ANL/ET/CP-103188

MODELING THE ELECTROCONSOLIDATION® PROCESS

Raymond R. Fessler
BIZTEK Consulting, Inc.

F.-C. Chang
Argonne National Laboratory

Brian D. Merkle, Joshua M. Borton, and W.M. Goldberger
Superior Graphite, Co.

Presented at 2000 International Conference on Powder Metallurgy & Particulate Materials, New York, NY, May 30-June 3, 1000.

Work at Argonne National Laboratory was supported by the U.S. Dept. of Commerce, NIST Advanced Technology Program.

MODELING THE ELECTROCONSOLIDAITON® PROCESS

Raymond R. Fessler, BIZTEK Consulting, Inc.;

F.-C. Chang Argonne National Laboratory;

Brian D. Merkle, Joshua M. Borton, and
W. M. Goldberger, Superior Graphite Co.

ABSTRACT

Electroconsolidation® is a process for densifying complex-shaped parts using electrically conductive particulate solids as a pressure-transmitting medium. The part is immersed within a bed of the particulate medium contained in a die chamber. Heating to sintering temperature is achieved by resistive heating of the medium while applying compaction pressure. The process is capable of ultra-high temperatures and short cycle times, and it offers the potential for low processing costs.

Control of the process as well as selection of process conditions require knowledge of the temperatures within the die. Temperature gradients will exist because of the high heating rate and because of variations of density and electrical resistivity of the medium due to the presence of the part. Direct measurement of temperature with thermocouples or other conventional means is impractical because of the high temperatures, high currents, and high pressures. Therefore, a computer model was developed to predict the temperature as a function of time and applied voltage for any location in the die. The computer model contains three parts: a geometrical model to approximate the density and resistivity variations in the medium, a finite-element model to calculate the rate of resistive heating within each element, and a finite-difference model to calculate the temperature distribution based upon solution of the heat-transfer equations. Predicted temperatures have been shown to be in excellent agreement with measurements.

INTRODUCTION

Electroconsolidation® is a new process for rapid, pressure-assisted densification of near-net-shape powder preforms. In the process, heat is generated by passing an electric current through a particulate bed (usually powdered graphite) that surrounds the preform and also transmits pressure to the preform. The process is described in more detail elsewhere in this conference [1].

The ability of the process to heat complex-shaped parts rapidly under pressure without canning provides the potential for considerable cost savings compared with other near-net-shape powder-consolidation processes [2]. However, the speed and simplicity of the process also produce challenges with respect to controlling it. Temperatures gradients exist in the system due both to the high heating rates and to the fact that the rate of resistive heating depends upon the resistivity of the particulate medium, which varies from place to place because of the presence of the preform and friction between the medium and the die walls and the preform. The resistivity of the medium decreases with increasing compaction.

Normal methods of measuring temperature are not available for determining temperatures within the Electroconsolidation® die. Except for testing during the development phase, thermocouples cannot be placed inside the die. Not only would this be physically difficult, they would hinder the loading and unloading operations. Moreover, thermocouple assemblies would have to withstand severe mechanical stresses as well as extreme temperatures, and they would have to be able to operate accurately in the presence of high electric fields. Other direct sensors of temperature would have the same problem. Optical methods might be used for locations outside of the die, but providing line of sight into the die chamber would interfere with operations as in the case of thermocouples.

Because of the impracticality of direct temperature measurements, a control strategy was devised that is based upon a computer model that predicts temperature as a function of location and time. Innovative, nonintrusive methods of sensing the temperature are then used to check or calibrate the model. One method of checking the temperature involves placing small pellets of known melting point at several locations in the die and using radiography to determine when they melt. Another method involves measuring the time of flight of ultrasound through the bed and correlating that with the average temperature. Although neither of these methods can give continuous measurements of temperature at all locations, they can be used to verify the accuracy of the computer model.

This paper describes the computer model and compares its predictions with experimental measurements.

ELEMENTS OF THE ELECTROCONSOLIDATION® SYSTEM

A schematic diagram of the Electroconsolidation® system is shown in Figure 1. Typically, the outside diameter of the die is 8 inches (20 cm) and the inside diameter can be 3 to 5 inches (7.6 to 13 cm).

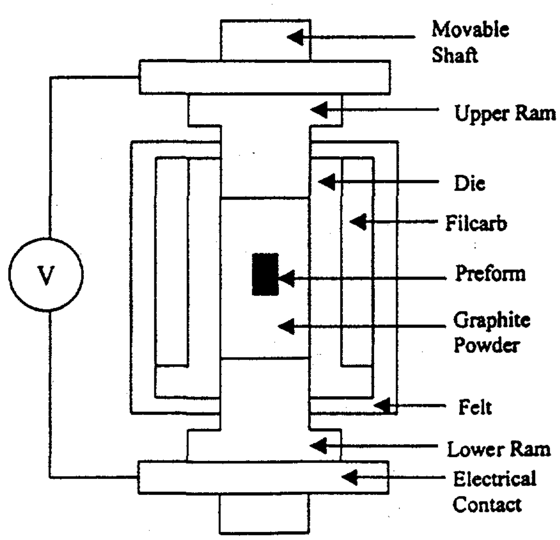


Figure 1. Schematic diagram of the Electroconsolidation® apparatus.

Pressure is applied with a 200-ton, double-acting hydraulic press, and current for heating comes from a 10,000-amp dc power supply. Electrical insulation can be used between the rams and the die and between the medium and the die if one wants to direct all current flow to the medium. Thermal insulation, usually graphite felt, may be used around the die to minimize heat loss and thermal gradients. Typically, the die and rams are constructed of solid graphite. For very high operating pressures, a fiber-reinforced sleeve (Filcarb) sometimes is used to support the graphite die.

APPROACH

The computer model for temperature prediction was developed in three modules. The first module calculates the density and resistivity of the particulate medium as a function of position in the die. The resistivities at each position are fed into the second module, where the rate of resistive heating is calculated. That information along with heat transfer calculations are used by the third module to predict the temperature at any location in the system as a function of time.

In developing any model, a number of simplifying assumptions must be made. In this project, an attempt was made to achieve an optimum balance between achieving adequate accuracy of the predictions and minimizing computational time. The accuracy of the predictions were verified against direct temperature measurements, which primarily were made with thermocouples at low and moderate temperatures.

DENSITY MODULE

The relationships among electrical resistivity, density, and pressure for the graphite particulate medium were determined experimentally using a large-diameter die to minimize wall-friction effects. Those relationships are shown in Figure 2.

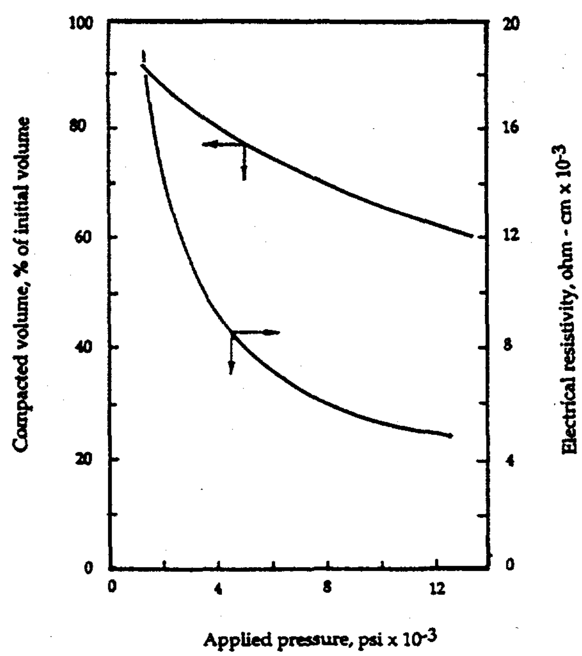
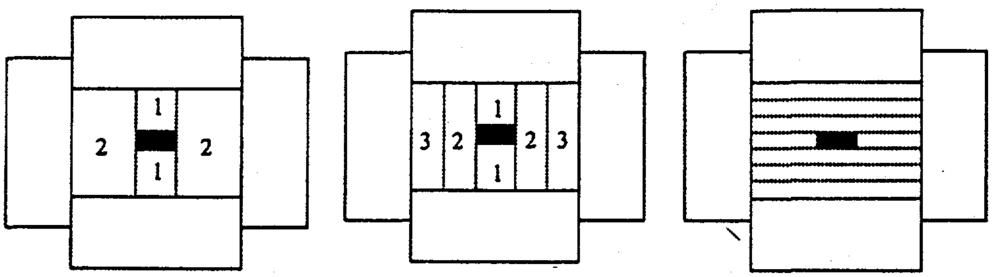


Figure 2. Effect of applied pressure on compacted volume and electrical resistivity

The biggest source of pressure variations within the die is the preform, which is relatively incompressible compared with the graphite medium. The graphite particles will tend to lock together at relatively low pressures, and, therefore, the medium will be more compressed above and below the preform than off to the sides. Another source of pressure variations is the friction between the medium and the die walls, which will cause the pressure to be somewhat elevated near the walls.

Several approaches for estimating the pressures (and thus the densities and resistivities) within the medium were evaluated. The simplest approach, which is the one that was finally adopted, was based upon geometrical considerations and an assumption that the graphite particles would not move horizontally in the die. Wall-friction effects also were assumed to be negligible. This situation is illustrated in Figure 3a, where the density - - and thus the electrical conductivity - - above and below the preform (Region 1) would be higher than in Region 2. The degree of compaction in each region can be calculated from the original and final positions of the rams, and the resistivity for each region can then be determined from Figure 2.



(a) (b) (c)

Figure 3. Schematic diagram of zones of equal density for (a) simple geometrical model, (b) simple geometrical model with high-density zone along die wall, and (c) Coulomb friction model.

Two approaches were used to estimate the effect on the temperature predictions of the higher density of the particulate medium along the die walls due to friction between the medium and the walls. The first approach involved a modification of the simple geometrical model, where a thin cylinder of higher-density medium (Region 3 in Figure 3b) was assumed to exist adjacent to the die walls.

The second approach (illustrated in Figure 3c) for estimating the effect of die-wall friction involved treating the powder as a cohesive material that obeys Coulomb's criterium for slip of the particles along the wall [3, 4]. A mean axial pressure, P_a, is introduced in place of the local axial pressure. A force balance for two horizontal planes separated by dz gives the following equation:

$$P_a \pi D^2/4 = P_f \pi Ddz + (P_a + dP_a) \pi D^2/4 \quad (1)$$

where D is the inside diameter of the die and

$$P_f = \eta P_r \quad (2)$$

where P_r is the radial pressure at the die wall and η is the coefficient of friction between the powder and the die wall. The radial pressure is related to the axial pressure by the Janssen constant κ (which is approximately equal to 0.175 for an unlubricated die wall) according to the following equation:

$$P_f = \kappa P_a \quad (3)$$

Thus

$$dP_a/P_a = (4\eta\kappa/D)dz \quad (4)$$

The mean axial pressure at any level can be obtained by integrating Equation 4. The axial and radial pressures are then used to calculate the compaction pressure, from which the density and electrical conductivity can be determined.

Figure 4 shows the results of temperature calculations for a position adjacent to the preform using the three approaches for estimating densities and resistivities throughout the bed. Because all three approaches give virtually the same results, the simplest was chosen for subsequent calculations.

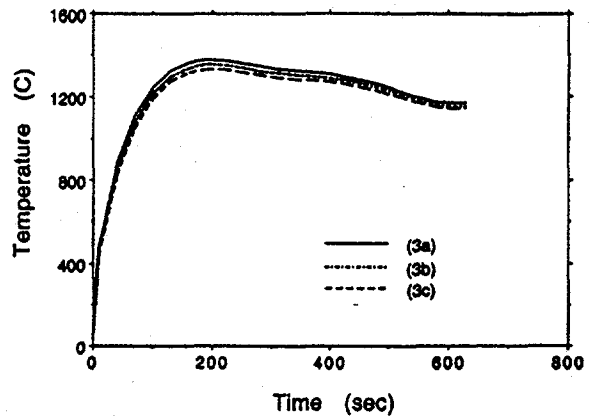


Figure 4. Temperature predictions based upon the three density models illustrated in Figure 3.

RESISTIVE-HEATING MODULE

A commercial finite-element code, ELEKTRA, was used to calculate the rate of resistive heating at all locations within the system. Published data were used for the electrical conductivity as a function of temperature for the rams, die, insulation, and preform. For the particulate medium, the electrical conductivity was treated as a function of density and pressure. Other input parameters are the physical dimensions of each component in the system and the applied voltage or current versus time.

In addition to providing numerical values for the rate of heating at each location, the program also can provide graphical output showing the magnitude and direction of current flow at each location, which can provide valuable insights into the reasons behind the ultimate temperature distributions.

HEAT-FLOW MODULE

A finite-difference computer model was developed at Argonne National Laboratory to calculate the temperature as a function of time for each location in the system. For each internal element, the rate of temperature change is proportional to the rate of heat generation by resistive heating (from ELEKTRA) plus the rate of heat conducted into the element from neighboring elements. For elements at exposed surfaces, heat loss by convection and radiation also is accounted for.

Published values for thermal conductivity and specific heat as functions of temperature were used for the die, rams, and insulation, as were published values for the density. For the graphite powder, published values were used for specific heat, but none were available for thermal conductivity as a function of density and temperature. The thermal conductivity, k_p, of the graphite powder as a function of temperature and density was estimated from the following relationship:

$$k_p = [0.0003 1/\gamma] [k_s (T)/k_s (25C)]$$

Where γ is the electrical resistivity in ohm-cm, $k_s(T)$ is the thermal conductivity of solid graphite at temperature T, and , $k_s(25C)$ is the thermal conductivity of solid graphite at 25°C, the latter two values being available from the literature. The constant 0.00031 is an empirical proportionality factor that relates thermal conductivity in cal/cm-sec-^{0}C and electrical conductivity at room temperature for solid graphite.

SAMPLE CALCULATIONS

Excellent agreement was obtained between predicted temperatures and those measured with a thermocouple in the medium. A typical example is shown in Figure 5, which also shows the applied voltage versus time and the position of the thermocouple. A pressure of 2000 psi was applied to the 3-inch (7.6-cm)-diameter die, and the rams were electrically isolated from the die. Figure 6 illustrates how the computer program can be used to show temperature gradients throughout the system during heating and cooling.

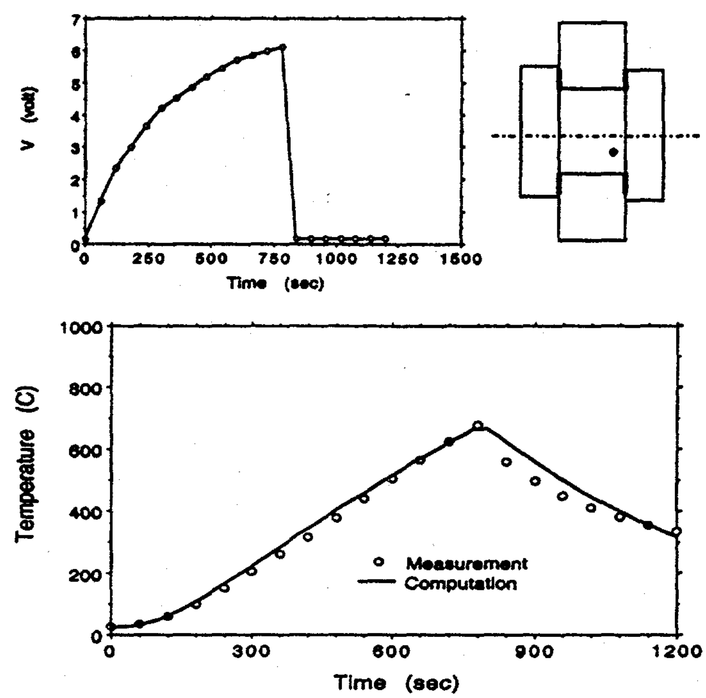


Figure 5. Comparison of calculated and measured temperatures in a 3-inch die.

Other typical comparisons between predictions and measurements are shown in Figures 7, 8, and 9. The conditions for Figure 7 are similar to those for Figure 5, except that a hold time has been added. Figure 8 shows the results for a larger die (5-inch [13-cm] diameter) surrounded by thermal insulation. Figure 9 shows results with a preform in the 5-inch (13-cm) die.

For small preforms, it would be economical to consolidate more than one at a time, provided that they all can be subjected to nearly identical cycles. The computer model can be used to predict temperature gradients in the die and temperature differences between preforms at various locations in the die. An example of this is shown in Figure 10, where it can be seen that reasonably uniform temperatures can be achieved for three preforms stacked vertically.

SUMMARY

A computer model has been developed to predict temperatures in an Electroconsolidation® die. With no adjustable or arbitrary input parameters, excellent agreement has been achieved between predictions and experimental measurements of temperature. Although density variations in the particulate medium affect the heating pattern, a simple approximation of those variations is sufficient to achieve accurate temperature predictions.

In addition to being the basis for an intelligent feedback control system for the Electroconsolidation® process, the computer model also can be used to predict power requirements for the process and to plan the placement of multiple preforms in the die.

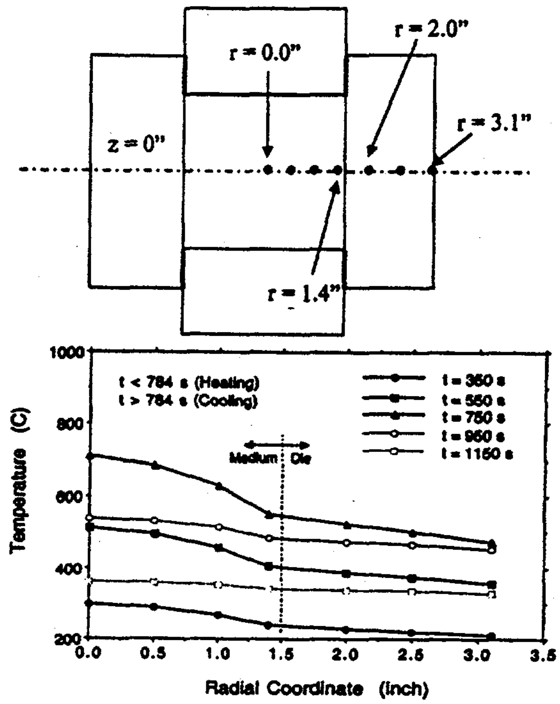


Figure 6. Temperature profiles during heating (solid points) and cooling (open points) in a 3-inch die.

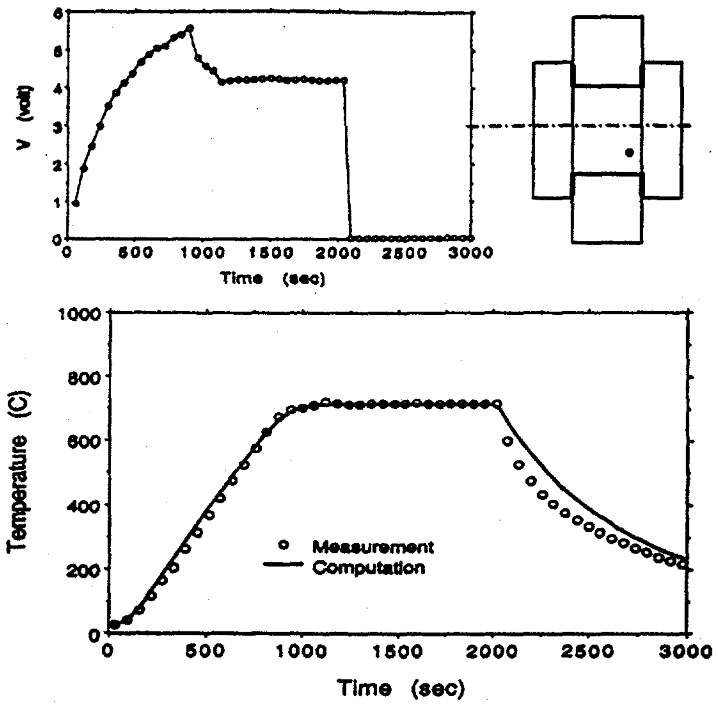


Figure 7. Comparison of calculated and measured temperatures with a hold time in a 3-inch die.

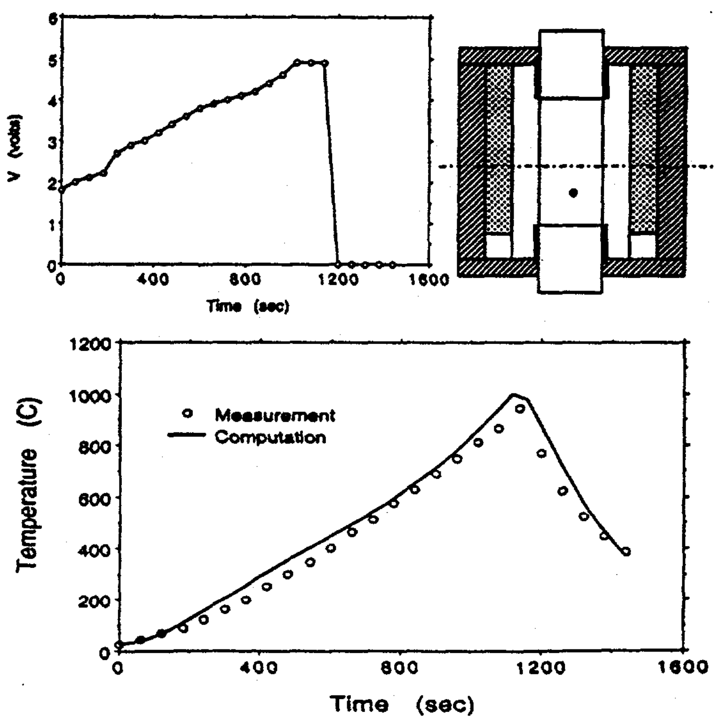


Figure 8. Comparison of calculated and measured temperatures in an insulated 5-inch die without a preform.

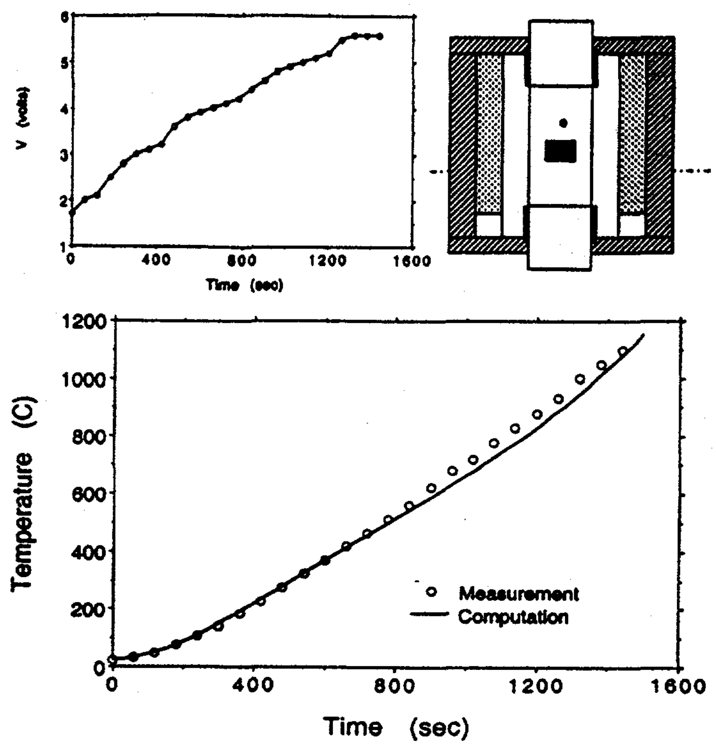


Figure 9. Comparison of calculated and measured temperatures in an insulated 5-inch die with a preform.

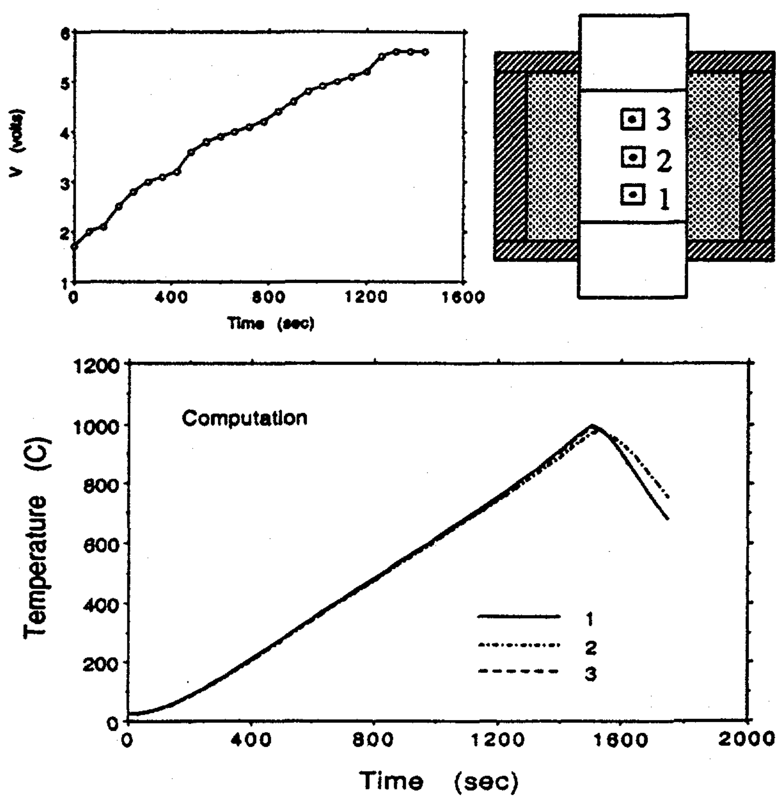


Figure 10. Predicted temperatures for 3 vertically separated preforms in a 5-inch die.

ACKNOWLEDGMENTS

The helpful suggestions and advice from Richard Valentin of Argonne National Laboratory and D. Lynn Johnson of Northwestern University are gratefully acknowledged. The authors are extremely grateful to the Department of Commerce, Advanced Technology Program and to members of the National Institute of Standards and Technology for their financial and technical support of this project.

REFERENCES

1. B. D. Merkle, J. M. Borton, and W. M. Goldberger, "Development of the Electroconsolidation® Process", *International Conference on Powder Metallurgy and Particulate Materials*, New York, NY, May 30-June 3, 2000.

2. W. M. Goldberger and R. R. Fessler, "The Development of Non-Intrusive Methods of Sensing and Control of Densification During Electroconsolidation®", *Advances in Process Measurements for the Ceramic Industry*, The American Ceramic Society, 1999, pp. 337-348.

3. Y. Li, H. Liu, and A. Rockabrand, "Wall Friction and Lubrication During Compaction of Coal Logs", *Powder Technology*, Vol. 87, 1996, pp 259-267.

4. Q. Deng and H. Liu, "Analysis of Coal Log Ram Extrusion", *Powder Technology*, Vol. 91, 1997, pp 31-41.