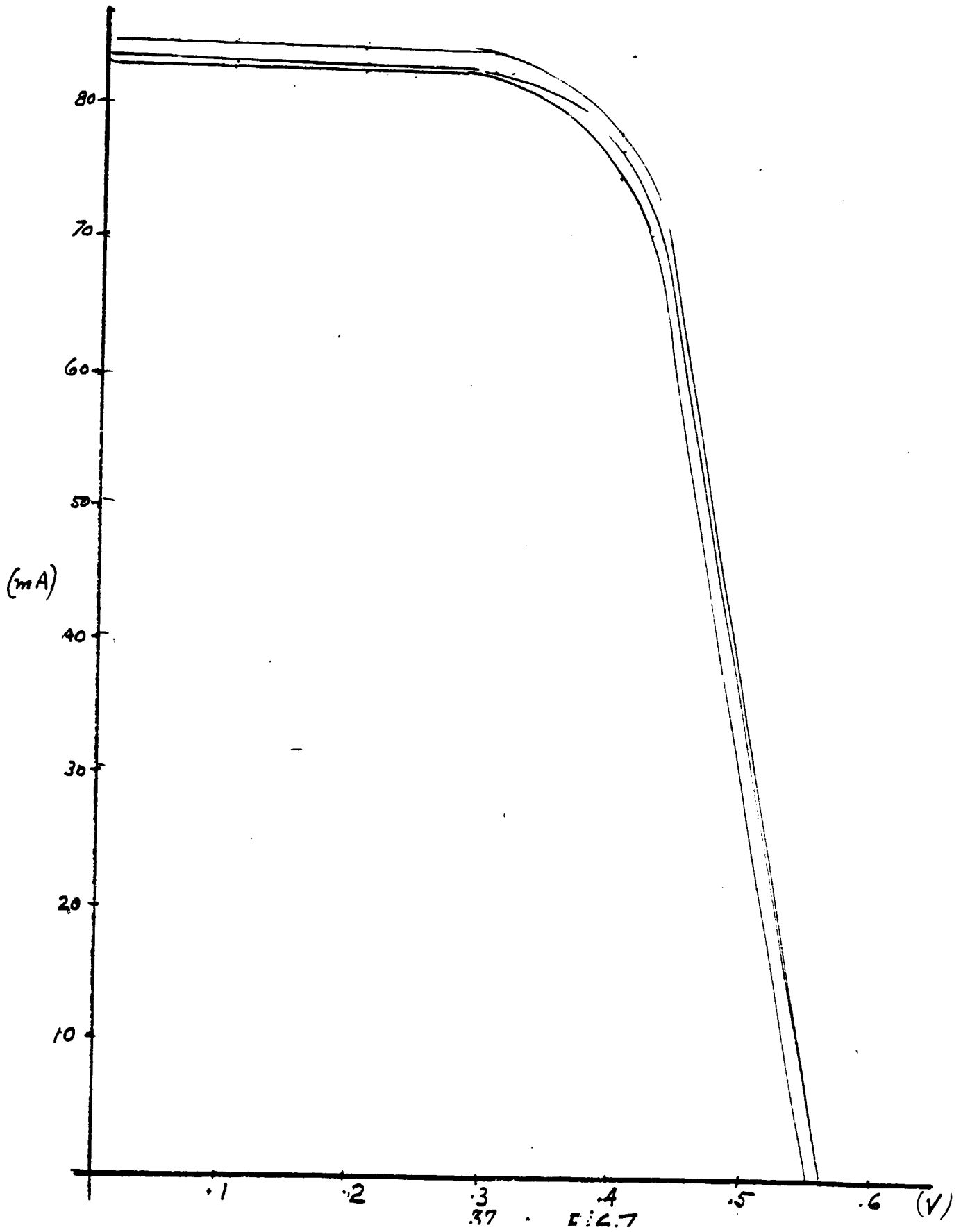
IN BOTH CASES THE RESULTS WERE WITHIN A CLOSE RANGE, ONLY THE CELLS FIRED AT LOW TEMPERATURE CYCLES HAD MECHANICALLY WEAKER BONDS.

THE CELLS PRIOR TO SCREENING WERE CLEANED BY 10% NaOH SOLUTION FOR 1 MINUTE.

IN OTHER EXPERIMENTS 1 MINUTE DIP IN CONCENTRATED HF WAS USED.

NO DIFFERENCE WAS OBSERVED IN OBTAINED RESULTS.

V-I CHARACTERISTICS OF
2 cm x 2 cm CELLS



III. 12. ENVIRONMENTAL TEST OF METALLIZED CELLS.

THE EVALUATION OF THE MO-SN SYSTEM AND CONTACT INTEGRITY UNDER ENVIRONMENTAL STRESSES INCLUDED THE HUMIDITY EXPOSURE AND SUBJECTION TO TEMPERATURE EXTREMES.

THE ATTEMPT HAS BEEN MADE TO ESTABLISH SOME STANDARD CRITERIA FOR THIS TEST.

INQUIRIES WITH JPL AND NASA-LEWIS RESEARCH CENTER IN CLEVELAND HOWEVER REVEALED THAT THERE ARE NO ESTABLISHED STANDARD TEST PROCEDURES WHICH WOULD APPLY TO TERRESTRIAL PHOTOVOLTAIC CELLS AND THE EXISTING TESTS ARE USED FOR SPACE CELLS WITH MODIFICATIONS SPECIFIED BY CUSTOMERS.

BASED ON SUGGESTIONS OBTAINED FROM THE NASA-LEWIS RESEARCH CENTER THE FOLLOWING TESTS WERE USED TO EVALUATE THE CONTACT:

A: SUBJECTION OF THE CELLS WITH ATTACHED LEADS TO A CONSTANT TEMPERATURE WATERBATH OF 45°C FOR ONE WEEK AND

B: SUBJECTION OF THE CELLS TO TEN THERMOSHOCK CYCLES BETWEEN 100°C (BOILING WATER) AND -45°C (DRY ICE-ACETONE).

THE DWELL TIME AT EACH EXTREME WAS 5 MINUTES.

THE CONTACT WAS EVALUATED THEN BY THE VALUE OF THE PULL-TEST AND ELECTRICAL CHARACTERISTICS.

THE CELLS FOR THIS TEST WERE PREPARED BY BORON DIFFUSION IN .5 OHM-CM N-TYPE CRYSTAL, USING BCL_3 SOURCE.

THE ELECTRODE PATTERN WAS SCREENED USING MoO_3-Sn INK FROM THE BATCH PREPARED FOR JFL.

THE WAFERS WERE CLEANED BY 10% $NaOH$ SOLUTION OR 60N.HF FOR 1 MINUTE PRIOR TO SCREENING.

THE SCREENED CELLS, AFTER DRYING AT $90^\circ C$, WERE SUBJECTED TO $400^\circ C$ HEAT IN AIR IN A MUFFLE FURNACE TO BURN OUT THE ORGANIC BINDER FROM THE INK. AFTER THAT THE CELLS WERE DIVIDED INTO 2 GROUPS OF 12 UNITS EACH AND SUBJECTED TO THE FIRING-METALLIZATION CYCLE IN FORMING GAS ATMOSPHERE (60% N + 40% H).

THE FIRST GROUP WAS FIRED AT $550^\circ C$ FOR 15 MINUTES THE SECOND GROUP WAS FIRED AT $560^\circ C$ FOR 10 MINUTES, FOLLOWED BY 1 MINUTE AT $700^\circ C$ AND 15 MINUTES AT $450^\circ C$.

SILVER RIBBON LEADS, .050" WIDE AND .005" THICK, WERE SOLDERED TO THE CELLS AND THE CELLS WERE TESTED ON V-I CHARACTERISTICS. THE MECHANICAL STRENGTH WAS DETERMINED BY A PULL TEST AND VARIED FROM 50 GR.

FOR CELLS FIRED AT LOW TEMPERATURE TO 200-PLUS GR. FOR CELLS FIRED AT HIGHER TEMPERATURE.

THE CELLS WERE SUBJECTED TO CONSTANT TEMPERATURE WATER BATH AT 45°C FOR ONE WEEK AND THE ELECTRICAL CHARACTERISTICS WERE MEASURED AGAIN.

THE RESULTS SHOWED THAT THE CELLS FIRED AT LOW TEMPERATURE (550°C) DEGRADED BY LOWERED SHUNT RESISTANCE IN THE V-I CHARACTERISTICS AS SHOWN IN THE ATTACHED GRAPHS. (FIG. 8 & FIG. 9)

THE CELLS FIRED AT HIGH TEMPERATURE DID NOT EXHIBIT SUCH DEGRADATION.

FURTHER EXPERIMENTS CONDUCTED FOR THE PURPOSE OF EXPLAINING THE DEGRADATION REVEALED THAT THE CELLS FIRED AT LOWER TEMPERATURE AND DEGRADED BY EXPOSURE TO 45°C HUMIDITY CYCLE, RECOVERED TO THE ORIGINAL V-I CHARACTERISTICS AFTER A 15 MINUTE HEAT TREATMENT IN FORMING GAS.

FOLLOWING THE HUMIDITY TEST THE CELLS WERE SUBJECTED TO 10 THERMO-SHOCK CYCLES CONSISTING OF EXPOSURES TO 100°C AND -45°C.

THIS TEST WAS DONE BY USING BOILING WATER FOR THE HIGH TEMPERATURE AND DRY ICE-ACETONE MIXTURE FOR THE LOW TEMPERATURE ENVIRONMENT.

THE CELLS WERE MAINTAINED IN EACH ENVIRONMENT FOR 5 MINUTES.

THE TRANSFER OF THE CELLS FROM ONE TEMPERATURE
EXTREME TO THE OTHER WAS DONE WITHOUT ANY TIME
DELAY.

ONLY CELLS FIRED AT 700°C PEAK TEMPERATURE, FOLLOWED
BY 15 MINUTES ANNEAL AT 450°C, WERE SUBJECTED TO
THIS ABOVE DESCRIBED TEST.

NO MECHANICAL BREAKAGE NOR ELECTRICAL CHANGE WAS
OBSERVED AFTER THE TESTS.

V-I CHARACTERISTICS OF
CELLS FIRED AT 550°C

ILLUMINATION: 100 mW/cm² - AM=1

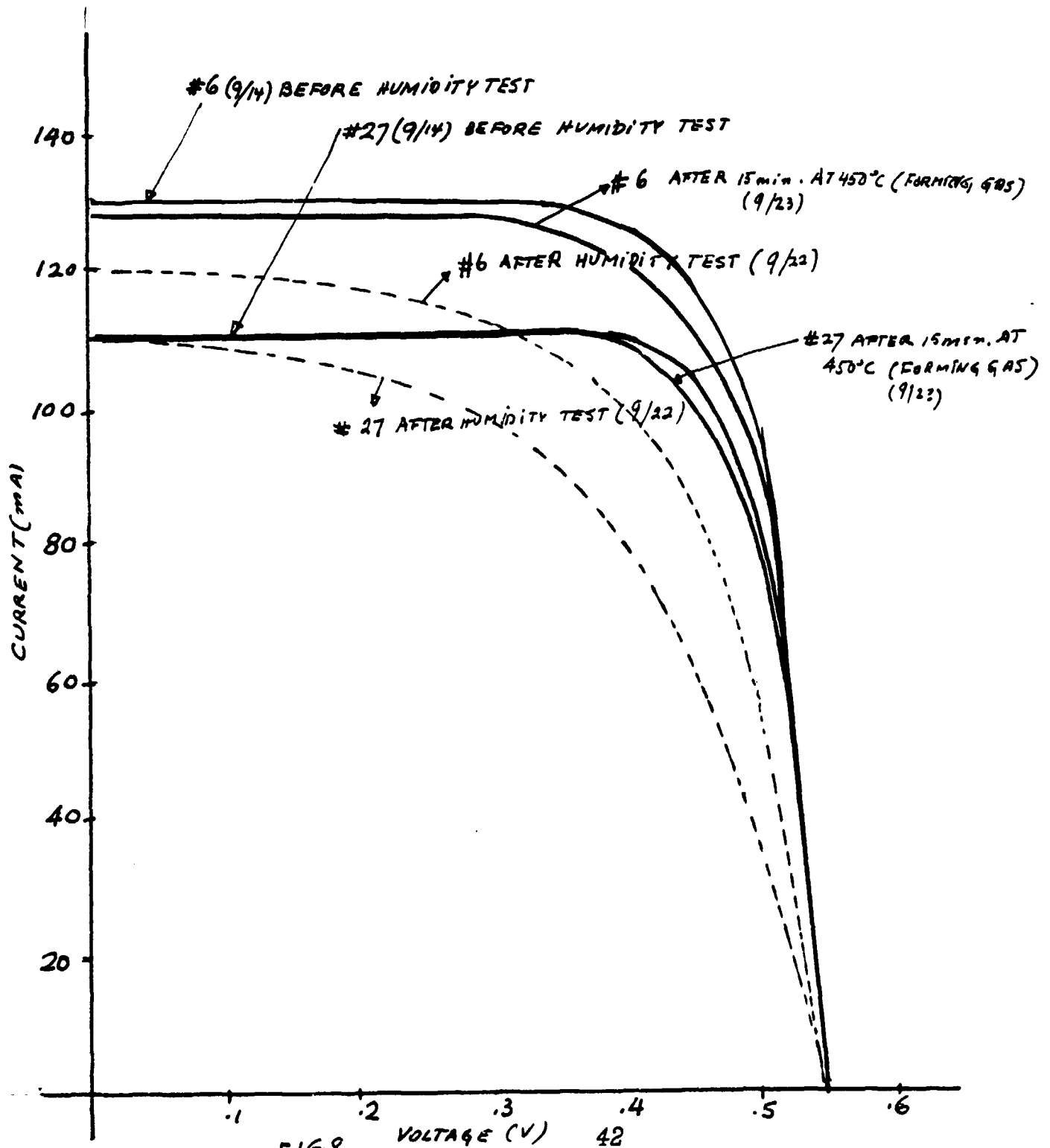


FIG 8

VOLTAGE (V) 42

V-I CHARACTERISTICS OF
CELLS FIRED AT 700°C (PEAK)
+ 450°C ANNEAL (15min)

ILLUMINATION : 100 mW/cm² - AM=1

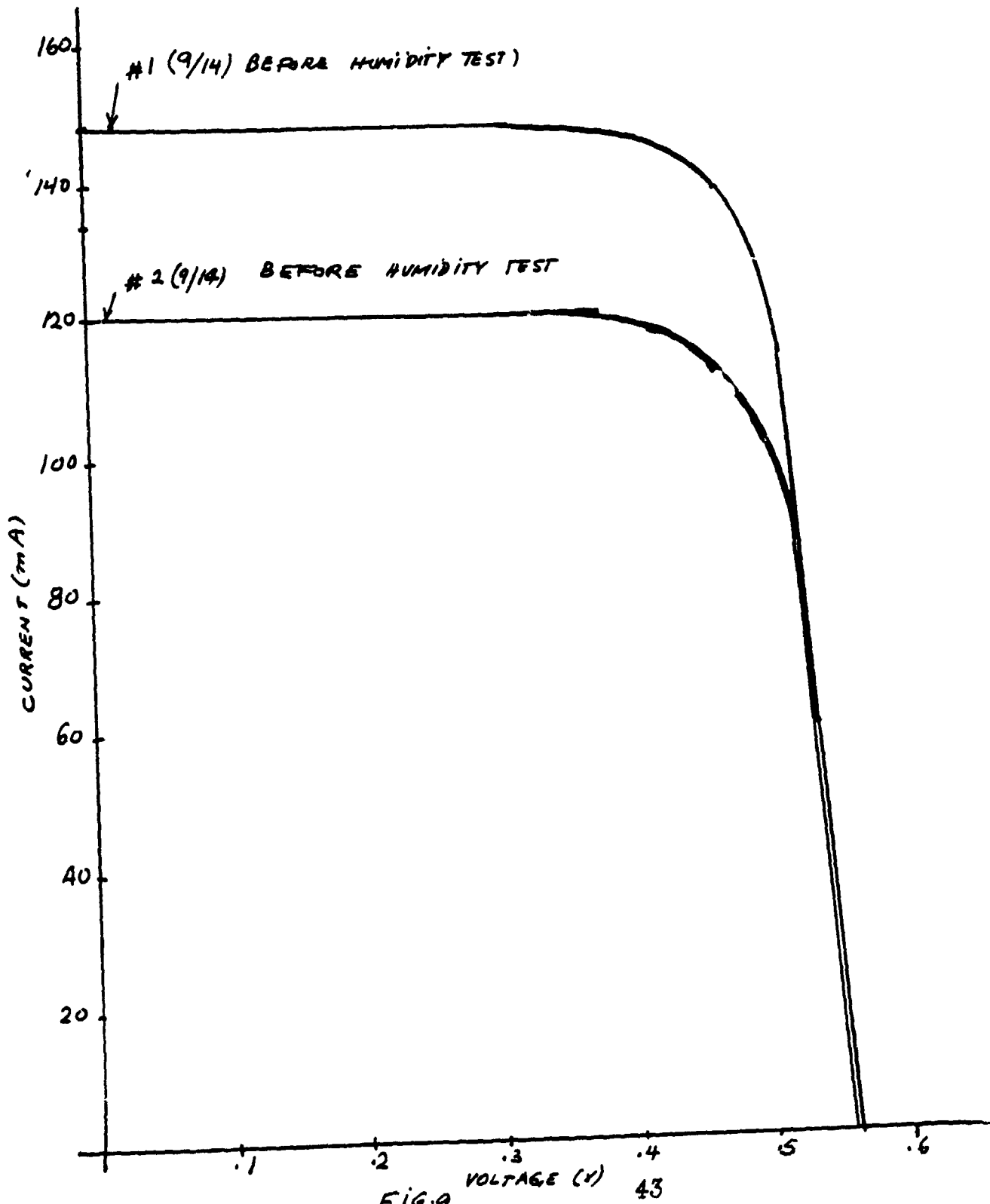


FIG. 9 43

III. 13. METALLIZATION OF N/P SOLAR CELLS BY Mo-Sn INK.

FIFTY DIFFUSED WAFERS, 2 X 2 CM - PRODUCED BY ANOTHER COMPANY WERE USED FOR THIS TEST.

THE WAFERS HAD AL BACKSURFACE FIELD.

THE CELLS WERE TESTED FOR OPEN CIRCUIT VOLTAGE PRIOR TO SCREENING BY MECHANICAL CONTACT.

THE VALUES OF THE OPEN CIRCUIT VOLTAGE RANGED BETWEEN .572 AND .585.

TEN CELLS FROM THIS LOT WERE SCREENED WITHOUT ANY PRELIMINARY TREATMENT OR CLEANING, USING THE STANDARD GRID FOR THE ACTIVE SIDE.

THE SCREENED CELLS, AFTER DRYING, WERE BURNED-OUT IN A MUFFLE FURNACE IN AIR AT 400°C PEAK TEMPERATURE FOR 10 MINUTES.

THE METALLIZED CYCLE WAS CONDUCTED IN THE TUBE FURNACE USING FORMING GAS ATMOSPHERE (60% N & 40% H).

THE CYCLE CONSISTED OF 5 MINUTES PURGE BY N, FOLLOWED BY FIRING IN FORMING GAS AT 525°C FOR 10 MINUTES, 700°C FOR 2 MINUTES AND 470°C FOR 15 MINUTES.

THE CELLS TESTED AFTER THE FIRING CYCLE WERE SHORTED AND ALL OF THEM SHOWED SPECKS, DARK IN APPEARANCE, ON BOTH SIDES OF THE WAFERS.

IT WAS SPECULATED THAT THESE SPECKS WERE AL PARTICLES

ADHERING TO THE SURFACE AFTER THE REMOVAL OF AL FROM THE BACK SURFACE AND CAUSED THE SHORTING OF THE JUNCTION WHEN HEATED TO 700°C.

IN ORDER TO CONFIRM THIS ASSUMPTION, ANOTHER LOT OF TEN WAFERS WAS PROCESSED THE SAME WAY, BUT WITH THE PEAK TEMPERATURE OF THE CYCLE LYING BELOW THE AL/SI EUTECTIC (577°C).

THIS SECOND LOT WAS FIRED FOR 15 MINUTES AT 525°C, FOLLOWED BY 15 MINUTES AT 470°C.

THE RESULTS OF THE SECOND TEST WERE AS LISTED ON THE ATTACHED TABLE. (TABLE III)

NO DARK SPECKS WERE PRESENT ON THE SURFACE.

III.14.

COMPARISON OF Mo/Sn WITH Ti/Ag EVAPORATED CONTACT.

TO COMPARE THE V-I CHARACTERISTICS OF CELLS WITH Mo/Sn AND Ti/Ag METALLIZATION, CELLS WERE USED WITHOUT AR COATING AS WELL AS CELLS WITH AR COATING.

THE RESULTS ARE SHOWN ON THE ATTACHED GRAPHS. (GRAPHS 10 & 11)

IN ORDER TO APPROACH THE CHARACTERISTICS OF THE AR COATED CELLS, CELLS METALLIZED WITH Mo-Sn INK WERE COATED WITH Ti RESINATE, DILUTED IN 10 TO 1 RATIO WITH ALCOHOL AND DRIED AT 110°C.

THE RESULTS ARE SHOWN ON THE ATTACHED TABLE AND GRAPH.

TABLE III.

VOC AND I_{sc} VALUES OF N/P CELLS (2x2cm)
 (Mo-Sn INK, 525°C PEAK.)

CELL #	Voc (V)		I _{sc} (mA)	
	BEFORE AR COATING	AFTER AR COATING	BEFORE AR COATING	AFTER AR COATING
1	.575	.581	84	120
2	.580	.585	86	116
3	.581	.582	84	116
4	.576	.578	85	118
5	.572	.575	85	120
6	.576	.576	85	118
7	.575	.578	87	120
8	.576	.580	84	118
9	.580	.581	87	120
10	.575	.576	86	116
Ti/Ag #1	.582	.582	85	118
Ti/Ag #2	.582	.582	86	122

V-I CHARACTERISTICS OF NYP CELLS
METALLIZED BY Mo-Sn. (525°C PEAK)
(NO AR COATING)

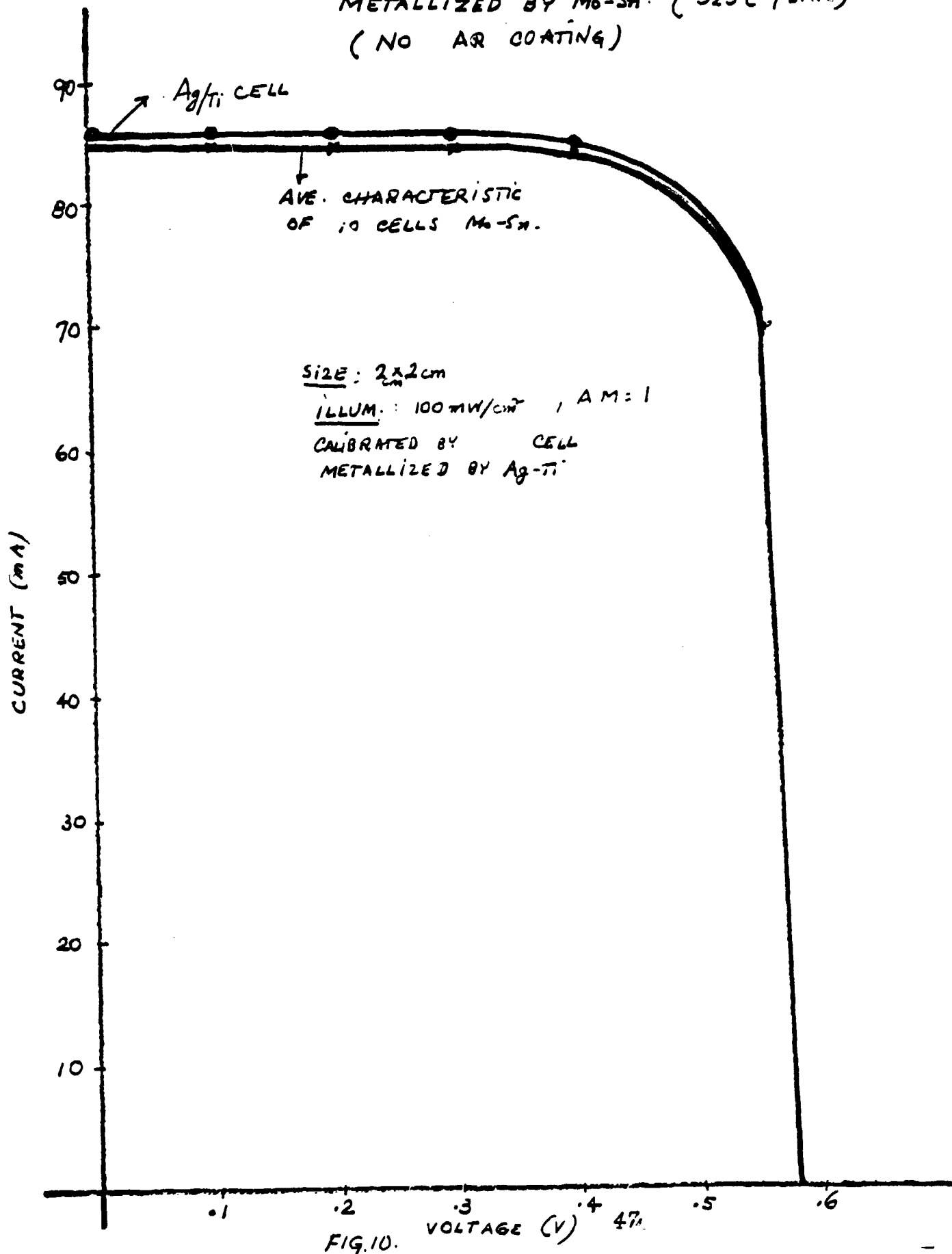
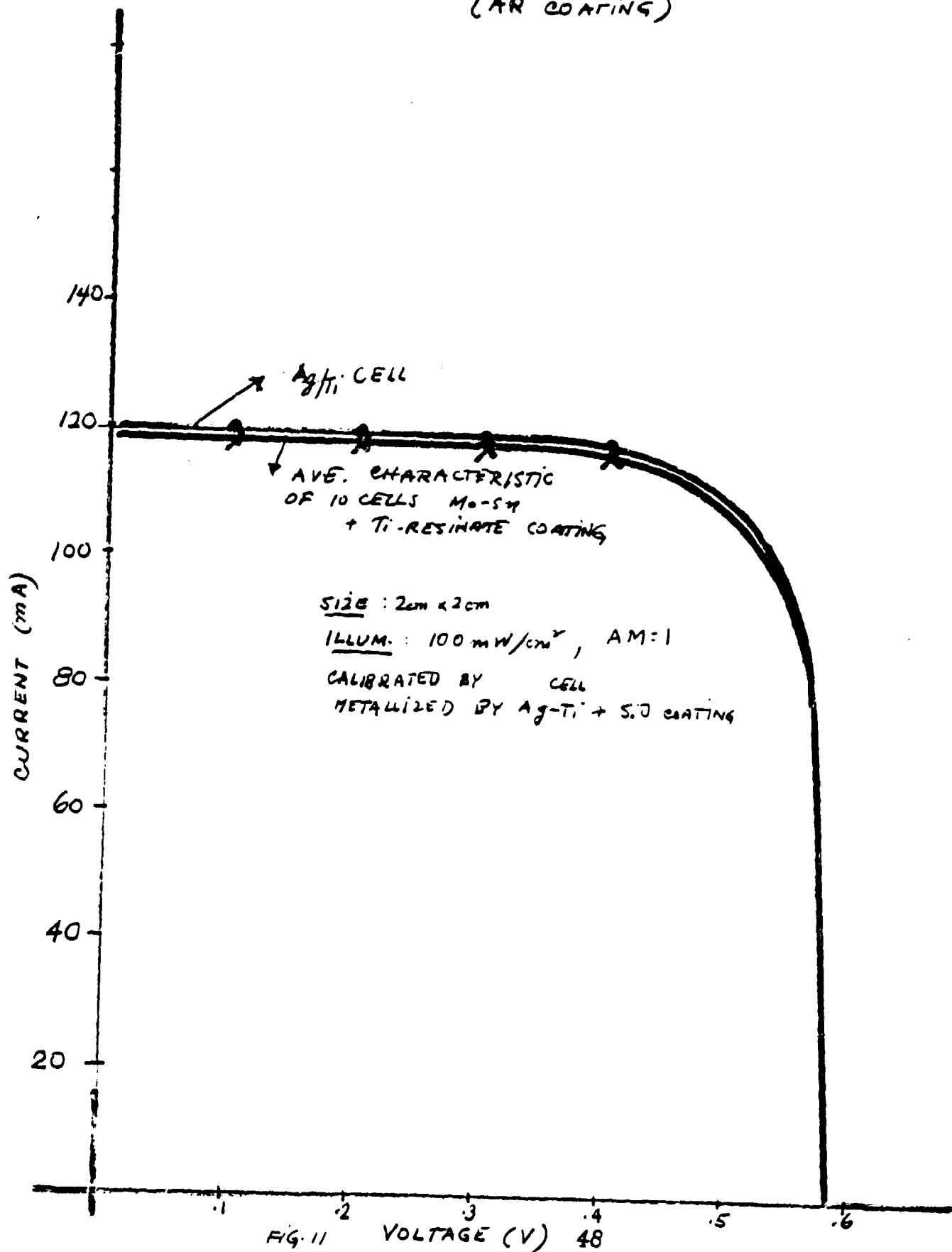


FIG. 10.

V-I CHARACTERISTICS OF N/P CELLS
METALLIZED BY MO-SN (525°C PEAK)
(AR COATING)



THE CONCLUSION OF THIS TEST WAS THAT THE MO-SN METAL-
LIZATION WAS ELECTRICALLY EQUIVALENT TO AG-TI METAL-
LIZATION.

MECHANICALLY, HOWEVER, THE MO-SN CONTACTS FIRED AT 525°C
PEAK TEMPERATURE WERE WEAKER THAN THE TI-AG CONTACTS.
A CLEANING PROCESS STILL REMAINS TO BE WORKED OUT TO
REMOVE LOOSE AL PARTICLES FROM WAFERS WITH AL BACK-
SURFACE FIELD, SO THAT A HIGHER TEMPERATURE FIRING
CYCLE CAN BE EMPLOYED IN ORDER TO IMPROVE THE MECHA-
NICAL BOND.

IV. COST ANALYSIS OF Mo-Sn METALLIZATION PROCESS.

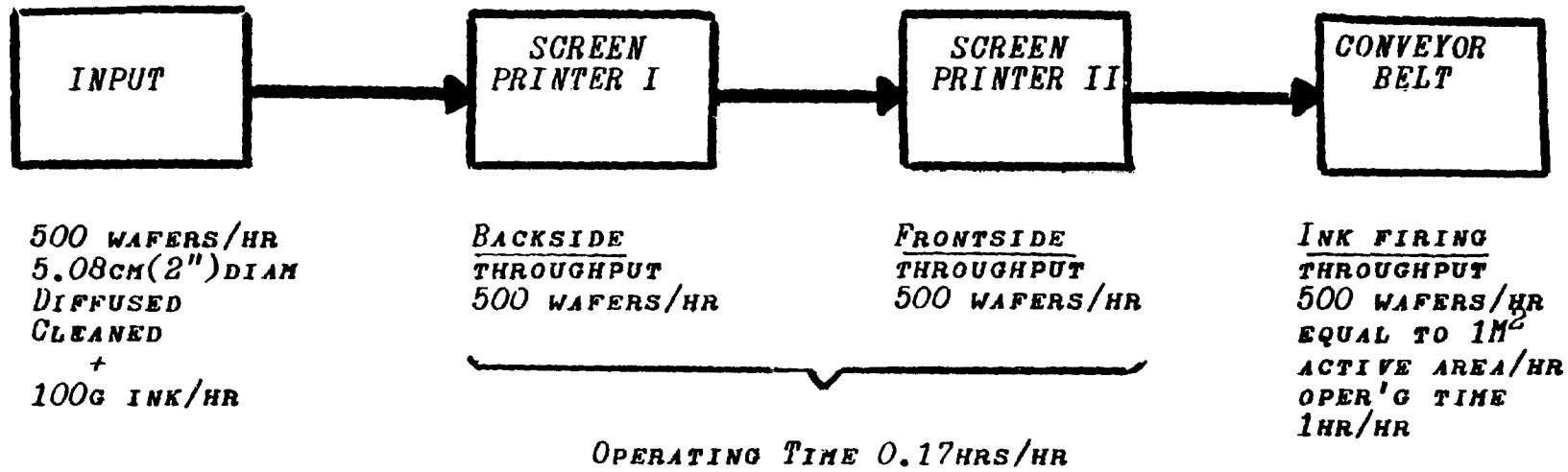
THE COST ANALYSIS OF THE NEW METALLIZATION METHOD IS BASED ON A CONTINUOUS MANUFACTURING PROCESS, USING AUTOMATIC SCREEN PRINTERS AND A CONVEYOR BELT FURNACE.

THE THROUGHPUT FOR THIS CALCULATION IS 500 DIFFUSED WAFERS/HR, 5.08 CM (2") DIAM. ON A FULL YEAR (8280 HOURS) MANUFACTURING SET-UP.

THE COST FOR MATERIAL AND EQUIPMENT ARE ACTUAL CURRENT VALUES, OBTAINED FROM SOURCES OF INDUSTRIAL SUPPLIERS AND MANUFACTURERS.

THE OUTPUT OF 500 CELLS/HR OF 5.08 CM (2") DIAM CORRESPONDS TO 1 M²/HOUR OF SOLAR CELL ACTIVE AREA AND BASED ON 10 TO 12% EFFICIENCY GENERATES APPROX 1 MEGAWATT PER YEAR.

METALLIZATION PROCESS FLOW



IV. 1. INPUT SPECIFICATIONS

MATERIAL:

P/N OR N/P DIFFUSED WAFERS

SI-SINGLE CRYSTAL, 1-1-1, OR 1-0-0 ORIENTATION

DIMENSIONS:

5.08 CM (2") DIAM.

0.0254-0.0304 CM (0.010-0.012") THICKNESS

QUANTITY REQUIRED:

500 WAFERS/HOUR

UNIT COST:

\$ 2.10, BASED ON THE PRESENT PRICE FOR 2" DIAM.

SINGLE CRYSTAL SILICON WAFERS

INPUT COST:

\$ 1050.00/HOUR

WAFERS ARE CLEANED BY THE 2-WAFER CLEANING PROCESS

OUTPUT RATE:

500 WAFERS/HOUR

IV. 2. DIRECT MATERIAL

TYPE:

Mo/SN SCREENABLE INK (FOR 500 WAFERS)

QUANTITY REQUIRED:

100G/HOUR

UNIT COST:

\$ 0.03/g

COST:

\$ 3.00/HOUR

INK COMPOSITION

DIRECT MATERIAL COSTS FOR MAKING INK.

MATERIAL	% BY WT.	DENSITY (G/CM ³)	% BY VOL.	COST		SOURCES	COST (\$) IN 100G INK
				\$/LB	\$/KG		
SN (-325 MESH)	62.4	7.3	29.6	8.00	17.60	ALCAN METAL POWDER, INC.	1.10
MoO ₃	17.6	4.5	13.0	4.46	9.81	CLIMAX MOLYB- DENUM Co.	0.173
ETHYL- CELLULOSE	1.2	1.1	3.2	3.50	17.70	DOW CHEMICAL	0.009
TRICHLORO- ETHYLENE	4.0	1.47	8.0	0.34	0.748	L.A. CHEMICAL Co.	0.003
TERPINEOL	14.0	0.935	44.0	1.60	3.54	ORANGE COUNTY CHEMICAL Co.	0.05
CARBITOL- ACETATE	0.8	1.0125	2.4	0.68	1.50	ORANGE COUNTY CHEMICAL Co.	0.0012
TITANIUM- RESINATE	1 PT/ 50000 MoO ₃ +SN	---	TRACES	31.80	70.00	ENGELHARD IN- DUSTRIES	TRACES

TOTAL DIRECT MATERIAL COSTS IN 100 GRAMS OF INK

\$ 1.34

MANUFACTURING CHARGE FOR INK FORMULATION OBTAINED BY METZ METAL-
LURGICAL CORP.: \$ 0.40/OZ OR \$ 1.42/100G

COST OF 100G OF INK: \$ 2.76

DIRECT MATERIAL COSTS PER CELL

BASED ON 2" DIAM. WAFER

COVERAGE: 100% BACK SIDE
10% ACTIVE SIDE

TOTAL AREA COVERED BY INK: 22 cm²

SCREENED THICKNESS : 0.00254 cm (0.001")

TOTAL VOLUME OF SCREENED INK/CELL: 0.0558 cm³

AMOUNT OF MATERIALS IN 0.0558 cm³ INK.

		Costs (\$)
SN	0.12g	2×10^{-3}
MoO ₃	0.03g	2×10^{-4}
ETHYLCELLULOSE	0.0007g	5×10^{-7}
TRICHLOROETHYLENE	0.0147g	1×10^{-5}
TERPINEOL	0.019g	6.7×10^{-5}
CARBITOL ACETATE	0.0012g	1.8×10^{-6}
TITANIUM RESINATE (TRACES)		TRACES
WEIGHT OF INK PER CELL	0.1856g	TOTAL <u>0.002278 \$/CELL</u>

IV. 3. INDIRECT MATERIAL

TYPE:

FORMING GAS, 60% N TO 40% H, MIXED FROM INDIVIDUAL GASES.

SPECIFICATIONS:

NEEDED CONTINUOUSLY FOR CONVEYOR BELT FURNACE
TO FIRE METALLIZATION INK.

QUALITY REQUIRED:

900 LITERS/HOUR OF N & 600 LITERS/HOUR OF H
(STANDARD FORMING GAS FLOW: 25 L/MIN
I.E. 15 L/MIN N & 10 L/MIN H)

UNIT COST:

H - \$1.35/100 FT³ (2832 L) + \$500/MO TANK RENTAL
N - \$0.66/100 FT³ (2832 L) + \$270/MO TANK RENTAL

COST:

H - \$ 0.98/HOUR
N - \$ 0.59/HOUR

TOTAL COST: \$ 1.57/HOUR

COST INFORMATION PROVIDED BY AMWELD INC.

IV. 4. EQUIPMENT

IV. 4.1 TYPE: 1 THICK FILM SCREEN PRINTER FOR BACK-SCREENING

MODEL: AEROLIX TECHNICAL CORP. OR AFFILIATED

MANUFACTURERS.

CAPACITY: 3000 WAFERS/HOUR

COST: \$ 15,000

THROUGHPUT: 500 WAFERS/HOUR

PLANT OPERATING TIME: 8280 HOURS/YEAR

MACHINE OPERATING TIME: 1375 HOURS/YEAR OR

16.6% OF PLANT OPERATING TIME

USEFUL LIFE: 7 YEARS

CHARGE RATE: 21.35% OF COST/YEAR

CAPITAL COST: \$ 3202.50/YEAR

PARTS OR OUTSIDE SERVICE: \$ 2000/YEAR

SALVAGE VALUE: \$ 3,000 (20% OF COST)

TOTAL COST: \$ 0.63/HOUR

IV. 4.2 TYPE: 1 THICK FILM SCREEN PRINTER FOR FRONT-SCREENING

MODEL: AEROLIX TECHNICAL CORP. OR AFFILIATED

MANUFACTURERS.

CAPACITY: 3000 WAFERS/HOUR

COST: \$ 15,000

THROUGHPUT: 500 WAFERS/HOUR

PLANT OPERATING TIME: 8280 HOURS/YEAR

MACHINE OPERATING TIME: 1375 HOURS/YEAR OR

16.6% OF PLANT OPERATING TIME

USEFUL LIFE: 7 YEARS

CHARGE RATE: 21.35% OF COST/YEAR

CAPITAL COST: \$ 3202.50

PARTS OR OUTSIDE SERVICE: \$ 2000/YEAR

SALVAGE VALUE: \$ 3,000 (20% OF COST)

TOTAL COST: \$ 0.63/HOUR

IV. 4.3 TYPE: CONVEYOR BELT FURNACE

MODEL: THERMO OR WATKINS JOHNSON

COST: \$ 30,000

INSTALLATION COSTS: INCLUDED

THROUGHPUT: 480 WAFERS/HOUR

PLANT OPER'G TIME: 8280 HOURS/YEAR

MACHINE OPERATING TIME: 7838 HOURS/YEAR OR

95% OF PLANT OPERATING TIME

USEFUL LIFE: 7 YEARS

CHARGE RATE: 21.35% OF COST/YEAR

CAPITAL COST: \$ 6400/YEAR

PARTS OR OUTSIDE SERVICE: \$ 2000/YEAR

SALVAGE VALUE: \$ 6,000 (20% OF COST)

TOTAL COST: \$ 1.01/HOUR

IV. 5. ENERGY

TYPE:

ELECTRICITY

A) SCREEN PRINTERS: 800 WATTS (400 WATTS EA)
17 % DUTY CYCLE

QUANTITY REQUIRED: 0.136 KWATTS

UNIT COST: \$ 0.06/KWH

COST: \$ 0.0081/HR

B) CONVEYOR BELT FURNACE: 20 KWATTS, CONTINUOUS.

QUANTITY REQUIRED: 20 KWATTS

UNIT COST: \$ 0.06/KWH

COST: \$ 1.20/HOUR

TOTAL ENERGY COST: \$ 1.21/HOUR

IV. 6. DIRECT LABOR

IV. 6.1. CATEGORY: SEMICONDUCTOR ASSEMBLER

ACTIVITY: OPERATION & MAINTENANCE OF SCREEN PRINTERS

RATE: \$ 5.00/HR AMOUNT REQUIRED: 0.17HOURS/HOUR

LOAD: 36%

COST: \$ 1.15/HR

IV. 6.2. CATEGORY: SEMICONDUCTOR ASSEMBLER

ACTIVITY: OPERATION & MAINTENANCE OF SCREEN PRINTERS

RATE: \$ 5.00/HR AMOUNT REQUIRED: 0.17HOURS/HOUR

LOAD: 36%

COST: \$ 1.15/HR

IV. 6.3. CATEGORY: SEMICONDUCTOR ASSEMBLER

ACTIVITY: OPERATION AND MAINTENANCE OF CONVEYOR BELT
FURNACE

RATE: \$ 5.00/HR AMOUNT REQUIRED: 1HOUR/HOUR

LOAD: 36%

COST: \$ 6.80/HR

IV. 6.4. CATEGORY: MAINTENANCE MECHANIC

ACTIVITY: SERVICE & REPAIR

AMOUNT OF TIME REQUIRED: 0.1 HOUR/HOUR

RATE: \$ 8.00/HR

LOAD: 36%

COST: \$ 1.10/HR

TOTAL DIRECT LABOR COST: \$ 10.20/HR

INDIRECT LABOR

TAKEN AS 25% OF DIRECT LABOR

COST: \$ 2.55/HR

SUB-TOTAL COST OF DIRECT + INDIRECT LABOR: \$ 12.75/HR

OVERHEAD ON LABOR: 5.26%

COST: \$ 0.67/HR

SUB-TOTAL LABOR COSTS: \$ 13.42/HR

IV. 7. FACILITIES

IV. 7.1 TYPE: FURNACE AREA

FLOOR AREA: 30 M²

CHARGE RATE: \$ 179.13/(M².YR)

(THIS INCLUDES ENERGY COSTS FOR HEATING, AIR-
CONDITIONING & LIGHTING)

COST: \$ 5373.90/YR --- \$ 0.65/HR

IV. 7.2 TYPE: SCREEN PRINTERS AREA

FLOOR AREA: 4 M²

CHARGE RATE: \$ 179.13/(M².YR)

COST: \$ 716.52/YR --- \$ 0.26/HR

SUB-TOTAL FACILITIES COST: \$ 0.91/HR

IV. 8. PROCESS COST COMPUTATION.

MATERIALS (DIRECT + INDIRECT)	\$ 4.57/HR
ENERGY	\$ 1.21/HR
LABOR (DIRECT + INDIRECT)	\$ 13.42/HR
EQUIPMENT + FACILITIES	\$ 3.18/HR
TOTAL	\$ <u>22.38/HR</u>
THEORETICAL LOSS IN PROCESS (BREAKAGE) (1% OF \$ INPUT/HR)	\$ <u>10.50/HR</u>

TOTAL COST \$ 32.88/HR

IV. 9. OUTPUT SPECIFICATIONS

ITEM: PHOTOVOLTAIC SOLAR CELL

DIMENSIONS: 5.08 CM (2") DIAM, 0.025-0.03 CM

(0.01-0.012") THICKNESS

MATERIAL: HIGH PURITY SILICON

V. SUMMARY

IT HAS BEEN PROVEN EXPERIMENTALLY DURING THIS PROGRAM THAT THE OHMIC CONTACT TO SILICON SOLAR CELLS CAN BE OBTAINED FROM Mo/Sn METAL SYSTEM BY REDUCTION OF MoO_3 -Sn MIXTURE.

IN ORDER TO LOWER THE CONTACT RESISTANCE WHILE MAINTAINING THE PEAK TEMPERATURE AROUND 800°C , THE BASIC MoO_3 -Sn FORMULATION WAS MODIFIED BY AN ADDITION OF TITANIUM IN THE FORM OF TITANIUM RESINATE.

A VERY CRITICAL PART IN THE METALLIZATION PROCESS WAS THE RATE OF COOLING DURING THE FIRING CYCLE, SPECIFICALLY AROUND 450°C .

THE IMPLEMENTATION OF THIS METHOD TO THE AUTOMATED CONVEYOR FIRING PROCESS WILL REQUIRE THEN THE DETERMINATION OF THE FIRING CYCLE WITH A SPECIAL ATTENTION TO THE COOLING RATE.

THE CHARACTERISTICS OF CELLS METALLIZED WITH THIS METAL SYSTEM WERE COMPARABLE WITH THE CHARACTERISTICS OF CELLS METALLIZED WITH THE STANDARD METHODS (PLATED Ni, SCREENED Ag AND EVAPORATED Ti/Ag).

SINCE THE PROCESS USES STANDARD MANUFACTURING EQUIPMENT SUCH AS USED IN THE ESTABLISHED SILK SCREENING PROCESSES, THE ADVANTAGE OF THIS NEW METHOD RESULTS FROM THE COST SAVING ON MATERIALS USED IN THE FORMULATION OF THE INK IN ADDITION TO THE TECHNICAL ASPECT

OF USING STRICTLY A METALLIC SYSTEM FOR THE FORM-
ATION OF THE CONTACT.

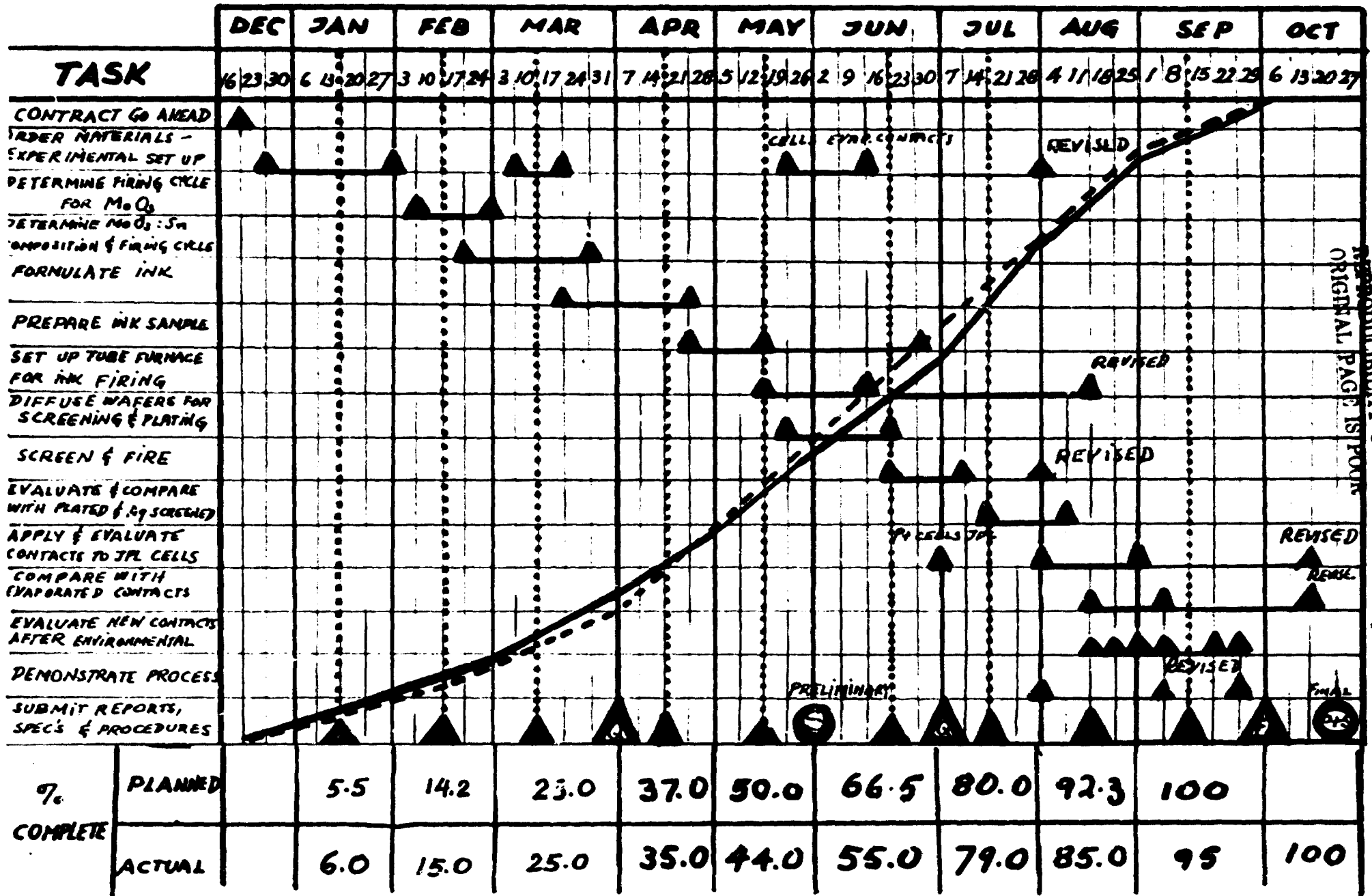
CONTRACT No: 955318

PROGRAM PLAN

FOR

SOL/LOS INCORPORATED
 2231 S. CARMELITA AVE.
 LOS ANGELES CA. 90064
 (213) 880-5181
 12/27/78

A NEW METHOD OF METALLIZATION FOR SILICON SOLAR CELLS



REPRODUCIBILITY OF THE ORIGINAL PAGE IS POOR