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PROCESSING EUTECTICS IN SPACE

BY

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SUMMARY

Experimental work has been directed toward obtaining interface shape control while a numerical thermal analysis program was being made operational. An experimental system was developed in which the solid-liquid interface in a directionally solidified aluminum-nickel eutectic could be made either concave to the melt or convex to the melt. This experimental system provides control over the solid-liquid interface shape and can be used to study the effect of such control on the microstructure.

The SINDA thermal analysis program, obtained from Marshall Space Flight Center, has been used to evaluate experimental directional solidification systems for the aluminum-nickel and the aluminum-copper eutectics. This program was applied to a three-dimensional ingot, and has been used to calculate the thermal profiles in axi-symmetric heat flow[6]. The results have shown that solid-liquid interface shape control can be attained with physically realizable thermal configurations and have indicated the magnitudes of the required thermal inputs.

This work has been supported by NASA Contract NAS8-29669-S/A2.

INTRODUCTION

Processing of materials in a weightless environment as provided by an orbiting facility such as Skylab and the planned Spacelab/shuttle is of interest because such processing will be independent of the effects of gravity. Density differences cause, on earth, such effects as gravity-driven convection, segregation, and inhomogeneities due to buoyancy in liquid systems. It has been shown by experiments run in Skylab (1) that removing the effects of density during the processing of certain materials taken to the molten state and allowed to resolidify can result in improved material in some respects. In some cases, the effects of other driving forces are magnified, such as surface tension, whose effects are often suppressed by the density effects. For example, it was observed by Larson (see ref. 1) that the weightless Skylab processing environment allowed the evolution of metallic gas within molten metal samples. Earth processing did not produce this because the hydrostatic pressure head within the molten metal itself suppressed the evolution of the gas.

While the processing of alloys has some attractive features, the Skylab programs demonstrated the importance of a rigorous consideration of each process parameter during space processing. Such an approach requires a sustained effort of investigation of earth processing parameters in order to fully anticipate the requirements for effective space processing. This approach has been taken in the present program under NASA sponsorship in which eutectic alloys have been used to provide systems which respond in easily measurable ways to processing conditions. When solidified, they exhibit a micro-duplex structure which is related in characteristic size to the rate of solidification, and in direction to the direction of the local thermal gradient. Other structural features, such as grains, cellular structure, and banding reveal heat flow directions, segregation effects, and thermal fluctuations during the solidification process.

The regular, repetitive structures obtained in directionally solidified normal eutectics have been the subject of many investigations, as noted in the bibliographic references of Ref. 2. Theoretical as well as extensive experimental investigations have been conducted. The 1972 Conference on In-Situ Composites - I, (3) provides a compendium of recent activity in the study of eutectic structures[6] and their applications. The extent of on-going activity is reflected in the recent call for papers for the second Conference on In-Situ Composites - II, September 1975.

As directional solidification techniques have improved, complex shapes such as turbine blades have been produced as well as cylindrical specimens using multicomponent alloys. In the case of eutectic alloys, structural defects such as bands arise from growth fluctuations which, when sufficiently severe, result in structure terminations with subsequent renucleation. Other structural faults occur when the direction of growth is not substantially parallel to the direction in which the growth is desired, so that, for example, the growth direction proceeds from the interior of the piece to the surface, terminating there, or alternatively, begins at the surface and terminated in the interior of the piece. Since the direction of growth is parallel to the thermal gradient at the solid-liquid interface, these defects are associated with the gross curvature of the interface.

It has been found, in general, that there are always faults in the microstructure of even carefully grown eutectic specimens, although regions of highly perfect structure have been reported in the literature (see Ref. 2). Jackson and Hunt (4) have provided a theoretical framework for understanding the origin and nature of eutectic microstructure and its faulting modes. In addition, they have provided an elegant study of eutectic structure using organic eutectics in thin cells coupled with phase-contrast microscopy to illustrate the conditions of growth and faulting in eutectic microstructures (5). Figures 1-5 are reproductions of photographs obtained by Hunt and Jackson illustrating the way in which defects occur during growth of eutectic systems. Figure 6 illustrates non-coupled growth of eutectics. Clearly, the faceted-faceted systems will never yield the uniform microstructure which may result in useful non-structural applications of eutectics.

A major problem standing in the way of experimental solidification studies has been the lack of thermal analysis programs applied to the calculation of heat-flow paths and isotherm contours in the solidification of three-dimensional shapes. While such calculations have been of continuing interest to engineers and mathematicians, useful program calculations applicable to the solidification of alloys have not been available in the general literature.

It has been the objective of the present study to develop an experimental thermal processing system and an analytical thermal analysis program in which the parameters affecting eutectic microstructural perfection could be independently controlled. Studies of eutectic microstructure could then be made with a degree of independent control over the processing variable of solidification rate, liquid-solid interface gross curvature, and thermal gradient at the liquid-solid interface. From these studies, control requirements for processing to minimize faulting in the microstructure could be developed.

BACKGROUND

NASA programs have been directed toward exploring the nearly weightless environment provided by orbiting spacecraft for conducting experiments which will lead to product manufacture in space for use on earth. A variety of studies are required to determine which materials would derive most benefit from production in a weightless environment.

Previous studies such as "Spherical Forming and Composite Casting in Zero-G" by A. E. Wechsler, J. Berkowitz-Mattuck, P. C. Johnson, and L. B. Griffith, A. D. Little, Inc., Cambridge, Massachusetts (6) have suggested that monotectic and eutectic composites should be studied as candidate materials for processing in a weightless environment. A monotectic system was suggested for formation of superconducting materials, such as Nb_3Sn-Sn, or the Cu-Pb system, while the study of Al-$CuAl_2$ was suggested to determine if the absence of thermal convection would result in more perfect microstructures. Improvements in the perfection of the microstructure are important for non-structural applications of eutectic compositions.

The results of the space processing experiment M-566 (7), in which the Al-$CuAl_2$ eutectic was directionally solidified under weightless conditions, indicated that the condition of weightlessness alone does not provide improvements in the eutectic microstructure without control over other process parameters. The Skylab IV specimens indicated improvement in structure compared to earth grown specimens, while Skylab III specimens were not as good. Trends in structure perfections correlate with the degree of convection associated with each experiment. Thus, any advantages in processing which might accrue from weightless processing will only be found in a carefully designed experiment in which all variables are controlled.

NASA Contract NAS8-29669 was carried out at UARL during the period from July, 1974 to December, 1974. The systems Al-Al_3Ni and Al-$CuAl_2$ were used as model eutectics with modest melting points to determine the effects of solidification rate and interface shape control on the defect structure in these systems. Thin sheets (less than 25 micrometers thick) of lead-tin were processed and found to contain small areas of quite perfectly aligned lamellar microstructure. Examinations were made of the degree to which eutectic microstructures could be expected to tolerate rapid changes in growth direction without breakdown. Finally, off-eutectic solidification using high thermal gradients was examined in the lead-tin system, where it was found that eutectic microstructure was retained at compositional deviations up to about eight percent by weight from the eutectic composition, on both the lead-rich side and on the tin-rich side (8). The object of these studies was to determine those experiments for weightless processing which would best elucidate the improvements to be gained by space processing of eutectics.

Hunt and Jackson (5) using optically transparent organic eutectic systems as analogs of the general classes of eutectics were able to observe the growth of separated solid phases from a homogeneous liquid directly, using thin films of material. Cline (9) observed the growth of lamellae in the Pb-Sn system using a hot-stage microscope and thin films. In these cases, a high degree of perfection often occurred because the thickness of the film was dimensionally less than the average length associated with the formation of instabilities in bulk samples. In addition, Guinnier, et. al. (10) reported the growth of highly perfect regions of Pb-Sn eutectic using thin films of material. This work suggested the thin film study carried out in Ref. 8. These results indicate that structural perfection can be obtained when one or more dimensions of the sample parallel to the growth direction is small, such as in films or in small diameter rods. The dimension to be considered "small" appears to be a function of the characteristic length associated with the formation of instabilities in the solidifying surface. To extend the degree of perfection observed in small samples to larger sample dimensions thus clearly requires a high degree of control over the growth process in terms of maintaining stable conditions. In addition, control must be available to set the growth velocity, thermal gradient, and solid-liquid surface curvature independently (within limits) in the regions of interest.

ACCOMPLISHMENTS

Experimental Work

During this five-month investigatory period, the Al-Al_3Ni eutectic system was used as model material having a high conductivity and a rod-matrix structure for experiments designed to determine the conditions necessary to independently vary the solidification parameters of rate, gradient, and interface curvature. The furnace configuration used consisted of two resistance heated units to provide two independent sources of power, an insulated region, and a quench region. The system is shown in Fig. 7. The Al-Ni material was placed in 13 mm O.D. by 10 mm I.D. aluminum oxide tubes closed on one end, 45 cm long.

Since the material, when melted and being processed for unidirectional solidification is always hotter on top than below, there is little, if any, convective stirring. The thermal conductivity of the liquid is significantly smaller than that of the solid, so that increasing the heat flow from the liquid to the solid (and thence to the quench) would require liquid temperatures high enough to cause significant reaction with the crucible if the heat were added uniformly to the molten material. The appropriate region to add heat, therefore, is in a restricted region near the solid-liquid interface. This was accomplished by the small auxiliary heater in Fig. 7. The ideal power distribution into the system should produce the desired temperature distribution as shown in Fig. 8.

Several different auxiliary furnace designs and structures were tried in order to produce the power distribution indicated in Fig. 8. The successful design used stainless steel in the configuration detailed in Fig. 9. The main problem was to obtain an adequate power density. Wire-wound furnaces are limited in the watt-density capacity of the wire so that small zones result in low power levels. Even the design successfully used is limited by the strength and oxidation resistance of the stainless steel at the 1000°C temperature required to radiate sufficient power to the specimen to obtain some control over the shape of the solid-liquid interface. It is planned to replace the auxiliary furnace with one of essentially the same design using INCONEL 713C, a higher temperature capability alloy. In addition, an induction heated radiator is under consideration, but a control system for maintaining a very stable power level would be required.

The successful result of using the furnace system of Fig. 7 is illustrated by the ingot sections shown in Fig. 10. While the degree of curvature reversal of the interface is small, it shows that the goal of interface shape change can be reached with the present design using high thermal conductivity alloys.

Analytical Work

The SINDA (Systems Improved Numerical Differencing Analyzer) computer program(11) was obtained from Marshall Space Flight Center and made operational on the United Aircraft Research Laboratories UNIVAC 1110 computer system. The program was used to solve a series of problems of increasing complexity, exercising the various thermo-physical options available in the program for thermal analysis work. These are summarized in Appendix I. An interesting result of this approach occurred when the freezing of a 0.5 meter long slab of water initially at 20°C was investigated. The base was brought into contact with a temperature reservoir at -20°C, and the propagation of the interface was determined. In this problem, the phase-change option was exercised. The results were compared to the solution published in Ref. 12. Initially, a disagreement was found with the published work which was traced to the apparent inclusion of only one-half the latent heat in the published work. When the SINDA program was rerun for this problem with one half latent heat, excellent agreement was obtained.

The analytic capability of the SINDA program has been applied to the furnace system shown in Fig. 7. The model, illustrated in Fig. 11, takes into account the radiation heating, the aluminum oxide crucible with its heat capacity and temperature dependent thermal conductivity, and the thermo-physical properties of the $Al-Al_3Ni$ eutectic alloy, including its phase change. The axial symmetry of the ingot-furnace system is used to simplify the problem to a two-dimensional analysis. The SINDA grid system used is shown in Fig. 12.

The results obtained have shown that the running time of the program is strongly dependent on the initial thermal profile chosen. This is required because the program must operate from a "history" so that each pass through the problem constitutes one step in a converging solution. In the present problem, beginning with a uniform ingot temperature and initiating the quench at time $t = 0$ results in a long computing time. Therefore, the initial condition to be chosen is one with a uniform temperature gradient in the ingot.

CONCLUSIONS AND RECOMMENDATIONS

As a result of work performed under Contracts NAS8-29669 and NAS8-29669-S/A2, a resistance-heated furnace processing system for independent control of solidification rate, interface curvature, and thermal gradient has been designed and implemented for directional solidification of high thermal conductivity eutectic alloys with melting points up to 700°C. The use of an isothermalizing liner and temperature controlled auxiliary heating of a narrow zone will contribute significantly to the thermal stability of the system, shown to be a prerequisite for the degree of perfection which can be attained in eutectic microstructures. In addition, the SINDA computer program has shown the capability of being applied to thermal design and analysis of working experimental processing systems for directional solidification, including the phase change on solidification of the eutectic alloys.

Review of the literature on the solidification of the eutectic alloys has shown that perturbations in solidification conditions at the solid-liquid interface cause defects to occur in the eutectic microstructure. Therefore, the emphasis, in studying the improvements which can be made in microstructural perfection, must be on the control of the conditions at the interface. The literature also establishes that the use of small-dimensioned containers, such as in the growth of thin films, provides an enhanced interface stability which makes it much easier to obtain perfect microstructures.

It is recommended that further studies, aimed at investigating the conditions which will produce improved microstructures and the role that space processing could play in obtaining those conditions, should be a combination of thermal analysis of the system and experimental work with the directional solidification apparatus. The investigation of directional solidification of eutectics in thin film form could provide the more direct achievement of perfect microstructures by making use of the observed stability enhancement.

APPENDIX I*

The SINDA program was developed by NASA under a series of contracts to provide a general thermal analysis program. It was decided to adopt SINDA for calculations relating to the directional solidification of eutectic alloys for two reasons, in addition to its availability and developmental status. One was its 3-dimensional computational capability; the second, more important reason was the availability of a sub-routine which accounts for the latent heat involved in a phase change.

The sub-routine LQSLTR of the SINDA program accounts for the phase-change energy by using the constructed mesh-node network with the inclusion of the phase-change energy as a capacitance, or storage, effect. The network solution sub-routines are allowed to calculate incorrect answers based on capacitance effects representing heat capacity only; when the solution reaches the temperature region of the phase change, LQSLTR performs a corrector operation that accounts for the phase change to produce the correct temperature.

Other attractive features of SINDA were the versatility with which variable thermophysical properties can be handled, and the ease in boundary-value input.

The program was exercised by solving a set of simple problems for which analytical solutions were available. These were:

1. a one-dimensional slab with all boundary condition options;
2. a two-dimensional slab with the following boundary conditions:
    a. specific energy flux
    b. specified temperature
    c. specified transient energy flux
    d. radiation on a surface.

In all cases the solution agreed with the analytical results within the limits of the models used.

Although phase-change problems have been of interest to mathematicians for some time, little progress has been made at solving them because of the non-linear nature of the equations. Analytical solutions exist for two classes or problems:

1. the infinite one-dimensional slab;
2. wedges, a general class of infinite two-dimensional slabs.

The numerical techniques for solving the more general phase change problems check their solution procedures by comparing their answers for solidification or liquefaction in a finite slab or wedge with those of infinite dimension for a short time period.

*This work was carried out by Jim Fitzpatrick of the Mathematical Analysis Group of UARL.

Three examples of Class 1 were exercised. Figure A-1 illustrates the configuration and the boundary conditions. The one-dimensional slab of water for the first example was subject to the boundary conditions of contact with a temperature reservoir at one end, a length of 0.5 meters, and insulated along all remaining boundaries. The initial condition specified that a constant energy flux was flowing out of the slab, initially at the freezing point with no ice present. The solution yielded a solidification front moving down the slab at a constant rate, as expected. Conservation of energy was satisfied.

In example 2, the initial condition of constant energy flux was changed to contact with a constant temperature reservoir. In this case, the phase front propagates proportional to the square root of the time, as shown in Fig. A-2. The results obtained from SINDA using 41 node points were compared with the results of Ref. 12 and found to agree if only half the latent heat value of the water-ice phase change was used. Careful examination of Ref. 12 showed that while the latent heat was distributed over the temperature range of $\pm 0.5^{\circ}$ around the freezing point, the calculational process only operated at temperatures up to the freezing point, thus not taking into account the portion of the latent heat distributed from the freezing point T_f to $T_f + 0.5^{\circ}$, or half the latent heat.

The third example considered used the conditions of example 2 except the initial water temperature was placed above the freezing point. To compare this case with the analytical solution for a semi-infinite body as performed in Ref. 12, the calculations were stopped at time when the temperature at the end of the slab started to change appreciably. The results agreed well with those of Ref. 12 (corrected).

The second class of problems used to confirm SINDA was the solidification of water in an internal corner of a square with the surface of the (square) wedge maintained at equal temperatures lower than the freezing temperature of the liquid. The physical problem is illustrated in Fig. A-3, with part of the SINDA model shown in Fig. A-4. Two cases were run for this class:

1. the liquid temperature was at the fusion temperature;
2. the liquid was initially at a temperature higher than the fusion temperature.

Computed locations of the interface were compared to the value published in Ref. 13. The shapes in all cases were in good agreement, although the values were not in good agreement. This difference was attributed to differences in thermophysical properties, and the results were judged acceptable.

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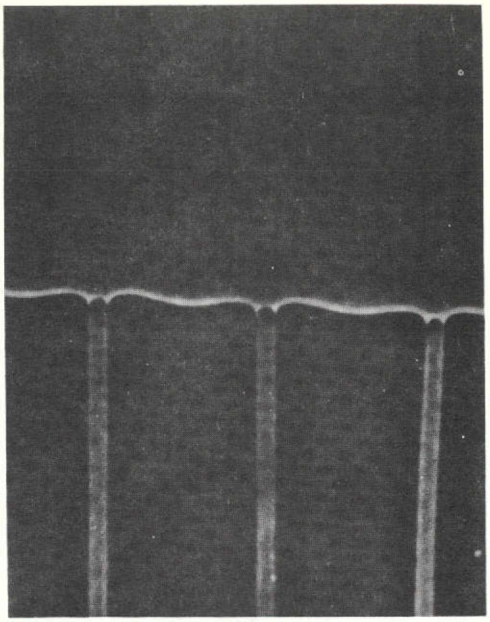
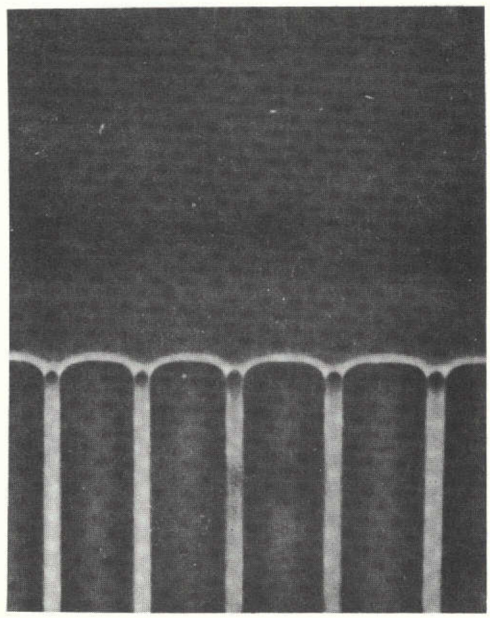
FIGURE 1. "PHASE DIAGRAM" OF THE CARBON TETRABROMIDE–HEXACHLOROETHANE SYSTEM SHOWING COUPLED MICROSTRUCTURAL GROWTH AROUND THE EUTECTIC COMPOSITION (REF. 5)



A) ALLOY SLIGHTLY RICH IN CARBON TETRABROMIDE



B) ALLOY OF ALMOST EUTECTIC COMPOSITION

FIGURE 2. THE SHAPE OF THE SOLID–LIQUID INTERFACE IN D.S. EUTECTIC ALLOYS OF CARBON TETRABROMIDE–HEXACHLOROETHANE (REF. 5)

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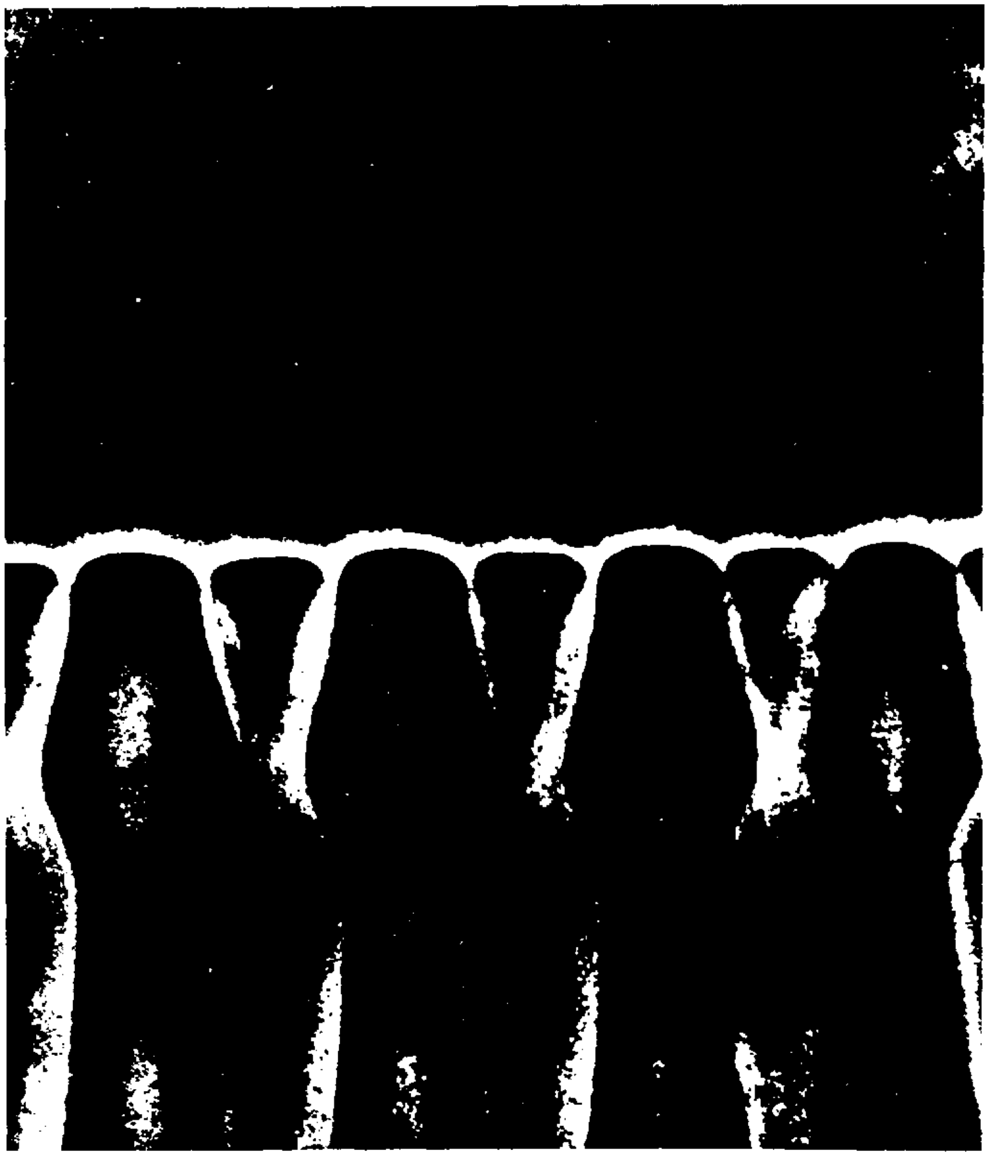


FIGURE 3. DEFECT LINE DUE TO FLUCTUATION IN GROWTH RATE – CARBON TETRABROMIDE–HEXACHLOROETHANE (REF. 5)

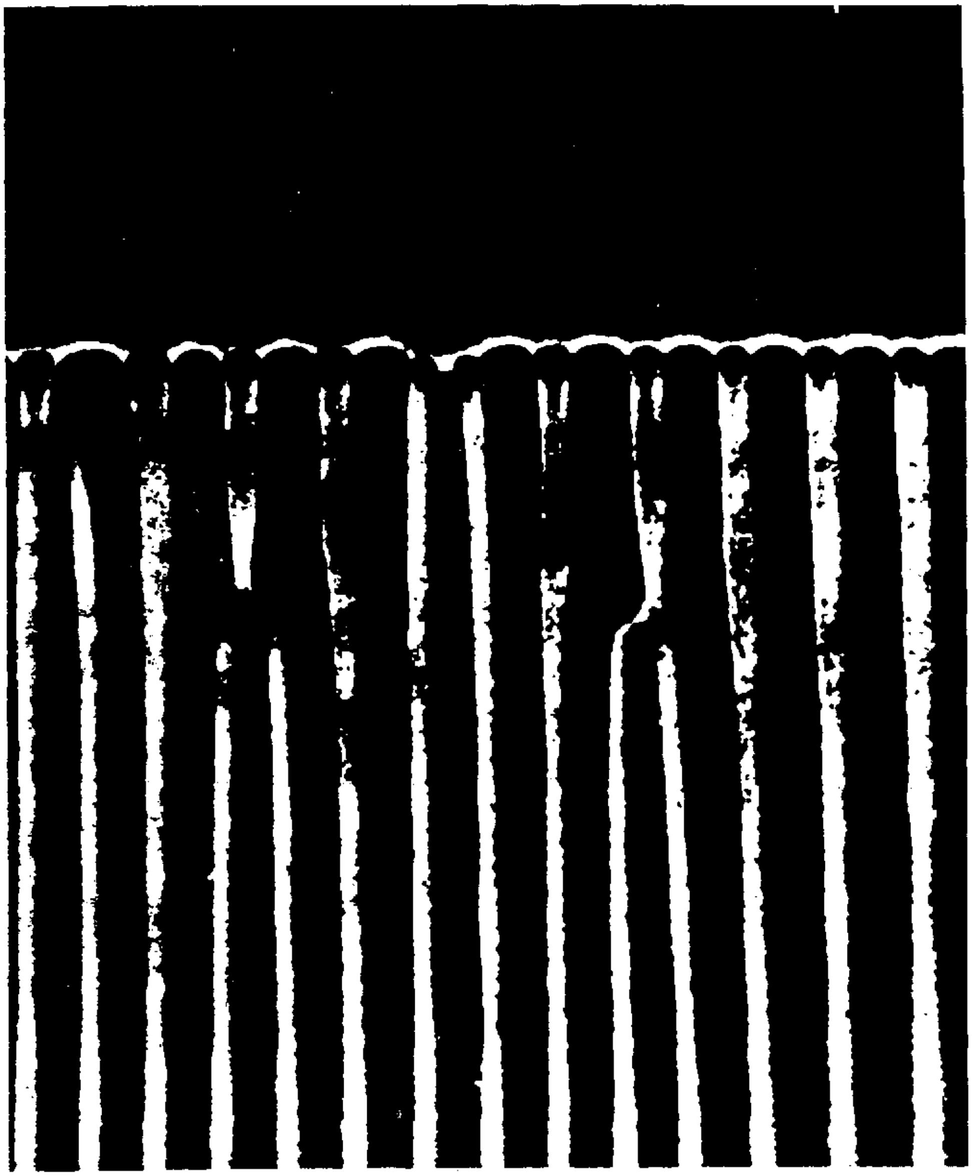
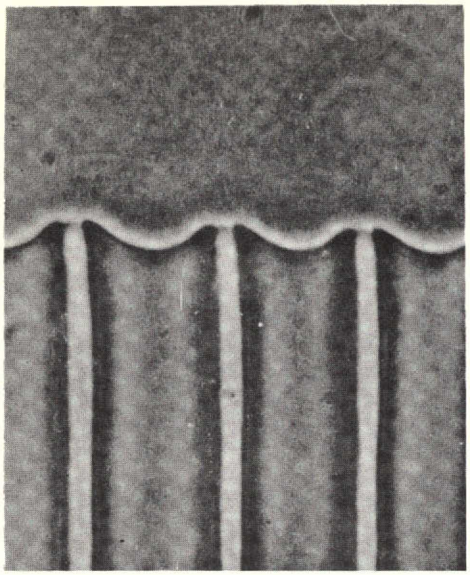
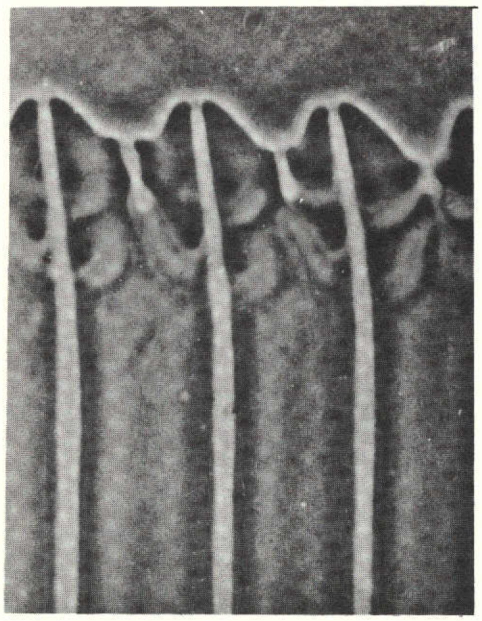


FIGURE 4 . THE GROWING OUT OF LAMELLAE UPON INCREASE IN GROWTH RATE (REF. 5)



A) A DEPRESSION FORMING IN THE CENTER OF THE WIDE LAMELLAE DUE TO INCREASED GROWTH RATE



B) INSTABILITY RESULTING IN NUCLEATION OF NEW LAMELLAE

FIGURE 5. THE FORMATION OF NEW LAMELLAE (REF. 5)

FIGURE 6. NON–COUPLED EUTECTIC GROWTH – AZOBENZENE–BENZIL EUTECTIC (REF. 5)

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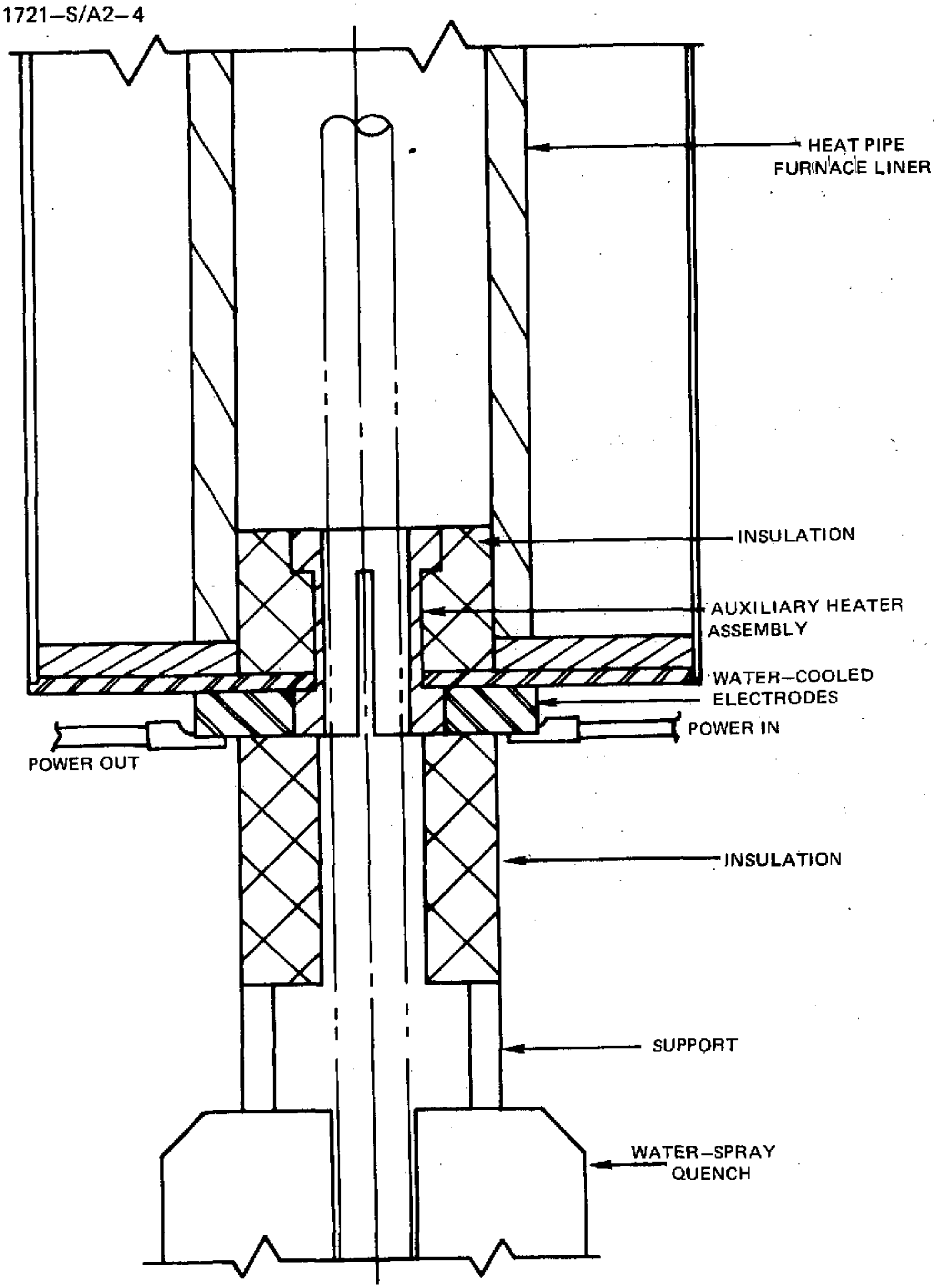

FIGURE 7. RESISTANCE-HEATED BRIDGMAN FURNACE FOR DIRECTIONAL SOLIDIFICATION

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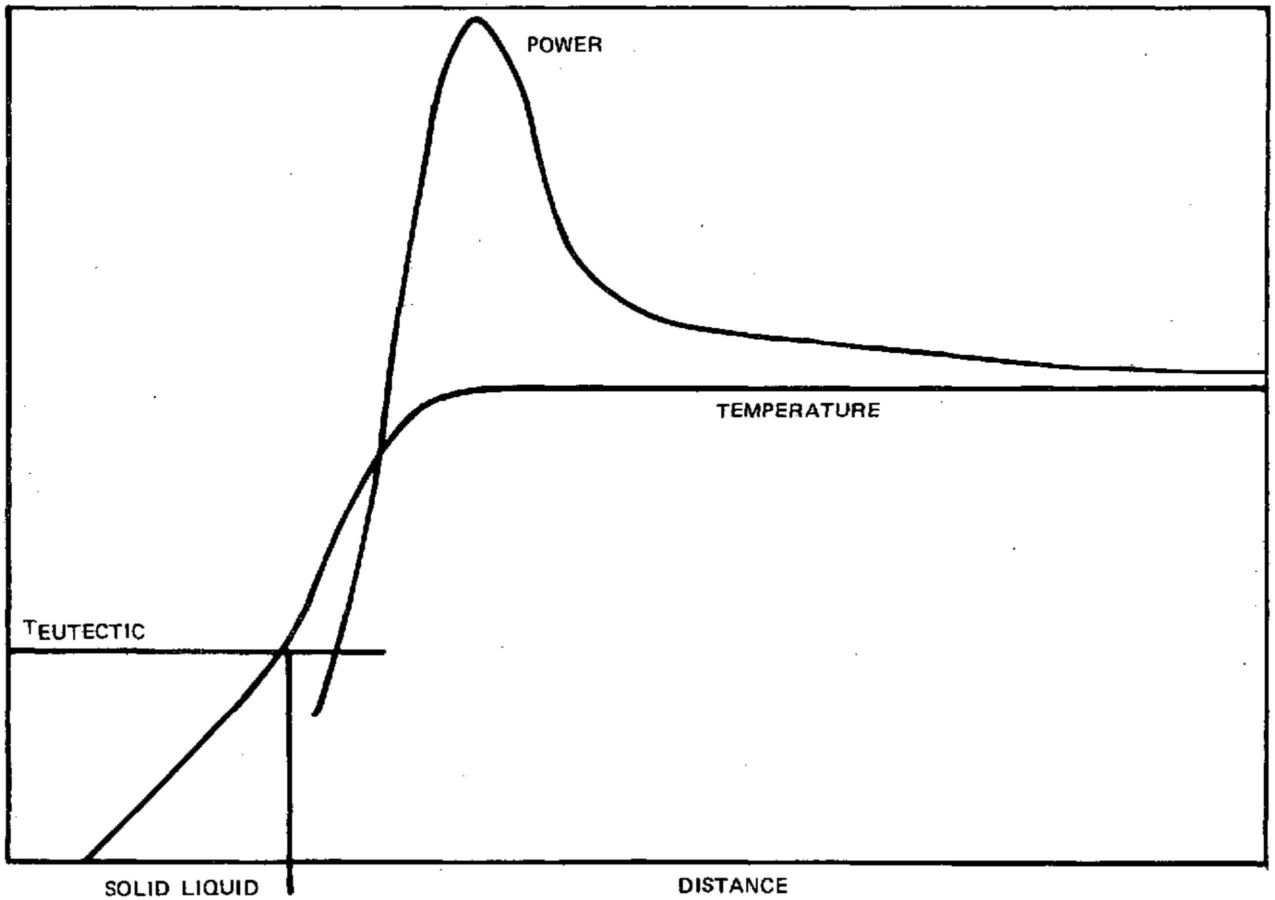


FIGURE 8. IDEAL POWER AND TEMPERATURE DISTRIBUTIONS ALONG A DIRECTIONALLY SOLIDIFYING INGOT DURING PROCESSING (ASSEMBLY DETAIL)

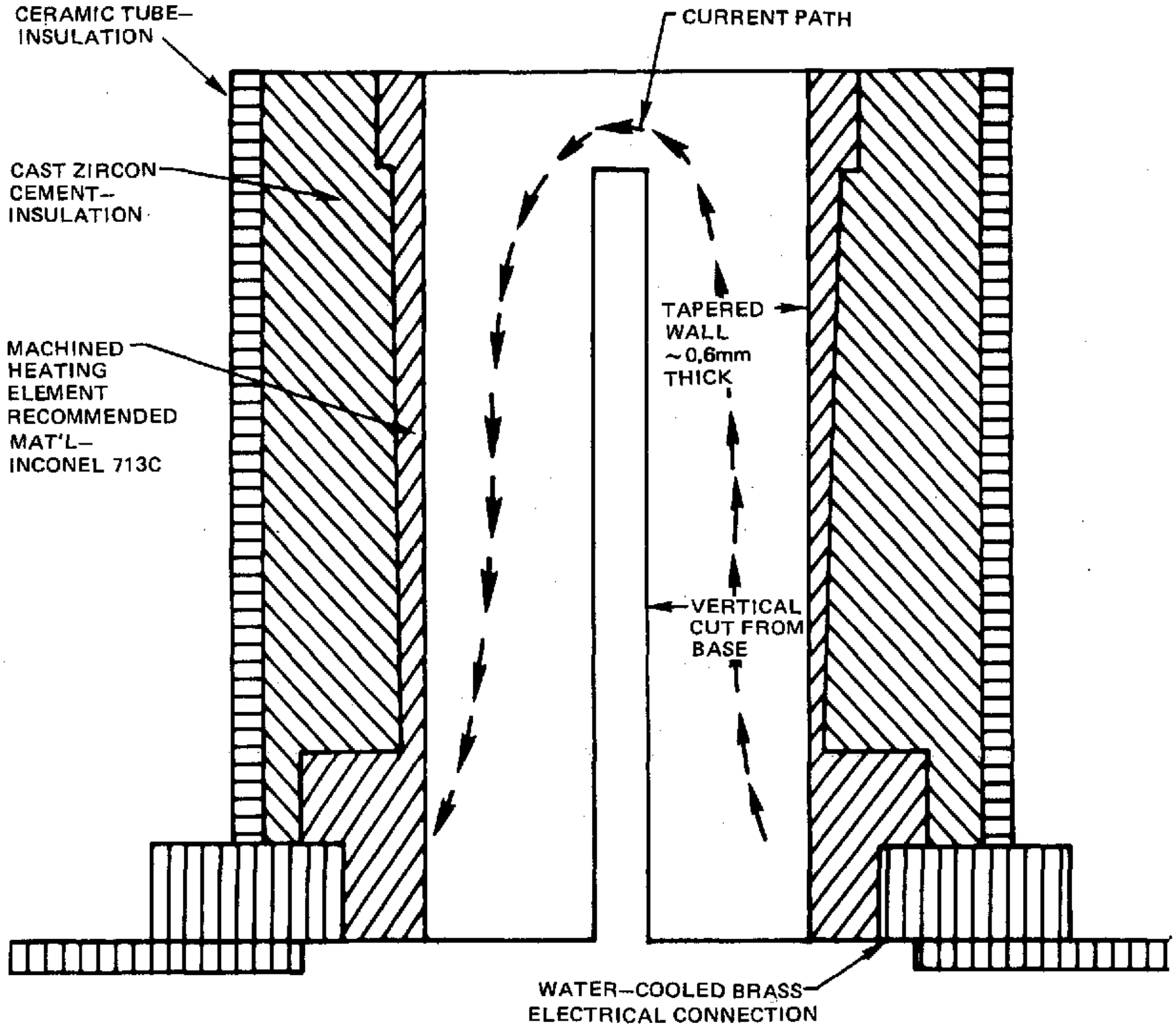
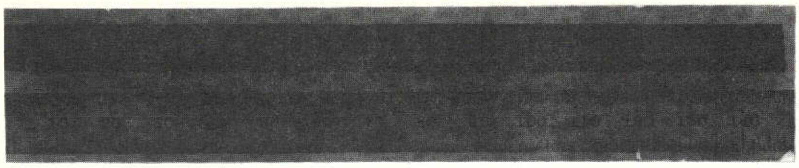


FIGURE 9. AUXILIARY FURNACE ASSEMBLY–DETAIL (SHOWN IN VERTICAL SECTION)



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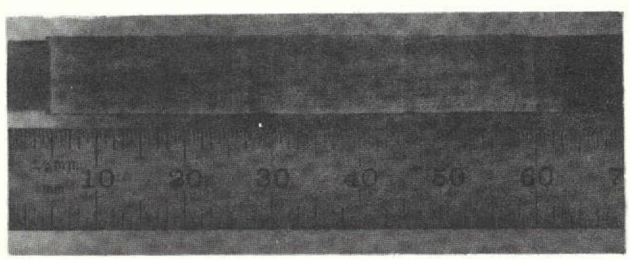

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FIGURE 10. Al–Al_3Ni EUTECTIC RODS SOLIDIFIED TO OBTAIN BOTH CONVEX AND CONCAVE SOLID–LIQUID INTERFACE CURVATURES

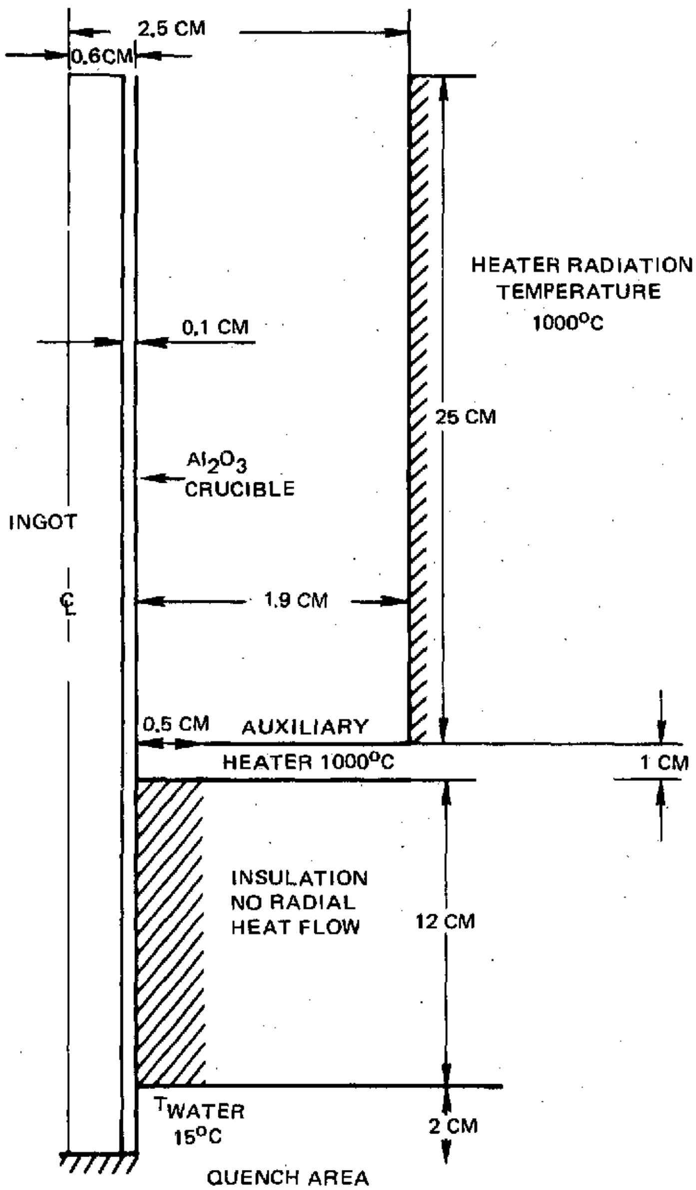

FIGURE 11. SYSTEM MODEL USED FOR SINDA THERMAL ANALYSIS

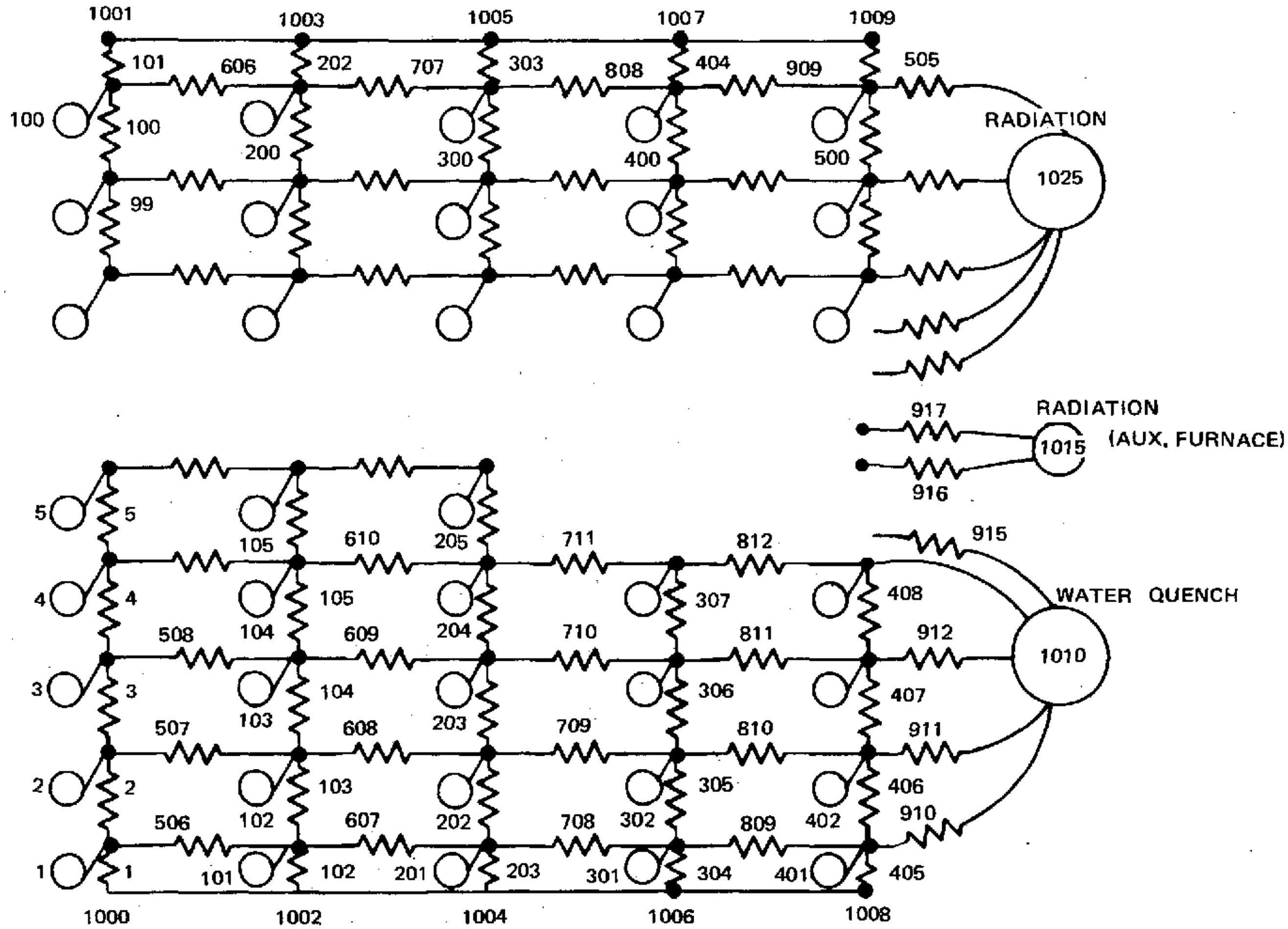

FIGURE 12. SINDA GRID SYSTEM FOR THE MODEL IN FIGURE 11

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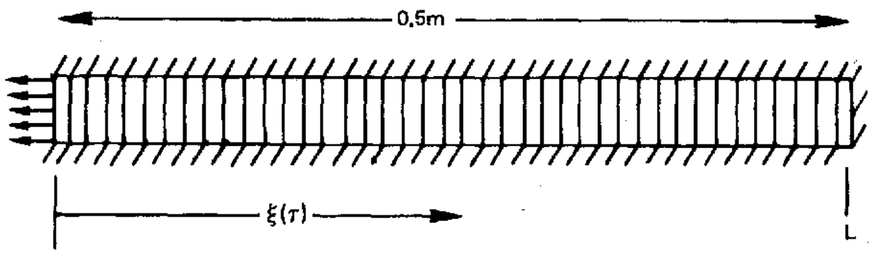
Symbols τ = time
ξ = interface position
x = length
T = temperature



ENERGY EXTRACTION AT THE BASE

Example 1: Insulated finite slab with constant energy extraction from one end

Boundary Conditions:

$$\left.\partial T(x,\tau)/\partial x\right|_{x=0} = \text{constant}, \quad \left.\partial T(x,\tau)/\partial x\right|_{x=L} = 0 \quad \text{for all } \tau > 0$$

Initial Condition: Slab is water at the freezing point; no ice present

Example 2: Insulated finite slab in contact with a temperature reservoir at one end

Boundary Conditions:

$$\left.T(x,\tau)\right|_{x=0} = Ta = \text{constant}, \quad \left.\partial T(x,\tau)/\partial x\right|_{x=0} \quad 0 \quad \text{for all } \tau > 0$$

Initial condition: Slab is water at the freezing point, no ice present

Example 3: Insulated finite slab in contact with a temperature reservoir at one end

Boundary Conditions: - Same as Example 2

Initial condition: Slab is water at a uniform temperature to above the freezing point.

FIGURE A–1. ONE DIMENSIONAL SLAB OF WATER 0.5 METERS LONG WITH VARIOUS BOUNDARY CONDITIONS FOR THE CLASS 1 PROBLEMS

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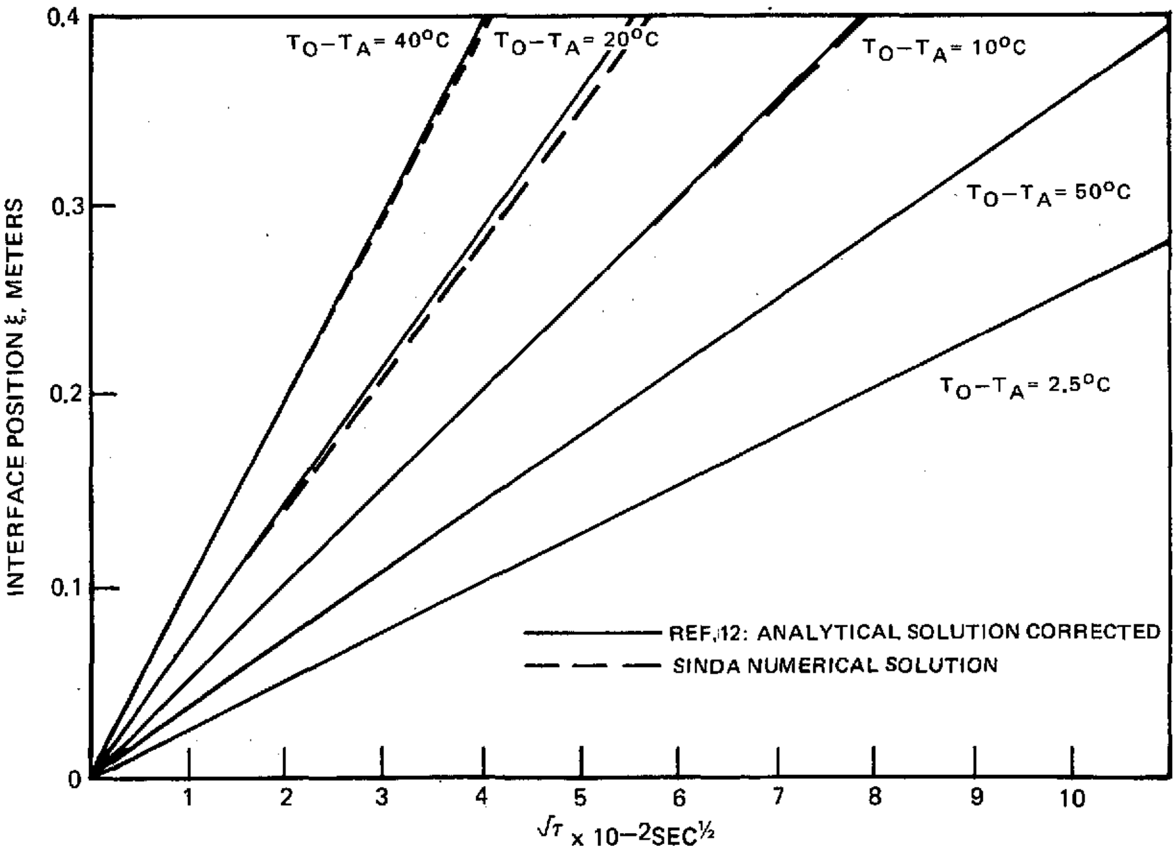
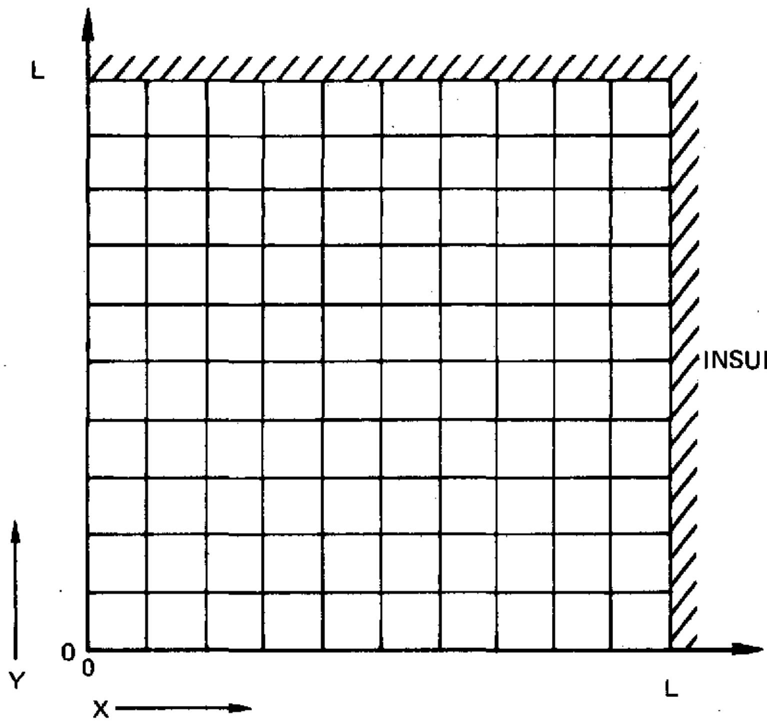


FIGURE A–2. RESULTS FOR EXAMPLE 2– PROPAGATION OF THE ICE WATER INTERFACE IN A ONE–DIMENSIONAL SLAB CONTACTING A CONSTANT TEMPERATURE RESERVOIR

N12–72–4



$$\partial T(x,y,\tau)/\partial x\Big]_{x=L} = 0$$

$$\partial T(x,y,\tau)\,\partial y\Big]_{y=L} = 0$$

$$T(x,y,\tau)\Big]_{x=y=0} = T_A \text{ (CONSTANT)}$$

FOR ALL $\tau > 0$

FIGURE A–3. ILLUSTRATION OF THE 2–DIMENSIONAL PROBLEM OF FREEZING WATER FROM THE CORNER OF A SQUARE WEDGE

N12–72–5

N911721–S/A2–4

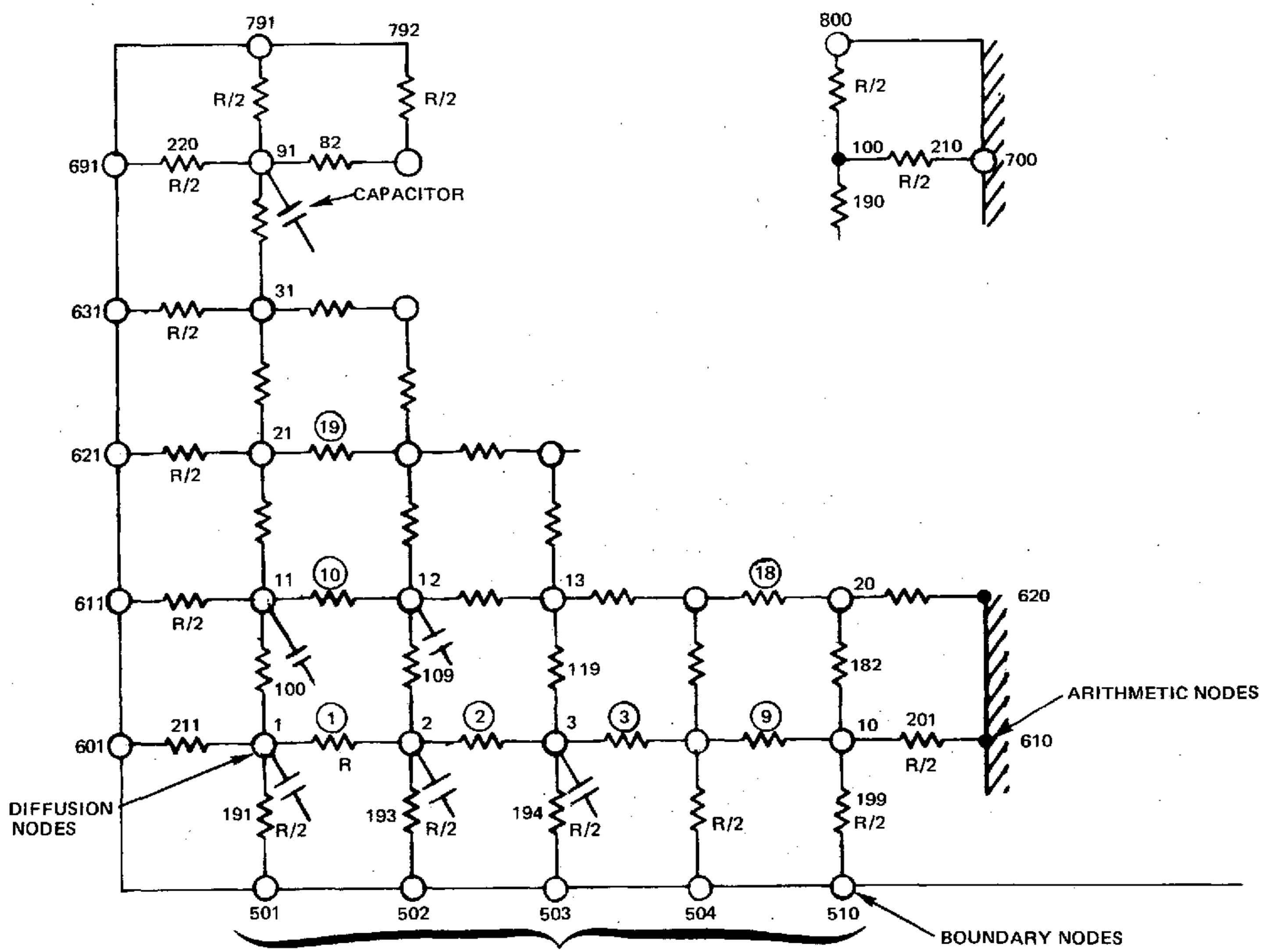


FIGURE A–4. SINDA GRID SYSTEM (PARTIAL) FOR EXAMPLE 3

N12–72–10