# Thermal Characterization of the Universal Multizone Crystallizator

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## Abstract

The Universal Multizone Crystallizator (UMC) is a special apparatus for crystal growth under terrestrial and microgravity conditions. The use of twenty-five zones allows the UMC to be used for several normal freezing growth techniques. The thermal profile is electronically translated along the stationary sample by systematically reducing the power to the control zones. Elimination of mechanical translation devices increases the systems reliability while simultaneously reducing the size and weight. This paper addresses the UMC furnace design, sample cartridge, typical thermal profiles and corresponding power requirements necessary for two normal freezing techniques: dynamic gradient freeze and zone melting crystal growth.

## 1. Introduction

The Universal Multizone Crystallizator (UMC) has been developed and patented by a team at the University of Miskolc for crystal growth of long duration space missions [1]. As a result of a joint agreement between scientists of the University of Miskolc and the NASA Microgravity Science and Applications Division, a test project was contracted this year. On this basis a collaborative study of the UMC was undertaken at the Marshall Space Flight Center (NASA/MSFC), involving the participation of both US and Hungarian investigators. The overall objective of this project is to evaluate the UMC characteristics so as to assess the possibility of pursuing further collaborative activities.

Figure 1 shows the schematical layout of the UMC furnace and support equipment. The furnace consists of 25 independent resistance heated elements separated by fibrous ceramics. The outer furnace shell is held at a constant temperature by a water coolant loop. The furnace operates in a vacuum environment at $10^{-6}$ torr made by a CTI-Cryogenics micro-processor based cryopump [2]. The furnace is capable of processing ten samples by utilizing an automatic cartridge exchange mechanism controlled by an onboard computer. The cartridge depot can be removed from the furnace and returned to earth. Therefore, reducing mission costs by allowing the furnace to remain in space to receive further cartridges. A detailed description of the UMC's power, cooling and control systems are described elsewhere [3]. Table 1 lists the main advantages and capabilities of the UMC.

## 2. Containment cartridge and experimental setup

In order to fulfill the different experiment requirements and taking into account the safety requirements of space flight missions, special cartridges have been developed for thermal testing and crystal growth.

The standard cartridge is shown in figure 2. It consists of a metallic tube with a wall thickness of 1 mm. A graphite liner which can be instrumented with thermocouples is placed inside the metal tube. This liner is used to tailor the thermal profile in the furnace and can be exchanged with other materials as dictated by required thermal gradients. The sample to be processed is loaded into the graphite liner where graphite ampoule supports ensure that the growth axis is aligned parallel with the residual gravity vector. A maximum of 15 thermocouples can be used to monitor the crystal growth process. After the sample is loaded into the graphite liner, an alumina insulator section filled with fiber-frax is pinned to the bottom of the quartz liner. The insulator section limits the heat transfer to the thermocouple connection preventing erroneous measurements. Next, a stainless steel thermocouple feed through is pined to the alumina insulator. A keying mechanism is located on this feed through which allows one to determine the sample's circumferential location in the furnace bore. The cartridge is sealed by bolting a stainless steel alignment/locking assembly to the metallic cartridge. Lastly, thermocouples are connected to a temperature compensated connector located on the alignment/locking assembly.

For temperatures less than 1000 °C, a stainless steel cartridge is utilized. A tungsten cartridge is used for temperatures above 1000 °C. As the operating environment is vacuum, no cartridge coatings are required to prevent oxidation.

## 3. Thermal characterization

A low temperature test cartridge equipped with an ampoule simulator was utilized in a series of experiments to calibrate the furnace controller, establish the zero-point compensation references and to characterize the thermal environment.

*Noise and Power Checking Experiment..*-To calibrate the PID control constants and the control thermocouples, a thermal gradient of 3 °C/mm was established in the UMC. This gradient was then translated at 4.2 μm/sec by varying the power to the heaters. Throughout the experiment, the furnace thermocouples experienced temperature fluctuations from 2-10 °C. These fluctuations were also recorded by the thermocouples located on the graphite liner in the test cartridge. As the graphite liner acts as a thermal leveler, the fluctuations were less significant. The largest being 3.5 °C. By adjusting the PID constants for each zone controller, the furnace control thermocouples were controlled to within 1 °C and the cartridge thermocouples were within 0.1 °C. See figure 3 for details.

*Dynamic gradient freeze or electronic Bridgman-Stockbarger.*-Directional solidification is a long established method of preparing crystals from the melt. A technique pioneered by Bridgman [4], involved the use of a vertical mounted tubular furnace through which an ampoule was lowered. This method was later modified by Stockbarger [5]. He utilized two furnaces to provide a upper and lower temperature zone. The upper zone was set at a temperature sufficient to melt the charge and the lower zone set point was chosen to optimize crystal growth. With the introduction of multizone furnaces, a typical Bridgman-Stockbarger temperature profile can be produced. However, the profile is translated by systematically reducing the power to the individual heater zones. Hence, the name electronic Bridgman-Stockbarger crystal growth.

The criteria to avoid constitutional supercooling [6] was used to establish the growth parameters necessary for the Bridgman-Stockbarger solidification of Gallium doped Germanium. An instumented ampoule simulator was utilized in conjunction with the standard test cartridge to determine the temperature gradient at the centerline of the furnace. The ampoule simulator consisted of a four-holed alumina rod inside a quartz ampoule in which four thermocouples were placed. This configuration (alumina rod, quartz ampoule) allows one to measure a more realistic axial gradient as seen by the melt. An additional 10 thermocouples were spaced axially along the outside of the quartz ampoule. The axial spacing of all thermocouples on the quartz ampoule corresponded with the axial locations of the furnace control thermocouples and the thermocouples in the alumina rod (sample simulator). This spacing was chosen to allow direct comparisons between each thermocouple and to enable the radial temperature gradient to be measured.

Figures 4 and 5 show the thermal gradients in the furnace bore and the ampoule simulator, respectively. The temperature spike seen in figure 4 was due to a faulty control thermocouple. For this test, the isothermal hot zone temperature was 1000 °C and the cold zone temperature was 850 °C. This produced a gradient in the furnace bore of 3 °C/mm and a gradient in the ampoule simulator of 1 °C/mm. This gradient was translated electronically at a speed of 4.2 µm/sec by varying the power to the control zones. The UMC was able to reproducibly position the thermal profile relative to the thermocouples located on the quartz ampoule. Thereby, demonstrating the capability of melting back a portion of a seed. A critical requirement in seeded growth situations.

*Zone melting*.-Another method to directional solidify crystals is by zone melting [7,8]. In this technique only a small region or zone of the crystal is melted. The zone is then traversed either mechanically or electrically along the sample. This technique is widely used to purify starting materials for other growth techniques [9].

The standard cartridge instrumented as previously described, was used to characterize a typical float zone melting profile produced by the UMC. The peak zone temperature was set to be 1000 °C with the lower temperature of 900 °C. Figure 6 shows the thermal gradients in the furnace bore. The peak isothermal temperature inside the ampoule was 950 °C over a 1.5 cm zone and was traversed at 4.2 µm/sec. The UMC demonstrated the ability to tailor the peak temperature and zone length to meet specific experiment objectives.

*Power requirements*.-One of the main design constraints imposed on the UMC was to limit the power consumption. The maximum steady state power required during the above experiments at 1000 °C was 450 w. Compared to other microgravity crystal growth systems [10, 11], the UMC requires approximately one-half the power for the same operating temperature. Figure 7 shows the power consumption for various set point temperatures. At an operating temperature of 1500 °C, the extrapolated power required is 850 w.

## 4. Summary

The thermal profile and translation characteristics of the Universal Multizone Crystallizator were quantitatively studied. Thermal profiles were characterized for the electronic Bridgman-Stockbarger and zone melting growth techniques. For the Bridgman-Stockbarger technique, the hot zone was set at 1000 °C and the cold zone was set at 850 °C. These set points produced a maximum thermal gradient in the insturmented ampoule simulator of 1 °C/mm at a translation rate of 4.2 μm/sec. The profile could be accurately positioned to allow for precise melt back during seeded growth. These thermal settings have been used in the growth of seeded (111) Gallium doped Germanium single crystal. A detailed characterization of the grown crystal will be published at a later date

A typical float zone thermal profile was also produced with the UMC. The peak isothermal temperature inside the ampoule was 950 °C over a 1.5 cm zone. The UMC demonstrated the ability to tailor the peak temperature and zone length to meet specific experiment objectives.

## Acknowledgments

The authors wish to declare that the UMC was invented and developed through many years by P. Barczy, G. Buza, G. Czel, J. Fancsali, P. Makk, C. Raffy, A. Roosz, B. Tolvaj who are members of the Miskolc University. So the presented instrumental parameters and results are achieved by the inventors. It is also necessary to express our appreciation to Curtis Bahr, Rens Ross, Don Lovell and Bob Cannon who are members of the Engineering Support Staff at the Marshall Space Flight Center. Without their uncompromising efforts, cooperation and enthusiastic support this collaborative study of the UMC would not of been possible.

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Table 1
Operational specifications for the Universal Multizone Crystallizator.

| | |
|---|---|
| Sample Dimension | $\phi$ (5–45) mm X 250 mm |
| Temperature range | 1500 C max. |
| Translation rates | 0.02–10 µm/sec |
| Furnace atmosphere | vacuum |
| Typical growth cycle | 1–10 days |
| Furnace control | computerized |
| Cartridge depot | 10 cartridges |

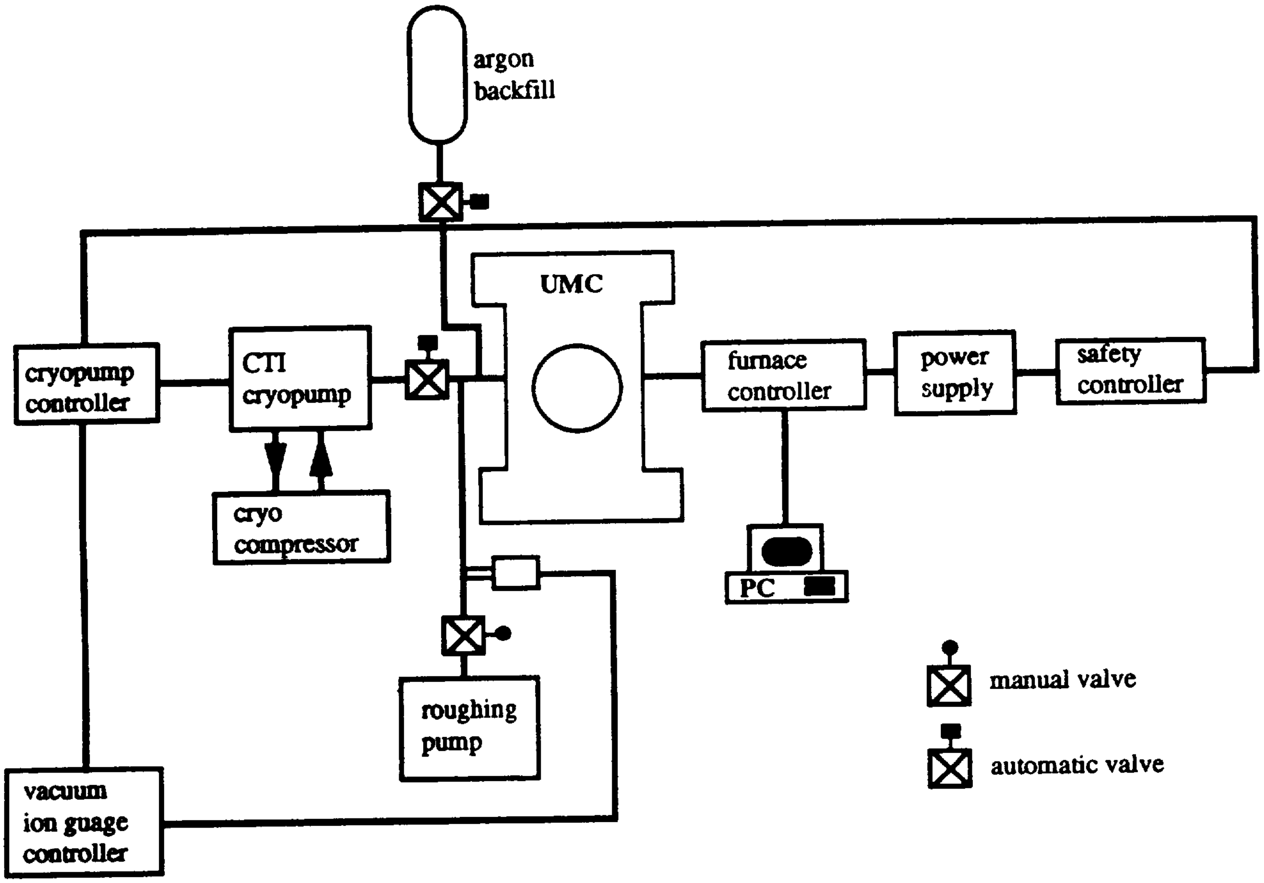

Figure 1. Schematic layout of UMC and support equipment.

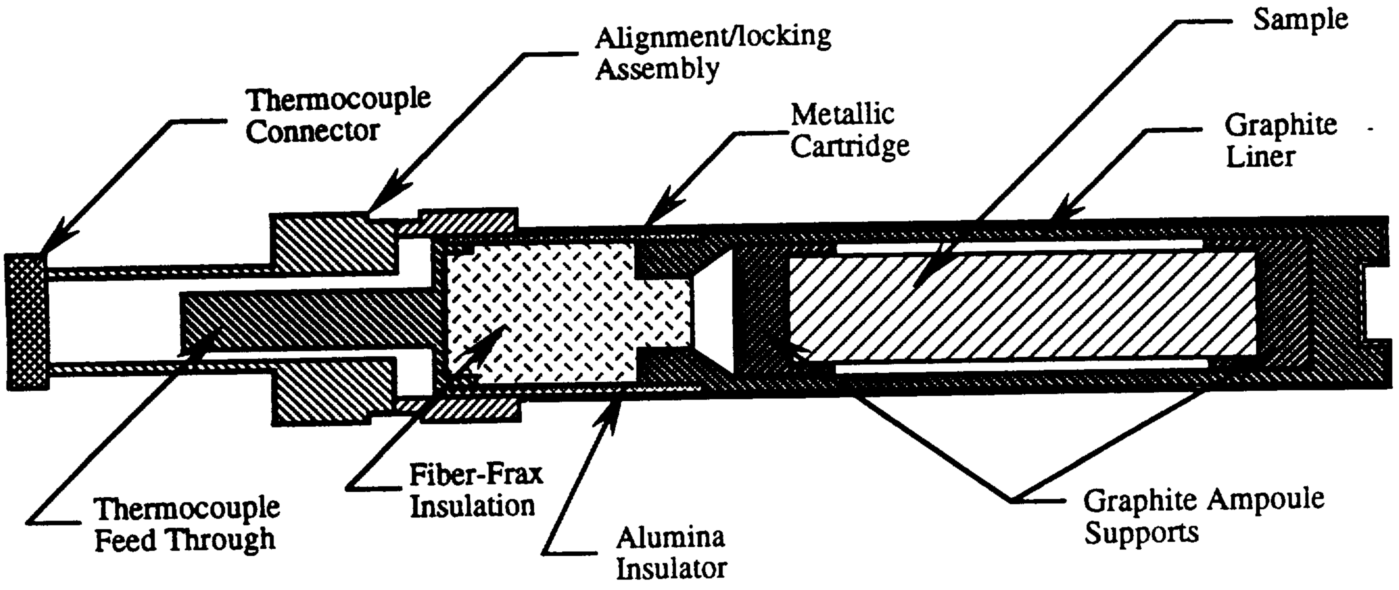

Figure 2a. Standard processing cartridge with graphite liner.

Figure 2b. X–ray of standard cartridge with instrumented ampoule simulator.

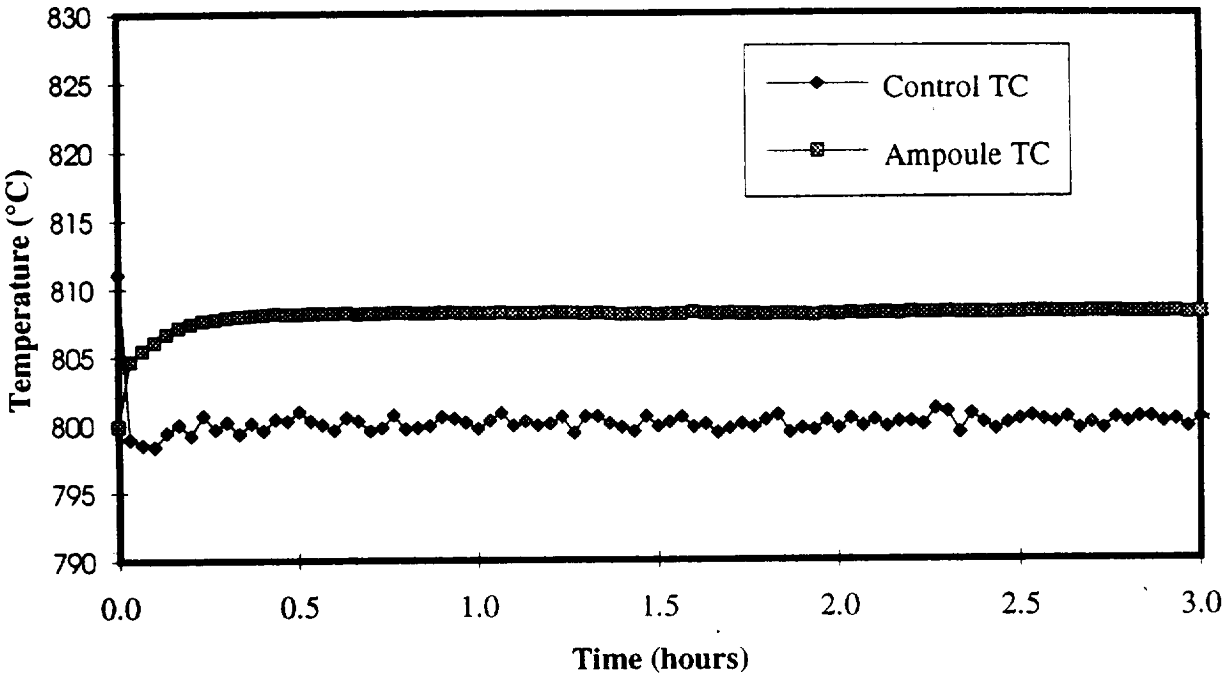

Figure 3. Temperature stability after PID adjustments.

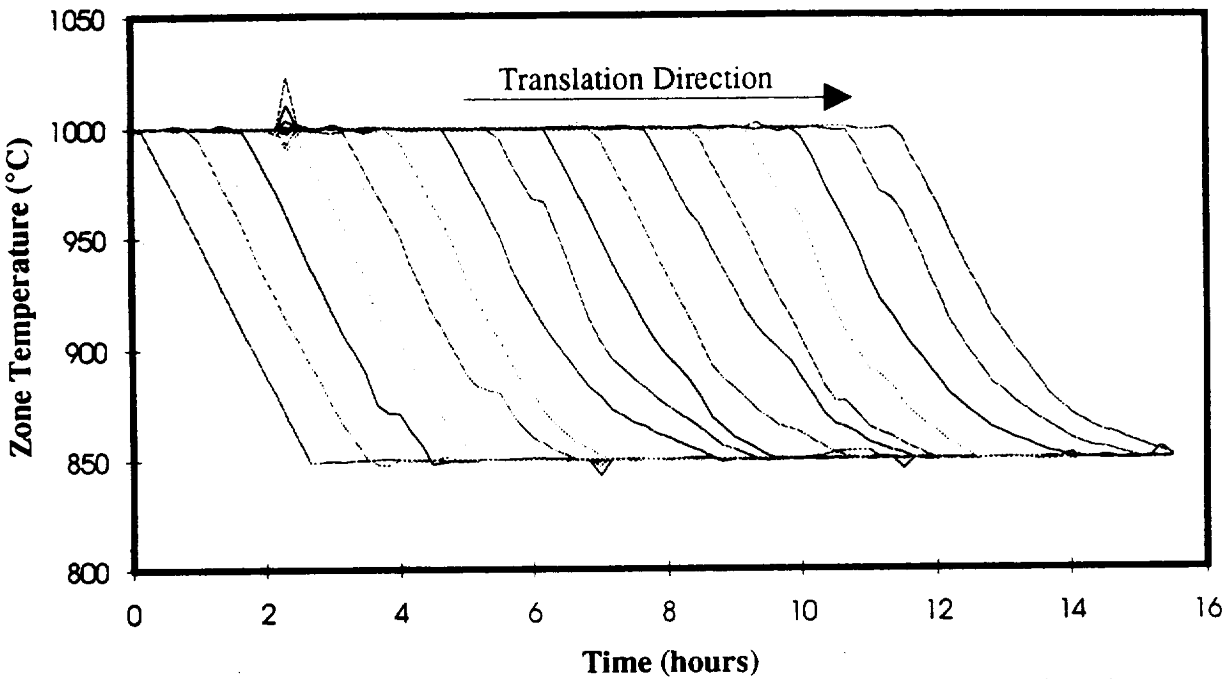

Figure 4. Thermal profile in furnace bore. Translation rate 4.2 µm/sec.

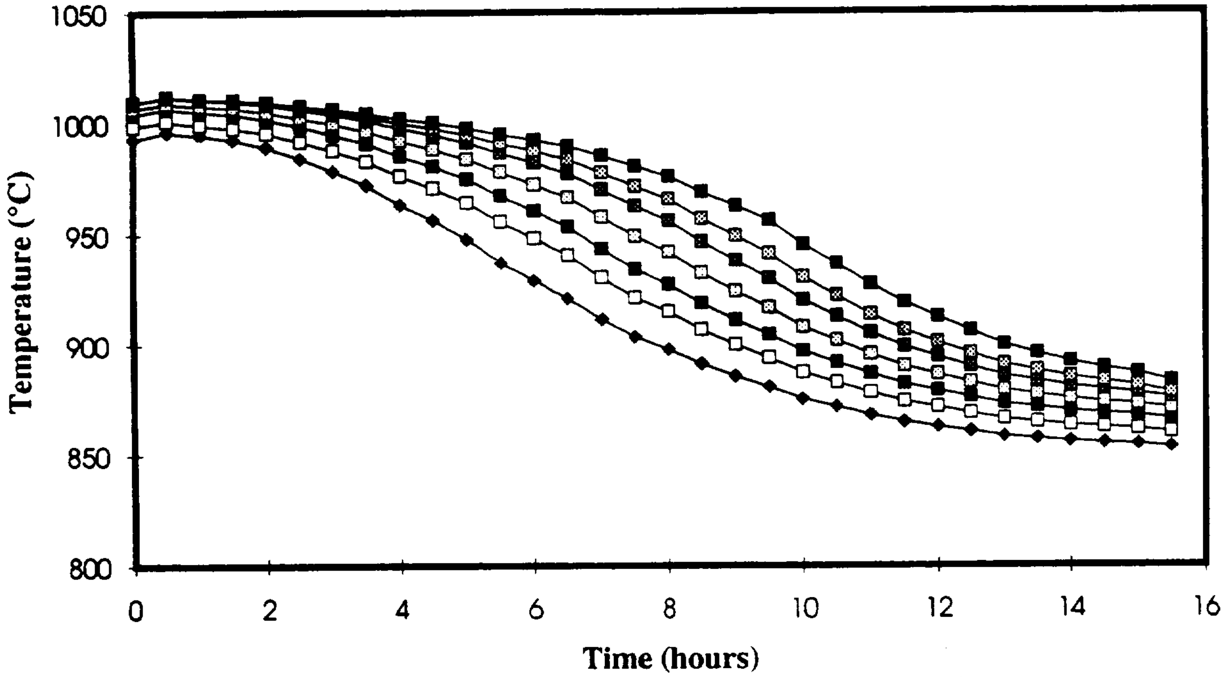

Figure 5. Thermal profile in the ampoule simulator.

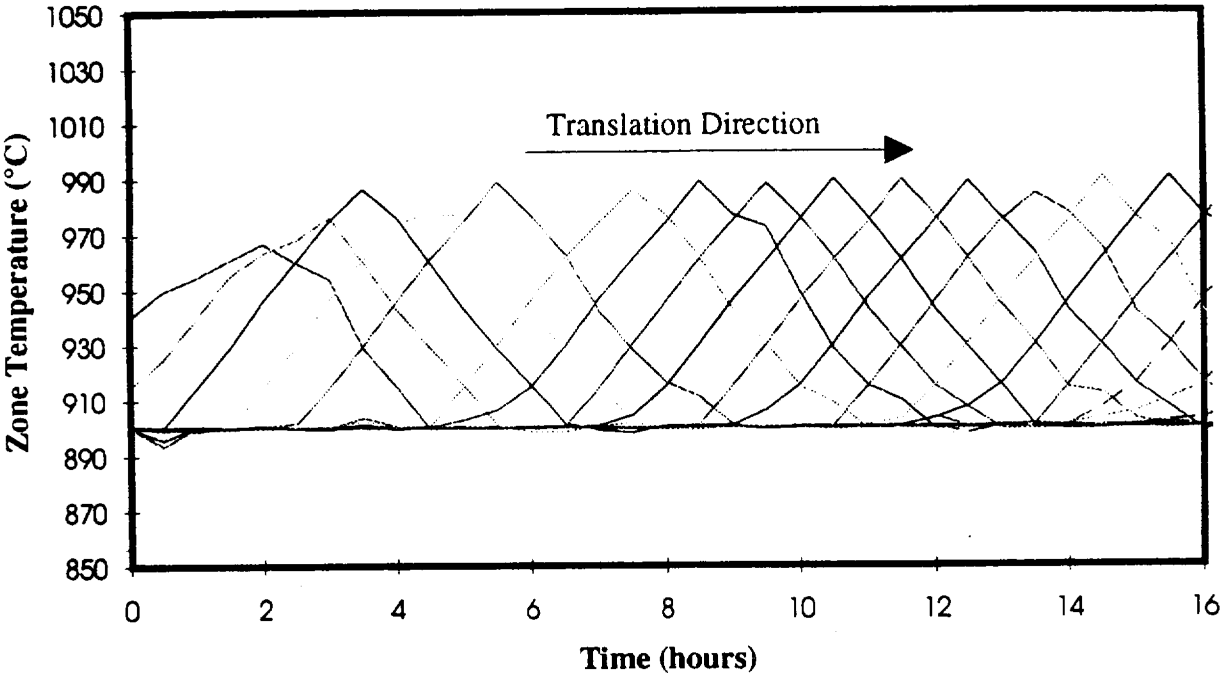

Figure 6. Thermal profile in furnace for zone melting profile.

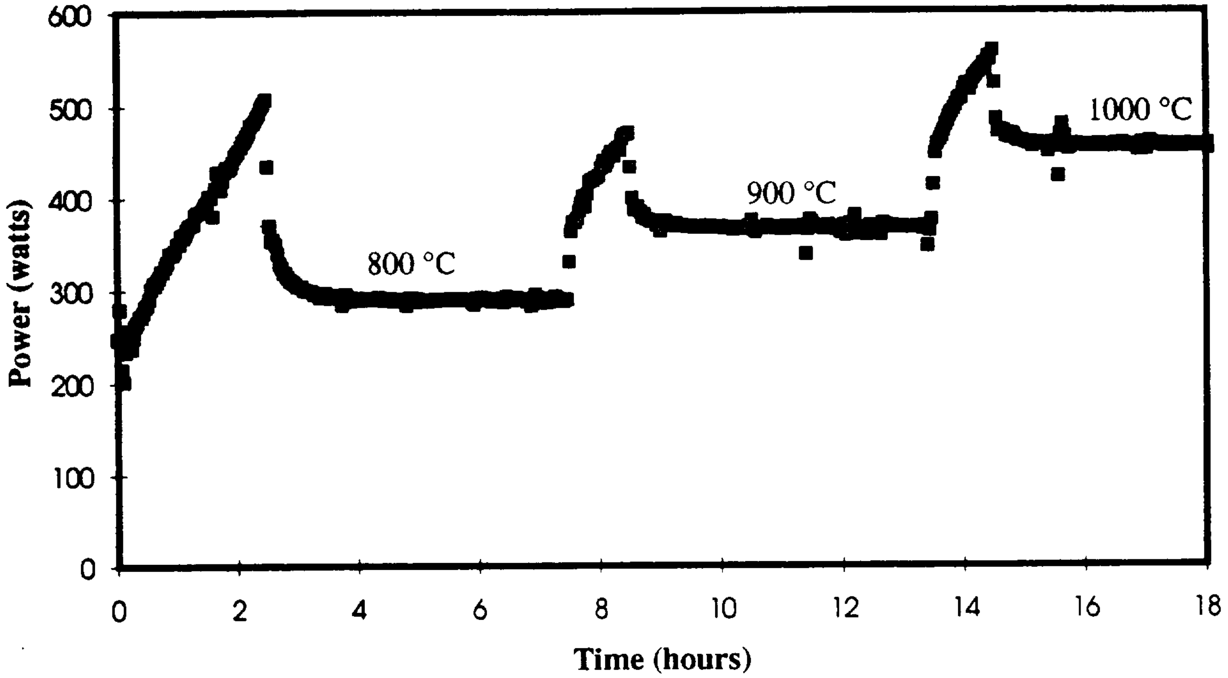

Figure 7. Power requirements for isothermal set point temperatures.