Report No. AAMU-NAS-002

(NASA-CR-149672) THE STUDY OF CRYSTALS FOR SPACE PROCESSING AND THE EFFECT OF O-GRAVITY (Alabama Agricultural and Mechanical Coll.) 43 p HC A03/MF A01 CSCL 20L G3/76
N77-18921
Unclas
17276

FINAL REPORT

on

NASA grant NSG-8033

THE STUDY OF CRYSTALS FOR SPACE PROCESSING AND THE EFFECT OF O-GRAVITY

February, 1977

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NAME OF THE INSTITUTION:	Alabama A. and M. University Normal, Alabama 35762
TITLE OF GRANT:	The study of crystals for space processing and the effect of 0-gravity.
TYPE OF REPORT AND PERIOD COVERED:	Final Report; Jan. 15, 1976 till Feb. 14, 1977
NASA GRANT NUMBER:	NSG-8033
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REPORT NO.:	AAMU-NAS-002

FOREWORD

The present report, No. AAMU-NAS-002, dated February, 1977, of Alabama Agricultural and Mechanical University, Normal, Alabama 35762, is the final report for NASA grant NSG-8033, entitled, "The Study of Crystals for Space Processing and the Effect of Zero-Gravity." This report covers a period from Jan. 15, 1976 till Feb. 14, 1977.

The work reported herein was done under the technical direction of Mr. Tommy C. Bannister and Mr. Charles C. Schafer of NASA/MSFC. Their useful comments are gratefully appreciated.

The author is thankful to Dr. Bessie W. Jones and Dr. Jerry R. Shipman for their interest in the work and to Mr. Collins Ikeakamnonn and Mr. Vincent I. Orizu, undergraduate students working under the project, for help in collecting the data.

ABSTRACT

The purpose of this investigation is to study the mechanism of crystal growth by solution technique and how it will be affected by space environment. Also investigation has been made as to how space processing methods can be used to improve the promising candidate materials for different devices.

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I. INTRODUCTION

The promise shown by results of skylab I and II and ASTP experiments on material science as discussed and presented in meetings[1,2] at NASA/MSFC are in confirmation of the possibility of processing of materials in space on a routine basis. These results have logically placed an emphasis on simple crystal systems. The results of flight experiments to date have shown excellent promise and should prompt a closer look at the opportunities for more complex, and more valuable, compound materials.

Many technologies are dependent on single crystal materials to varying degrees. The materials can be processed for making efficient semiconductors to use in the field of communications, materials which will make better super-conductors for control and distribution of energy, materials for dielectric-elastic-magnetic crystals and materials for non-linear optical devices. The eventual processing of materials in space is likely to become a major economic use for space technology. The scarcity of space flights in the decade of 70's prior to the space shuttle have made this project timely in providing information on which to base research and development programs in the field of crystal growth, and specially for materials needed for different device application.

In an earlier work,[3] a "handbook of materials," was compiled for materials which are useful from the technological point of view and have potential for improvement in the low-gravity environment.

II. OBJECTIVES

The objective of the work reported herein is to undertake a more detailed investigation of how space processing methods could be utilized to improve the most promising candidate materials identified in the earlier work.[3] The mechanism of crystal growth which may be affected by space environment has been investigated in relation to the most promising candidate materials. The literature on crystal growth from solution, vapor phase techniques has been reviewed and the physical phenomena important for crystal growth in low-gravity environment has been analyzed. Also it has been investigated as how space processing methods could be used to improve the most promising candidate materials identified in the earlier work.[3]

III. CRYSTAL GROWTH PROCESSES RELEVANT TO SPACE ENVIRONMENT

The crystal growth of solid state materials from melt, the vapor phase, and from solution is an art of many years. The temperature profile in a crystal growth system, is of great importance for understanding the transport and growth phenomena in general, and for morphological stability in particular. In growth from melts, the role of interfacial temperature gradients, as well as temperature fluctuations is rather well understood. A good deal of experimental data as well as proven theoretical models are available relating compositional variations with growth rate fluctuations that are induced via natural or forced convective temperature instabilities.[4-6] In solution growth where the interfacial kinetics is in general more complex than growth from melts, it has been reported,[7] that short term temperature instabilities of few millidegrees can lead to structural in homogeneities, such as solvent occlusion. A thorough review of chemical vapor deposition (CVD) systems were put forward by Curtis and Dismukes, and later Rosenberger et. al,[9] reported in detail the heat transfer and temperature oscillations in CVD. The positive effects of micro-gravity on crystal growth and fundamental properties of vapor transport reactions were established by analyzing the results of GeSe and GeTe vapor transport experiments performed on Skylab and ASTP by Weidmier et. al.[1,2]

The techniques of growth from Melt, Float Zone, Vapor phase were discussed earlier in another report.[10]

IV. CRYSTAL GROWTH FROM SOLUTION

Crystal growth from solutions is simple in principle and has many applications. The technique is particularly useful for growth of materials which have high vapor pressure or decompose irreversibly at the melting point. Crystals will be grown from solution if the solution is supersaturated, i.e. it contains more of solute than it can hold in equilibrium with the solid. The growth methods,[11] are based on solvent used, because the equipment, range application, problems and approach are to a large extent determined by the choice of the solvent. However, the more fundamental delineation of the methods could be made on the basis of the methods of producing supersaturation. The growth methods can be divided on this basis.

1. Isothermal methods (constant temperature methods)

   A. Solvent evaporation or solvent concentration charge (mainly used in aqueous and molten salt growth)

   B. Temperature differential (mainly used in hydrothermal, aqueous and molten salt growth; also includes temperature-gradient zone melting when the gradient is moved through the sample).

   C. Chemical or electrochemical reaction (mainly used in aqueous growth)

2. Non-isothermal methods (temperature variation methods)

   A. Slow cooling (mainly used in aqueous, liquid metal solvent, and molten-salt growth)

   B. Temperature-gradient zone melting (when the gradient is imposed over the whole sample).

IV. 1. Advantages of Growth from Solution

In general solution growth can be accomplished at temperatures considerably below the melting point of the material, and the use of lower temperature alleviates many of the problems associated with the melt growth process. The main advantages are listed below.

1) The most important advantage of crystal growth from solution is the control that it provides over the temperature of growth. This makes it possible to grow crystals that are unstable at their melting points or that exist in several crystal forms depending upon temperature.

2) A second advantage is the control of viscosity, thus permitting crystals that tend to form glasses when cooled from melts to be grown.

3) Crystals grown from solution usually have well defined faces as compared with those grown from melts.

4) It avoids strains, reduces vacancy concentration, and sometime reduces dislocations and low angle grain boundaries associated with high temperature growth.

5) Solution growth or low temperature growth is experimentally more convenient. Higher temperature processes are often more demanding on equipment and difficult to control, and harder to keep clean so that products are pure.

Solution growth has had its main success in the preparation of bulk crystals.

There are some disadvantages of a polycomponent growth and they are enumerated below.

1) The additional component will be a contaminant and will have solid stability in the grown crystal.

2) Elimination of the additional component at the growing interface will set-up an upper limit on rate of growth. Diffusion will be important in this process. This may be an advantage in low-gravity and will be a

dominant factor.

3) Because of the concentration gradient at the growing interface, constitutional supercooling will often occur, facets effects, cellular growth and dendritic growth can thus be problems.

IV. 2. Effect of Impurity Adsorption on Kinetics of Crystal Growth From Solution.

Recently it has been reported by Davey,[12] that a presence of a third component can often have dramatic effects on the crystal growth kinetics (third component may be an impurity). Adsorption of impurities onto the crystal faces changes the relative surface free energies of the faces and may block sites essential to the incorporation of new solute into the crystal lattice. These effects may result in changes in growth kinetics and hence, habit modification of the crystalline phase.

From the studies of Davey (loc. cit) it seems worth noting the following points in relation to the mechanism of growth rate reduction by impurity adsorption.

a) Impurity adsorption results in the blocking of key sites on the crystal surface.

b) Impurities which bear a structural resemblence to the crystallizing component may be most effective in kink and step sites, while impurities which are structurally dissimilar to the crystallizing component may be limited to ledge sites.

The following requirements of an experimental study may be elucidated.

1. Substantiation of growth mechanism for pure solution.
2. Measurement of growth rate as a function of impurity concentration at a fixed supersaturation.
3. Observation of step systems on faces growing in pure and impure solutions.

4. Measurement of adsorption isotherm of the impurity onto the crystal faces under consideration.
5. The selection of experimental system in which the structural nature of the impurity in solution is known.

The experimental data should then be correlated with different available models of crystal growth in solution.

V. HOW SPACE PROCESSING METHODS COULD BE USED TO IMPROVE SOME PROMISING MATERIALS

One of the objectives of this study is to undertake an investigation of how space processing methods could be used to improve the most promising candidate materials identified in the work, reported earlier.[3]

In the next few sections different potential candidate materials will be discussed in relation to their potential use.

V. 1. Compounds with High Dissociation Pressures at their Melting Point.

In this category the most important material is gallium Arsenide GaAs which has many applications for different technological usage and has a great potential to be improved in low gravity environment.

V. 1. A Gallium Arsenide (GaAs)

Large single crystals of GaAs can be routinely prepared by several techniques, including czochralski, floating zone, solution growth, horizontal Bridgeman procedure and ribbon technique.

Optimum performance of solid state devices in the microwave frequency range depends upon accurate control of nearly every parameter accessible to the crystal grower. GaAs is the semiconductor with the greatest scope and promise in the microwave device field.[13] The greatest potential of GaAs gunn diodes or transferred electron devices is also well documented. There is still a significant gap between the predicted limits, i.e. 30% efficiency for gunn devices compared with the best published experimental results of 12.5% for gunn diodes.[15]

The relatively low experimental values are due to a great extent to shortcomings in circuit designs and heat sinking, but the quality of expitaxial layers and the contacting technology are also import limiting factors. Quality includes such factors as, crystalline perfection, minimum amount of unwanted impurities and deep level traps, smooth doping profile and exact carrier concentration and thickness. In the case of avalanche devices < 0.5μm thick layers are required with a thickness control of ± 0.5μm and carrier concentration ± 5% or better.

Because of higher band gap energy GaAs is theoretically capable of yielding solar cells with slightly higher efficiency than silicon (24% vs 21%) and is capable of higher operating temperatures. However, the technology of producing GaAs solar cells is more difficult and much greater costs are involved. Interest in the GaAs solar cells has been reviewed as a result of some success achieved with the (AlGa) As - GaAs liquid phase epitaxy (LPE) heterojunction technology which overcomes some key technological limitations encountered with homojunction solar cells. So far the efficiency of such small area (p-p-n) heterojunction solar cells,[16] (13-15%), has been comparable to efficiencies obtained with large silicon cells. It is, however, likely that the silicon results may be surpassed with further work and better grown crystals and films in the low gravity environment.

V. 1. B. Discussion of Growth Methods

Liquid phase expitaxy (LPE) has been an established technique,[17] for the synthesis of a number of superior devices using GaAs. The discovery of near ideal (AlGa) As - GaAs heterojunction gave the LPE technology its greatest impetus since no other synthesis technique has so far been successfully used for heterojunction device preparation. The impact of LPE process has been particularly strong in the Al - containing crystals since it avoids the use of highly reactive Al compounds needed in the vapor phase epitaxy (VPE).

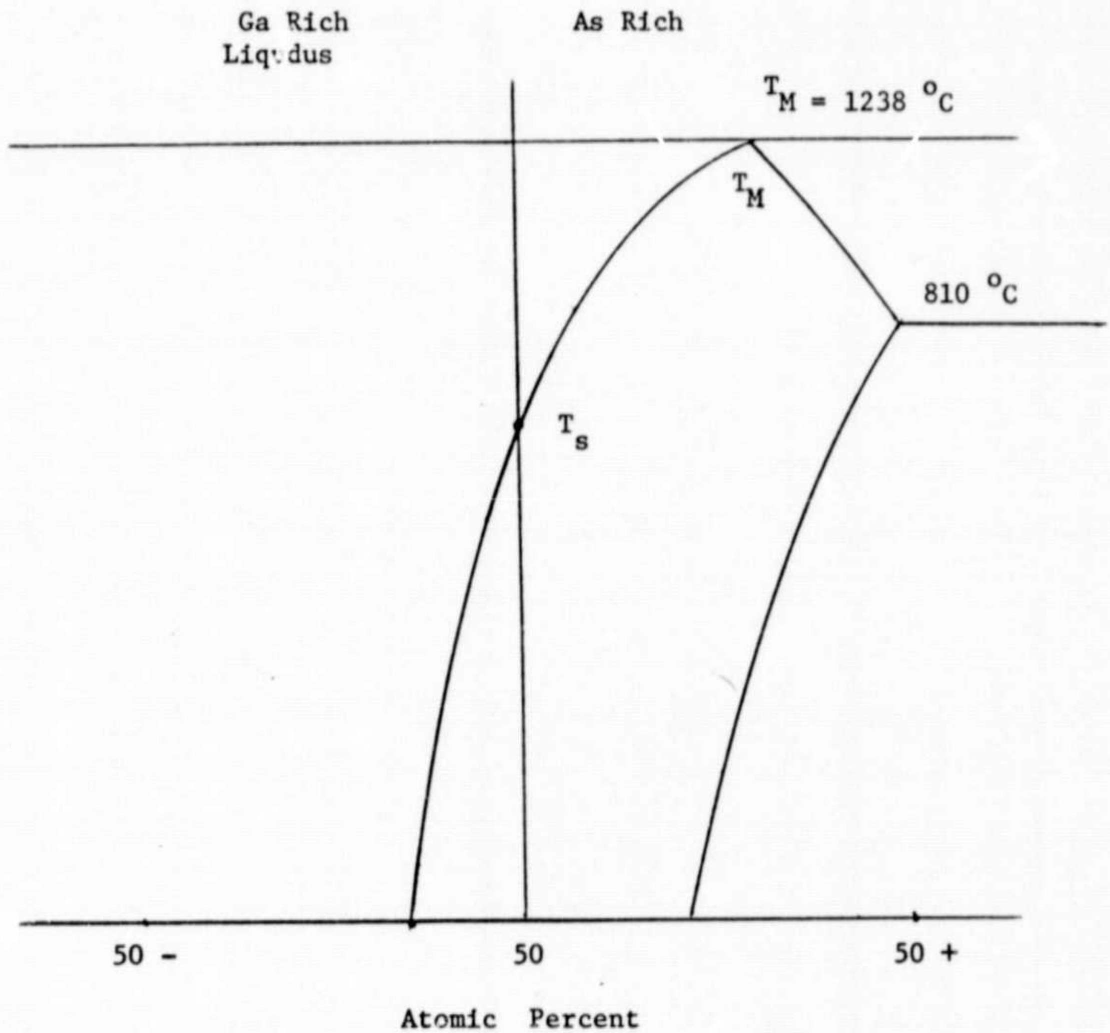

Fig. 1

(Taken from. H.Kressel, J.Electronic Mat. 6, 747(1974)

Three basic growth techniques are used in LPE: the tipping technique in which solution-substrate contact is achieved by tipping the furnace; the dipping technique, in which the substrate is dipped vertically into the solution; and the multibin system, in which layers are sequentially grown by bringing the substrate into contact with different solutions.

Important differences exists between GaAs prepared from LPE, VPE (ref. 18), or growth from melt.

Fig. 1 shows an expanded and idealized GaAs phase diagram,[19] which can serve as the basis for a discussion of the dependence of the crystal stoichiometry on the growth conditions. When grown at its melting temperature T_M, from a Ga - As melt, GaAs crystals usually contain a large concentration δ, of Ga vacancies indirectly estimated to be as high as 10^{17} - 10^{19} cm^{-3} (ref. 20). While the vacancy concentration reduces during the cooling of the crystal, a significant density remains, particularly vacancies associated with impurity atoms. It is evident from fig. 1 that the gallium vacancy concentration can be reduced by growing the crystal below T_M from a GaAs solution. At a growth temperature T_s (T_s $\sim$ 700-800°C) the Ga vacancies are almost zero, while some Ga and As vacancies of relatively low concentration are formed as required by thermodynamic considerations.

The temperature range used for LPE growth (700-800°C) is therefore most desirable from the point of view of stoichiometry. VPE growth also is carried out in the same temperature range but differs from LPE in that the crystal stoichiometry is very sensitive to the relative pressures of the gaseous constituents. Crystals grown below T_s, As vacancies will predominate. Therefore, dopants which can replace either Ga or As in the crystal lattice will be incorporated differently in GaAs as the growth temperature decreases.

LPE is most widely used today for the preparation of GaP, GaAs, (AlGa) As, as well as magnetic bubble garnets because of some unique advantages of LPE over VPE. In general LPE is advantageous because of the fact that:

1) The equipment is simpler than required for VPE.

2) Higher deposition rates can be achieved, thus allowing the growth of thick epitaxial layers in convenient time periods.

3) The choice of dopants for LPE is wider than for VPE, and unusual properties can be obtained in some cases because of stoichiometric differences.

4). III-V alloys containing Al can be grown more conveniently by LPE because the corrosive gases needed in VPE are avoided.

The limitations of LPE technique are however severe under certain circumstances. Reproducible ternary or quarternary alloy growth is difficult where the distribution coefficients of one or more constituents vary widely. Furthermore, when LPE growth occurs over a wide temperature interval, homogeneity in the growth direction is not easily attained. Also LPE is difficult to use when substrate and deposited layers have grossly dissimilar lattice constants.

In the environment of low gravity, convection and sedimentation become negligible. Diffusion should be the predominant mixing mechanism. Experiments to test this hypothesis were performed in space during the Apollo-Soyuz Test Project (ASTP).[21] The results proved the feasibility of the solution growth technique and with further refinements, should yield superior crystals of variety of materials. In particular it seems reasonable to predict that LPE technique to grow GaAs epilayers in low gravity environment can significantly improve the layer quality and in turn the technology of GaAs devices.

V. 1. C. Gallium Phosphide

Because of high melting point and dissociation pressure, the growth of GaP is so difficult that good large crystals are difficult to reproduce. GaP has a large potential for high efficiency low current light emitting diodes (LED) in the commercial market. Growth in low gravity environment to produce large area substrates will be a potential improvement in the technology. It has been reported earlier[3, 22, 23, 24,] that GaP is well suited for electro-optic modulators and can serve as an extension of GaAs which has been successfully used for electro-optic modulation of infra-red radiation.

The preparation of acceptable high purity GaP may depend on development of low temperature solution methods and the adaptation of vapor or chemical methods to the growth of sizable crystals or single crystals.

V. 2. Compounds with Low Dissociation Pressures at their Melting Point.

V. 2. A. InSb

In this category the most important and potentially useful material is indium antimonide (InSb). InSb crystals have a large potential use for infra-red filters and detectors. Normally on earth grown crystals thermal gradients necessary for crystal growth lead to the thermal convection which in general causes uncontrolled variations in the solidification rate and in diffusion boundary layer thickness and macroscopic segregation in homogeneities. It was established by Witt, et. al[1, 2,] that ideal diffusion controlled steady state conditions, never accomplished on earth, were achieved during the growth of Te-doped InSb crystals. Surface tension effects were found to establish non-wetting conditions under which free surface solidification took place in confined geometry. The results obtained prove the advantageous conditions provided by outer space.

V. 3. Materials for Radiation Detectors

There has been an increasing interest in detectors requiring less cooling or no cooling at all, even if they have lower performance, which may lead to systems with greater cost-effectiveness. The two detectors in this class are the pyroelectric thermal detectors using triglycine sulphate (TGS) and photoconductive detectors using (HgCd)Te optimized for 3-5μm region at -80°C and above. It has been shown by Morten[25], that performance of TGS is better for frequencies below 1 KH_{21} and that of (HgCd)Te better above that frequency. The low frequency performance of TGS is of particular importance in space applications where modulation in provided by rotation, or where the provision of high frequency modulation consume too much power.

V. 3. A. <u>Triglycine Sulphate</u>

Present aqueous solution growth technique on earth results in crystals with flaws and inclusions of solvent. Also cutting and polishing of crystals introduces strain and defects which modify the ferroelectric properties and degrade the device behavior. The growth of this material in low-gravity environment should yield large, flawless crystals. The benefits of the solvent methods in contrast to melt methods are important for substances which decompose before melting, for example KDP or TGS. So the micro-gravity environment of space is believed to offer considerable promise for substantial increases in crystal perfection when crystals are grown from aqueous solution. Essentially quiescent conditions at the growth interface are highly desirable for the growth of high perfection crystal. If the need of stirring is also eliminated, the complexity of apparatus is greatly reduced. Solution growth will of course, require longer duration for actual production of bulk crystals. All this can be accomplished on Shuttle Flights in the 1980's. It is recommended that TGS may be grown by aqueous solution method in the low-gravity environment. The increase in stability at the interface without convection should allow growth at higher supersaturations of solute in solvents. Also improvements in the performance of TGS for the above applications by doping the crystal by l-alanine has been recently reported by Bye. et. al[26] and White et. al[27].

V. 4. Materials for Magnetic Memory Devices

In 1967, Boebeck[28], presented a new memory and logic device using magnetic bubble domains in thin platelets of transparent magnetic single crystals. Experimental magnetic bubble memory systems have then been assembled and tested and are in great demand of improved single crystal magnetic films and substances for widespread use[29].

The restrictions on the magnetic film are severe and are indicated in several articles[30, 31]. Materials with very low defect densities are required. The initial step in achieving high quality magnetic films for bubble domain memory devices is the preparation of a suitable substrate free from defects which would interfere with the subsequent growth of epitaxial film.

For magnetic single crystal growth, the possible benefits of space processing must be investigated in terms of solvent growth. It is reasonable to expect that growth from solvent will become increasingly important for future materials. The kinetics at the liquid-solid interface are of utmost importance. A stable interface region is required to eliminate compositional gradients and to ensure uniform growth rates. Without gravity, it may be possible to grow very uniform thickness films by LPE.

The specific materials which have potential for improvement in their crystal quality and perfection by growing in low gravity environment are: $Sm_{0.55}Tb_{0.45}FeO_3$, $FeLiO_8$, $Tb_3Fe_5O_{12}$, BiCaVlG, YCaVlG, $Y_{2.5}Gd_{0.5}GaFe_4O_{12}$.

The non-cubic garnets are extremely attractive because of the feasibility of growing thin film by liquid epitaxial growth or by hydrothermal epitaxy.

V. 5. Materials for Energy Conversion Devices

In this time of rising concern over energy, it will be appropriate to consider the growth of materials needed for efficient energy conversion devices. A possible benefit of micro-gravity will be to grow single crystal films for these devices. The general objective at present is to develop low-cost, long lived, reliable photovoltaic conversion systems to be commercially available for a variety of terrestrial and other applications. The more specific objective is to reduce the cost of solar cell arrays made from silicon wafers by a factor of more than 10 and to investigate the growth possibilities of other materials for solar cell technologies.

As useful as solar cell have been in space program, their potential importance for large scale power generation to meet earth's energy need is even greater.

The silicon solar cell has proved its merit on many space flights. A fraction of the incident optical energy, typically 10 to 12 percent, is converted directly to electrical energy. For large scale application in the U. S. energy economy, the problem is entirely one of cost[32]. According to Claassen[33], solar cell arrays now cost \$20,000 to \$80,000 per peak kilowatt compared to approximately \$500 per kilowatt for conventional steam electric generators (which, of course, require fuel). Cost reduction of two orders of magnitude calls for revolutionary approaches to system design, materials for solar cells, and methods of manufacturing.

One approach is to replace the silicon cell with material or materials that have appropriate semiconducting properties and yet can be deposited as thin films with suitable properties. Materials of this type that have been actively studied include heterojunction material (Cds: Cu_2S, GaAlAs) nomojunction materials such as GaAs, Cds, CdTe.

V. 5. A. GaAs and $Ga_{1-n}Al_nAs$-GaAs

As discussed in article V. 1. A., GaAs homojunctions and heterojunctions are promising candidates for growth in low gravity environment. The most successful method for producing GaAs and $Ga_{1-n}Al_nAs$ for high efficiency devices is the LPE technique[34, 35]. The dopant can be added directly to the melt and will be incorporated the growth in accordance with the segregation coefficient. This will be accomplished in a better way in growth in micro-gravity environment.

The growth of GaAs ribbons is still mostly in the planning stages. The method proposed[36], involves encapsulation of GaAs during the growth process to prevent volatilization at the needed high temperature. It is possible that edge-defined, film fed growth (EFG) may be used, but it would have to be in a closed system to maintain the As pressure at 1 Atm to ensure stoichiometry. The cost advantage obtained with Si ribbon (to be discussed later) would not be obtained with GaAs ribbon of comparable thickness.

V. 5. B. Silicon Solar Cells

The crystal growth methods of most interest for solar cell work are czochralski, float zone, and ribbon methods and chemical vapor deposition. The two techniques which are interesting from the point of view of growth in micro-gravity are float zone and ribbon technique.

In float zone technique the regrown Si crystal emerges with high purity, due both to absence of a crucible in contact with the melt and to the purifying action of the molten zone in preventing impurities with low distribution coefficients from entering the growing crystal. The unique feature of micro-gravity can be of utmost importance of processing of Si - crystals by this technique.

There is an increasing interest in solar cell structures produced using silicon grown in the form of thin ribbons by EFG technique, on a means of reducing the cell cost. In this technique, a thin ribbon-like crystal is pulled from Si - melt at a very fast rate (in/min). Currently, the ribbon technique is probably the least expensive method for producing Si crystals, and it has a high potential for making solar energy conversion using solar cells cost competitive with other methods of generating large amount of electrical power[37, 38]. It has been shown by Kressel et. al[39], that epitaxial layers grown on polycrystalline Si ribbons are crystallographically improved than the substrate and are capable of providing substantially improved solar cells compared to cells produced by direct diffusion. Processing of Si - ribbons in future NASA flights will present a tremendous challenge towards the improvement of Si - solar cells.

V. 5. C. Organic Solar Cells

Solar Cells made from organic materials have received little attention in the past, mainly due to their low measured efficiencies (0.1% or less). The ease of fabrication of solar cells made from these materials together with their possible low cost indicates a closer look at their potential as useful devices. Organic solar cells have been made from many materials, like, anthracene, tetracene, and phthalocyanine[40, 41, 42]. In all these devices, the power conversion efficiency has been limited by low quantum efficiencies and not by low voltage output or fill factors. The poor quantum efficiencies in turn are probably due to a very high trap density, which lowers the lifetime, mobility and diffusion length to poor values. Since the diffusion lengths in organic materials are quite low, the materials with highest absorption coefficients over the visible region are most likely to have the highest quantum efficiencies photocurrents. Materials such as phthalocyanine have broad absorption in the 5000 - 9000 Å range of wavelength and films of about 1000 Å will absorp most of the sunlight in this range. The photosensitivity of Mg-phthalocyanine films is strongly enhanced by doping the films with oxygen and possibly with Al also[43]. The low gravity environment is unique for growing doped organic solar cell films which may have high efficiencies.

V. 6. Materials for Acousto-optic Devices

Acousto-optic devices are gaining more importance in the field of optical information processing and display. This will depend upon the availability of better transducers and improvements in the growth of acousto-optic materials. The two most important material requirements are: high figure of merit and low acoustic loss.

The guidelines for the materials as discussed by Pinnow[44], indicate that $PbMoO_4$ and $LiNbo_3$ are very promising materials with $PbMoO_4$ having a higher figure of merit. Both these materials can be grown by Czochralski method. To grow crystal from melt, the compound must melt congruently; that is, the solid and liquid must have the same composition. Also some portion of the solid-liquid interface is in contact with the crucible. Any irregularities on the boat surface will affect the growth and may cause spurious nucleation.

According to Utech[45], the mechanism to account for the origin of dislocations in crystals grown from melt are:

a) Introduction from seed
b) Externally applied stresses
c) Stresses of thermal origin
d) Concentration gradients (thermal convection)
e) Condensation of vacancies
f) Trapping of inclusions.

The results of skylab experiments indicate that possible causes (a, b, c, and d) are substantially eliminated in low-gravity environment. The remaining mechanism seems unlikely to cause any large concentration of dislocations and defects in the crystals. It was established by Witt, et. al.[46] that ideal diffusion controlled steady state conditions, never accomplished on earth were achieved during the growth of Te-doped InSb crystals in Skylab.

The possible benefits of space processing must be investigated for growth of $LiNbO_3$ and $PbMoO_4$ crystals by Czochralski method.

V. 7. Materials of Electro-optic Devices

Electro-optic materials are needed for optoelectronic devices which convert electrical energy into optical radiation or vice versa, and for those which detect optical signals, like semiconductor lasers, photovoltaic devices, electro luminescent devices and photodetectors.

The most promising materials in this category were identifed in an earlier report (ref. 3) and they are: ZnO, $LiIO_3$, $LiNbO_3$, GaAs, GaAs-films, GaP, Alloys of III-V compounds, YAG crystals, $CaCo_3$, and Bismuth Titanate $Bi_4Ti_3O_{12}$.

Without gravity, it will be possible to grow very uniform thickness films by LPE. Low gravity environment may be extremely useful to grow films of Titanates, ZnO and GaAs by LPE.

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