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THIN FILMS OF GALLIUM ARSENIDE ON LOW-COST SUBSTRATES

Quarterly Technical Progress Report No. 5 and
Topical Report No. 1, October 2—December 31, 1977

By
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MASTER

January 1978

Work Performed Under Contract No. EY-76-C-03-1202

Rockwell International
Anaheim, California

U.S. Department of Energy

Solar Energy

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for the period

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January 1978

PREPARED FOR THE DIVISION OF SOLAR TECHNOLOGY, UNITED STATES
DEPARTMENT OF ENERGY, UNDER CONTRACT NO. EY-76-C-03-1202

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ABSTRACT

The fifth quarter of work on the contract is summarized. The metalorganic chemical vapor deposition (MO-CVD) technique has been applied to the growth of thin films of GaAs and GaAℓAs on inexpensive polycrystalline or amorphous substrate materials (primarily glasses and metals) for use in fabrication of large-area low-cost photovoltaic device structures. Trimethylgallium (TMG), arsine (AsH_3), and trimethylaluminum (TMAℓ) are mixed in appropriate concentrations at room temperature in the gaseous state and pyrolyzed at the substrate, which is heated in a vertical reactor chamber to temperatures of 725-750°C, to produce the desired film composition and properties.

The substrates used during the quarter included composite structures of sputtered Mo films on various glasses, composites of CVD (GeH_4 pyrolysis) Ge films on these same glasses, large-grained annealed bulk Mo sheet, large-grained bulk polycrystalline GaAs, and one grade of high-purity high-density graphite. The glasses used were Corning Codes 0317, 7059, and 1723, with most of the composites involving the 0317 type. Preliminary results with graphite substrates were not encouraging, but the available substrates used were not of adequate purity or surface quality.

Both single-layer and multilayer structures of polycrystalline GaAs were grown on the above substrates, but the emphasis was on preparation of polycrystalline multilayer structures of p^+/n, n^+/p, n^+/n, and $p^+/i/n^+$ configurations. A number of experiments involving growth of single-crystal GaAs and GaAℓAs films were also carried out to verify Zn doping data using diethylzinc as the dopant source and to establish conditions for the growth of heavily doped p^+ films. Doping concentrations in excess of 10^{19} cm^{-3} in epitaxial layers of GaAs:Zn can now be obtained routinely, and concentrations greater than $1x10^{18}$ cm^{-3} have been achieved in $Ga_{1-x}A\ell_xAs$ alloy layers for $x = 0.8$, indicating that p^+ GaAℓAs window layers can be successfully grown in thin-film solar cell structures by the MO-CVD process.

The polycrystalline p-n junction structures involved Zn doping in a range of concentrations in the p-type layers. Examination of stained angle-lapped multilayer samples failed to produce any evidence of Zn diffusion along grain boundaries out of the p-type regions and into (or through) the n layers. When step-etching techniques were employed to remove an upper layer and expose the layer below the junction, tests indicated the proper conductivity type and at least approximately the expected carrier concentration. However, I-V characteristics indicated the junctions were consistently leaky or, in some cases, completely shorted. Such I-V characteristics were found even when highly asymmetric doping concentrations (i.e., p^+/n and p/n^+) were used in forming the junctions.

Encouraging results were obtained, however, with polycrystalline junction structures grown in the p^+-i-n^+ configuration on substrates of large-grained bulk Mo sheet and also sputtered Mo films on Corning Code 0317 glass; I-V characteristics indicated considerably lower junction leakage.

Plans for the work of the next quarter are outlined.

CONTENTS

ILLUSTRATIONS

1. INTRODUCTION

The long-range objective of the National Photovoltaic Program is to ensure that photovoltaic conversion systems play a significant role in the nation's energy supply by stimulating an industry capable of providing approximately 50 Gwe of installed electricity generating capacity by the year 2000.

One of the specific shorter-term objectives of the national program is to conduct research and advanced development on thin-film materials, cell structures, and advanced concentrator material systems to show the feasibility of reducing photovoltaic array costs to $300 per peak kwe by 1986, and to establish the viability of this advanced technology by the year 1990.

The Research and Advanced Development activity of the national program has among its specific technical goals the demonstration of at least 10 percent AM1 cell efficiency in more than one thin-film solar cell material or configuration by the end of FY80, with continued emphasis on reaching the longer-term goals. The latter consist primarily of (1) achieving flat-plate module or concentrator array prices (1975 dollars) of less than $0.50 per peak watt (electric) by 1986 with an annual production rate of at least 500 peak Mw of arrays/modules, and (2) achieving flat-plate module or concentrator array prices (1975 dollars) of $0.10 to $0.30 per peak watt (electric) by 1990 with an annual production rate of such arrays/modules of at least 10-20 peak Gw by the year 2000.

The work of this contract is directed toward those goals. The overall objective of the contract is the performance of intensive studies that constitute an initial step in DoE's program to overcome current problems and deficiencies in design and fabrication of arrays of extremely low-cost thin-film photovoltaic cells of relatively high efficiency and long life. Acquisition of further important knowledge of thin-film deposition methods and thin-film technology for polycrystalline GaAs on various substrates is also anticipated.

The existing contract (originally No. E(04-3)-1202) was modified on September 29, 1977 (although made retroactive to September 5, 1977), and the modified Statement of Work specifies the following areas of technical activity:

1. Evaluate the use of glass, glass-ceramic and graphite as low-cost substrates and perform the detailed analysis and experimental studies necessary to grow thin films of GaAs with enhanced grain size on these substrates by the metalorganic vapor deposition technique.

2. Perform the proposed parametric studies and measurements to determine the pertinent mechanical, electrical, and optical properties of the films, with special emphasis on adherence, morphology, uniformity, doping, carrier concentration, mobility, lifetime, properties of the electrical contact adjacent to the substrate, and the properties of grain boundaries and the role they have in determining the microscopic properties of the polycrystalline films.

3. Investigate epitaxial growth, diffusion, ion implantation, ITO, and thin Au Schottky barriers for the formation of collecting junctions in polycrystalline GaAs.

4. Develop the optimal solar cell design and fabrication techniques for the fabrication of thin-film GaAs photovoltaic devices with a conversion efficiency greater than 10 percent, and evaluate the electrical and optical performance of the cells.

5. Determine estimated cost, quality, and requirements of the feedstock materials as appropriate for scaled-up production of 50,000 Mw/year.

6. Beginning with the inception of the contract, supply a minimum of 4 cm^2 of representative samples of current production of thin films each month to DoE, for whatever purpose DoE deems suitable.

In addition, various required reports are to be prepared and submitted, as specified in the modified contract.

1.1 GENERAL TECHNICAL APPROACH OF CONTRACT WORK

The general technical approach used in this program has involved the metalorganic chemical vapor deposition (MO-CVD) technique for the growth of thin films of GaAs and GaAℓAs on inexpensive polycrystalline or amorphous substrate materials in configurations permitting fabrication of photovoltaic devices. The parameters of the CVD process have been chosen to maximize the chances of achieving the required properties in the deposited films and the efficiency goal of the photovoltaic devices (10 percent AM1), subject to the constraints imposed by the properties of the selected substrate material(s).

As applied in this program, the MO-CVD process involves the mixing of a metaloganic compound of a Group III element with a hydride or metalorganic compound of a Group V element, and pyrolysis of this mixture or its reaction product under appropriate conditions to produce the Group III-Group V semiconductor. Thus, trimethylgallium (TMG) and AsH3 are mixed at room temperature in the gaseous state and pyrolyzed at established temperatures in a cold-wall reactor to form GaAs according to the following simplified equation:

$$(CH_3)_3\,Ga + AsH_3 \longrightarrow GaAs + 3CH_4.$$

By mixing TMG in the gas phase with trimethylaluminum (TMAℓ) and AsH_3, GaAℓAs is obtained upon pyrolysis at appropriate temperatures; the composition of the alloy is controlled by the ratio of the reactants. The organic byproduct, methane (CH_4), is stable at film growth temperatures. In similar fashion, AℓAs can be prepared from TMAℓ and AsH_3, if desired. Many such compounds and alloys have been prepared by the MO-CVD process (Ref 1).

The MO-CVD process has several attributes that are important to the application involved in this program:

1. The process is completely free of halides, thus eliminating competing etching reactions, reducing generation of unwanted impurities by reactions with the low-cost substrate materials, and minimizing complication of the heterogeneous film-growth process involved.

2. Only a single high-temperature zone is required, greatly simplifying the apparatus and the necessary control systems, and allowing the deposition chamber walls to remain relatively cool because only the pedestal and the samples are heated.

3. The reactants used are either liquid or gaseous at room temperature, facilitating their handling and introduction into the reactor system carrier gas upstream from the deposition chamber, and allowing control of composition of the deposited film by means of flowmeter adjustments.

4. Impurity doping of the deposited films can be achieved by introduction of appropriate dopant compounds (liquid metalorganic and/or gaseous hydride sources) into the primary reactant gas stream, again with doping levels controllable by means of flowmeter adjustments.

5. The growth process can be observed directly by the operator, since the reactor walls are transparent and unobstructed, thus allowing changes in growth conditions to be made during an experiment if it is desired or necessary.

6. Large-area, uniform surface coverage can be achieved in a single growth sequence, using the same type of commercially available apparatus that is used for epitaxial growth of elemental semiconductors (e.g., Si).

7. The process requires neither single-crystal GaAs material nor semiconductor-grade (ultrahigh-purity) polycrystalline GaAs for its application, since only compounds of Ga and of As are used in the reaction, thus eliminating the expensive and energy-wasteful processes of producing melt-grown GaAs source material that is required for other crystal growth and/or film deposition techniques.

1.2 SUMMARY OF FIRST YEAR'S ACTIVITIES

The first year of work on this contract verified some of the strengths of the MO-CVD process and, at the same time, defined further some of the technical problems that remain to be solved (Ref 2). A summary of that work follows:

Ten candidate materials were initially selected for further experimental investigation, on the basis of a set of qualification criteria and initial experimental tests. Those evaluated most extensively included certain glasses, polycrystalline aluminas, and metals, as well as composite substrates of Mo metal films and Ge films on insulating substrates. Some of the glasses were found to be physically and/or chemically unsuited to the growth of GaAs films in H_2, as were the Kovar-like (Co-Ni-Fe alloy) metals. Mo and Ge intermediate layers deposited on Corning Code 0317 glass substrates appeared to be compatible with the MO-CVD growth of GaAs. The large-grained alumina and bulk metal substrates are considered too expensive to meet the future cost goals of the DoE program, but have been used for experimental film growth that provides a comparison with growth on amorphous (or very small-grained) substrates.

A new dedicated reactor system was designed and constructed for use in this program; it was completed in the second quarter of the contract and has been used as one of two MO-CVD reactor systems employed in these investigations. MO-CVD experiments undertaken in these two reactor systems established that good control and reproducibility of the doping of GaAs:Se, GaAs:Zn, and GaAℓAs:Zn single-crystal films can be achieved. Doping experiments with polycrystalline GaAs films on low-cost insulating substrates established that a conducting intermediate layer will probably have to be employed to allow adequate contact to the back surface of a polycrystalline GaAs solar cell grown on such substrates. Such intermediate layers of CVD Ge and sputtered Mo films have been used in the growth of polycrystalline GaAs n/n^+ films for the fabrication of Schottky-barrier solar cells and for the growth of polycrystalline GaAs p-n junction structures.

The single-crystal and polycrystalline films grown in the first year of the contract were evaluated by a variety of characterization techniques. The electrical properties of the polycrystalline GaAs films were analyzed by van der Pauw measurements, and the resistivities were found to be 2-3 orders of magnitude greater than that of single-crystal material of the same input doping level. No difference was observed in the resistivity of n-type and p-type GaAs of comparable doping levels. However, because it is possible to dope p-type GaAs more heavily than n-type material the lowest resistivities were achieved in p-type GaAs films.

The limits of applicability of C-V measurements for determining impurity concentration were explored. Only when a back-plane contact to the films was available could these measurements be successfully applied. In addition, films doped higher than 10^{16} cm^{-3} were not successfully measured, owing to excessive leakage in the Schottky barriers.

The physical and structural properties of the polycrystalline GaAs films were examined by SEM and x-ray diffraction analysis. The films all showed similar general surface features, with apparent grain sizes of 2-10 μm. The grain size most typically seen for films ~5 μm thick was ≤ 5 μm in apparent horizontal dimension. Films grown on refired Vistal alumina substrates exhibited 1mm-size grains that had grown epitaxially on the large individual $A\ell_2O_3$ grains in the alumina. The preferred orientation of GaAs grown on most of the substrates was $\{111\}$, but the preferred orientation of those grown on glasses was shown to vary with temperature of deposition.

Techniques for determining the Aℓ concentration in GaAℓAs films have been established. Epitaxial films grown on GaAs were analyzed by electron microprobe x-ray techniques and conventional x-ray lattice constant measurements to establish a working calibration curve for film composition. These data are to be used for further studies of Aℓ concentration in polycrystalline GaAℓAs films.

Techniques for the determination of grain size in polycrystalline films were explored in considerable detail, especially during the fourth quarter of the program. EBIC-mode measurements on Schottky barrier devices on polycrystalline films in the SEM demonstrated the presence of localized non-radiative recombination regions that in many cases coincided with apparent grain boundaries in the polycrystalline films.

Both single-crystal and polycrystalline solar cells were grown by MO-CVD, fabricated, and evaluated. Contact technologies were explored and apparently suitable ohmic contacts were developed. Single-crystal GaAℓAs/GaAs p-n junction cell structures were prepared with thin (~500Å) $Ga_{1-x}A\ell_xAs$ ($x \approx 0.8$) windows and GaAs:Zn-GaAs:Se junction regions. Efficiencies as high as 12.8 percent under simulated AM0 illumination with no AR coating were obtained, indicating the high quality of the semiconductor layers grown by the MO-CVD technique. Extrapolation of these results to those expected from similar devices with AR coatings indicates that efficiencies as high as those attained by LPE growth methods can be achieved with material made by the MO-CVD process.

Films with grain sizes in the ~5 μm range were used for fabrication of polycrystalline film solar cells on a variety of substrates. Most attention was devoted to Schottky-barrier cells. The best results obtained with these devices was an efficiency of 2.25 percent for AM0 illumination and no AR coating, for n/n^+ GaAs films grown on a composite Mo/glass substrate. Short-circuit current densities of up to 12.5 mA/cm^2 were achieved in such devices. This particular substrate combination was found to be a good low-cost baseline reference with which to compare other results. The GaAs-Mo interface was found to be ohmic as long as the donor impurity concentration in the GaAs film exceeds $\sim 10^{18}$ cm^{-3}.

P-n junctions in polycrystalline GaAs films were also formed by growing alternate p and n layers by MO-CVD. The electrical properties of these junction structures generally showed excessive leakage currents, resulting in soft and sometimes shorted characteristics for p on n devices and considerably less leaky characteristics for n on p devices. It is thought that diffusion of Zn along grain boundaries may be responsible for the leaky device characteristics in these structures.

Finally, preliminary analyses of material and processing costs associated with the fabrication of two different conceptual designs of GaAℓAs/GaAs heterostructure solar cells - one involving the conventional configuration of an opaque substrate and one involving an inverted configuration utilizing a transparent substrate - were developed. Some consideration was also given to future material requirements and costs of producing such cells in large quantity. The principal conclusions reached from these analyses are as follows:

1. Reduction by a factor of 10 to 15 in the cost of the input semiconductor materials relative to present values would probably permit thin-film GaAℓAs/GaAs cells to meet the 1986 DoE cost goals.

2. The substrate cost remains as the predominant factor in the overall materials costs for thin-film GaAℓAs/GaAs solar cells; glasses still appear as potentially the best substrate material prospect for allowing total array costs to come within the established DoE goals.

3. Added-value costs of ~\$17 per m^2 for fabrication of large-area films of GaAs and a similar figure for cell and array fabrication, "borrowed" from the DoE/JPL LSSA Project, appear to be reasonable cost goals for 1986 for GaAs low-cost solar array fabrication, as well.

4. At least in terms of deposited thin-film GaAℓAs/GaAs solar cells on substrates of materials other than single-crystal GaAs, there appears to be enough recoverable Ga metal in projected future bauxite ore supplies to meet the DoE solar array production goals for the years 1986 and 2000, especially if improvements in Ga recovery technology are introduced to increase process efficiencies by relatively modest amounts.

5. The MO-CVD process for fabrication of GaAs cells appears to be adaptable to continuous or semi-continuous manufacture of large-area thin-film cells in the quantities required for meeting the DoE 1986 and 2000 production goals, primarily through design of the necessary scaled-up apparatus and through development of companion processes for producing appropriate substrates at sufficiently low cost.

Thus, the CVD technique is still viewed as potentially the best method for achieving large areas of solar cells at sufficiently low cost to meet both the performance goals and the economic goals of DoE's long-range program. The results of the first year's work demonstrated that the MO-CVD technique is capable of producing materials of quality sufficient to meet the goals of the photovoltaic conversion program, and that the technique is also a strong candidate for eventual use in fabrication of single-crystal GaAℓAs/GaAs cells for high-efficiency applications (e.g., space power supplies), with or without concentrator systems.

1.3 SPECIFIC TASKS OF SECOND-YEAR PROGRAM

The continuation of these studies will emphasize further substrate development as well as improved understanding of the properties and effects of grain boundaries in the polycrystalline films. In addition, further attention will be devoted to junction formation techniques and techniques which yield larger grain size for equivalent Ga content. Further development of solar cell fabrication technology, including contacts and AR coatings, will also be undertaken.

To pursue the remaining technical problems and to meet the objectives of the modified contract the work of the second year has been organized into five main technical tasks, as follows:

Task 1. Experimental Evaluation of Low-cost Substrates and Methods for Obtaining Enhanced Grain Size in MO-CVD Films

Task 2. Evaluation of Film Properties and Grain Boundary Effects and Correlation with CVD Growth Parameters

Task 3. Investigation and Development of Barrier Formation Techniques

Task 4. Development of Photovoltaic Device Designs and Fabrication Techniques

Task 5. Analysis and Projection of Cell Material Requirements and Fabrication Costs.

In addition, the required program management activity and the preparation of technical reports and documentation, technical data, and review and briefing information, as well as the preparation and delivery of the required representative samples each month, are carried on in conjunction with the above technical tasks.

This is the fifth Quarterly Technical Progress Report (including the Annual Report (Ref 2)) for this contract* and covers the period October 2 through December 31, 1977. The technical activities for that period are described by task in the following section. Conclusions of the quarter's work are given in Section 3, and plans for the next quarter's activities are outlined in Section 4. Section 5 contains references used in the body of the report.

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*Note, however, that the first three Quarterly Project Reports and the Annual Report were identified with the original contract number - No. E(04-3)-1202.

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2. TECHNICAL PROGRESS

The work of the first quarter under the modified contract (fifth quarter of the program) is described by task in this section. The individual technical tasks are identified in the Introduction (Section 1).

2.1 TASK 1. EXPERIMENTAL EVALUATION OF LOW-COST SUBSTRATES AND METHODS FOR OBTAINING ENHANCED GRAIN SIZE IN MO-CVD FILMS

According to the Statement of Work of the modified contract, this task is to evaluate the use of glass, glass-ceramic and graphite as low-cost substrates and perform the detailed analysis and experimental studies necessary to grow thin films of GaAs with enhanced grain size on these substrates by the metal-organic chemical vapor deposition technique.

The emphasis during this fifth quarter of the program was on use of composite substrates of sputtered Mo metal films on various glasses and composite substrates consisting of Ge films grown by chemical vapor deposition (GeH_4 pyrolysis) on glass, although selected graphite substrates were also employed in some experiments. Specifically, the candidate composite substrates used involved Corning Code 0317 glass, Corning Code 7059 glass, or Corning 1723 glass. Polycrystalline films of Mo or Ge were obtained on these glasses in all cases. The Ge was deposited in the GaAs reactor in a separate experiment prior to the GaAs growth experiment. The Mo was deposited in completely separate apparatus.

The high-purity polished Poco Grade DFP-3 graphite substrates ordered many months ago for use on this program were finally received in October but still required repolishing to provide an acceptable finish. This work was undertaken at Rockwell and resulted in improved surfaces, but small pores remained in the material and were found to trap cleaning liquids or other materials that contacted the graphite. Some of the replished graphite substrates were returned to Poco for repurification, to be used later as experimental substrates for GaAs film growth by the MO-CVD technique. However, the repurified substrates were not received from the vendor during the quarter covered by this report.

Some of the repolished substrates that had been retained by Rockwell were treated in H_2 at ~1000°C in the MO-CVD reactor and then used for growth of undoped GaAs at ~725°C, using the standard deposition procedure developed during the first year of this contract program. The films were fairly reflective after light etching in a Br_2-MeOH solution and appeared to have large (10-20 μm) grains. Unfortunately, the film adherence was poor; the Scotch-tape test for adherence resulted in film removal, and some of the films simply peeled away from the substrate during normal handling. Because of the poor results obtained with the repolished but not repurified substrates no further film growth experiments with that material were done during the quarter. Arrangements were made, however, with Carbone-Lorraine Industries Corp. (Boonton, NJ) to obtain samples of their highest density graphite for evaluation as a possible substrate for GaAs growth. This material has been reported by other investigators (Ref 3) to be superior for this application. Additional work with graphite substrates will be carried out during the coming quarter.

As a result of the recent successful use of vacuum-deposited Aℓ films on glass as composite substrates for the growth of InP films in Rockwell's DoE contract on polycrystalline InP films for low-cost solar cells (Ref 4), similar composite substrates were prepared for initial evaluation in this program. First experiments with these will probably be carried out early in the next quarter.

Deposition experiments late in the quarter involved substrates of CVD Ge on Corning Code 0317 glass, sputtered Mo on 0317 glass, and large-grained annealed bulk Mo. Polycrystalline GaAs films (n and p type) were also grown on large-grained bulk polycrystalline GaAs substrates, to provide a comparison with the smaller-grained polycrystalline films obtained on the above substrates. In each case a GaAs:Si single-crystal substrate was included as a control wafer. Results of evaluation of these films are discussed in the following section.

No new substrate materials with properties especially suited to the growth of enlarged-grain GaAs films were found during the course of this quarter's activities. Attempts to locate and/or develop such materials are continuing on a routine basis, however. Specific experimental studies of techniques intended to produce grain-size enhancement in polycrystalline films grown on the available substrate types now being used are to be undertaken in the coming quarter.

2.2 TASK 2. EVALUATION OF FILM PROPERTIES AND GRAIN BOUNDARY EFFECTS AND CORRELATION WITH CVD GROWTH PARAMETERS

The modified contract Statement of Work stipulates that this task will involve the performance of the proposed parametric studies and measurements to determine the pertinent mechanical, electrical, and optical properties of the films with special emphasis on adherence, morphology, uniformity, doping, carrier cnncentration, mobility, lifetime, properties of the electrical contact adjacent to the substrate, and the properties of grain boundaries and the role they have in determining the microscopic properties of the polycrystalline films. In particular, it involves the improvement of film growth and doping procedures, routine evaluation of film properties, and study of grain boundary effects in the polycrystalline films grown on various low-cost substrates.

The immediate goals of this task are (1) to provide rapid feedback on the properties of films grown on low-cost substrates so that correlations between the properties relevant to photovoltaic performance and the growth conditions used in preparing the films can be established, and (2) to perform detailed studies of the effect of grain boundaries on the photovoltaic properties of polycrystalline GaAs.

2.2.1 Film Growth and Doping Procedures

Both single-layer and multilayer structures of polycrystalline GaAs were grown on various substrates during the quarter, although the emphasis was on multilayer structures including p^+/n, n^+/p, n^+/n, and $p^+/i/n^+$ (the i layer being n-type). As indicated in the previous section, the low-cost substrates used for these experiments included composites of Mo or Ge films deposited on various glasses (Corning Codes 0317, 7059, or 1723). In several instances bulk polycrystalline GaAs substrates were also used, as were graphite substrates and annealed Mo sheet substrates in a few preliminary experiments. Additionally, some single-crystal thin-window GaAℓAs/GaAs heteroface solar cell structures were grown on single-crystal GaAs substrates, and other single-crystal films (GaAs/Ge and Ge/GaAs) were grown for the purpose of examining the GaAs/Ge interface. Results obtained with some of these structures are discussed later in this report.

A series of polycrystalline p-n junction structures was grown with varying doping concentration in the p-type Zn-doped GaAs layer. Usually a Si-doped GaAs single-crystal substrate was also included in the run to serve as a control wafer and to provide information on the properties of the simultaneously grown single-crystal p-n junction.

In some cases a two-step growth sequence was used. In those cases, a deposition run was first made to grow a GaAs:Se n-type film ~4 μm thick on the surface of the Mo/glass or the Ge/glass composite substrate. The resulting film was then etch-polished with Br_2-MeOH (1% solution) to a final thickness of ~2 μm. This produced a very smooth surface, thus making it possible to grow the upper (p-type) layer of the junction structure on a smooth substrate. This upper layer was then grown, Zn-doped to the desired concentration level. In some cases the p-type layer was also etch-polished in a similar fashion.

In order to monitor the impurity doping concentration obtained in each layer of these two-step junction structures a GaAs:Cr high-resistivity substrate was sometimes used in each of the runs of the two-step growth sequence. Measurements of carrier concentration in the companion epitaxial samples then provided the desired data.

Many of these polycrystalline p-n junction structures were angle-lapped (3-deg bevel) to permit measurement of the layer thicknesses. The thicknesses determined by this technique generally agreed well with the thicknesses measured on the single-crystal companion samples using the same measurement procedure. This angle-lapping and staining process failed to provide any evidence of detectable Zn diffusion along the grain boundaries from the upper Zn-doped layer into the polycrystalline bottom layer (Se-doped, n type), although it is possible that such Zn diffusion had, in fact, occurred but was simply not detectable by this relatively insensitive procedure.

A number of experiments involving growth of single-crystal GaAs films were also carried out during the quarter, mainly to provide further baseline reference data on film properties and doping characteristics that could be used in interpreting results being obtained with the doped polycrystalline films. These experiments included the growth of GaAs:Zn and GaAℓAs:Zn films 6-10 μm thick on GaAs:Cr single-crystal substrates, to verify previously established Zn doping curves using diethylzinc (DEZn) as the Zn source and to establish conditions for the growth of heavily doped p-type GaAs:Zn and GaAℓAs:Zn films.

Doping concentrations in excess of 10^{19} cm^{-3} for GaAs:Zn layers (epitaxial) have now been grown routinely. Heavily doped layers of several $\text{Ga}_{1-x}\text{A}\ell_x\text{As}$ alloy compositions, with $p > 10^{18}$ cm^{-3}, have also been grown for two different temperatures of the DEZn source. These results are shown in Figure 2-1, in which the open data points (circles and squares) correspond to the same DEZn flow conditions into the reactor chamber but are for two different DEZn source temperatures (0°C and 24°C). The solid data points (dots) are for a somewhat lower DEZn flow rate and for a DEZn source temperature of 0°C. Clearly, as the Aℓ content in the alloy increases, especially above about $x = 0.8$, it becomes more difficult to obtain heavily doped (and thus high-conductivity) p-type material.

Other single-crystal GaAs films grown during the quarter included a group of undoped layers grown on (100)-oriented Ge substrates for use in the study of the role of intermediate layers of Ge formed on low-cost substrates as a means of influencing the growth habit and/or grain size of subsequently deposited GaAs films. The nominally undoped GaAs films exhibited evidence of some Ge incorporation, based on point-contact rectification (PCR) measurements that are indicative of net carrier concentration in the film under test; the PCR behavior of these films was compared with that of undoped GaAs films grown directly on GaAs:Cr single-crystal substrates. Further study of epitaxial films in the GaAs/Ge configuration will be carried out in the coming quarter to determine optimum growth procedures for reducing the Ge content in the GaAs layer.

In addition, a group of single-crystal Ga-doped Ge films were separately grown by the CVD process by adding TMG to the reactant gas stream during growth of Ge from GeH_4. These films were found to be heavily doped p type and were very conductive. It thus appears that such films could probably be used as p-type intermediate layers for the growth of n-p polycrystalline GaAs junction structures. This possibility will be investigated further.

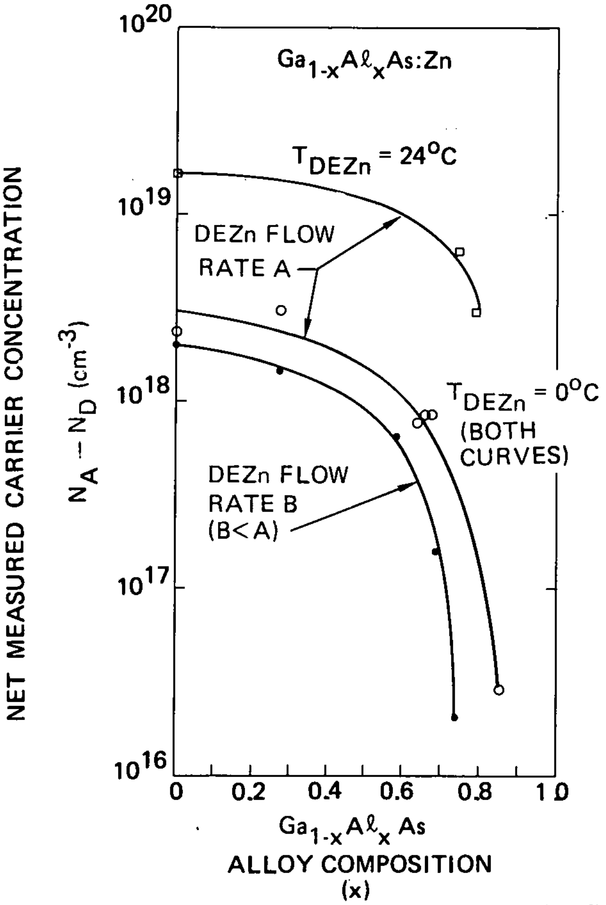

Figure 2-1. Net Measured Carrier Concentration in $Ga_{1-x}A\ell_xAs$ Films Grown by MO-CVD as Function of $A\ell$ Content for Three Different Growth Conditions

Finally, several single-crystal large-area (~4 cm^2) GaAℓAs/GaAs thin-window heteroface solar cells were grown, primarily for use in further development and evaluation of contacting materials and procedures for such large-area cells. (See Task 4 discussion.)

2.2.2 Evaluation of Film Properties

The characterization work during this report period was concentrated on studying the properties of multilayer structures to determine the effects of subsequent GaAs growth on the final properties of the various layers as well as on routing evaluation of GaAs and GaAℓAs film properties. In addition, considerable effort has been expended, at no direct cost to this program, to automate the present apparatus for measuring the Hall effect in thin-film samples. A brief description of this system is given in this report since it is expected to impact considerably the results of this program, both in time saved for routine measurements and in the possibility of performing more detailed experiments in a timely manner.

Several techniques were applied to the study of multilayer solar cell structures, one purpose of these studies being to determine if the properties of the individual layers showed any relationship to the junction transport properties of the structures. As indicated in the preceding section, angle-lapping techniques were commonly used for thickness determination. This procedure revealed planar junctions, generally, with no detectable deviations from planarity that would be attributed to Zn diffusion along grain boundaries, as mentioned earlier. However, no firm conclusion was possible on the basis of these experiments because of scratches and other defects resulting from the angle-lapping procedure used. The high degree of planarity of the junctions somewhat surprising in view of the fact that the morphology of these films typically becomes quite rough after ~5 μm of growth. More detailed studies are planned to determine the evolution of the surface morphology as growth proceeds.

Selective-area step-etching was also used to study film properties, most often in conjunction with PCR measurements to determine the electrical properties of the various layers. This has proven to be a most valuable technique, as will be described in Task 3. In general, Br_2:MeOH was used for the etchant; layer thicknesses predicted by growth-rate calibration have been at least grossly confirmed by this technique, also.

The PCR measurements are performed by applying two tungsten probes to the surface of the material. The first probe is charged with a Tesla coil to break down the surface potential for ohmic conduction, and the second probe is then placed in contact with the surface at a nearby location. A curve tracer is connected across the two probes, and the voltage polarity and the breakdown voltage give a measure of the conductivity type and carrier concentration. The voltage breakdown readings were "calibrated" during the first year of the contract by van der Pauw measurements on many samples of both n- and p-type polycrystalline GaAs material. However, the result was found to be somewhat substrate-dependent and sharply dependent on doping, so that only approximate doping concentrations can be derived by this convenient method. Except for the highest doping levels ($\geq 10^{18}$ cm^{-3}), however, it provides a quick and useful indication of doping type.

A new and completely automated Hall-effect apparatus was made operational during this quarter and is shown schematically in Figure 2-2. The system is based on a Hewlett-Packard 3052 data acquisition system, and consists of a Hewlett-Packard 9825A calculator as the system controller and data processor. The acquisition of data is accomplished by controlling the functions of two switching interfaces. One is a Hewlett-Packard low-leakage relay-scanner that is directly interfaced to the calculator. This scanner determines which of the legs of the van der Pauw sample the current is directed through and which ones are connected to the voltage-measuring apparatus. The current is controlled by the use of a Keithley picoampere current source, and the voltages are measured by a Hewlett-Packard high-impedance DVM. The magnetic field is switched by an interface designed and built at Rockwell. This interface is also controlled directly by the calculator, and is software-variable.

The system as it presently exists is capable of measuring samples with impedances up to 10^{10} ohms at current levels ranging from 10^{-12} to 10^{-3} amperes, depending on sample resistance. It performs the entire measurement in less than 2 min and has been found to be reproducible to better than 1 percent. Present plans call for the design and construction of a temperature-control interface, to allow program control of temperature-variable measurements. In the interim, manual control is being employed.

2.2.3 Study of Grain Boundary Effects

Many of the multilayer structures grown during the quarter (see Section 2.2.1) were prepared not only for study of the properties of the resulting barriers themselves but also for examination of various grain boundary effects, such as possible Zn diffusion along the grain boundaries as discussed above. Possible grain boundary effects observed in the junction studies are taken up in the next section (Task 3). Other experiments directed toward producing grain size enhancement during GaAs film growth are planned for the coming quarter.

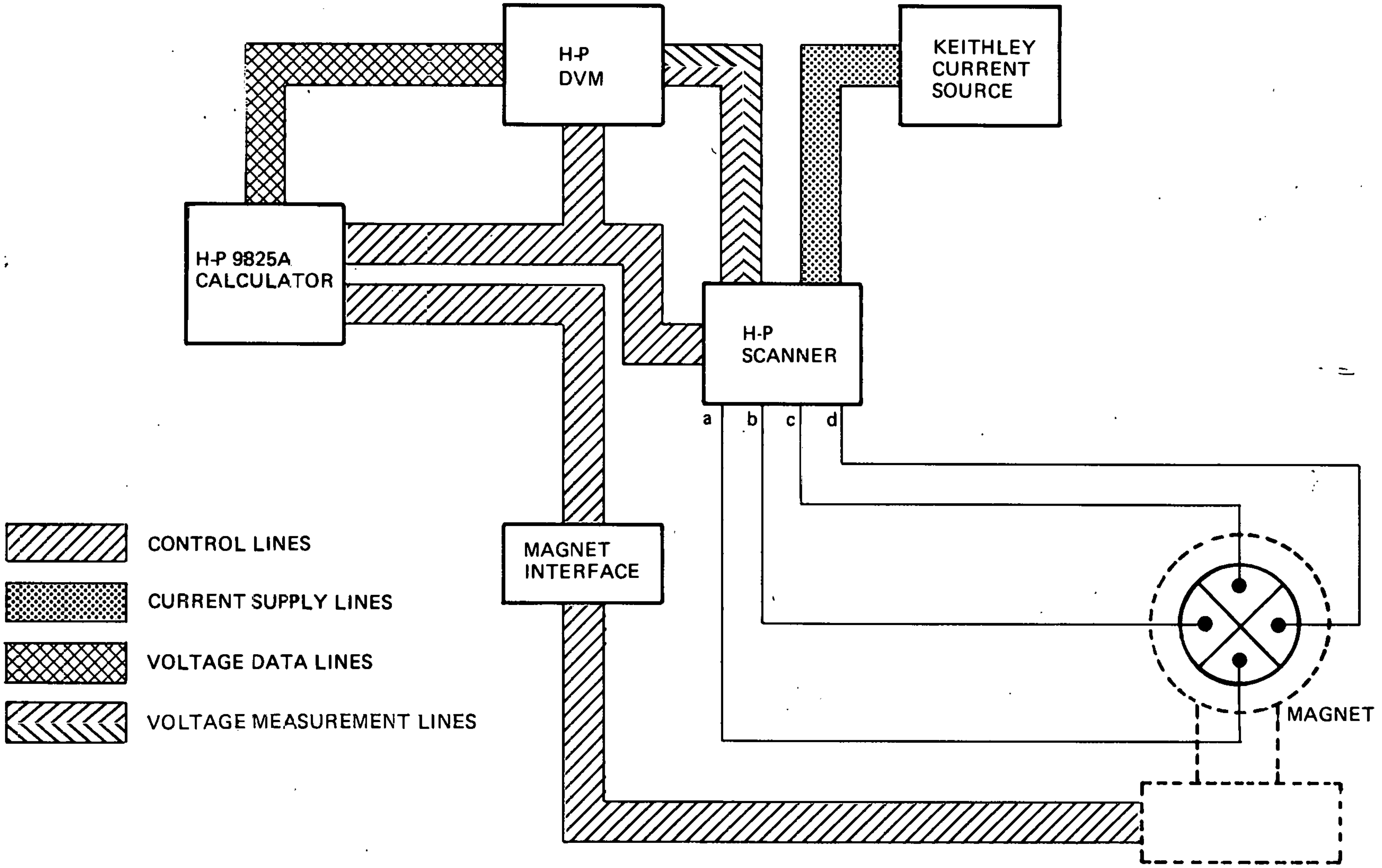

Figure 2-2. Schematic of Automated Hall-effect Apparatus

2.3 TASK 3. INVESTIGATION AND DEVELOPMENT OF BARRIER FORMATION TECHNIQUES

According to the modified contract Statement of Work this task involves investigation of direct layer growth by MO-CVD, thermal diffusion, ion implantation, the use of deposited ITO layers, and the use of thin Au Schottky barriers for the formation of the charge collecting barriers in polycrystalline GaAs thin-film photovoltaic devices.

The principal method to be investigated is the growth of p-n junctions in situ in the MO-CVD reactor chamber by successive deposition of suitably doped layers of GaAs, with a variety of substrate materials, deposition sequences, impurity doping concentrations, substrate preparation procedures, and other controllable parameters being included in the study. Also of major, but somewhat less, interest is the use of Au (or other metal) Schottky barriers for the charge collection function in polycrystalline devices.

Indium-tin oxide (ITO) layers may be found to be of considerable potential value in the GaAs solar cell materials system, either as one component of a heterojunction device (ITO/GaAs) or as a transparent ohmic contact on GaAs devices having the barrier formed by some other process, and will be evaluated as part of the work of this task as well as of Task 4. Other potentially useful barrier-formation procedures — in particular, ion implantation and conventional thermal diffusion of the appropriate impurities into polycrystalline GaAs films after their formation by the MO-CVD process — are considered to be alternative methods of probable second-order importance, to be investigated later in the year unless the work with the processes of primary interest indicates the necessity of resorting to such alternatives at an earlier time.

Emphasis of the work of this task during the quarter was on investigation of the properties of p-n junctions formed by in situ doping during deposition. This approach was stressed in an attempt to obtain a better understanding of the cause for the excessive leakage currents observed in most deposited polycrystalline p-n junction structures made to date in this program.

Three composite substrates were used for growth of most of the polycrystalline p-n junction structures studied during the quarter — 1) sputtered Mo (~0.2 μm thick) on Corning Code 0317 glass, 2) sputtered Mo (~0.2 μm thick) on Corning Code 7059 glass, and 3) Ge deposited by CVD (GeH_4 pyrolysis) on Corning Code 0317 glass. Both n/p and p/n structures were investigated. The doping concentration of the bottom layer (adjoining the substrate) was typically varied slightly in the 10^{17} - 10^{18} cm^{-3} range. Layer thicknesses were also varied slightly, ranging from ~2.5 μm each to ~5.0 μm each.

The junction structures were prepared for evaluation by one of two procedures. In the first, a lightly alloyed contact of In was formed on the top (p-type) layer, followed by etching of the edges of the composite sample around the entire sample periphery, to remove any edge-shorting effects. This procedure is quite simple but it does suffer from the uncertainty in the resulting area of the device and the possibility that the In might alloy completely through the upper layer. The second procedure, intended to avoid the latter possibility, involved a point contact in place of the alloyed contact. For the heavily doped top layers used in most of the devices examined to date such a contact was found to be sufficiently ohmic to permit adequate qualitative determination of the junction properties. However, such a contact is of limited value in determining the properties of large-area junctions at high current densities because of the high sheet resistance of the polycrystalline GaAs. Nonetheless, the point-contact method has been useful as a non-destructive screening procedure for use with polycrystalline materials, as discussed in Section 2.2.2.

The typical procedure for evaluating a polycrystalline junction has been to step-etch the sample, to reveal a portion of each layer in some region of the sample, and then to obtain an indication of the doping concentration in each layer by the point-contact reverse-breakdown (PCR) measurements described earlier (Section 2.2.2), and to obtain the junction I-V characteristics on a curve tracer by making point contacts to the top layer and to the conducting layer of the composite substrate. In most cases the doping level indicated by the PCR measurement was as intended and expected on the basis of the growth parameters used.

The samples prepared early in the quarter involved mainly substrates with Ge intermediate layers. When the resulting junctions consistently showed shorted or excessively leaky characteristics at ~10 mA/cm^2 measurement current densities the use of Ge intermediate layers was stopped. While it is not clear that the presence of Ge creates any additional problems, the removal of any additional degrees of uncertainty was considered necessary.

Later in the quarter most of the junction work centered on the use of Mo-coated substrates. Initial samples were grown with junction doping profiles that simulated those of earlier single-crystal cells. Subsequent samples involved variations in both n- and p-layer doping levels. In all cases, the junctions showed excessive leakage currents and I-V characteristics that could best be described as that of an ohmic resistance.

The two most likely reasons for these observed junction properties appeared to be (1) a film growth morphology that was such that the top layer (usually p type) did not fully cover the bottom layer, and (2) diffusion of Zn from the p-type top layer along the grain boundaries of the polycrystalline structure so as to short the bottom n-type layer.

The first possible cause appeared to be ruled out by the flat and continuous p-type layer that was repeatedly observed in the angle-lapped sections made on junction samples having p layers ~5 μm thick, as indicated earlier (Section 2.2). Also, the fact that similar junction structures grown in two separate deposition experiments, by depositing a p-type Zn-doped layer on the smooth etch-polished surface of a previously grown n-type layer, were also shorted tended to argue against the validity of the first possible cause.

The second possible cause, that of Zn diffusion along grain boundaries, seemed far more likely. To examine the possibility further, p-n junctions with asymmetric doping concentrations (i.e., p^+-n and p-n^+ structures) were grown and characterized. Again, for both structures, excessively leaky junction properties were observed. In addition, it was found that the voltage breakdown properties of n layers before and after the growth of p layers on top of them were unchanged. This indicated that, most likely, there were no shorting regions extending from the junction to the conducting substrate as would be expected if Zn had diffused down the grain boundaries. This point will be tested further by the deposition of Schottky barriers on etch-exposed n regions of p-n junction structures so that the Schottky barriers will make intimate contact with grain boundary regions; should shorting p regions be present the characteristics of the Schottky barriers will show the effect of these regions.

Near the end of the quarter some experiments were done with junctions in which one side was very lightly doped, and it was found in the few samples that were evaluated prior to the end of the quarter that the junction leakage was reduced considerably. These p^+-i-n^+ structures, in which the undoped i region is n type, were deposited on substrates of large-grained bulk Mo and also sputtered Mo films (~0.2 μm thick) on Corning Code 0317 glass. The middle (i) layer was intentionally undoped in an attempt to eliminate the possibility of forming a tunnel junction in the grain boundaries. Such tunnel junctions could be formed as a result of impurity segregation in the grain boundaries, especially since relatively high doping concentrations were being used in the polycrystalline p-n structures to obtain as low a series resistance as possible. As a result of these improved properties additional effort will be directed toward preparing and characterizing additional p^+-i-n^+ (as well as p^+-p-i-n^+ and other similar) structures in the coming quarter.

However, in view of the discouraging overall results obtained in the evaluation of deposited polycrystalline p-n junctions grown to date in this program it is clear that much more detailed information about the nature of grain boundaries in polycrystalline GaAs is necessary to understand the behavior of these junctions. An example of the inconsistencies observed in the experimental results obtained to date is that whereas essentially all small-grained polycrystalline p-n junctions with doped p and n regions grown on low-cost substrates have shown very leaky I-V characteristics, p-n junction structures similarly grown on large-grained bulk polyscrystalline GaAs substrates have exhibited quite good rectifying I-V characteristics and were very light-sensitive. In the latter devices a small-area (50 mil x 50 mil) Au-Zn-Au contact was applied to the top of the p-type polycrystalline layer and alloyed to form an ohmic contact. The n-type contact was made directly to the substrate. The top contacts were then used as masks for the etching of mesa diodes into the polycrystalline GaAs layer structure. The possibility that different types of grain boundary properties are involved in the fine-grained films grown on low-cost substrates and the large-grained films grown on bulk polycrystalline GaAs wafers needs further examination.

Thus, in the next quarter considerably greater emphasis will be placed on fundamental studies having the goal of an improved understanding of the properties of grain boundaries in polycrystalline GaAs films.

2.4 TASK 4. DEVELOPMENT OF PHOTOVOLTAIC DEVICE DESIGNS AND FABRICATION TECHNIQUES

This task involves the development of optimal solar cell design and fabrication techniques for the preparation of thin-film GaAs photovoltaic devices having conversion efficiencies greater than 10 percent, and the evaluation of the electrical and optical performance of cells so fabricated. Specifically, further development of appropriate contact materials and procedures for applying them to the various polycrystalline device structures prepared in this program is to be undertaken, including the investigation of indium-tin oxide (ITO) films and thin Au layers as transparent ohmic contacts. Also included is investigation and development of suitable antireflection (AR) coatings for the polycrystalline GaAs cells; initial attention is expected to be given to TiO_2 layers deposited by chemical pyrolysis or other methods. It is intended that additional single-crystal thin-film GaAs solar cells, grown by MO-CVD on single-crystal substrate materials, will also be fabricated from time to time to provide baseline reference data against which to compare the performance of the polycrystalline GaAs cell structures that are of primary concern in this program.

Relatively little work was done on this task during the quarter, the emphasis being placed on the problems of Tasks 2 and 3. However, some attention was given to several alternative contact systems to those used in the contract work to date. The contact metalization typically used for both the single-crystal and the polycrystalline solar cells prepared in this program has consisted of Au/Zn/Au for p-type layers and Au-Ge for n-type layers. Both of these contact materials require sintering in the temperature range 400-500°C.

This type of processing may not be best for polycrystalline p-n junctions because of the possibility of diffusion of Zn or Ge along grain boundaries. Consequently, examination of various possibilities for non-sintered ohmic contacts was begun. However, each of the alternatives considered to date requires a heavily doped "cap layer." These alternatives include (1) ITO, (2) thin Au, (3) Cr/Au, (4) Pd/Ag, (5) Ti/Pt/Au, and (6) Aℓ. Factors such as ultimate cost and preferred deposition techniques have ben weighed in comparing the various possibilities. Experimental verification of acceptably low contact resistance will be required in selecting the best combination of material and method of application, and that work is expected to be pursued further in the next quarter.

Late in the quarter a number of large-area (~5 cm^2) single-crystal GaAℓAs/GaAs thin-window (~400Å) solar cell structures were grown. These multilayer samples had a range of thicknesses for the p-type GaAs layer, but all other structural parameters were kept constant. These samples will be fabricated into finished cells in the coming quarter, and their photovoltaic performance will be evaluated to permit a determination of optimum p-type layer thickness and to provide up-to-date baseline reference data for single-crystal cells made by the MO-CVD process.

2.5 TASK 5. ANALYSIS AND PROJECTION OF CELL MATERIAL REQUIREMENTS AND FABRICATION COSTS

This task is intended to continue the analysis and projection of estimated costs, material quality, and material quantity requirements associated with scaled-up production of 5×10^4 Mwe (peak) solar array generating capacity per year (the DoE program goal for the year 2000) using the MO-CVD process.

The program plan does not include any work on this task for the quarter covered by this report. Effort on this task is to be resumed in the next quarter.

3. SUMMARY AND CONCLUSIONS

The second year of work on application of the MO-CVD process to the preparation of polycrystalline GaAs solar cell structures on low-cost substrates has begun, with the program consisting of work on (1) experimental evaluation of low-cost substrates and methods for obtaining enhanced grain size in MO-CVD films; (2) evaluation of film properties and grain boundary effects and correlation with CVD growth parameters; (3) investigation and development of barrier formation techniques; (4) development of photovoltaic device designs and fabrication techniques; and (5) analysis and projection of cell material requirements and fabrication costs. The work of the quarter covered by this report consisted mainly of activity in the first three areas.

The substrates used during the quarter included composite structures of sputtered Mo films on various glasses, composites of CVD (GeH_4 pyrolysis) Ge films on these same glasses, large-grained annealed bulk Mo sheet, large-grained bulk polycrystalline GaAs, and one grade of high-purity high-density graphite (Poco Grade DFP-3). The glasses used were Corning Codes 0317, 7059, and 1723, with most of the composites involving the 0317 type. No new substrate materials not previously available were obtained during the quarter. The preliminary results with graphite substrates were not encouraging, but the available substrates were not sufficiently purified nor was adequate attention given to surface preparation. Additional experiments with this material will be undertaken in the next quarter.

Both single-layer and multilayer structures of polycrystalline GaAs were grown on the above substrates during the quarter, but the emphasis was on preparation of polycrystalline multilayer structures of p^+/n, n^+/p, n^+/n, and $p^+/i/n^+$ configurations, principally on composites of Mo films or Ge films on glass. A number of experiments involving growth of single-crystal GaAs and GaAℓAs films were also carried out during the quarter, primarily to verify Zn doping data using diethylzinc as the dopant source and to establish conditions for the growth of heavily doped p^+ films.

Doping concentrations in excess of 10^{19} cm^{-3} in epitaxial layers of GaAs:Zn can now be obtained routinely, and concentrations greater than $1x10^{18}$ cm^{-3} have been achieved in $Ga_{1-x}A\ell_xAs$ alloy layers for $x = 0.8$. These results indicate that p^+ GaAℓAs window layers can be successfully grown in thin-film solar cell structures by the MO-CVD process. Some additional single-crystal films of GaAs were grown on Ge single-crystal substrates as part of the study of the GaAs-Ge interface, relative to the continuing interest in the use of Ge intermediate layers on low-cost substrates for growth of polycrystalline GaAs cell structures.

The polycrystalline p-n junction structures grown during the quarter involved Zn doping, in a range of concentrations, in the p-type layers. Examination of stained angle-lapped multilayer samples failed to produce any evidence of Zn diffusion along grain boundaries out of the p-type regions and into (or through) the n layers. Also, when step-etching techniques were employed to remove an upper layer and expose the next lower layer (of opposite conductivity type, below the junction) all tests indicated the proper conductivity type and at least approximately the expected carrier concentration. However, the I-V characteristics indicated the junctions were consistently leaky or, in some cases, completely shorted. Leaky I-V characteristics were found even when highly asymmetric doping concentrations (i.e., p^+/n and p/n^+) were used in forming the junctions.

Somewhat encouraging results were obtained, however, with polycrystalline junction structures grown in the p^+-i-n^+ configuration (the undoped i region is n type) on substrates of large-grained bulk Mo sheet and also sputtered Mo films on Corning Code 0317 glass. The I-V characteristics of those samples examined just prior to the end of the quarter indicated considerably lower junction leakage. However, the continuing problems encountered in preparing grown p-n junction structures in small-grained polycrystalline GaAs films on low-cost substrates demonstrate the importance of obtaining a more complete understanding of the fundamental structural and electrical properties of these films, and the contract work in the next quarter will emphasize studies directed toward achieving such information.

4. PLANS FOR NEXT QUARTER

Contract activities in the sixth quarter will follow the Management Plan as given in revised form in the Monthly Technical Status Report for December 1977. By task, the planned activities include the following:

Task 1

1. Continue evaluation of graphites of various grades and surface finishes as substrate material for GaAs deposited by the MO-CVD process
2. Continue use and evaluation of conducting composites (especially those involving Mo films) and of composites with Ge intermediate layers as substrates for polycrystalline GaAs films
3. Begin study of grain size vs thickness in polycrystalline MO-CVD GaAs films.
4. Begin investigation of effects of presence of HCℓ vapor during film growth on grain size and other film characteristics
5. Commence examination of effects of *in situ* and post-growth annealing on properties of polycrystalline MO-CVD GaAs films
6. Continue attempts to obtain new or improved substrate materials, beyond those already in use.

Task 2

1. Begin investigation of transport properties and impurity segregation effects in polycrystalline GaAs films on low-cost substrates
2. Continue study of morphology of film growth surface as MO-CVD growth process proceeds
3. Continue efforts to improve film growth and doping procedures, and prepare single- and multilayer structures as required for other Tasks
4. Perform routine characterization measurements to determine structural and electrical properties of polycrystalline films, and continue to explore improved methods for deterimining grain size and carrier diffusion lengths in these materials.

Task 3

1. Conduct additional investigations of p-i-n and similar (e.g., p+-p-i-n+) junction structures in polycrystalline GaAs films grown on low-cost substrates by MO-CVD process

2. Undertake detailed examination of properties of Schottky barriers on n-type polycrystalline GaAs regions exposed by etch-removal of Zn-doped p-type upper layers

3. Begin investigation of ITO/GaAs heterojunction formation techniques and properties of resulting structures

4. Conduct preliminary evaluation of other alternative barrier formation techniques (ion implantation, thermal diffusion).

Task 4

1. Investigate alternative contact material systems with reduced sintering-temperature requirements for use on polycrystalline GaAs films

2. Revise design of photomasks for fabrication of complete polycrystalline cell structures, including contacts, as required

3. Investigate deposited ITO layers as possible contacts for polycrystalline GaAs thin-film solar cells

4. Begin study of formation and properties of deposited TiO_2 films for use as AR coatings for GaAs solar cells

5. Fabricate and characterize photovoltaic properties of polycrystalline and single-crystal window-type GaAs solar cells.

Task 5

1. Resume analysis of present total costs of fabricating complete thin-film GaAℓAs/GaAs solar cells using MO-CVD process on laboratory scale

2. Update projections of future materials requirements for fabricating large quantities of cells of present design(s) on selected low-cost substrates.

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