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RUGGED THIN GAS SOLAR CELL DEVELOPMENT PHASE I(U)

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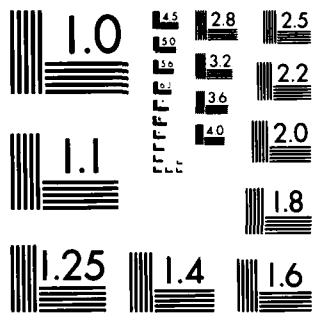
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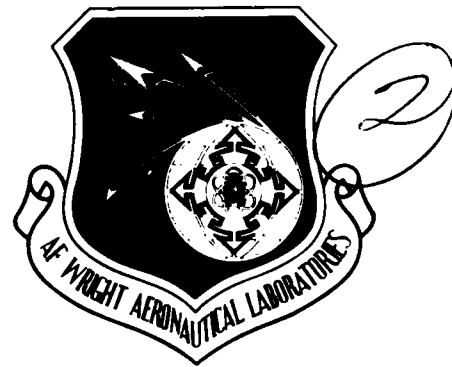
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**RUGGED, THIN GaAs SOLAR CELL DEVELOPMENT**

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## PHASE I - CELL FABRICATION

### 1.0 INTRODUCTION

The overall objective of this program is to develop the technology for fabricating thin layer, high efficiency GaAs solar cells atop thin germanium substrates (3 mils). The specific goals for the program are:

- An Air Mass Zero (AMO) conversion efficiency of 18% for beginning-of-life (BOL) at  $27 \pm 2^{\circ}\text{C}$ .
- An AMO conversion efficiency of 13.5% at  $27 \pm 2^{\circ}\text{C}$  after exposure to  $5 \times 10^{15}$  (MeV) electrons/cm<sup>2</sup>.
- A cell weight not to exceed 0.05 grams per square centimeter of cell area.

The overall program consists of three phases:

- 1) Phase I - Cell Fabrication
- 2) Phase II - Cell Optimization
- 3) Phase III - Cell Testing

### 2.0 SUBSTRATE EVALUATION

Since this program is oriented to develop the manufacturing technology for mass producing rugged thin GaAs solar cells, suitable substrate material must be available, and key properties such as resistivity, dislocation density, crystal orientation, dopant element, size of wafer, thickness of wafer, price and delivery schedule are all of concern. We contacted four U.S. vendors (Atomergic Chemicals Corp., Materials by Metron Inc., Eagle-Pitcher and Semi-Alloys, Allied Electronic Components) to survey the availability of germanium wafers.

Semi-Alloys of Allied Electronic Components indicated that they no longer make single crystal germanium. The other two vendors (Materials By Metron Inc., and Atomergic Chemicals) could not give competitive quotations. During the course of contacts, we learned that the germanium market in the U.S. is dominated by one single vendor - Eagle Pitcher.

Therefore, realistically, we will deal with Eagle-Pitcher to obtain the needed germanium wafers unless there are any new developments in germanium supplies. In their quotation, Eagle-Pitcher indicated that to purchase germanium in the ingot form will cost \$4,253,40/kg in quantities of 5-kg and \$4,132.80/kg in quantities of 10-kg. Hence, we can see from this quotation that the price can be made for quantities of 5-kg or larger. We believe this is not a true price structure. The price is artificially fixed by the vendor due to the lack of true market demand this moment in time. By comparing the price per sliced wafer vs. price per kg, we found that it is more expensive to buy the material in ingot form to produce wafers (for a sliced wafer 0.05cm thick with assumed 0.05cm kerf loss during sawing) than to purchase sliced wafers directly. This discrepancy is due to the fact that Eagle-Pitcher is able to reclaim the germanium in the saw dust and reuse it.

In order to have good material utilization and to obtain good throughput for the OM-CVD growth system, rectangular-shape wafers are most desirable. Eagle-Pitcher indicated that at a quantity of 100,000 2.5cm x 4.5cm x 0.05cm - wafers per year, the price per wafer will be \$8.99.

To obtain additional germanium needed for this project, we have discussed with Eagle Pitcher the possibility of obtaining germanium materials of different sizes (3-in diameter round wafer, 2.5cm x 2.5cm wafer, 4.5cm x 4.5cm wafer, 3-in round ingot and 2.5cm x 4.5cm ingot) in small quantities.

A typical quotation for small quantities of Ge is:

A. The unit costs per germanium wafer are as follows (15-20 mil thick):

1. 3-Inch Diameter Wafers:

\$62.15 per wafer @ quantity of 10

\$53.44 per wafer @ quantity of 20

2. 2x5cm x 4.5cm Wafers:

\$21.99 per wafer @ quantity of 20

\$18.01 per wafer @ quantity of 40

\$16.68 per wafer @ quantity of 60

\$16.01 per wafer @ quantity of 80

3. 4.5cm x 4.5cm Wafers:

\$40.23 per wafer @ quantity of 10

\$31.71 per wafer @ quantity of 20

B. The costs of germanium ingots are as follows:

1. 3-inch Diameter Ingots:

\$758.41 for 10cm length.

\$1,359.42 for 20cm length.

2. 2.5cm x 4.5cm Ingots:

\$339.64 for 1cm length.

\$521.16 for 2cm length.

\$702.67 for 3cm length.

\$884.19 for cm length.

After studying the price structure and our needs for substrates at this time, we placed an order with Eagle-Pitcher Industries, Inc. for 60 single crystal germanium wafers,  $2.5 \pm 0.02\text{cm} \times 4.5 \pm 0.02\text{cm}$ , 15-20 mil thick, and for a single crystal germanium ingot,  $2.5 \pm 0.02\text{cm} \times 4.5 \pm 0.02\text{cm}$ , 4cm thick. Both germanium materials were to be oriented (100) off  $2 \pm 1$  deg. toward (110) direction, resistivity 0.02-0.8 ohm-cm and EPD (etch-pit-density) less than  $5000\text{cm}^{-2}$ . The reason for purchasing germanium ingots, in addition to the wafers, was to test the feasibility of producing very thin germanium wafers by slicing. This will reduce effort for subsequent cell thinning.

After some delays, a suitable supply of substrates was obtained, to allow the necessary tests.

### 3.0 SUBSTRATE THINNING

We started working on tests of etch-thinning of thick Ge substrates, concentrating on the use of different chemical etchants to provide planar thinned Ge substrates. The etch rate and the influence of the surface finish of the Ge was investigated, while parallel work was proceeding on the feasibility of combining mechanical lapping with chemical thinning.

Early experiments used single sided polished Ge substrates, 1cm x 1cm area and 12 mil thick. Two etchants were tried. The first etchant consisted of 10% by volume of 100ml  $\text{H}_2\text{O}_2$  and 8gm of NaOH. The mixed solution was heated up to  $70^\circ\text{C}$  using a hot plate. Another etchant used was "CLOROX" at  $40^\circ\text{C}$ . The polished front surface of the Ge substrates were coated with CVD  $\text{SiO}_2$  (5000A). This  $\text{SiO}_2$  layer was used for protecting the specular front surface from chemical etching and corrosion. Each sample was examined by visual

inspection followed with thickness measurement after every 5 minutes etching time. It was found that the NaOH and  $H_2O_2$  mixed solution had an etch rate of 2.5 $\mu$ m/min. during the first 5 minutes of etching. The etchant also removed the  $SiO_2$  layer and destroyed the mirror-like front surface after 20 minutes etching. The "CLOROX" solution had approximately the same etching rate as the first solution, but it did not attack the  $SiO_2$  protective layer. Hence, the front surface was kept intact during the etching process. Later, it was determined that a combination of mechanical and chemical polishing technique is best suited for thinning to 2-3 mils thickness while maintaining surface flatness.

### 3.1 Etch Polishing

Published work describes several polishing or thinning chemical etches, including mixtures of HF,  $HNO_3$  and  $CH_3COOH$  (acetic acid), called CP4A, CP6, CP8. When Br is added to CP4A, it is called CP4. Also NaOH/ $H_2O_2$  mixtures at 70°C and diluted NaOCl at 40°C have been used, but we found the etch rate of these etchants were too slow.

Our experience with Si etch-polishing suggested that a fairly high etch-rate is needed to provide good polish. We therefore tested polish-etch solutions used successfully for Si (Ge and Si are both group IV elements, the chemical properties should be reasonably similar), mainly mixtures of HF,  $HNO_3$  and  $CH_3COOH$ . A mixture of 2HF:15  $HNO_3$ : 5 $CH_3COOH$  used for Si polishing was found not too satisfactory. However, after testing several mixture ratios, we found that a 1:2:2 mixture of HF: $HNO_3$ :  $CH_3COOH$  was promising (etch rates of 14-18 micrometers/min. per face) and a 1:2:1 mixture (etch rate about 14 micrometers/min. per face) was slightly better.

At this stage, another important variable was involved, the ratio of Ge surface area to the volume of acids. When this ratio increased, the etchant temperature increased, leading to faster etch rates (we reduced localized hot spots by vigorously agitating the slices in the etchant). Also, we generally started with as-sawn Ge surfaces which gave high etch rate, this rate decreasing as the surfaces became more polished.

Using the 1:2:2 mixture we had some success in providing some germanium slices about 250-300 micrometer thick with a fairly specular finish (with orange-peel texture), and also in thinning (and polishing) some germanium slices to 75-100 micrometers thickness.

In addition, we tried mechanical polishing of some germanium slices (with variable polish over the surface). We modified a machine used to chemically-mechanically polish Si to accept rectangular Ge slices. The modification involved first, the design and purchase of plastic templates of different thicknesses, with rectangular slice holding pockets, and later, the use of different polishing media. For the latter, we first tried the standard "Syton" (colloidal  $\text{SiO}_2$ ) method; later we tried other materials because the system did not give a good polish.

Using "**CLOROX**" and selected polishing pads, after empirical tests of solution concentration, pad material and polishing pressures, we achieved good surface finish after removing about 75 $\mu\text{m}$  from as-received Ge wafers. However, there were still ripples and dimples on the surface.

Later modifications of pads and lower concentration "CLOROX" solutions gave good polish. Three groups of germanium samples were prepared by such a polishing technique, with 75-micrometer, 100-micrometer and 125 micrometer surface layers being removed. All three groups of germanium samples had excellent mirror-like surface with no ripple or haze when viewed by 200X dark-field microscope. The reasons for removing different surface layer thicknesses were two fold: 1) to find how much material need be removed from the surface of the germanium to remove the surface damage due to ingot slicing; and 2) to check whether the additional thickness being removed from the surface will introduce excessive surface rounding or other undesirable effects. The results indicated that additional material could be removed without severe sounding effects. This is significant because it indicates that we will be able to thin the wafer thickness down either pre- or post-growth of GaAs solar cell structure, which offers us greater flexibility in producing the final cell structure.

#### 4.0 OMCVD GROWTH ON SUBSTRATES

By using the substrate thinning techniques above, we prepared a few germanium wafers ranging in thicknesses from 75 micrometers to 300 micrometers. Subsequently, p on n GaAs structures were grown on those samples. The intention here was not to produce a solar cell structure, but to evaluate the hetero-epitaxial layer growth of GaAs on Ge. We successfully produced samples of GaAs on Ge with surface finish closely resembling the initial surface of germanium.

After these promising results, progress was slowed while new working areas were being constructed near the OMCVD system. Following this delay we have made a p on n AlGaAs/GaAs heteroface homojunction layer structure growth on

germanium wafers obtained by using the technique described above. The intended thicknesses for the p-AlGaAs window layer, GaAs emitter and n-GaAs buffer layer were about 0.1 micrometer, 0.45 micrometer and 9 micrometers, respectively. The doping concentration for the p-GaAs emitter and the n-GaAs buffer layer were about  $4 \times 10^{18} \text{ cm}^{-3}$  and  $4 \times 10^{17} \text{ cm}^{-3}$ , respectively.

We later found that these Ge substrates were  $4^\circ$  off (100) rather than the specified  $2^\circ$ . Those substrates gave excellent surface morphology.

Subsequently, 2x2cm AlGaAs/GaAs/Ge solar cells were fabricated by using these wafers. The AMO energy conversion efficiency of greater than 15% was observed for the best, thus achieving the goal for Phase I. (Details of the cells are given below in Section 6.)

Later tests, making p on n cell structures on Ge wafers  $2^\circ$  off (100) towards (110) exhibited poor surface morphology. Under microscope observation, it appears that we experienced domain growth on those germanium wafers. It has been reported <sup>(1)</sup> that domain growth should not occur for crystal orientation of  $2-3^\circ$  off (100) toward (110) or (111) direction for GaAs on Germanium structure. We believe that the domain formation was due to the error in the cutting angle of the germanium wafers, which caused the orientation shift to less than  $2^\circ$  off (100) direction. To avoid such problems in the future, we promptly requested the germanium supplier to change the specification to  $4^\circ$  off (100) toward (110) direction for all future substrates.

Further tests showed epitaxial layers with excellent surface morphology when the orientation was more than  $2^\circ$  off (100).



## 5.0 LAYER EVALUATION

The hetero-epitaxial p-GaAs/n-GaAs/n-Ge wafers grown by OMCVD were evaluated using a Polaron C-V Profiler. The doping profile of the front p-layer was found to be about  $3 \times 10^{18} \text{cm}^{-3}$ , which is close to the target value. Similarly, the doping concentration of the n-GaAs layer was found to be about the same as our target value of  $2-4 \times 10^{17} \text{cm}^{-3}$ . More importantly, when we profiled the n-GaAs layer well into the GaAs/Ge interface, we saw no evidence of any significant auto-doping piling-up effect or any p-type auto doping effect. This is promising for making our final cell structure growth.

A carrier concentration vs. depth profile of the n-GaAs grown on n-Ge substrate is shown in Figure 1. From this figure, we can see that the interface between n-GaAs and n-Ge is at a depth of about 9 micrometers. The doping level change is very mild with the peak doping concentration of  $2.7 \times 10^{17} \text{cm}^{-3}$  (at about 7.2 micrometer) as compared to the minimum value of about  $1.4 \times 10^{17} \text{cm}^{-3}$ . The average doping concentration is very close to the targeted value of  $2-4 \times 10^{17} \text{cm}^{-3}$ .

Additional effort is described in Section 8 (Iterative Testing).

## 6.0 COMPLETED CELLS (CONVENTIONAL CONTACTS)

We processed the AlGaAs/GaAs/Ge wafers into completed solar cell structures. The cell evaluation results are discussed in 7.0. To date, the same solar cell processing sequence is applied to both conventional AlGaAs/GaAs solar cells and AlGaAs/GaAs/Ge solar cell. All process details, such as contact and coating design were similar, the contact to the back surface of the substrates was Au-Ge-Ni-Au-Ag, to the P-GaAs (through slots in the AlGaAs) was Au-Zn-An-Ag.

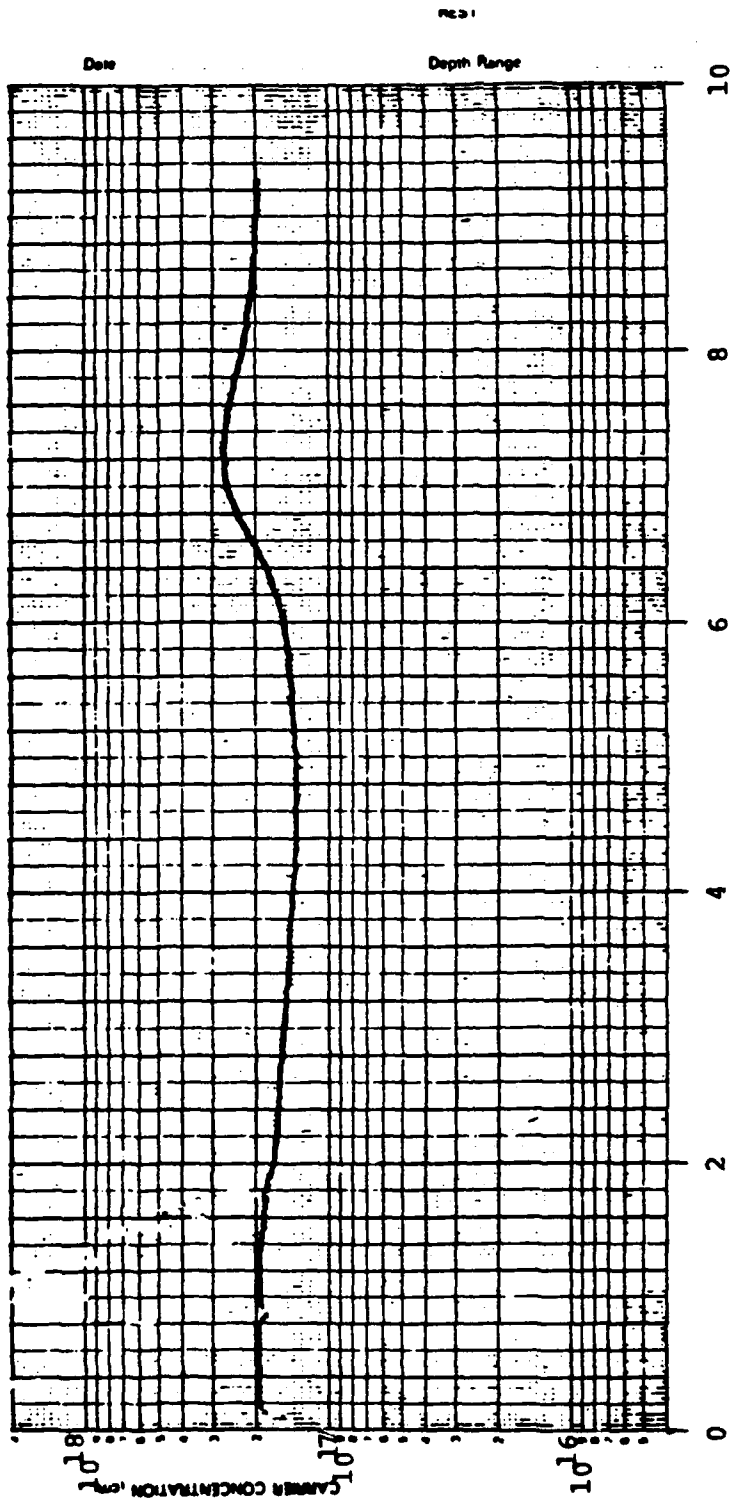


FIGURE 1  
n - GaAs/n-Ge PROFILE

The AR coating was two dielectric layers ( $\text{TiO}_x$ ,  $\text{Al}_2\text{O}_3$ ) deposited over the AlGaAs.

Despite problems with the AR coating machine, we obtained a few 2x2cm cells 15% efficient; this met the minimum goal, and further increases are described in Section 8.0.

## 7.0 CELL EVALUATION

Photovoltaic data was obtained when AlGaAs/GaAs/Ge wafers were processed into solar cells of 1cm x 2cm size. However, the first Ge samples were incorrectly oriented (see-above) and the cell results were poor; Voc was extremely low (270-330mV), and Jsc was also low,  $19\text{mA}/\text{cm}^2$  (AMO spectrum).

Using the  $4^\circ$  off-oriented Ge, we then made 2x2cm cells (AlGaAs/GaAs/Ge) with efficiencies 14%. Excellent open circuit voltages (greater than one volt) were obtained on those solar cells. Good short circuit current densities (about  $29\text{mA}/\text{cm}^2$ ) were also obtained. The AMO energy conversion efficiencies were about 14%. The light I-V characteristics of the best 2x2cm AlGaAs/GaAs/Ge solar cell in this group is shown in Figure 2. The AMO efficiency was 14.8%, which is close to the target efficiency of 15% AMO for Phase I. The other photovoltaic data associated with this cell are as follows: open circuit voltage 1.12 volt, short-circuit density -  $29.2\text{mA}/\text{cm}^2$ , and fill-factor - 0.615. The cell was 10 mil thick.

With these results we expect values for 1x1cm cells to be higher. However, additional runs gave even better results for 2x2cm cells.

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2x2cm

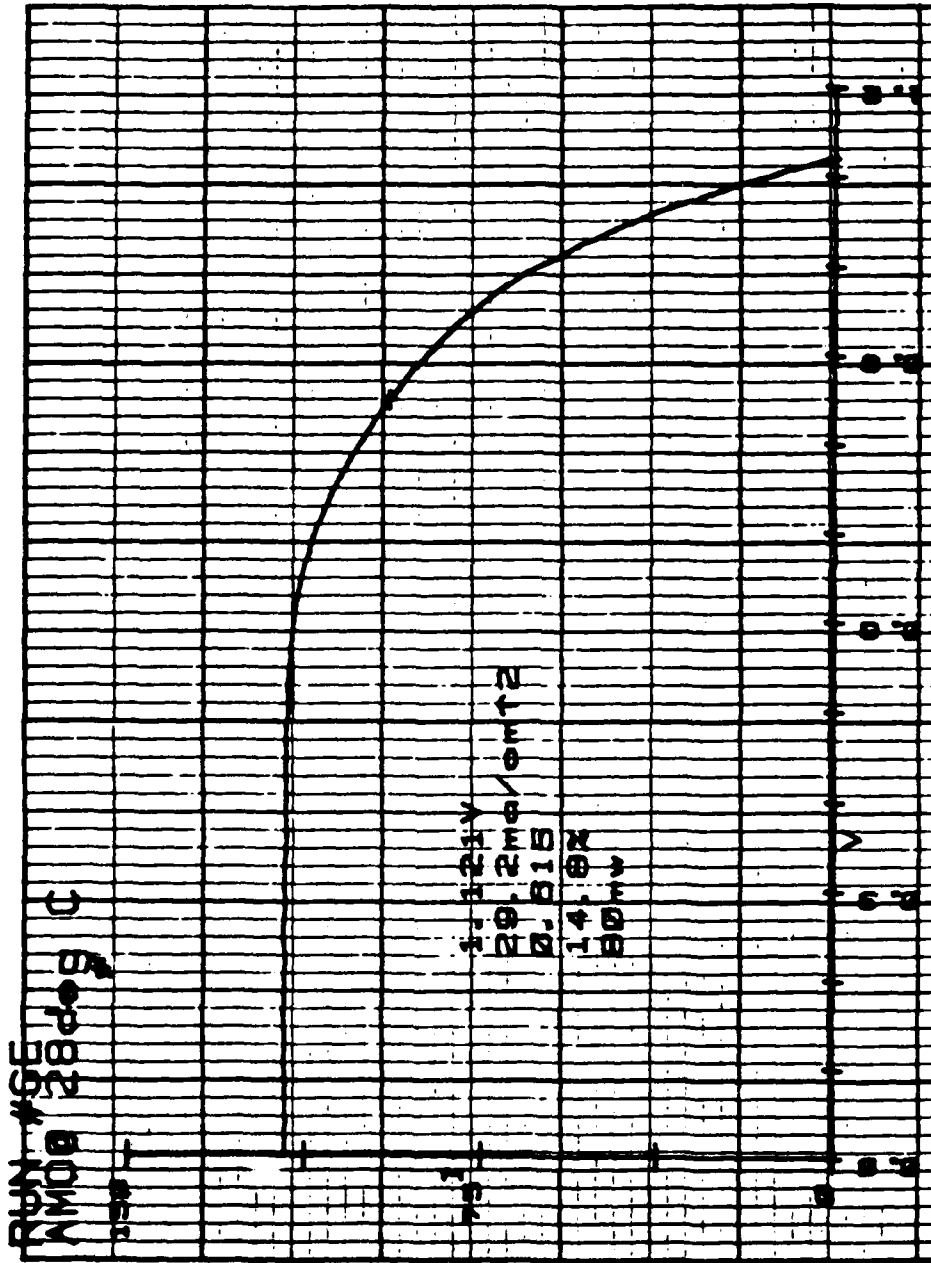


FIGURE 2  
AlGaAs/GaAs/Ge SOLAR CELL

The best AMO efficiency achieved in these later groups was 15.2%, which exceeded the target efficiency of 15% AMO. The cell was 8 mil thick. The other photovoltaic data associated with this cell were as follows: open-circuit voltage - 1.123V, short-circuit current density -  $24.7\text{mA}/\text{cm}^2$ , and fill-factor - 0.738 (see Figure 3a). The improvement in efficiency over that above was primarily due to the better fill factor value (0.738 vs. 0.615). The donor profile is shown in Figure 3b.

In the next test, photovoltaic data were obtained when the AlGaAs/GaAs on Ge wafers were processed into solar cells of  $2\times 2\text{cm}$  size before AR coating. Again, very good open-circuit voltages were obtained on those solar cells. Most (75%) of the solar cells tested have high open-circuit-voltage values. In order to rule-out an unexpected second cascade germanium junction in the AlGaAs/GaAs/Ge structure, we performed spectral response measurements in the wavelength region including photon energies below the bandgap of gallium-arsenide and greater than that of the bandgap of germanium. We observed no response in that wavelength region. Hence, we concluded that the observed high voltage value is not due to a cascade germanium junction. The high voltage obtained may be due to the better bulk crystalline material of germanium as compared to the bulk gallium-arsenide material, therefore, a better p-n junction with lower leakage current can be formed.

The average short-circuit-current-density before AR coating was  $22.5\text{mA}/\text{cm}^2$  for these cells. The projected current-density (after AR coating) was  $29.5\text{mA}/\text{cm}^2$ . This current-density is similar to values for GaAs cells made on gallium-arsenide wafers. The light I-V curve of one of these  $2\times 2\text{cm}$

AMO, 28 deg C, 2X2 CM

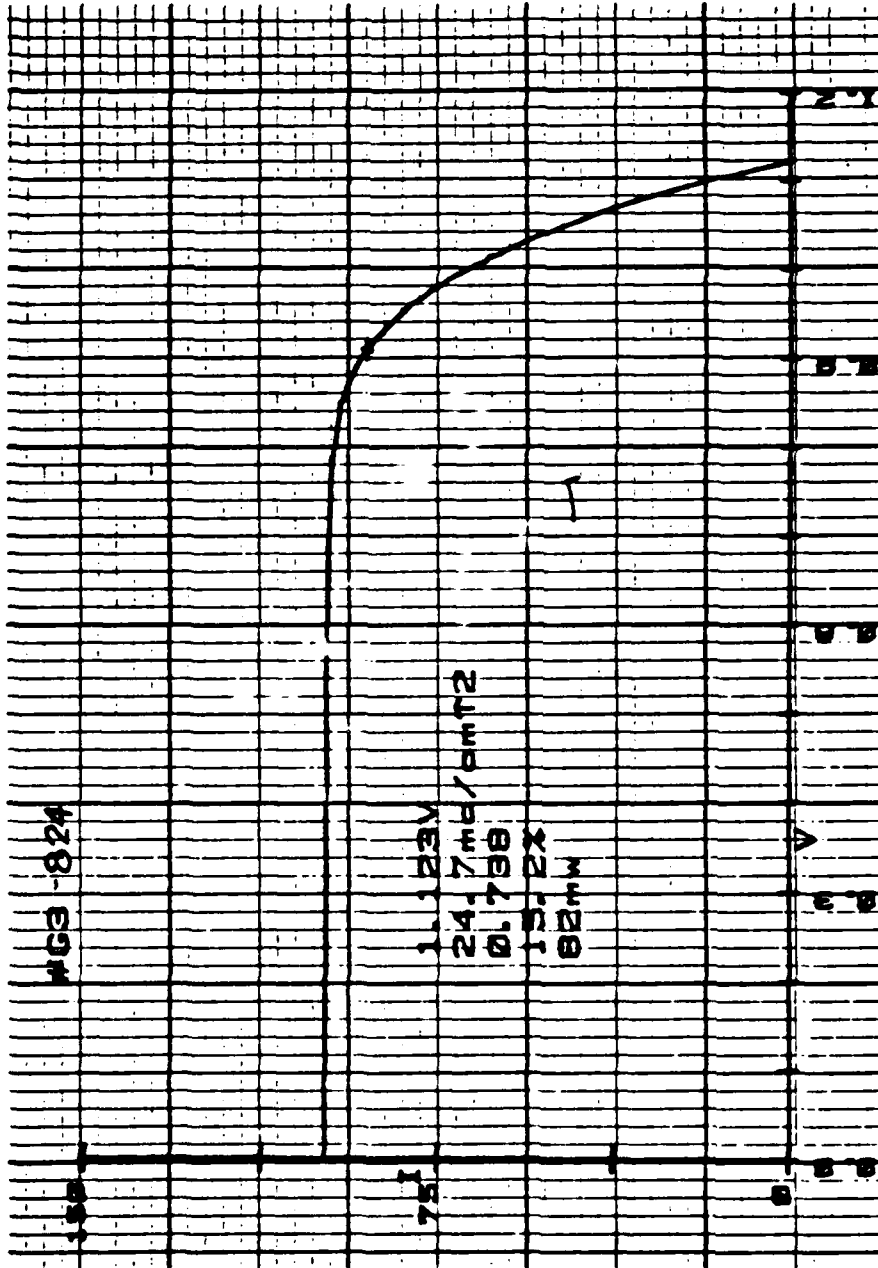
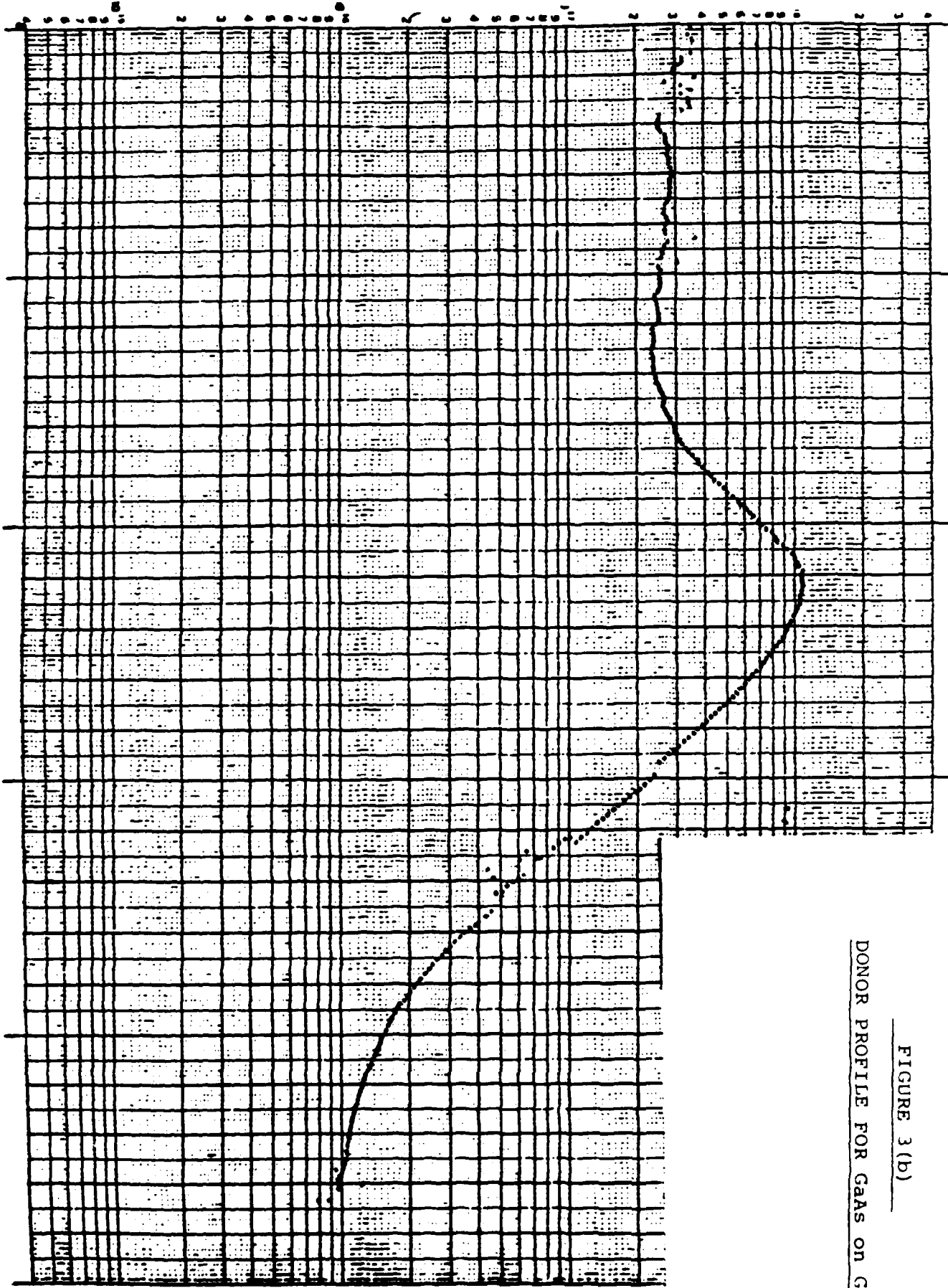


FIGURE 3 (a)

AlGaAs/GaAs/Ge SOLAR CELL



DONOR PROFILE FOR GaAs on Ge

FIGURE 3 (b)

AlGaAs/GaAs/Ge solar cell is included as Figure 4. The photovoltaic data associated with these cells were as follows:

- Open Circuit Voltage: 0.972V
- Short Circuit Current Density: 22.7 mA/cm<sup>2</sup>
- Fill Factor: 0.706
- AMO Efficiency: 11.5%

(All parameters before AR coating).

The projected efficiency after AR coating was 15% (mainly due to the current gain and partly due to the voltage gain).

Next, we deposited an AR coating on these solar cells. In addition, we made a few more 2x2cm size AlGaAs/GaAs/Ge solar cells from new GaAs growth runs on Ge substrates. After applying the AR coating layer on these cells, we made a few 2x2cm size solar cells with AMO efficiency greater than 14.6% (14.6% is the efficiency needed for the bare solar cell to satisfy the 15% glass-covered cell efficiency requirement, since the AR coating is designed to match the index of refraction of the cover glass rather than that of air). The light I-V characteristics of one of these solar cells is included as Figure 5. The photovoltaic data associated with this cell were:

- Open Circuit Voltage: 1.089V
- Short Circuit Density: 28.9mA/cm<sup>2</sup>
- Fill Factor: 0.640
- AMO efficiency: 14.9% (No Cover Glass)



AMO, 28 deg C, 2x2cm

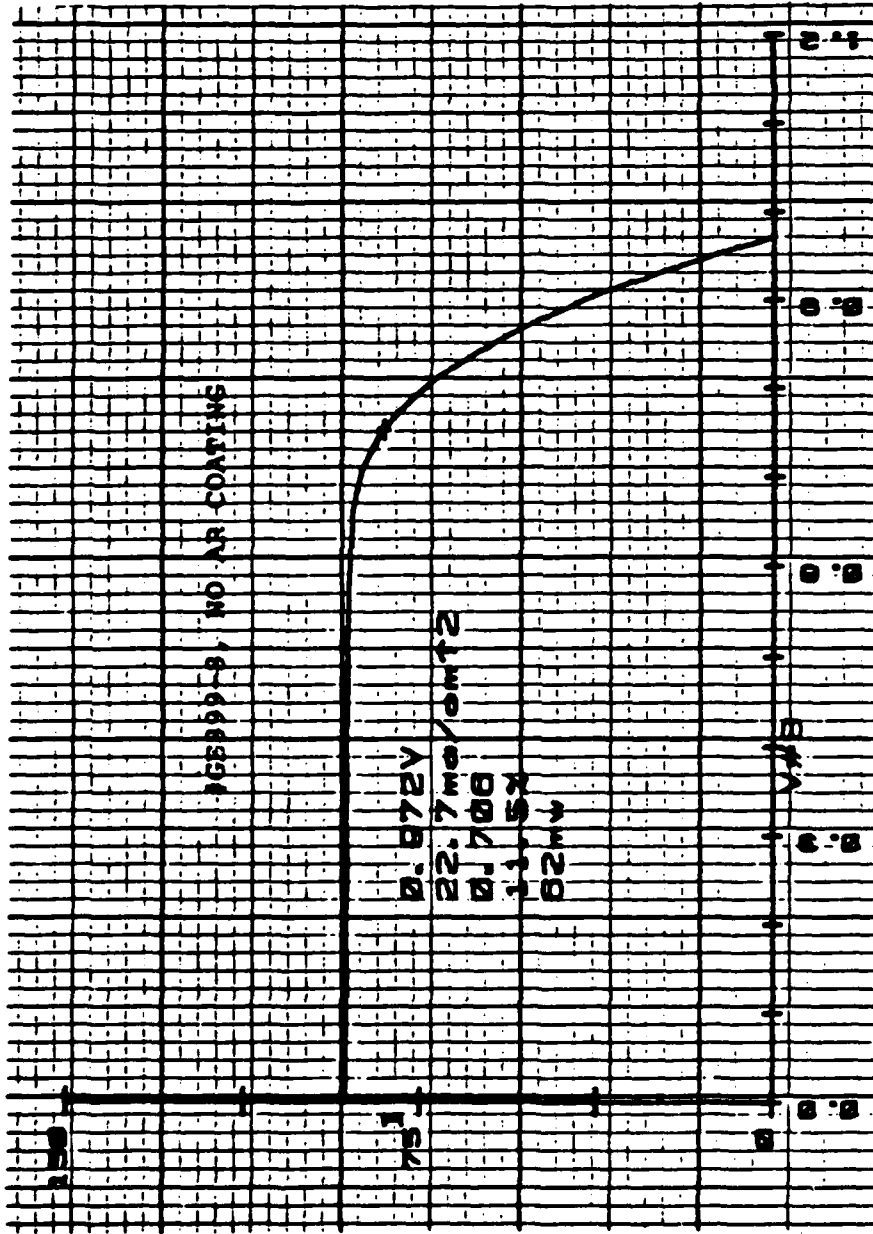


FIGURE 4

AlGaAs/GaAs/Ge SOLAR CELL

AMO, 28 deg C, 2x2cm

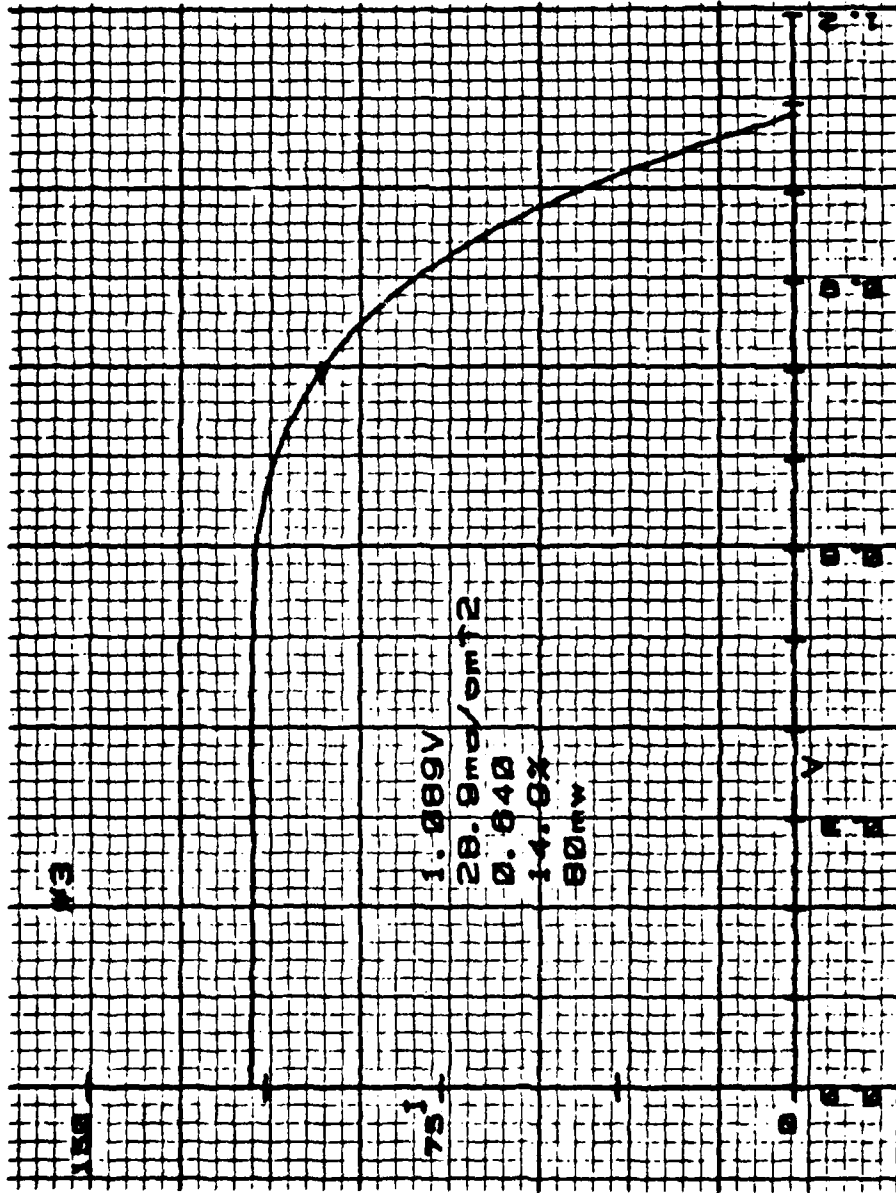


FIGURE 5

AlGaAs/GaAs/Ge SOLAR CELL

The projected efficiency after applying a cover glass was about 15.3% AMO.

Some later results on cells are given in Tables 1 and 2, the latter group with no AR coating.

Also, in Figure 6, we show I-V curves for some cells with efficiency 16%, illustrating some high Voc values.

## 8.0 ITERATIVE TESTING

Because the 2x2cm cells gave good performance, we resumed work on thinning cells down to 50-75um.

NOTE: Some extra delays were caused at this period because of reactor problems, but later those were remedied.

Our next test used Ge substrates thinned to 190um. Also, we tested two techniques to thin wafers to 50-75um (simulating actual thinning of cell/Ge structures). The first method used chemical thinning (with the active layers protected by resist); however, the etch thinning for 175um to 75um caused excessive roughness, with some holes.

In the second method the wafer was mounted on a thick flexible backing material, and the substrate was thinned from the back using the chemical-mechanical polishing techniques described above for Ge wafers. We are able to produce wafers 75um thick, with 50% yield. These wafers are presently being processed into cells, in the beginning of Phase 2.

TABLE 1  
EARLY GaAs/Ge CELL DATA (AMO, 28°C)

Cell #	Voc (V)	Isc (mA)	CFF	EFF (%)	Vm (V)	Im (mA)	R <sub>□</sub> (Ω/□)	Cell Thickness (Mils)
Ge-1	1.066	116.2	0.596	13.64	0.78	94.6	856	12
Ge-2	1.070	117.4	0.635	14.73	0.78	102.2	645	10
Ge-3	1.053	117.0	0.635	14.46	0.796	98.3	3872	10

TABLE 2  
CELL DATA ON 8 CELL LOT (BEFORE AR COATING)

Cell #	Voc (V)	Isc (mA)	CFF	EFF (%)	Vm (V)	Cell Thickness (Mils)
1	1.034	93.9	0.63	11.4	0.772	8-10 ↓
2	1.068	92.0	0.615	11.2	0.816	
3	1.06	90.8	0.68	12.1	0.800	
4	1.012	90.1	0.67	11.2	0.772	
5	1.043	78.7	0.67	10.2	0.800	
6	1.047	89.8	0.62	10.8	0.784	
7	0.603	92.2	0.26	2.7	0.36	
8	0.972	91.3	0.71	11.6	0.752	

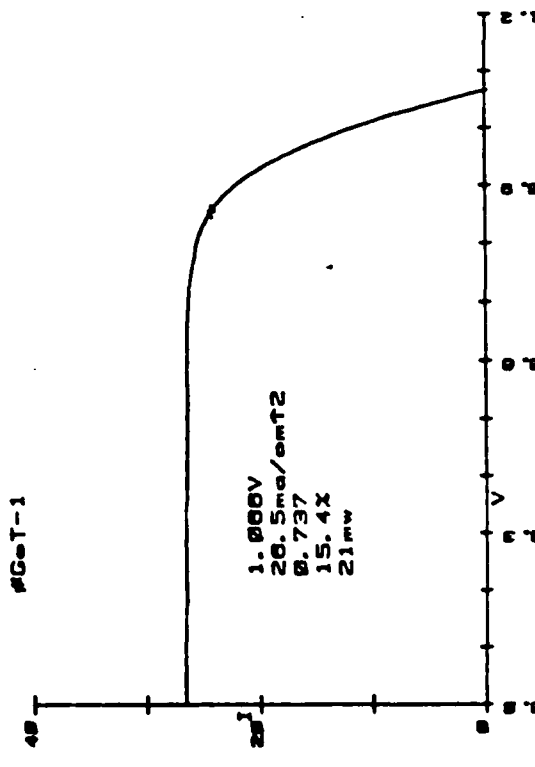
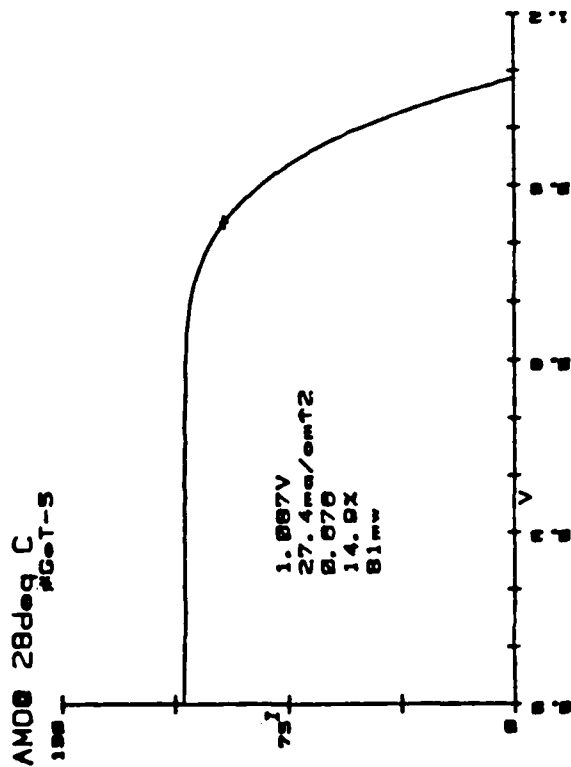
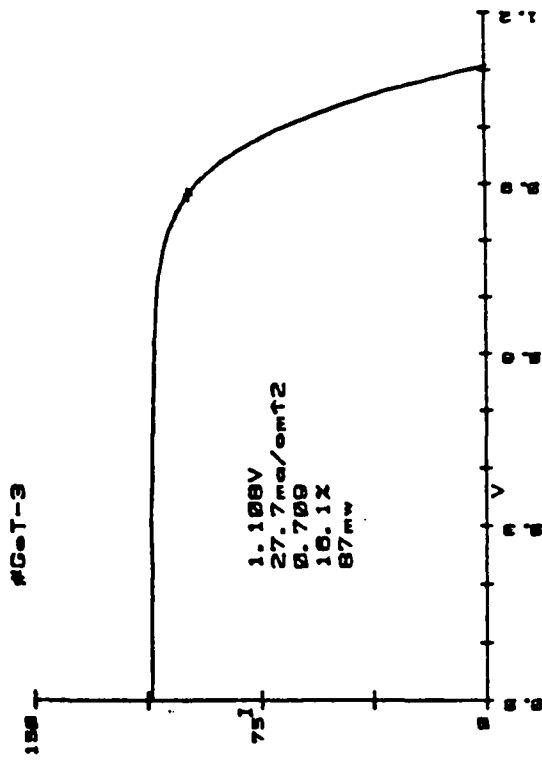
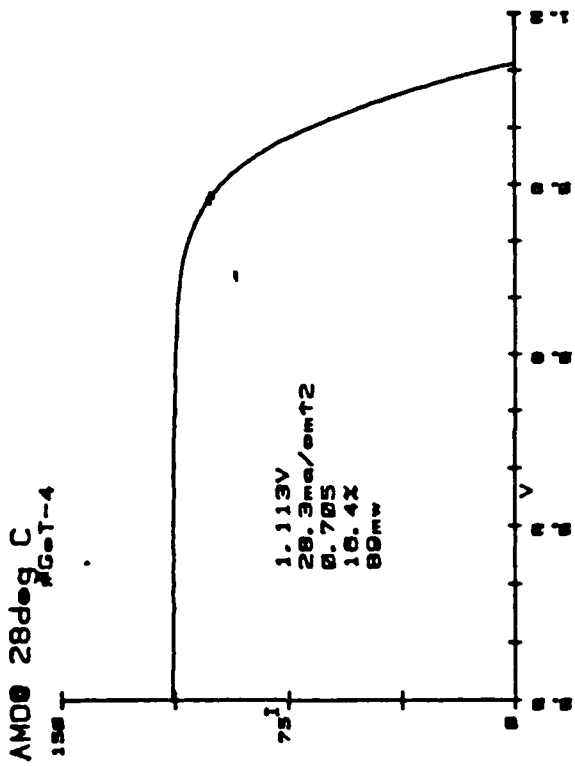


FIGURE 6

I-V DATA FOR 16% GaAs/Ge CFIL

## 9.0 TASK SCHEDULE

The completed schedule for Phase 1 is shown in Figure 7.

Several state-of-the-art cells were delivered to the AF for evaluation. Also two oral reviews were presented (at ASEC) to the project monitors.

## 10.0 CONCLUSIONS

We have successfully developed techniques to polish, both thick and thin Ge wafers. Using OMCVD we have grown good performance GaAs cells on those substrates ( $\sim 16\%$ ). We have achieved high Voc values ( $\sim 1.1V$ ) and are presently investigating the reason for low CFF in these structures.

We are working to combine all those techniques. These results were achieved during a period when the Ge substrate supply was restricted to one supplier, and several mechanical problems were encountered in the OMCVD reactor. These problems included both unforeseen mechanical problems and predictable problems caused by severe facility modifications, and physical relocation and retrofitting of the MOCVD reactors.

We have developed some promising thinning/polishing techniques to apply to finished cells. Our modeling has been extended to understand the experimental results and the work completed has allowed movement into Phase 2 of the contract.

## 11.0 REFERENCES

1. "Efficient GaAs/Ge/Si Solar Cells", B-Y Tsaur et al, Proceedings 17th IEEE Photovoltaic Specialists Conference, 1982, p.1143.



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