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# A Simple Inexpensive Experimental Apparatus for Crystal Growth Studies*

R. VENKATACHALAM
*Department of Mechanical Engineering, Regional Engineering College, Warangal—*506 004, *India*
A. RAMACHANDRA REDDY
*Department of Physics, Regional Engineering College, Warangal—*506 004, *India*

*Single crystals, being highly stabilized materials, are important in scientific studies and electronic industries. They may be grown using crystal pulling techniques which require moving a seed crystal smoothly at very low and uniform velocities in an electric furnace. In this paper an inexpensive method is proposed which ensures the required slow movement. An experimental apparatus was designed and fabricated, attempts were made to grow single crystals, and the results were found to be good.*

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*Nomenclature*

| | |
|---|---|
| $A$ | area of plate |
| $B$ | buoyancy force |
| $C$ | constant, see equation (5) |
| $F$ | force of viscous resistance of Coulomb friction force |
| $h$ | thickness of the fluid layer |
| $m$ | mass of the body |
| $P$ | net driving force |
| $t$ | time |
| $T_0$ | melting point temperature of the melt, see Fig. 1. |
| $V$ | velocity of the plate |
| $v$ | velocity of fluid particles, $0 \leqslant v \leqslant V$ |
| $W$ | weight of the piston assembly including the weights added on the pan |
| $\tau$ | shear stress |
| $\mu$ | absolute viscosity |

## INTRODUCTION

A SINGLE crystal is one in which the structure of the material extends periodically throughout the material. In contrast, in polycrystalline materials, the periodicity is interrupted at the grain boundaries. The size of the grains in which the structure is periodic may vary from few angstroms to a macroscopic dimension. When the size of the grain becomes comparable with the size of the pattern unit, the periodicity of the structure can be disturbed completely and it is no longer either a single crystal or a polycrystalline material; it is called an amorphous substance.

Good single crystals are essential for a variety of scientific and commercial purposes. They are needed for scientific appraisal of crystallography, topography, and tensor properties of all crystalline materials. Being highly stabilized materials, the single crystals find their application in the electronic industry; examples include ruby, yttrium aluminium garnet (YAG), and titanium in laser applications, quartz for timers, permalloys in computer memory elements, etc.

The rate of crystal growth involves the control of a phase change. The three categories of the crystal growth process are:

(1) solid growth, which involves solid to solid phase transition;
(2) melt growth, which involves liquid to solid phase transition;
(3) vapour growth, which involves gas to solid phase transition.

This paper is concerned with melt growth. One widely used method, called 'the crystal pulling method' which was developed by Czochralski [1], is described in Fig. 1. The figure shows an electric

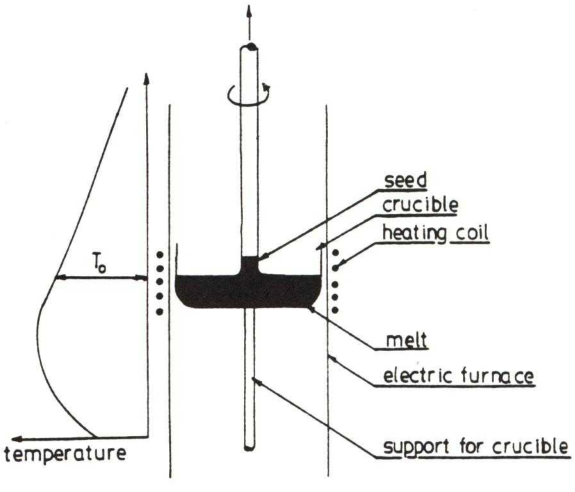

Fig. 1. The crystal pulling method.

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furnace, and a typical temperature variation within the furnace. A crucible containing the melt is kept at a position at which the temperature of the furnace is just above the melting point temperature $T_0$ of the melt. Into the melt is dipped the seed crystal which is withdrawn at a very slow rate to form an ingot which is often a single crystal. The seed crystal may be rotated slowly in order to keep the growing crystal uniform, and cylindrical in shape. Instead of rotating the seed crystal while it is being pulled up, one may simply withdraw the seed crystal and rotate the crucible.

The rate at which the seed crystal is pulled up is of order 5 to 10 mm per hour. This linear motion must be free from sudden movements or vibrations, as even a slight disturbance can spoil the growth of a crystal. Mechanisms involving speed-reduction gears are available for pulling the seed at very slow linear velocities. However, they are very expensive and it is very difficult to produce jerk-free motion. Attempts have also been made to make use of the falling level of water in a tank, in which the water is drained out slowly and uniformly [2–5]. The motion of a float on the water surface is passed to the seed via a string which is passed over pulleys. These attempts have been found to be not very successful because the motion obtained was not smooth due to friction in the bearings of the pulleys. Mercury has been used in place of water [2] which is particularly suitable for vacuum furnaces. However, this is also expensive as mercury is involved.

This paper describes an attempt made to achieve the required slow motion by creating motion of a body under the influence of a constant force in a viscous environment. The development of the final apparatus is described and the results presented.

## DEVELOPMENT OF THE BASIC PRINCIPLE

Figure 2 shows a plate of area $A$ which is separated from a stationary surface by a layer of viscous fluid of thickness $h$. The plate is moved with a velocity $V$ by applying a force $F$. The fluid particles adhering to the stationary surface have zero velocity while the particles adhering to the surface of the moving plate have velocity $V$, the same as that of the plate. The fluid particles within the layer have velocities $v$ ranging from zero to $V$. The figure also shows the velocity profile. Using Newton's law of viscosity [6] it may be written,

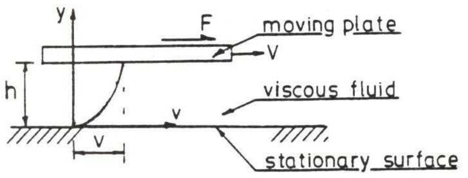

Fig. 2. A moving plate separated from a stationary surface by a viscous liquid.

$$\tau = \mu(\mathrm{d}v/\mathrm{d}y) \tag{1}$$

where $\tau$ = shear stress in fluid at a height $y$, $0 \leqslant y \leqslant h$, $\mu$ = absolute viscosity (N s m$^{-2}$), $\mathrm{d}v/\mathrm{d}y$= velocity gradient.

If the thickness of the fluid layer is very small ($h \to 0$), the variation of the velocity of the fluid particles may be assumed uniform, and the velocity gradient may be approximated as

$$\mathrm{d}v/\mathrm{d}y \simeq V/h \tag{2}$$

Hence, Eqn (1) may be written as,

$$\tau = (F/A) \simeq \mu(V/h) \tag{3}$$

or

$$F = CV \tag{4}$$

where

$$C = \mu A/h \ (\mathrm{N\,s\,m^{-1}}) \tag{5}$$

For a given configuration, $C$ may be treated as a constant. It may also be noted that when the fluid layer is of very small thickness, the force required to move the plate is proportional to the velocity of the plate [7].

Now consider a body moving in a viscous medium under the action of a constant force. Figure 3 describes a typical kinematic arrangement. A cylindrical body of mass $\boldsymbol{m}$ is kept in a cylindrical jar containing viscous fluid. The diameter of the body is smaller than the diameter of the jar providing an annular gap through which the fluid may pass from the bottom to the top of the body as the body travels down. The flow through this narrow annular gap provides viscous resistance while the weight of the body is a constant driving force. The conditions of flow of the liquid through the narrow annular gap is similar to that in Fig. 2. Hence, the model described by Eqn (4) may be employed.

Consider the free body diagram of the moving body, as shown in Fig. 4. The equation of motion may be written as

$$P - C\dot{x} = m\ddot{x} \tag{6}$$

The force $P$ is a constant driving force which in this case is due to the weight of the body. In the later discussion, the force $P$ will not be due to the weight of the body alone, but will include any additional weights that may be added to the body. Further,

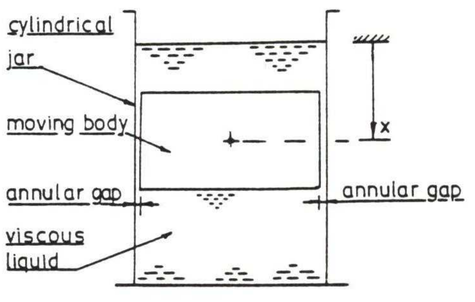

Fig. 3. A body moving in a viscous medium.

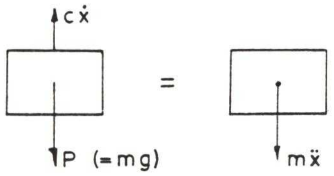

Fig. 4. A free body diagram.

*P*may also be effected by any possible Coulomb friction. Whatever the case, *P* is a constant force.

Equation (6) may be rewritten in terms of velocity $V(=\dot{x})$ as,

$$m\dot{V} = P - CV \tag{7}$$

With an initial condition, $V = 0$ at $t = 0$, the solution for Eqn (7) may be written as,

$$V = (P/C)\,[1 - \exp(-Ct/m)] \tag{8}$$

Figure 5 shows a typical building-up of velocity, to a constant value of $P/C$. This shows that when a body is in motion in a viscous medium, under the action of a constant driving force, the body attains a constant velocity. This may be explained physically as follows: while the constant force has a tendency to build up the velocity uniformly, the viscous resistance which is proportional to the velocity also builds up uniformly opposing the motion, and finally an equilibrium state of uniform velocity is attained. A practical example of this is falling raindrops, which, under the action of gravitational force in a viscous medium (atmospheric air), attain a constant velocity [8].

If the parameters, namely, the viscosity of the fluid, the narrow passage dimension, and the magnitude of the constant driving force, are so adjusted, it may be possible to obtain a desired low velocity. An example of this is a stone thrown into a drum containing tar which takes several days to sink.

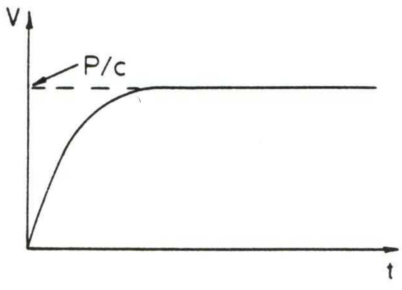

Fig. 5. A typical variation of velocity.

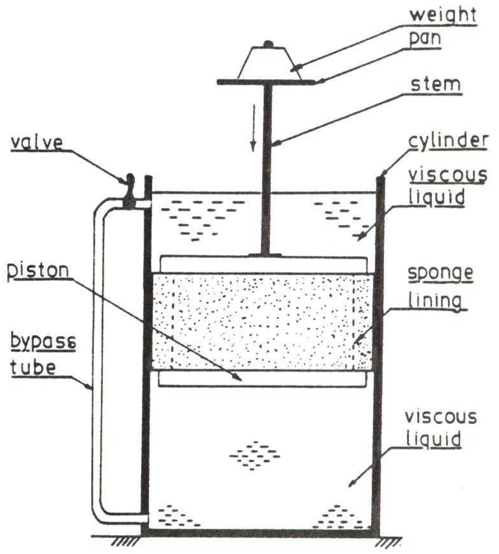

Fig. 6. The final apparatus for the feasibility study.

## FEASIBILITY STUDY

The feasibility of the principle was tested. The final apparatus arrived at by Bhushan Kumar *et al.* [9], after several trials, is shown in Fig. 6. This closely resembles the kinematic arrangement described in Fig. 3. A cylinder liner of an internal combustion engine (diameter 70 mm and length 110 mm) was used to serve as a cylinder as it had a perfect cylindrical bore. A wooden piston with a considerable amount of clearance (around 5 mm radial clearance) between the cylinder and the piston, was made. The piston had a sponge lining to fill the clearance between the piston and the cylinder. The diameter of the sponge lining could exceed the inner diameter of the cylinder by 10 to 20 mm before assembly. When the piston was assembled, the sponge layer was in a compressed state. The sponge lining was used for the following reasons:

(1) its pores contained narrow passages through which the viscous fluid could flow, giving rise to viscous resistance;
(2) it eliminated careful machining of both piston and cylinder surfaces, and could adjust itself to any minor irregularities on the surface of the cylinder;
(3) it provided uniform coulomb friction conditions over the entire surface of contact.

In view of the advantages offered by the sponge lining, even a worn out engine cylinder liner could be used. In fact, in the work reported in this article, the authors made use of used cylinder liners only.

The piston had a steel stem and a pan on which some weights were placed as shown in Fig. 6. the fluid used was glycerine because it is easily available and has high viscosity, and hence it offered more viscous resistance.

The piston is raised to near the top open end of the cylinder, with the viscous liquid below it. During the experiment, the piston travels downwards while the liquid flows through the pores of the sponge around the piston. Finally, when the bottom of the cylinder is reached, the piston stops. The entire experiment takes several hours depending on the velocity of the piston. In order to avoid the liquid having to pass back through the pores before the experiment can be repeated, a bypass tube is provided, as shown in Fig. 6. When the valve is open, the viscous liquid flows through it because it is the path of least resistance. With this arrangement, the piston is raised very easily. However, care must be taken to ensure that the valve is closed before the experiment is performed.

The net driving force $P$ may be expressed as,

$$P = W - F \tag{9}$$

where $W$ is the weight of the piston, the sponge lining, stem, pan and the weights carried by the pan, and $F$ is the Coulomb friction force between the sponge lining and the cylinder surface.

Table 1 shows different velocities obtained with different values of $W$. As expected, the velocity obtained increases with increase in the value of $W$. However, the variation is not linear.

Table 1. Results of a feasibility study

| $W$ (N) | 11.0 | 12.0 | 12.5 | 13.5 |
|---|---|---|---|---|
| Velocity of piston (mm/h) | 4 | 9 | 15 | 32 |

The present study has established that it is possible to obtain very low velocities which are suitable for crystal growth studies. Further, the motion obtained is very smooth and does not involve any sudden movements. This may be because the contact surfaces are very well lubricated and the chances of stick-slip motion [10] giving rise to jolts is greatly reduced. The slow motion may also be used to rotate solar collectors, booster mirrors, etc., in order to track the sun continuously.

## DEVELOPMENT OF APPARATUS SUITABLE FOR CRYSTAL GROWTH STUDIES

In crystal growth studies, the seed must be moved from the middle of a furnace to its top edge at a very low velocity. The apparatus described in the feasibility study may be used straightaway, with the small modification shown in Fig. 7. The downward motion of the piston can be converted to upward motion of the seed, by connecting them by a string which is passed over two pulleys. Though it offers a very simple modification, the motion produced will not be smooth because of friction at the pulley bearings. Hence, a modification is needed which involves fewer moving parts.

The final arrangement derived by Shyam Kumar *et al.* [11] is shown in Fig. 8.

The piston consists of tin sheet with sponge lining. The constant upward force is due to a buoyancy force produced by a closed hollow cylindrical box which is also made of tin sheet. The cylindrical box and the piston are connected by a steel stem whose extension carries a pan on which weights can be added. The net driving force may now be expressed as

$$P = B - W - F \tag{10}$$

where $B$ is the buoyancy force and $W$ and $F$ have the same meaning as before.

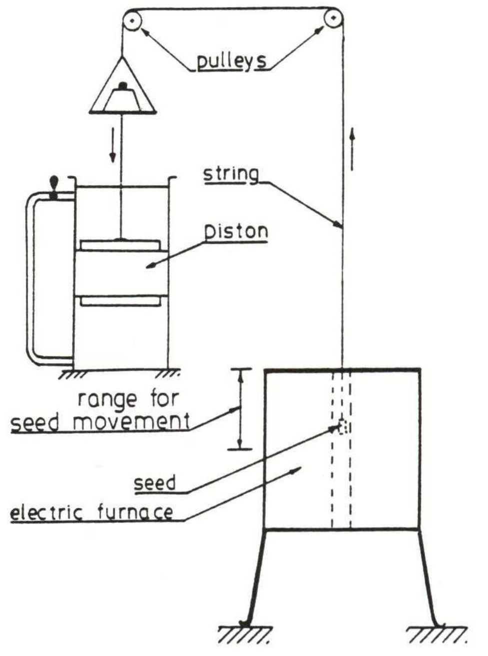

Fig. 7. The first experimental set-up for crystal growth studies.

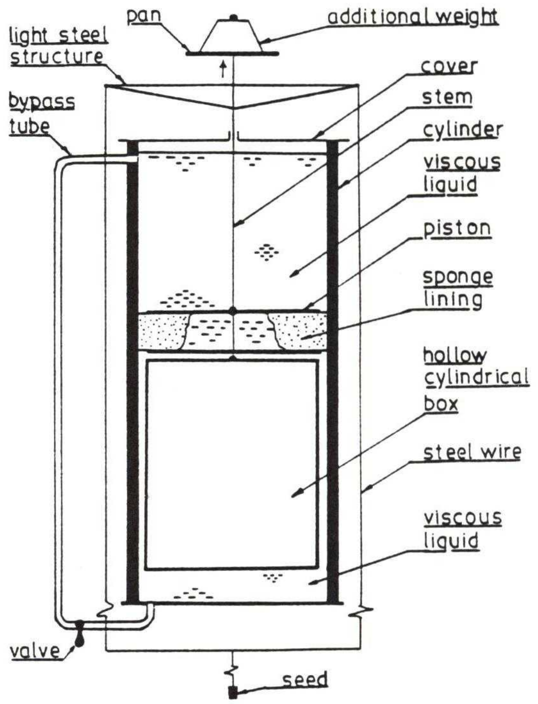

Fig. 8. The final apparatus for providing slow motion for crystal growth studies.

SAE 40 lubricating oil is used to provide the viscous medium. At the beginning of the experiment, the hollow cylindrical box is on the bottom of the cylinder. The oil is around the cylindrical box, and above and below the piston. The oil flows through the pores of the sponge from top of the piston to the bottom, and the piston-box assembly gradually rises at a desired low speed. This upward motion may be utilized directly through a light steel structure, to lift the seed inside the furnace as shown in Fig. 8. At the end of the experiment, the piston is near the top edge of the cylinder. A bypass tube and valve are provided so that the experiment can be easily repeated.

Figure 9 shows a photograph of the experimental apparatus. A used engine cylinder liner of a diesel locomotive, whose dimensions are 230 mm internal diameter and 560 mm length, is used. The piston separates the chambers of the cylinder, and provides a narrow passage for viscous liquid flow, as well as guides the movement of the piston; it is 50 mm high. The hollow cylindrical box should be as large as possible, in order to attain maximum buoyancy force, but care must be taken to ensure it does not touch the sides of the cylinder. The size of the hollow cylindrical box used was 210 mm in diameter and 260 mm in length.

With this apparatus, it is possible to achieve low velocity motions over a distance of 200 mm, which is quite sufficient for crystal growth studies.

It may also be noticed that the entire apparatus is kept on a stand. As shown in Fig. 10, the apparatus may be mounted over an electric furnace. A tray is placed below the cylinder to prevent any oil from falling on the furnace. The apparatus gives very smooth and constant vertical motions of velocities of 2 to 10 mm per hour. The apparatus may be set to give a specific velocity by altering the weights on the pan.

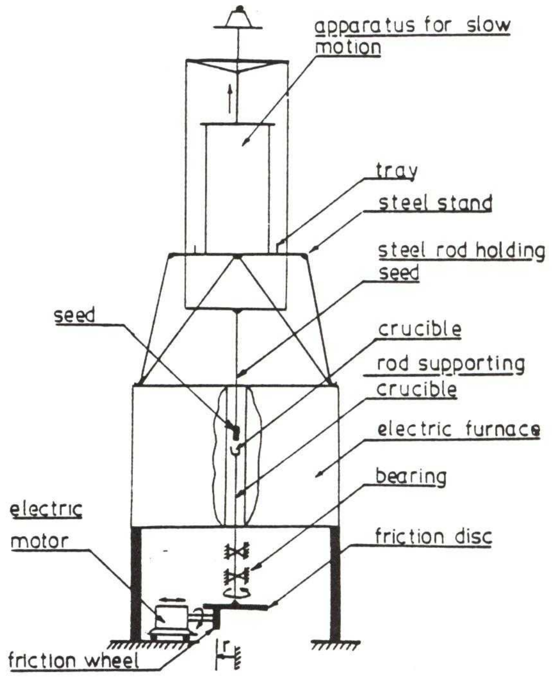

Fig. 10. Growing crystals using the apparatus developed.

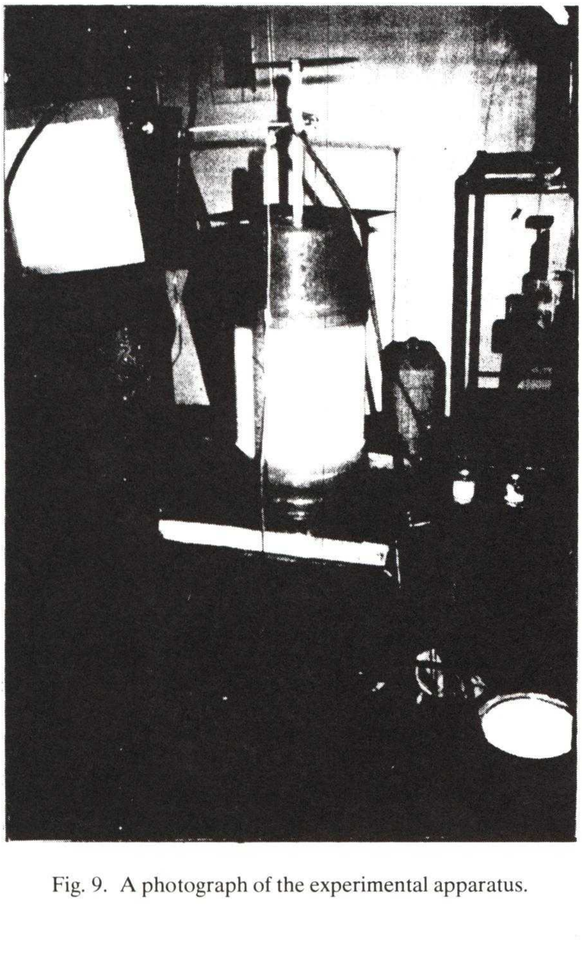

Fig. 9. A photograph of the experimental apparatus.

## EXPERIMENTAL STUDIES

Rakesh Sharma *et al.* [12] attempted to grow single crystals using the apparatus developed. The crucible containing the melt is supported by a rod connected to the bottom of the furnace as shown in Fig. 10. The crucible is rotated by the rod which supports it which is on two ball-bearings; the rotary motion is provided by an electric motor through a friction drive arrangement. The electric motor unit also has a reduction gearing arrangement and the minimum output speed is 10 rpm. The electric motor is mounted on rails and can be moved radially inwards or outwards. Hence, by controlling the distance *r*, it is possible to get even lower speeds for the friction disc and the rod holding the crucible.

In order to produce single crystals of materials like steel, the rods holding the seed and supporting the crucible, and the crucible must be of materials with very high melting point (more than 1600°C). One such material is platinum whose melting point is 1769°C. However, platinum is very expensive and so it was decided to grow crystals of low melting point metals such as lead, tin and zinc, as a first attempt. For these materials, stainless steel rods (melting point = 1540°C) could be used to support the crucible and hold the seed. The crucible used was silica.

Using the experimental apparatus, single crystals of tin were grown. The effect of linear motion of the seed was observed; as the velocity of removing the seed increased, the diameter of the crystal decreased. The single crystals were polished and their micro-structure examined under a microscope. The results were found to be good. The authors are currently trying to produce single crystals of high melting point materials, such as steel.

## CONCLUDING REMARKS

The development of the system described in this paper may be summarized as follows:

(1) A simple and inexpensive method for producing very low and uniform velocities is proposed and developed.
(2) The proposed method involves driving a body under a constant force against viscous resistance.
(3) After a feasibility study of the proposed method, an experimental apparatus is designed and fabricated, for the purpose of growing single crystals.
(4) The apparatus developed gave very slow and uniform velocities of 2 to 10 mm per hour. The motion is completely jerk-free and very smooth.
(5) As a first attempt, single crystals of tin were grown as it has a low melting point. The results were found to be good.
(6) The present set-up can also be used for growing crystals using the Bridgman Technique [13] in which a crucible containing a molten charge is moved slowly relative to a stationary temperature gradient for unidirectional solidification.
(7) The important parameters which effect the rate of growth of crystals are, the velocity of extraction of the seed and the angular velocity of rotation of the crucible. It becomes apparent that the growing of crystals is more of an art than a science.
(8) Since the proposed method does not involve any expensive equipment or construction, it appears to be very attractive.

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**Rapur Venkatachalam** was born in Visakhapatnam, India, on 28 April 1953. He received his B.E. in Mechanical Engineering from Andhra University College of Engineering, Waltair, India, in 1974. He obtained M.Tech. and Ph.D. degrees in Mechanical Engineering from the Indian Institute of Technology, Kanpur, India, in 1976 and 1980, respectively.

He joined the Regional Engineering College, Warangal, India in early 1981, where he is currently an Assistant Professor of Mechanical Engineering. His research interests are in the areas of attitude and orbital dynamics of satellites, control systems, optimization and vibrations.

**Anumula Ramachandra Reddy** was born in Chinna Kistapur, Andhra Pradesh, India on 16 March 1950. He obtained his B.Sc. and M.Sc. degrees in Physics from Osmania University, Hyderabad, India in 1974 and 1976, respectively. He received his Ph.D. in Physics from Regional Engineering College, Warangal, Kakatiya University, India in 1982.

He joined the Regional Engineering College, Warangal, India in 1977, where he is presently an Assistant Professor in Physics. His research interests include the areas of growth and characterization of garnets, crystal growth instrumentation and solar cells.