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INITIAL DEVELOPMENT OF A HIGH-PRESSURE CRYSTAL GROWTH FACILITY -- CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT (PROJECT NO. 87-25)

By F. R. Szofran, S. L. Lehoczky, S. D. Cobb, and D. C. Gillies

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A low-cost, flexible, high-pressure (600 psi) system for crystal growth and related thermophysical properties measurements was designed, assembled, and tested. The furnace system includes a novel, magnetically coupled translation mechanism that eliminates the need for a high-pressure mechanical feedthru. The system is currently being used for continuing crystal growth experiments and thermophysical properties measurements on several material systems including $Hg_{1-x}Cd_xTe$, $Hg_{1-x}Zn_xTe$, and $Hg_{1-x}Zn_xSe$.

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OBJECTIVES AND BACKGROUND OF THE PROJECT

This project initiated the development of a high-pressure facility for future flight experiments and commercial scale crystal growth to facilitate the measurement of thermophysical properties and growth of larger diameter crystals of semiconducting compounds having a very high vapor pressure at the growth temperature. Many of the compound semiconductors currently being developed to meet unique photonic and electronic materials requirements have vapor pressures of tens of atmospheres at the growth temperatures. These materials include all of the important narrow band-gap solid solution semiconducting alloy systems such as $Hg_{1-x}Cd_xTe$, $Hg_{1-x}Mn_xTe$, $Hg_{1-x}Zn_xTe$, and related compounds which are the subjects of several approved space flight experiments. Most III-V compounds such as GaAs, also the subject of a flight experiment, and InP and their alloys also have high vapor pressures. The crystal growth of any of these materials in commercially viable sizes can be done more efficiently, if at all, in a high-pressure furnace. For example, $Hg_{1-x}Cd_xTe$ alloys are typically grown in fused silica ampoules in diameters up to about 1.5 cm, but the vapor pressure prohibits growth of larger-diameter material in ambient pressure apparatus. Requirements are projected for diameters 4 to 5 times larger than the capabilities provided by presently available apparatus, and the long lead times for developing such apparatus require that development begin as soon as possible. Such a facility is being considered for inclusion in the Space Station Furnace Facility being developed for use on Space Station *Freedom*.

APPROACH AND DESCRIPTION OF THE APPARATUS

A high-pressure furnace in which the furnace shell is also the pressure vessel has been described by Ciszek and Evans [1]. A modified version of that furnace, shown in Figure 1, has been constructed. All necessary instrumentation is included for pressure control, temperature control, translation, and temperature measurement to carry out experiments in the furnace including crystal growth by directional solidification (Bridgman) or the quench-anneal method and differential thermal analysis for phase diagram determination of II-VI Hg compounds such as $Hg_{1-x}Zn_xTe$ and $Hg_{1-x}Zn_xSe$ which are materials being investigated in a current flight project.

The furnace has high- and low-temperature zones, a gradient zone, and a booster heater at the high-temperature end of the gradient zone to enable the achievement of higher temperature gradients. The actual bore diameter is 22 mm which will permit sample containers to be used with outside diameters up to 20 mm. This, in turn, will allow the growth of ingots up to 18 mm in diameter. An example temperature profile used to grow $Hg_{0.8}Cd_{0.2}Te$ is shown in Figure 2. An additional feature of the design is that the furnace core and insulation package are modular so that replacing the core to achieve additional thermal profiles is straightforward and nondestructive. Thus, additional cores could be constructed and easily interchanged as required for different experiments.

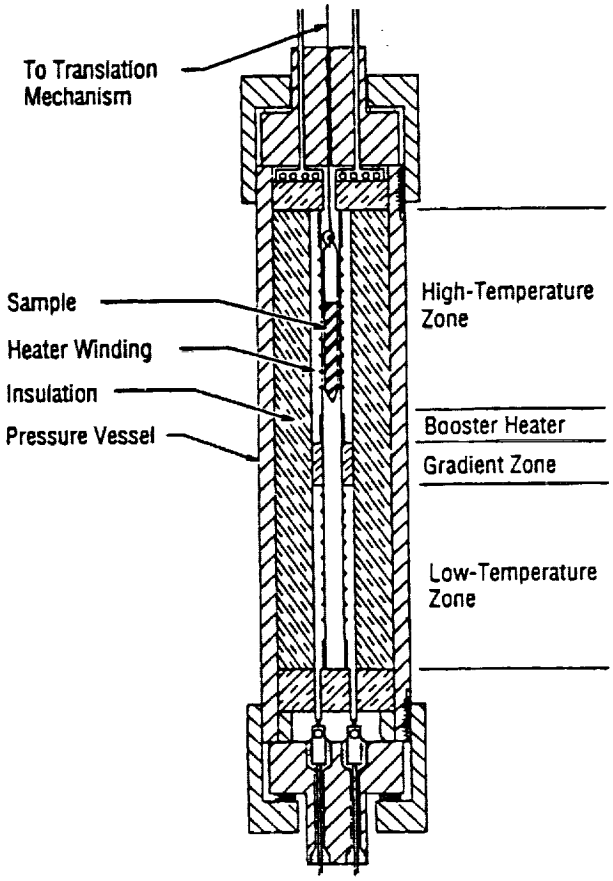

Figure 1. Schematic drawing of the high-pressure part of the furnace excluding the translation mechanism.

The furnace system includes a novel translation mechanism that uses magnetic coupling and which eliminates the need for a high-pressure mechanical feedthru. The translation rate range is determined by a combination of a dc motor and gear reduction box connected to a ball lead screw. Thus, any translation rate of interest for crystal growth can be achieved.

The argon gas handling system was safety tested up to 1100 psi but pressures above 600 psi will not be needed in the laboratory. The pressure inside the furnace is under closed-loop control and can be programmed to follow a pressure profile such as the pressure inside an ampoule during the temperature profile imposed during crystal growth or thermophysical properties measurements. A schematic diagram of the furnace control functions is shown in Figure 3. The pressure system has been used successfully during several $Hg_{0.8}Cd_{0.2}Te$ crystal growth experiments.

RESULTS

For a modest overall cost, a highly functional and safe high-pressure furnace system has been designed, assembled, and tested. In addition to the individual tests of the thermal and pressure subsystems, two $Hg_{0.8}Cd_{0.2}Te$ ingots have been directionally solidified in the furnace as a system test. The temperature profile is shown in Figure 2 and the pressure profile was designed to equalize the pressure on either side of the ampoule wall. The pressure inside the ampoule was calculated from the known pressure over $Hg_{0.8}Cd_{0.2}Te$ [2] and is approximately one-third the equilibrium vapor pressure of pure mercury. Other parameters are given in the following table.

Test No.	Ampoule o.d./i.d. (mm)	Translation Rate (mm/hr)	Length Grown (mm)
HP-1	12/8	0.35	33
14-1	14/8	0.35	129
14-2a	14/8	0.36	50
14-2b	14/8	0.29	48

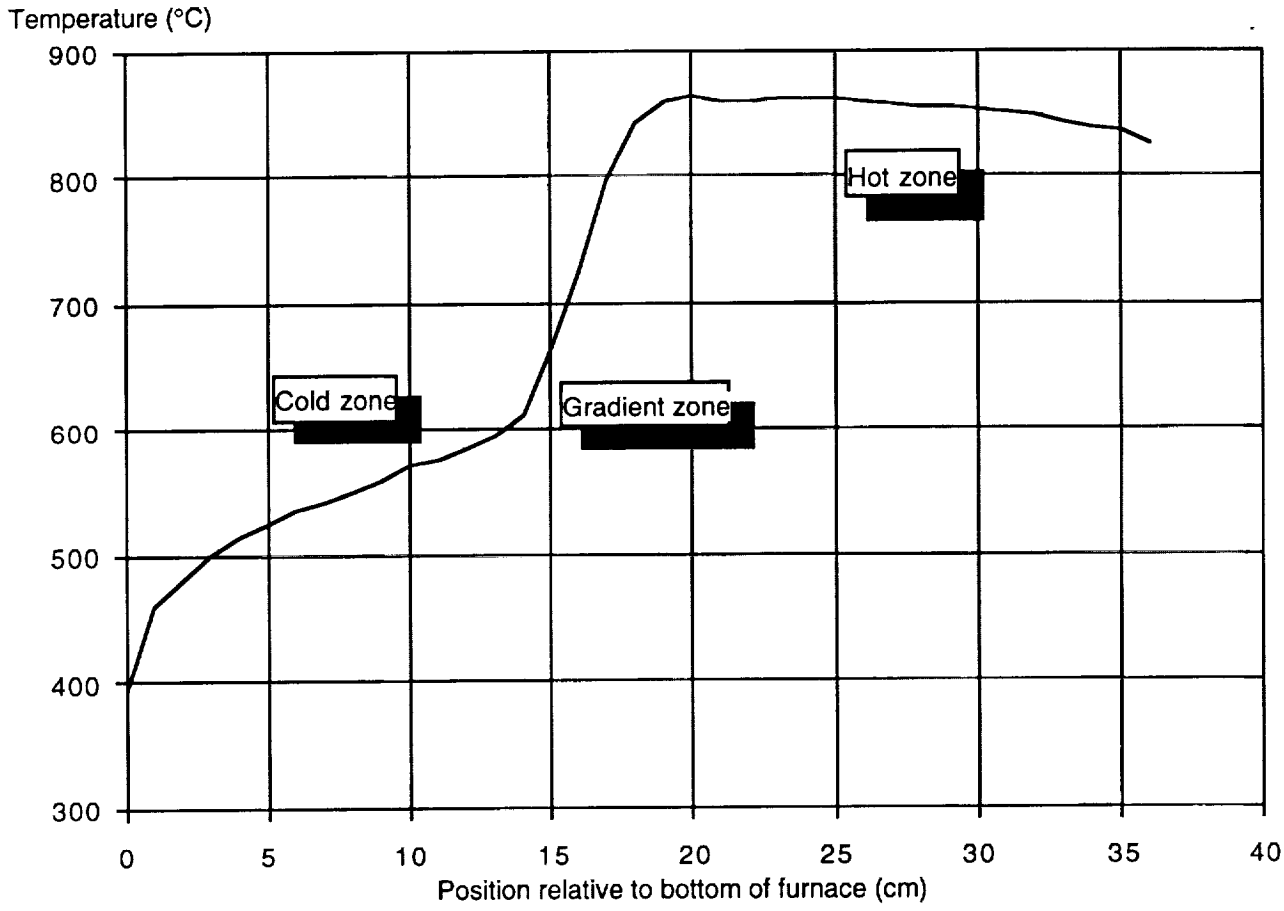

Figure 2. Furnace thermal profile suitable for growing $Hg_{0.2}Cd_{0.8}Te$.

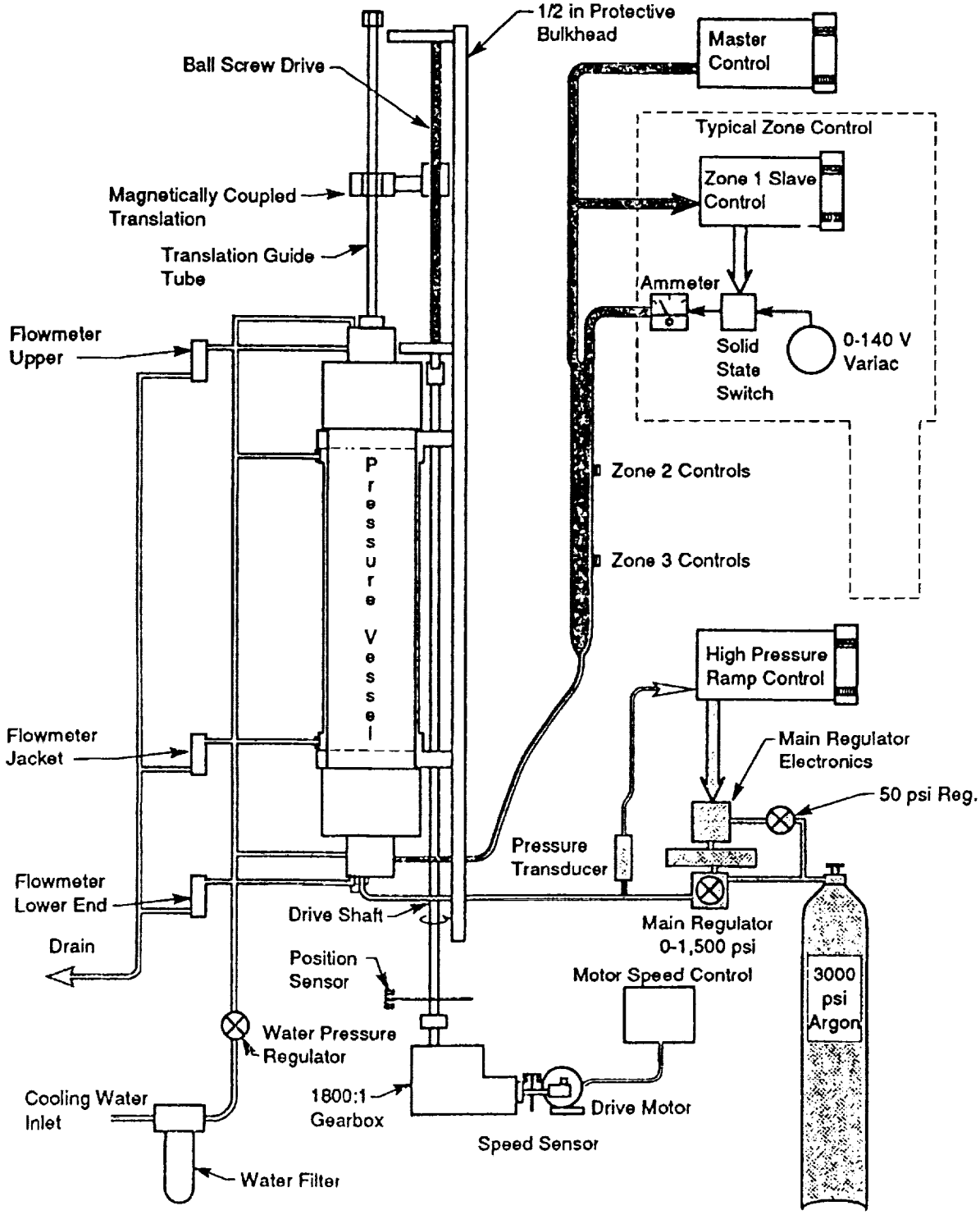

Figure 3. Overall schematic diagram of the high-pressure furnace system.

SUMMARY

The project has shown that a low-cost, flexible, high-pressure system for crystal growth and related thermophysical properties measurements can be assembled. The system is currently being used for continuing crystal growth experiments and thermophysical properties measurements on several material systems including $Hg_{1-x}Cd_xTe$, $Hg_{1-x}Zn_xTe$, and $Hg_{1-x}Zn_xSe$. A pending proposal would use the system for $Hg_{1-x}Mn_xTe$.

REFERENCES

1. Ciszek, T. F., and Evans, C. D., presented Seventh American Conference on Crystal Growth, Monterey, CA, July 12-17, 1987.

2. See, for example, Kelly, J. D., Martin, B. G., Szofran, F. R., and Lehoczky, S. L., J. Electrochem. Soc., **129**, 2360 (1982) (and references therein).

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APPROVAL

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This report has been reviewed for technical accuracy and contains no information concerning national security or nuclear energy activities or programs. The report, in its entirety, is unclassified.

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