



Non-Contact Temperature Measurement Requirements for Electronic Materials Processing

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## ABSTRACT

The requirements for non-contact temperature measurement capabilities for electronic materials processing in space are assessed. Non-contact methods are probably incapable of sufficient accuracy for the actual absolute measurement of temperatures in most such applications but would be useful for imaging in some applications.

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### 1. Introduction

In a terrestrial environment the crystal growth of electronic and electro-optical materials are significantly effected by gravitational forces. In particular, free-convection caused by density gradients resulting from thermal and compositional gradients in the fluid phase can alter the compositional distribution of components, increase the probability of incorporation of impurities leached from the container walls, and cause growth rate fluctuations that lead to an increase in the densities of intrinsic crystal defects.

It is generally believed that the reduction or elimination in space of gravity-induced, convective flows and hydrostatic pressure related phenomena will result in materials with improved electrical and optical properties due to concomitant improvements in compositional homogeneity and reductions in crystal defect density. Additionally, the elimination of the need for confinement for some of the solidification processes is expected to lead to a reduction in impurity concentrations and in the densities of stress-induced defects such as dislocations.

The materials of interest include inorganic semiconductors (Si, Ge, GeAs, CdTe, HgCdTe, PbSnTe, etc.), organic and inorganic non-linear materials, various conducting and insulating oxides and

selected magnetic and piezoelectric materials. The preparation methods include solidification from molten and vapor phases, crystallization from saturated solutions, and various solid-state compaction and crystallization processes. The latter are not expected to be effected by reduced gravity; thus, only the processes that involve solidification from a fluid phase will be considered further.

In general, the various solidification methods used do not depend on a precise knowledge of the material surface temperatures, and thus, usually do not employ non-contact temperature measurement data for process control and implementation. Nonetheless, some of the methods, e.g., Czochralski and float-zone, frequently use similar optical measurement techniques to assist crystal diameter measurement and control. In the near term, of the commonly used melt growth methods (Czochralski, Bridgman, Bridgman-Stockbarger, float-zone, traveling heater-zone, etc.) only the Czochralski and float-zone growth processes are expected to benefit significantly from advances in non-contact temperature measurement technology. Thus, the remainder of the paper will primarily address issues associated with these two melt growth methods.

Section two of the paper gives a brief description of the two processes. Section three gives an example of the use of an infrared television system for thermal imaging and diameter control for a

Czochralski-type crystal growth process. Finally in section four details of the specific requirements for future non-contact temperature measurements are discussed.

# 2. <u>Crystal Growth Methods and Related Non-Contact Temperature</u> <u>Measurement Problems</u>

The Czochralski and float-zone crystal growth processes are briefly described below. In the Czochralski growth process the source material is placed in an inert crucible and melted by using RF or resistive heating methods. A seed crystal is inserted into the melt and crystal growth proceeds by controlled withdrawal of the crystal from the melt. The crystallographic orientation of the growing crystal is determined by the orientation of the seed and its diameter is determined by the temperature distribution in the growth system, which is controlled by the power input into the heaters.

In the classical float zone method a molten zone is established at the juncture of a mono-crystalline seed and the source material. The zone width is controlled by the amount of heat input into the zone region. Similarly to the Czochralski method either RF or resistive heating techniques are used to establish and maintain the desired temperature distribution in the zone region. Additional process control can be derived from provisions for the rotation and relative axial displacement of the seed and source materials. Usually, for both growth processes, the particular vapor pressures of the materials at the growth temperatures are large and thus evaporation from the melts can result in deposition of the materials on the container walls

and the "viewing-port" windows. For some of the smaller vapor pressure electronic materials, e.g. Si, Ge, etc., the evaporation rate can be significantly reduced by the introduction of an inert gas into the process chamber. The processing of the higher vapor pressure materials, e.g., compound semiconductors, usually requires both large inert gas pressures (up to 100 atm.) and the use of liquid encapsulants, e.g., B<sub>2</sub>O<sub>3</sub>. Changes in either the window or encapsulant spectral transmissivities with time are expected to significantly impact the accuracy of temperature values derived from the measured intensity of the thermal radiation from the surface region of interest. Additional errors may result from the lack of knowledge of the pertinent emissivities as well as of their temperature, wavelength and composition dependences. A related problem is the potential for time-dependent changes in the surface compositions caused by either preferential surface segregation of minority components, e.g., impurities and dopants, or by preferential evaporation and thus surface depletion of some of the major components caused by differences in component chemical potentials at the phase boundaries. Finally, as the crystal grows, the relative volumes of the crystal and melt will change. Related changes in surface geometries will result in changes in the various geometrical view-factors leading to further temperature determination errors.

The extreme sensitivity of device yield and performance to variations in material properties necessitates a high degree of reproducibility in the material processing variables, in particular, thermal conditions. Because of the potential sources of error described above, we believe that in most cases, contact temperature measurements cannot provide accurate enough data to assure routine reproduction of the desired temperature values. Nonetheless, data provided by such measurements can be very useful for determining relative variations in surface temperatures and thus thermal gradients. Such data can be useful in establishing thermal boundary conditions for process modeling and optimization.

## 3. System for Thermal Imaging and Crystal Diameter Control

Non-contact measurement systems have been in use for diameter control in Czochralski growth systems for more than 20 years.[1] By 1972 non-contact imaging systems had already reached the fundamental physical limits discussed in Section 2. Improvements since that time have, therefore, been limited to faster processing and better graphical display of the information. The improved graphics capabilities are due in part to faster computers and in part to improvements in detector technology, especially detector arrays. For example, the earlier use of ordinary photovoltaic diode arrays has more recently given way to the significant use of charge coupled arrays. CCDs have higher sensitivity and inherent integration capability for noise filtering. The 1972 system of Kwap et al. [2] is described below. A current system for non-contact temperature measurements in a Czochralski system was described by Wargo at this Workshop.

The system of Kwap, et al. used an infrared TV camera to determine relative temperatures of the melt surface and growing crystal. The diode array in the camera had a range of 0.55 to  $1.15\mu$ m with peak sensitivity near 0.83  $\mu$ m. By computer analysis of the TV data, crystal diameter measurements were made and the power to the crystal puller was controlled by computer to regulate the diameter.

This system was used to grow oxides near  $1400^{\circ}$ C. The system could measure diameter to 0.1 mm and data were taken from the camera at the rate of 30 scans per second. By changing filters at  $300^{\circ}$ C intervals, the system could measure temperatures from  $300^{\circ}$ C to  $1500^{\circ}$ C. The output voltage sensitivity ranged from 4 to  $12 \text{ mV/}^{\circ}$ C so that temperature resolution for most applications would not be a problem. The limitations on temperature measurement accuracy were primarily due to the laws of nature and the lack of knowledge of the emissivities, as discussed in Section 2. These limitations remain unchanged.

## 4. Thermal and Optical Imaging Requirements

In summary, requirements exists for thermal and optical imaging of free or liquid encapsulated melt/crystal surfaces for the Czochralski-type and float-zone type crystal growth. The temperature range of interest is from 200 to 1600<sup>°</sup>C. Requirements also exist for thermal mapping of growth interfaces and real-time monitoring of solidification rates during vapor crystal growth. The shape/temperature/growth-rate data can be used for feedback control of thermal fields, crystal growth rates and crystal/ingot diameters.

The crystal growth of electronic materials, in most cases, requires the precise knowledge of the absolute values of the temperatures at selected locations on the sample/growth-system surfaces. Usually the desired absolute temperature measurement accuracies range from:  $1)\pm 1.5^{\circ}$ C for the 200 to 700°C range,  $2)\pm 2.5^{\circ}$ C for the 700°C to 1100°C range,  $3)\pm 3.5^{\circ}$ C for the 1100 to  $1350^{\circ}$ C and  $\pm 5^{\circ}$ C for the highest temperatures. Sampling rates of up to 5 samples per second are required. We believe that these accuracy requirements very likely cannot be met in most cases using non-contact temperature measurement techniques because of (1) the errors resulting from the lack of precise knowledge of some of the required thermophysical properties, e.g., emissivities, and (2) the various

inherent process-imposed limitations described in some detail in Section 2.

The requirements for temperature resolution measurements range from about  $0.2^{\circ}$ C to about  $4^{\circ}$ C. For imaging purposes the required sampling rates are of the order of 30 samples/second. The spatial resolution requirements range from about 0.1 to 1 mm. In contrast to the absolute temperature accuracies, for most cases, especially for the higher temperature ranges (>700°C), these imaging requirements probably can be met by using the combination of judiciously designed imaging optics and commercially available photovoltaic or CCD array cameras. The most stringent requirements will probably require the use of special cryogenically cooled detector arrays or scanning detector systems.

### 5. <u>References</u>

- 1. E.J. Patzner, R.G. Dessauer, and M.R. Poponiak, Solid State Technology, October 1967, p. 25.
- 2. T.W. Kwap, D.F. O'Kane, and L. Gulitz, in <u>Temperature, It's</u> <u>Measurement and Control in Science and Industry</u>, Vol. 4, Part 1, Instrument Soc. of America, Pittsburgh (1972) p. 541, and D.F. O'Kane, T.W. Kwap, L. Gulitz, and A.L. Bednowitz, J. Crystal Growth <u>13/14</u> (1972) 624.