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DEVELOPMENT OF PG/SiC COMPOSITE MATERIALS FOR  
ROCKET-NOZZLE APPLICATIONS

VOLUME III - COMPARISON OF DEPOSITION FURNACES AND A  
GENERIC PROCESS CONTROL SYSTEM SPECIFICATION

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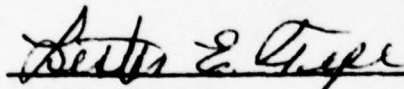
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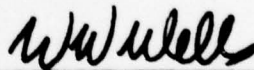
## FOREWORD

This report published in three volumes was submitted by Los Alamos Scientific Laboratory (LASL) of the University of California, Los Alamos, New Mexico, 87545, under the auspices of U.S. Energy Research and Development Administration Contract W-7405-ENG. 36, to the Air Force Rocket Propulsion Laboratory (AFRPL) under AFRPL MIPR agreement F04611-76-X-003, JON-305909JQ. The work was directed for LASL by Mr. T.C. Wallace and for AFRPL by Major J.G. Dean.

This report has been reviewed by the AFRPL Technical Information Office and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public. This technical report has been reviewed and is approved for publication; it is unclassified and suitable for general public release.




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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This is the final volume in a three volume set describing the pyrolytic graphite/silicon carbide (PG/SiC) coating development work at the Los Alamos Scientific Laboratory (LASL). In this volume we compare the injector deposition furnace with the channel flow furnace that was developed at LASL during this program. They are compared for ease of scaling to larger sizes and modeling with existing

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computer programs, and relative ease of implementing changes. A specification is given for a generic process control system for the PG/SiC deposition. A recommended step-by-step procedure to develop a coating furnace for a specified rocket-nozzle part is outlined and an example of its use is given.

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TABLE OF CONTENTS

Section	Page
I	INTRODUCTION . . . . . 1
	A. Background . . . . . 1
	B. Approach . . . . . 1
II	COMPARISON OF COATING FURNACES . . . . . 4
	A. Thermal Characteristics . . . . . 4
	1. Heatup and cooldown time . . . . . 4
	2. Relative stability at steady state . . . . . 5
	3. Relative ease of implementing changes . . . . . 5
	4. Scaling . . . . . 5
	B. Flow Characteristics . . . . . 6
	1. Scaling . . . . . 6
	2. Modeling . . . . . 6
	C. Deposition Characteristics . . . . . 7
	D. Process Control Requirements . . . . . 8
	1. Substrate surface temperature . . . . . 8
	2. Temperature gradient along the preheater . . . . . 9
	3. Inductive power supplied to the furnace . . . . . 9
	4. Mass flow rates of $N_2$ , $CH_4$ , and $CH_3SiCl_3$ . . . . . 9
III	CONTROL SYSTEM SPECIFICATION . . . . . 9
	A. General . . . . . 9
	B. Mass Flow Rate of the Reactants . . . . . 10
	1. General description . . . . . 10
	2. General requirements . . . . . 10
	3. Vaporizer/controller for MTS . . . . . 10
	4. Mass flow controller for $CH_4$ . . . . . 11
	5. Mass flow controller for $N_2$ . . . . . 12
	C. Substrate Temperature Control . . . . . 12
	1. General description . . . . . 12
	2. Deposition surface temperature measurements . . . 13
	a. General requirements . . . . . 13
	b. Pyrometer specifications . . . . . 13

TABLE OF CONTENTS (continued)

Section	Page
3. Furnace power controller . . . . .	14
a. General requirements . . . . .	14
b. Power controller specification . . . . .	15
4. Inlet wall temperature control . . . . .	15
IV DEPOSITION FURNACE DEVELOPMENT . . . . .	16
A. Preliminary Configuration . . . . .	16
B. Preliminary Model . . . . .	16
C. Final Design . . . . .	18
D. Refine Models . . . . .	18
E. Build Furnace and Run Tests . . . . .	19
V SUMMARY . . . . .	19
ACKNOWLEDGMENTS . . . . .	19
REFERENCES . . . . .	19
APPENDIX - EXAMPLE OF PRELIMINARY DESIGN FOR COATING A NOSE CAP . . . . .	21

LIST OF ILLUSTRATIONS

Figure		Page
1. Injector deposition furnace . . . . .		3
2. Channel flow deposition furnace . . . . .		3
3. Logic flow diagram . . . . .		17

## I. INTRODUCTION

### A. Background

In 1971 the Air Force Rocket Propulsion Laboratory began an effort to develop a continuously nucleated pyrolytic graphite (PG) with silicon carbide (SiC) additions.<sup>1</sup> This codeposited material, pyrolytic graphite/silicon carbide (PG/SiC), has the SiC phase deposited in a PG matrix as needles perpendicular to the deposition surface. The acicular SiC provides reinforcement in the c-direction and vastly improved shear strength between the a-b layers of the PG matrix. Additionally, it was found that the bond between the PG/SiC coating and an ATJ\* substrate was exceedingly strong.

A development program was then started with a major objective to demonstrate that the coating process for rocket-nozzle inserts could be scaled from a small-throat size (25 mm-i.d.) to a large-throat size (318 mm-i.d.). An additional requirement placed on the program was to demonstrate the capability to reproducibly coat nose caps that have a relatively complex configuration for incorporation into an advanced rocket-nozzle throat assembly. Unfortunately, the percentage of acceptably coated inserts decreased drastically as the throat size was increased.<sup>2</sup> An even lower acceptability rate was experienced with the nose cap configuration. Therefore, it was concluded that a more fundamental understanding of the deposition process was needed.

The Los Alamos Scientific Laboratory (LASL) was given the tasks of securing the requisite data base, developing an analytical model of the furnace and coating process, and specifying a fully automatic control system for the deposition process.

### B. Approach

To establish a relevant data base for validation of the analytical model and to document the operational characteristics of an existing PG/SiC coating furnace, a contract (LASL Order No. L66-17503-1) was made with Atlantic Research Corporation\*\* (ARC) to perform a series of engineering tests in their PG/SiC coating facility. The principal feature of the injector deposition furnace is that the flow into the furnace is an axisymmetric confined jet with high Reynolds ( $\sim 65000$ ) and low Mach numbers. The gas flow is characterized by a large

\*Trade name for a high-purity, high-density graphite manufactured by Great Lakes Carbon Company.

\*\*Atlantic Research Corporation, 5390 Cherokee Avenue, Alexandria, VA.

separated flow region with associated hot spot and a high level of turbulence (see Fig. 1). Test procedures and instrumentation were specified by LASL. Requisite deposition furnace modification for instrumentation, operation of the furnace during the tests, and a portion of the coat characterization were performed by ARC personnel. Volume I<sup>2</sup> describes these engineering tests, discusses the results, and compares these results with the analytical model.

A necessary characteristic of a deposition model is that it be an efficient and simple design tool for PG/SiC coating of nozzle parts. From a practical point of view, if the model is too complex, too expensive, or too demanding in terms of computer facilities for its general and routine application, its utility is greatly diminished. Early in the injector deposition furnace work, it became apparent that the recirculating flow within the deposition furnace was very complex and that the modeling of turbulent separated flows with heat addition, chemical reaction, and mass transfer is in an early stage of development. It became apparent that it would require much more time and resources than were available to develop a viable model for the injector deposition furnace.

A qualitative analysis of process change through the earlier scale-up program indicated that PG/SiC codeposited material with the requisite mechanical properties had been obtained from both separated and attached flow regions. Based on information developed to this point and the availability of a qualified computational fluid mechanics code that was capable of treating steady, two-dimensional, turbulent boundary flow with heat addition, chemical reactions, and mass transfer (but not capable of handling recirculation flow), a new deposition furnace configuration (channel flow furnace) was conceived that eliminated the unsolvable problems associated with the injector deposition furnace.

Design criteria for the new channel flow deposition furnace included an orifice configuration that permitted accurate specification of the fluid flow parameters at the start of the coating chamber, equivalent flow conditions for throat inserts and nose caps, better heat transfer to parts being coated, and other improved features.<sup>3</sup> In this configuration the process gas is injected tangentially in a large mixing plenum located beneath an annular coating chamber (see Fig. 2). The gas enters the annular flow passage with uniform velocity, temperature, and species concentrations. The rocket-nozzle part (substrate) is contained in the outer wall of the annular flow passage and is inductively heated. The flow over the heated substrate may be either laminar or turbulent but

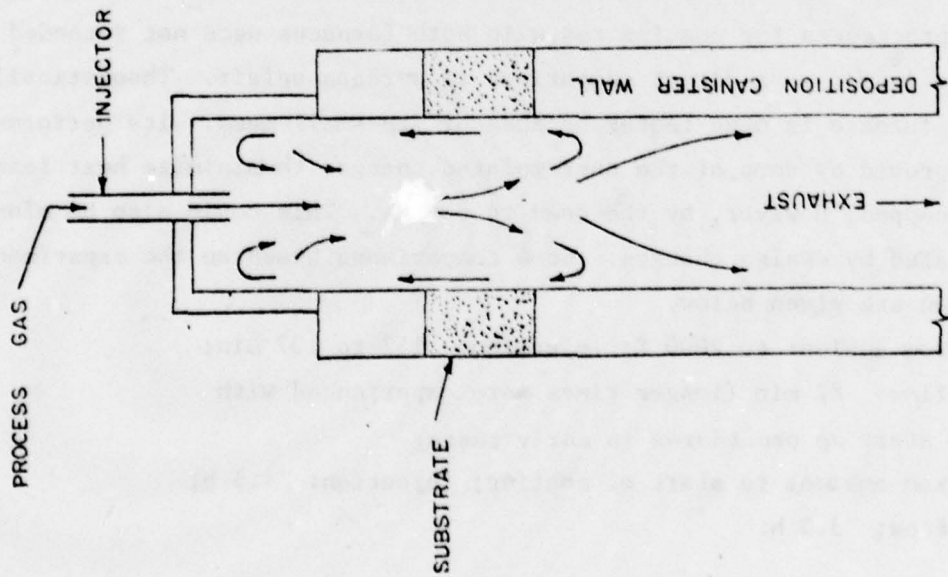


Fig. 1. Injector deposition furnace.

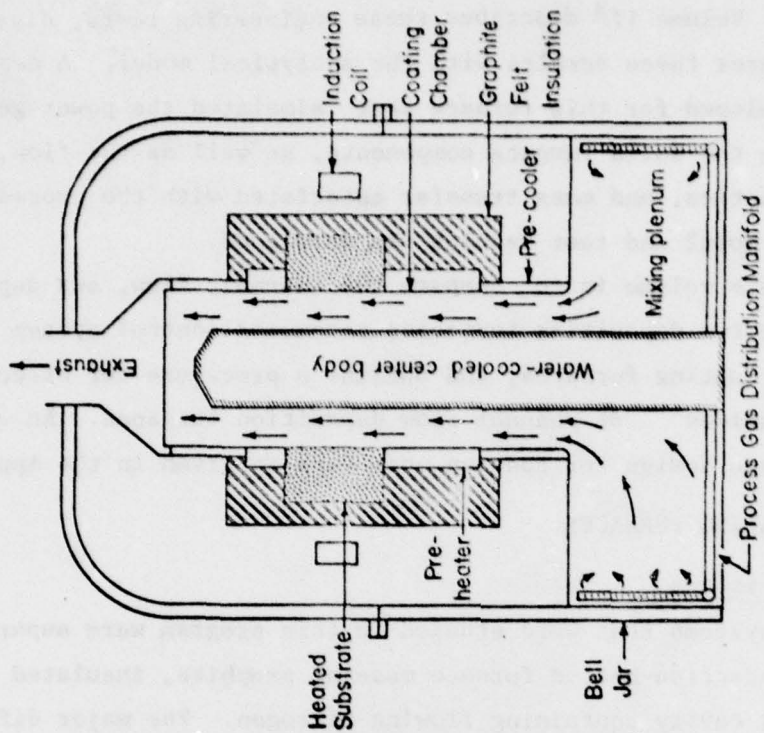


Fig. 2. Channel flow deposition furnace.

does not contain regions of separated flow. The deposition furnace was built and operated at LASL. Volume II<sup>3</sup> describes these engineering tests, discusses the results, and compares these results with the analytical model. A deposition process model was developed for this furnace that simulated the power generation and heat conduction in the solid furnace components, as well as the flow, heat transfer, chemical kinetics, and mass transfer associated with the process gas. The agreement between model and test results was very good.

The purpose of this volume is to compare the thermal, flow, and deposition characteristics of the two deposition furnaces; recommend control system specifications for generic coating furnaces; and outline a procedure for effective design and/or modifications of channel flow deposition furnaces. An example of a preliminary furnace design for coating nose caps is given in the Appendix.

## II COMPARISON OF COATING FURNACES

### A. Thermal Characteristics

The two coating systems that were studied in this program were superficially very similar -- an induction-heated furnace made of graphite, insulated on the outside with a central cavity containing flowing nitrogen. The major difference affecting modeling was the injection vs channel flow for the coating gases, discussed in Sec. II.C.

1. Heatup and cooldown time. The production rate of coated parts is dependent on the time to heat the furnace from ambient to its stable operating temperature and on the time to cool it after coating so that it can be disassembled. The test procedures for coating tests in both furnaces were not intended to minimize these items, so a direct comparison is perhaps unfair. Theoretically, the channel flow furnace is much faster because of its small mass. Its performance could be improved by some of the contemplated changes to minimize heat losses. It is handicapped, however, by the need to outgas. This could also be minimized or eliminated by design changes. Some comparisons based on the experiments that have been run are given below.

- Heatup from ambient to 2000 K; injection: 152 to 137 min;  
channel flow: 82 min (longer times were experienced with cautious start up procedures in early tests).
- Heatup from ambient to start of coating; injection: ~4.5 h;  
channel flow: ~3.3 h.

- Cooldown rate of substrate from coating conditions; injection: 0.12 K/s; channel flow: 0.92 K/s.
- Approximate time from power off to start of disassembly; injection: ~12 h; channel flow: ~ 0.5 h.

2. Relative stability at steady state. Because of its larger mass and its reduced water cooling, the injection furnace is inherently more stable. This is precisely the difference that allows the channel flow furnace to cool down faster. With an adequate control system, the ability to maintain constant temperatures in the channel flow furnace should not be hindered by its faster response. Some difficulty was encountered with the power controller in the channel flow furnace, which is unrelated to the design. Nevertheless, comparison of the data plots for substrate surface temperatures (T-7) in Refs. 2 and 3 shows that a reasonably constant temperature was maintained.

The exit temperatures from the cooling water loops gradually increased in the channel flow coating furnace. This was also true (to a lesser degree) of the cooling water exit temperature and fiberfrax o.d. temperatures in the injector deposition furnace. This is an indication that neither furnace was absolutely stable during the coating runs, although the measured internal temperatures, especially the substrate temperatures, were certainly stable enough for coating. The coating buildup was one of the reasons for increasing temperatures.

3. Relative ease of implementing changes. This is a subjective judgment to some extent. Access to the furnace interior is much better in the channel flow furnace because of the split in the bell-jar at the height of the substrate (Ref. 3, Figs. 1 and 3). In contrast, to reach the substrate in the injector deposition furnace, one must stand on a ladder and reach down inside the fiberfrax cylinder to about arm's length (Ref. 2, Figs. A-6 and A-11).

Changes are also facilitated by the relative ease in modeling the channel flow furnace. This enables more rapid, less expensive, and more accurate predictions of the effects of intended changes before they are implemented.

4. Scaling. The maximum diameter piece that can be coated in either furnace is limited by the diameter of the coils and, ultimately, the diameter of the fiberfrax or the bell-jar. The scaling is most affected by the ability to maintain the same coating conditions as the diameter increases. This is easily done in the channel flow furnace, but is difficult for the injector furnace (see Sec. II.C.).

There does not appear to be any other advantage for either design, except that the cost of a larger bell-jar is probably greater than the cost of a larger fiberfrax cylinder.

## B. Flow Characteristics

The major reason for developing the channel flow furnace was to eliminate the recirculating flow and the need for a complicated fluid dynamic model as in the injector deposition furnace. Figures 1 and 44 in Ref. 2 illustrate some of the complex flow fields in the injector furnace, and Sec. IV.B of Ref. 2 describes the development of the flow model. The flow model for the channel flow furnace is described in Sec. IV.B. of Ref. 2.

1. Scaling. The flow field of the channel flow furnace is directly scalable to larger diameters because the annular spacing between the part to be coated and the centerbody does not change. The flow field in this annular space does not change significantly with changes in diameter unless the diameter is reduced to less than 4 in. Although it cannot be stated with absolute certainty, the flow field in the injector furnace does not appear to be scalable without extensive computer modeling. The recirculating flow velocities depend on the diameters of the coating chamber and the injector, as well as the heat transfer at the wall. This study did not include experimental investigation of the effects of changes in coating furnace diameter. Previous studies<sup>4</sup> have shown that the heat transfer is a function of the ratio between upstream (injector) and downstream diameters. The heat transfer affects the local gas densities, which in turn affects the flow (Ref. 2, Sec. IV.B.2). It should be possible to increase both the coating furnace and injector diameters so as to keep their ratio constant and (hopefully) maintain the same flow field and heat transfer at the wall. However, we have not located any evidence among the sparse literature to show that this is true when both upstream and downstream diameters are changed. The mass flow rates of coating gases will increase as the square of the part diameter in the injector furnace, but linearly with the diameter in the channel flow furnace.

2. Modeling. The channel flow can be modeled using existing computer programs.<sup>3</sup> Because the coating chamber contains no adverse pressure gradients or abrupt area changes, boundary layer separation should not occur. Curved annular passages can be designed using the hodograph method<sup>5</sup> to prevent boundary layer separation. The numerical modeling of boundary layers is well developed.

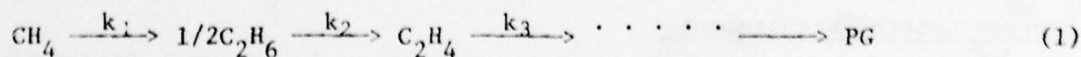
Fully separated internal flows are exceedingly complex with much higher levels of turbulence energy and stress than boundary layers. The full set of two-dimensional elliptic equations must be solved for the injector furnace. The turbulence was modeled using a mixing-length approximation.<sup>2</sup> The length-scale of the turbulence (or its equivalent) is essential to make the computational scheme suitable for design purposes. However, the proper turbulence mixing length is difficult to obtain. A more advanced kinetic-energy model would remove this difficulty, but current models neglect density variations and, in general, do not predict flows with large density variations very well.<sup>8</sup> In the injection coating furnace, the gas density varies with position by a factor of 7 because of the heating and velocity differences.

### C. Deposition Characteristics.

In the injector furnace tests, deposition onto the rocket-nozzle insert substrate occurred from an unattached region of flow; whereas, in the channel flow furnace, deposition occurred from an attached region of flow (Figs. 1 and 2). It is apparent that the thermal history of the coating constituent gaseous species in the injector furnace is very complex relative to that for the channel flow furnace.

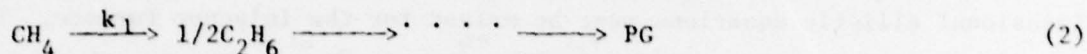
The optimum substrate surface temperature for the injector furnace is  $2050 \pm 50$  K compared with  $1850 \pm 50$  K for the channel flow furnace. Further, a more uniform substrate microstructure was obtained when the temperature across the substrate was constant ( $\pm 30$  K) or slightly increasing in the flow direction. When the substrate surface temperature is in the optimum temperature range for either furnace, the desired microstructure of the codeposited material was obtained for nitrogen flow rates greater than  $0.1 \text{ g/cm}^2\text{-s}$ , based on the cross sectional area of the flow passage. Reduced flow rates, at least with the channel flow furnace, resulted in the SiC being concentrated at the PG cone boundaries as coarse grained crystals.

When the temperature of a fluid element containing  $\text{CH}_4$  increases rapidly to greater than 1500 K, a series of consecutive-irreversible reactions occurs,<sup>9,10</sup>



where the  $k$ 's are first order reaction rate constants that are temperature dependent. The chemical and physical processes during the pyrographite deposition process are complex and are not well understood. However, in modeling the deposition process for the channel flow furnace, excellent agreement was

obtained between model and experiment if it was assumed that the rate limiting step in the chain was the decomposition of  $\text{CH}_4$  to  $\text{C}_2\text{H}_6$ ,<sup>3</sup> i.e.,



where  $k_1 = 10^{16} \exp(-103000/RT)$ ,  $\text{s}^{-1}$ . It is also reasonable to assume that this is the rate determining step in the injector furnace. Further, it was assumed that each  $\text{C}_2\text{H}_6$  molecule that diffuses to the heated substrate surface deposits as PG. Each  $\text{CH}_4$  molecule that diffuses to the wall is assumed to be reflected. The maximum deposition rate of PG was directly proportional to the initial concentration of  $\text{CH}_4$  in the process gas stream and independent of the substrate surface temperature in the range of 1800 to 1950 K. The axial variation of the PG deposition rate is dependent on the temperature gradient along the preheater section (Fig. 2) in general agreement with the rate limiting step proposed in Eq. 2. The PG deposition rate was constant ( $\pm 10\%$ ) for axial distances within 60 to 80 mm of the substrate's mid-point.

In the deposition model for SiC we assumed that all  $\text{CH}_3\text{SiCl}_3$  (MTS) that diffuses to the wall is deposited as SiC. From 1400 to 1900 K, the SiC deposition rate is directly proportional to the initial concentration of MTS in the process gas stream and independent of substrate surface temperature. Above 1900 K, the deposition rate appeared to drop drastically with increasing temperature, but later investigation with the analytical model showed this decrease in deposition rate to be caused by local depletion of MTS near the wall.

To obtain an acceptably coated rocket-nozzle insert, it is essential that the axial zone where the PG deposition rate is constant (for a given process gas  $\text{CH}_4$  concentration) coincides with the location of the part. Once this match is obtained, the weight percent of SiC codeposited with the PG can be controlled by the concentration of MTS in the process gas stream. This design effort can be effectively completed through the use of the deposition process model developed for the channel flow deposition furnace. This statement is not true for the injector deposition furnace.

#### D. Process Control Requirements.

The following process parameters are considered to be the most important for control of the coating furnace.

1. Substrate surface temperature. This temperature must be constant ( $\pm 20$  K) over the axial length of the part being coated. To achieve this, there must

be a means for measuring the surface temperature near the center of the part and heat transfer axially should be minimized relative to the radial direction. The temperature measurement may be done optically or with the placement of several thermocouples. If the temperature is measured optically, a two-color pyrometer should be used and the absorptivity of the coating gas in the configuration being used must be determined experimentally. If thermocouples are used, their installation and calibration must be carefully done to insure that the temperature being measured is representative of (or corrected to) the substrate surface. Because of the insulating property of the coating, this thermocouple-to-surface correction will be a function of the coating's thickness.

2. Temperature gradient along the preheater. Control of this parameter determines the extent to which the  $\text{CH}_4$  has been pyrolyzed in the region near the hot wall. It is also important to obtain a smooth transition between the PG deposition of the substrate and the adjacent graphite part. It can be measured in the same way as the substrate surface temperature. Measurements may not be necessary in a well-characterized furnace.

3. Inductive power supplied to the furnace. Control of this parameter is necessary in order to achieve the desired temperatures and maintain them during a coating run. The measurement should account for changes in the electrical properties of the furnace as it heats up.

4. Mass flow rates of  $\text{N}_2$ ,  $\text{CH}_4$ , and  $\text{CH}_3\text{SiCl}_3$ . The coating deposition rate is directly proportional to the concentration of  $\text{CH}_4$  and  $\text{CH}_3\text{SiCl}_3$  and the quality of the coating is dependent on the  $\text{N}_2$  flow rate. The controllers used in the coating tests<sup>2,3</sup> for these gases were very satisfactory and similar systems would be required in any new development.

### III. CONTROL SYSTEM SPECIFICATION

#### A. General.

There are six parameters to be controlled for the chemical vapor deposition of PG/SiC. The mass flow rates of gaseous nitrogen, methane, and MTS must be selectable and controlled. The temperature of the substrate must be controlled. This temperature measurement is greatly influenced by the configuration of the substrate and the chemical vapor deposition furnace. The configuration of the deposited part could dictate the requirement for two temperature measurements on the substrate. The induction power supplied to the furnace should be controlled and operate in a feedback loop with the substrate surface temperature. The wall

temperature gradient from the inlet to the substrate should be controlled. This last parameter could ultimately be a function of the furnace design with no actual controller required. The design base is for rocket-nozzle throat diameters in the range of 170 to 380 mm and associated nose caps.

## B. Mass Flow Rate of the Reactants.

At present the sizes and configurations of the rocket nozzle parts to be coated dictate the delivery rate of reactants. The large range in flow rates could require different flow meters and flow controllers for each reactant. This is especially true for the MTS. The specifications for these controllers follow.

1. General description. This system shall provide for the automatic control of liquid vaporization and mass-flow rate of MTS. By controlling the mass-flow rate of the carrier gas (helium) through the liquid MTS, the rate in grams per minute of liquid vaporization shall be controlled independent of liquid level, temperature, or pressure. This system shall also provide automatic control of the mass-flow rate of methane ( $\text{CH}_4$ ), and nitrogen ( $\text{N}_2$ ). A digital display shall be provided to indicate the mass-flow rate of MTS, He,  $\text{CH}_4$ ,  $\text{N}_2$ , and the ratio of MTS to He. All plumbing and valves necessary to interconnect, fill, purge, and control the system shall be included as part of this system. The bubbler tank<sup>2</sup> for the MTS shall also be included. The bubbler tank shall be capable of delivering  $0.2 \text{ m}^3$  (50 gallons) without refilling. The system shall perform within specification limits while subjected to the frequency field (3 to 10 kHz) produced by a 250 kW induction furnace.

### 2. General requirements.

System Input Power	115 $\pm$ 10 VAC, 60 $\pm$ 6 Hz
Electrical Connections and Cables	All interconnecting cables and connectors shall be included. The length of cable between the controllers and the display shall be 8 m minimum.

### 3. Vaporizer/Controller for MTS

Vaporized Liquid	$\text{CH}_3\text{SiCl}_3$ (MTS).
Carrier Gas	Helium
Measurement and Control Range	Seven to 200 grams of MTS per min. $\pm 4.0\%$ of full scale over entire operating range.
Regulation	$\pm 0.25\%$ of full scale for pressures from 69 to 276 kPa.
Command Setting	4 digit minimum resolution.

Control Point Repeatability	The correlation between the mass-flow rate of MTS and the command setting shall be repeatable to within $\pm 0.25\%$ full scale.
Operating Pressure Range	69 to 276 kPa.
Minimum Pressure Differential	69 kPa.
Gas Temperature Range	288-316 K (60-110°F).
Ambient Temperature Range	288-316 K (60-110°F).
Response	20 max to within $\pm 4\%$ of set point.
Indication	4-1/2-digit DVM and shall indicate in "the International System of Units (SI)"
Mounting	To be determined.
Gas Connections	1/4-inch Swagelok or equivalent compression type.

#### 4. Mass-flow controller for CH<sub>4</sub>

Measurand	CH <sub>4</sub> (gas)
Measurement and Control Range	0 to 0.3 m <sup>3</sup> /min
Inaccuracy	$\pm 1\%$ full scale over entire operating range. (The operating range includes the specified ranges of operating pressure, ambient temperature, and gas temperature).
Nonlinearity	$\pm 1\%$ full scale over entire operating range. (This operating range includes the specified ranges of operating pressure, ambient temperature, and gas temperature).
Regulation	Controller shall maintain preset mass-flow rate to within $\pm 0.25\%$ of setting when pressure drop is between 69 and 276 kPa.
Command Setting Adjustment	4 digit minimum resolution.
Control Point Repeatability	The correlation between the mass-flow rate and the command setting shall be repeatable to within $\pm 0.25\%$ full scale.
Operating Pressure Range	69-276 kPa.
Response	20s to within $\pm 2\%$ of set point.
Maximum Gas Pressure	1.03 MPa
Ambient Temperature Range	278-316 K (40-110°F)
Gas Temperature Range	278-316 K (40-110°F)

Indication	4 1/2 digit DVM and shall indicate in "The International System of Units (SI)"
Mounting	Mounting of unit in any position shall not affect accuracy.
Gas Fittings	1/4 inch Swagelok or equivalent compression type.

#### 5. Mass-flow controller for N<sub>2</sub>

Mesaurand	N <sub>2</sub> (gas).
Measurement Control Range	0.5-3.0 m <sup>3</sup> /min.
Inaccuracy	+ 1% full scale over entire operating range. (The operating range includes the specified ranges of operating pressure, ambient temperature, and gas temperature).
Nonlinearity	+ 1% full scale over entire operating range. (The operating range includes the specified ranges of operating pressure, ambient temperature, and gas temperature).
Regulation	Controller shall maintain preset mass-flow rate to within ± 0.25% of setting when pressure drop is between 69 and 276 kPa.
Command Setting Adjustment	4 digit minimum resolution.
Control Point Repeatability	The correlation between the mass-flow rate and the command setting shall be repeatable to within ± 0.25% full scale.
Operating Pressure Range	69-276 kPa.
Response	20 s to within ± 2% of set point.
Maximum Gas Pressure	1.03 MPa.
Ambient Temperature Range	278-316 K (40-110°F).
Gas Temperature Range	278-316 K (40-110°F).
Indication	4 1/2-digit DVM and shall indicate in m <sup>3</sup> /min of N <sub>2</sub> .
Mounting	Mounting of unit in any position shall not affect accuracy.
Gas Fittings	To be determined.

#### C. Substrate Temperature Control.

1. General description. It is necessary to control the temperature of the substrate by controlling the power to the furnace. This control specification will be described in two parts. The first part will describe substrate

temperature measurement and the second part, the power control function.

The number of temperature measurements and their measurement technique depend on the size and configuration of the substrate and the configuration of the furnace. Some shapes of rocket nozzle parts (such as the nose cap) could require two temperature measurements on the deposition surface or two measurements on the o.d. of the substrate, if the furnace configuration precludes measurement on the deposition surface.

## 2. Deposition surface temperature measurement.

a. General requirements. As stated above, either one or two temperature measurements could be required, depending upon the configuration of the substrate. It might become necessary to incorporate two heating coils for some configurations to control the temperature gradient across the substrate within the  $\pm 25$  K tolerance. Temperature measuring devices cannot contact the substrate surface, so an optical pyrometer is necessary to make these measurements. The pyrometer will be viewing the substrate through the reactant gases; therefore, a two-color type pyrometer similar to that used to measure the substrate temperature during the LASL experiments is required (Ref. 3, Appendix B). An infrared or total-radiation type pyrometer could be used in lieu of a two-color unit to measure the temperature of the substrate o.d. However, it is mandatory to use the two-color type pyrometer for the substrate surface, and these units can be used with equal versatility and accuracy elsewhere; therefore, only the two-color units will be specified.

### b. Pyrometer specifications.

Temperature Range	1200-3000 K min.
Inaccuracy	$\pm 1\%$ max. of temperature span.
Field of View (Resolution Angle)	8.7 milliradians $0.5^\circ$ max.
Focusing Range	600 mm to infinity.
Response Time	
(i) Output to Controller	Compatible with the requirements of the controller.
(ii) Meter	3 s max. to 95% of full scale.
Nonrepeatability	$\pm 0.25\%$ max. of temperature span.
Instability	
(i) Constant Temperature	$\pm 0.5\%$ max. of temperature span over a period of 24 h.

(ii) Line Voltage Variation	$\pm 0.5\%$ max. of temperature span for a change in line voltage from 90 to 135 <sup>o</sup> F.
Resolution	
(i) Meter	10 <sup>o</sup> K minimum.
(ii) Output to Controller	Compatible with the requirements of the controller.
Linearity	Compatible with the requirements of the controller.
Hysteresis	Compatible with the requirements of the controller.
Ambient Temperature Range	278-322 K (40-120 <sup>o</sup> F).
Power Requirements	115 $\pm$ 10 VAC, 60 $\pm$ 6 Hz.
Cables	All interconnecting cables and connectors shall be included. The length of cable between pyrometer and the controller shall be 8 m minimum.
Optical Head Mount	Tripod (1/4-20 thread).

### 3. Furnace Power Controller.

a. General requirements. The type of controller required to adjust the furnace power as a function of substrate temperature cannot be specified at this time. Many factors that dictate the controller design are involved, such as the exact size and configuration of the furnace, the power supply, and the shape of the heating coils. These will probably become a design trade off between the analytical model specification, the equipment availability, and the rocket-nozzle part configuration. The power supply may furnish furnace power at DC through RF frequencies. If induction heating is used, a control system to automatically adjust the power factor is desirable. The controller should include a program to ramp the power as a function of time or temperature. This feature limits the thermal shock to the furnace and substrate and should be used on the furnace cool-down as well as the heat-up cycle. It is suspected that this ramp function would be a characteristic of the furnace and substrate configuration, which means that it should be programmable. The type of power controller is intrinsically linked to the type of motor-generator set or DC rectifier used.

An additional problem exists should the power control be based upon the temperature of the substrate o.d. As the coating thickness increases and the insulation of the furnace degrades, the temperature of the substrate o.d. must be increased to maintain a constant temperature on the coating surface. Based upon available data, this time/power relationship would follow a curve defined by

$$y = e^{ax}$$

(3)

where

$x$  = a function of temperature\* and time.

$e$  = 2.71828

$a$  = a constant greater than zero

$y$  = desired temperature of substrate o.d.

The system time constant is directly related to the physical configuration of the furnace, its materials, and the efficiency at which it is heated. Each furnace configuration could then necessitate a different controller transfer function.

In view of the required versatility for this controller, a microprocessor would be well suited for this application. Specifications for a microprocessor will not be included.

b. Power controller specification.

General Requirements	The tolerances of the following parameters are system tolerances, i.e., they include the tolerances specified on the pyrometers.
Accuracy	$\pm 25$ K.
Temperature Regulation	$\pm 10$ K minimum
Control Stability	$\pm 10$ K over a 24 h period.
Command Setting	4 digit minimum resolution.
Control Point Repeatability	The correlation between the temperature and the command setting shall be repeatable to within 10 K.
Response	To be determined.
Controller Output	To be determined.
Ambient Temperature Range	278-322 K (40-120°F).
Power Requirements	115 $\pm$ 10 VAC, 60 $\pm$ 6 hz.

4. Inlet wall temperature control. The control of this temperature might be an inherent property of the substrate temperature control, should the furnace and heating coil design allow such latitude. However, in the event that explicit

\*This temperature is not the substrate temperature, but is a temperature measurement within the insulation of the furnace indicating the degradation of the insulation as a function of time.

and separate control is required, the specification outlined earlier for the measurement and control of the substrate temperature will govern. The only exception is the control temperature range, which is 1200 to 1900 K.

The required axial temperature gradient is almost entirely a function of the deposition gas velocity. The wall temperature control set-point depends upon such conditions as inlet tube diameter, gas flow rate, and measurement position. These are all part of the furnace development described in Section IV.

#### IV DEPOSITION FURNACE DEVELOPMENT

The furnace development outlined in the logic flow diagram (Fig. 3) is similar to that involved in developing any complicated equipment.

##### A. Preliminary Configuration.

The preliminary configuration evolves from consideration of the part specifications. Its shape will dictate the size of the coil and contour of the channel flow passage. The thickness of the coating might require some alteration in the flow annulus to allow for its growth. The coil will be sized and placed around the substrate so as to heat it evenly, and insulation will be added as needed to minimize energy loss and equalize temperatures. The shape of the coolant passage will dictate the configuration of the centerbody, which can be developed using hodograph methods<sup>5</sup> to prevent boundary layer separation. Cooling circuits can be added as required to insure that parts of the furnace are not overheated. The flow rate of coating gases will be dictated by the part specifications, process kinetics (Sec. II C), and flow passage geometry. When these steps are completed, a rough sketch of the furnace with major dimensions shown is produced.

##### B. Preliminary Model.

A preliminary model of the furnace is produced from the rough sketch using the required gas flow rates and estimates for the cooling water flow, boundary conditions, and power input. It is at this stage that many unique features of the computer programs GENMIX<sup>6</sup> and AYER<sup>7</sup> can be used to advantage.

The GENMIX program uses stream function and distance along the flow passage as basic independent variables rather than a fixed geometric coordinate system. Therefore, the model does not require any finer computing mesh due to the curvature in the annular flow passage because the stream lines tend to follow it.

If the flow geometry is simple enough that velocities can be estimated easily, the GENMIX program may be bypassed for the preliminary model. The AYER program

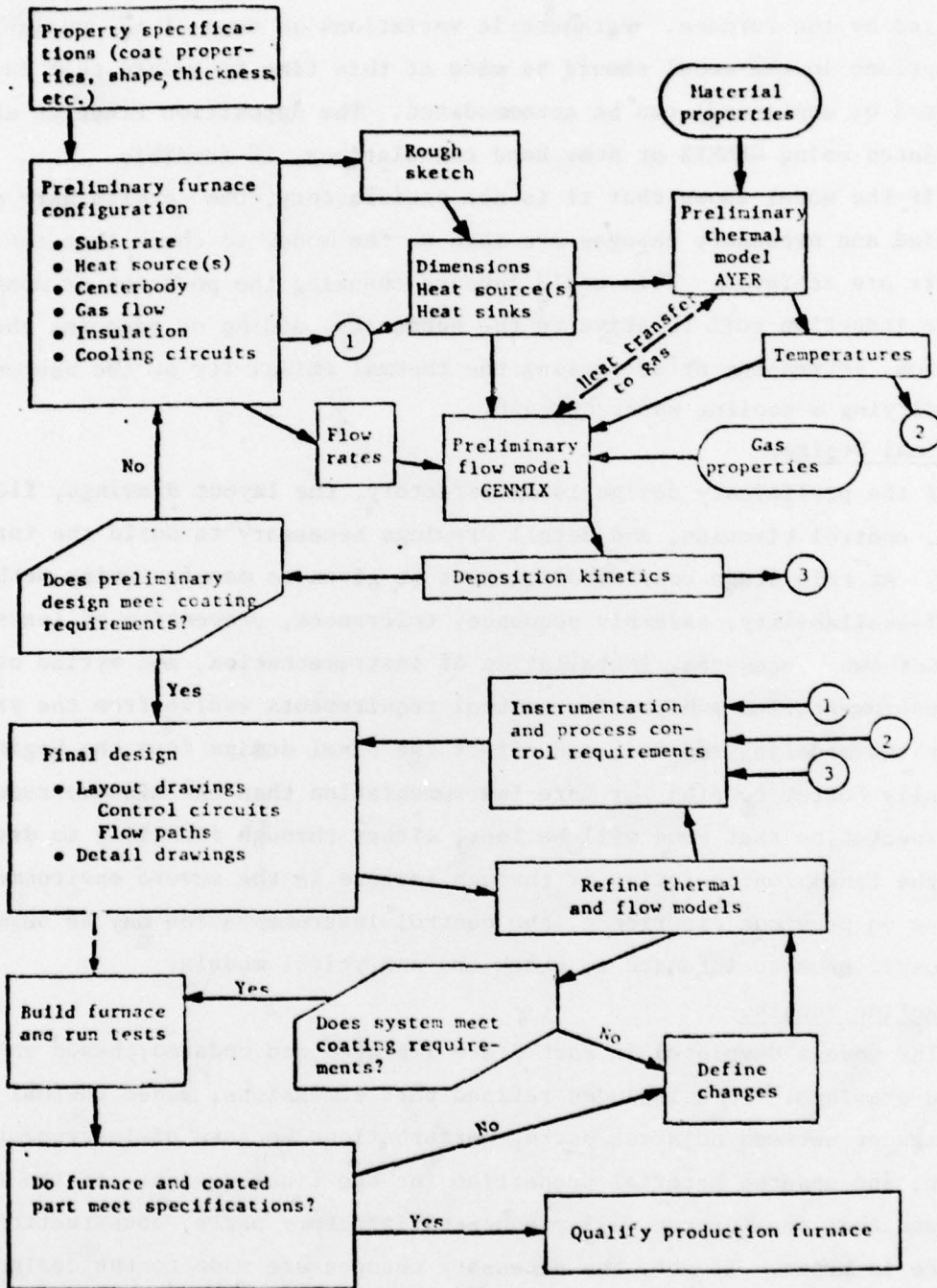


Fig. 3. Logic flow diagram.

includes terms involving energy transport by fluid motion, so that the flow passage can be included in the heat conduction model. Fixed temperature boundary conditions can be applied, which eliminates the need to guess at the total power required by the furnace. Parametric variations in some of the variables and assumptions in the model should be made at this time to insure that future changes (planned or unplanned) can be accommodated. The deposition kinetics should be calculated using GENMIX or some hand calculations, if feasible.

If the model shows that it is not satisfactory, the preliminary design is modified and necessary changes are made to the model to check that the desired effects are achieved. This could involve changing the position or configuration of the induction coil relative to the substrate, adding or removing thermal insulation, increasing or decreasing the thermal emissivity of the centerbody, or modifying a cooling water circuit.

#### C. Final Design.

If the preliminary design is satisfactory, the layout drawings, flow schematic, control circuits, and detail drawings necessary to build the furnace are begun. At this stage consideration must be given to manufacturing methods, material availability, assembly sequence, tolerances, prevention of leaks, start up and shutdown procedures, installation of instrumentation, and myriad other details. The instrumentation and process control requirements evolve from the preliminary design and modeling results, and affect the final design from the beginning. It is usually better to plan for more instrumentation than the minimum required in the expectation that some will be lost, either through inability to design it into the final configuration or through failure in the severe environment. Depending on previous experience, the control instrumentation may be augmented by diagnostic sensors intended to check the analytical models.

#### D. Refine Models.

The models developed in Part B are improved and updated, based on the completed drawings. This includes refined part dimensions, added thermal contact resistances between adjacent parts, perturbations because of instrument installation, and updated material properties for the final design. If the calculations indicate that the furnace will produce satisfactory parts, construction of the furnace is begun. If not, the necessary changes are made to the design. The model and resulting configuration are again subjected to the review process. It may be helpful at this stage to formalize the procedure, including a drawing sign-off where all parties to the design, operation, analysis, and instrumentation can

resolve conflicting (or overlooked) requirements.

#### E. Build Furnace and Run Tests.

The final step in the furnace development is to build and test it against the model predictions. If it performs satisfactorily, a production qualification would take place, verifying that it can produce the satisfactory parts repetitively. If the test shows a need for improvement, the possible changes can be examined in the model. This was done in the LASL channel flow furnace when it was found that the wall upstream of the substrate was too hot. Several potential "fixes" were modeled and it was found that adding a 13-mm thickness of felt insulation on the top and bottom of the substrate was practical and effective.

#### V. SUMMARY

The channel flow furnace was found to have advantages in ease of scaling and modeling and relative ease of implementing changes. A specification for a generic process control system and a recommended step-by-step procedure to develop a coating furnace have been described.

#### ACKNOWLEDGMENTS

We appreciate the interest and advice of Major John G. Dean, the Air Force Rocket Propulsion Laboratory Technical Project Officer for this program.

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## APPENDIX

### EXAMPLE OF PRELIMINARY DESIGN FOR COATING A NOSE CAP

The substrate design for the nose cap is taken to be that given on Atlantic Research Corporation (ARC) drawing No. ND 5640-029.\*

1. Using the drawing as a reference, lay out extended tangential lines in a smooth, gradual transition from the substrate surface to the desired inlet and exit configurations. To minimize the possibility of boundary layer separation, the flow should be accelerated along the flow annulus and, therefore, it is preferable that the flow direction be from the larger to the smaller diameter of the nose cap.
2. Given the desired  $N_2$  flow velocity over the nose cap surface (from prior work) and assuming a desired separation between the graphite nose cap and the water-cooled centerbody, calculate the required process  $N_2$  flow.
3. The surface contour of the centerbody can be estimated using various approximate methods and can be checked with GENMIX code. The idea is to keep the flow area, normal to the coating surface, constant or slightly decreasing with distance from the inlet. An alternative method, using the hodograph technique, is outlined in Ref. 5. The surface contour of the centerbody can be determined from approximate relations given below. For the lower half of the graphite substrate, construct a line normal to the surface. The radius of the centerbody,  $R_{cb}$ , lies on the normal at

$$R_{cb} = \sqrt{R_s^2 - \frac{A \cos \theta}{\pi}},$$

where  $R_s$  is the radius at the point the normal intersects the substrate,  $\theta$  is the angle between  $R_s$  and the normal, and  $A$  is the flow area as described in item 2. For the upper half of the substrate, construct a line normal to the surface. The centerbody intersects the normal at a distance,  $\ell$ , from the substrate,

$$\ell = A/2\pi R_s \cos(\theta - 90).$$

It may be desirable to decrease the area,  $A$ , as one moves around the nose cap to increase the flow velocity.

4. Referring to Fig. A-1, the configuration is now defined. For compatibility with the fluid mechanics code, the gas must be injected into the precooler at a known, relatively uniform velocity. This could probably be achieved by

\*Private communication (Proprietary) from ARC.

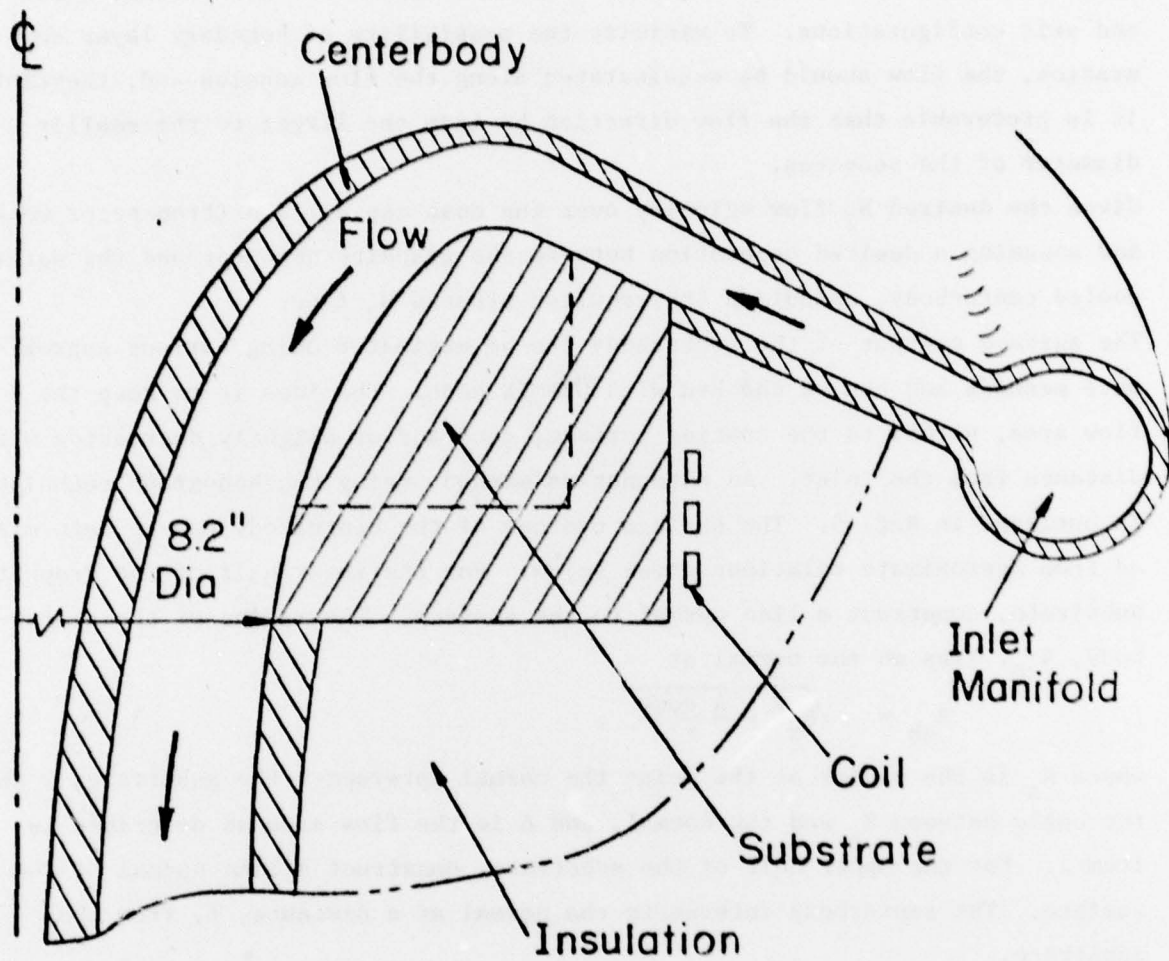


Fig. A-1. Coating chamber configuration.

injecting through a thin porous plate from the gas distribution manifold.

5. From the deposition kinetics, assume that a constant temperature (1920 K) around the nose cap surface is desired. Using the AYER code, vary the shape of the outside of the graphite substrate, placement of the induction coil, the emissivity of the water-cooled centerbody, and configuration of thermal insulation to obtain this constant surface temperature.

An example of this calculation is given in Figs. A-2 and A-3. The substrate sketched in Fig. A-1 is heated by the coil and loses heat by thermal radiation to the centerbody and by conduction through the surrounding insulation (the coating gas flow was omitted from the model). When the centerbody's thermal emissivity is uniform (at 0.31, typical of oxidized copper) the substrate surface temperature is nonuniform. Figure A-2 shows a "cold" spot (<1700 K) near the minimum radius section of the nose cap. When the thermal emissivity of the centerbody is varied\* along the contour, a much more uniform surface temperature can be achieved (Fig. A-2). The emissivity was reduced from 0.31 to 0.09 directly opposite the smallest radius section, and from 0.31 to 0.19 for a short distance on either side of this section.

6. With the calculated wall temperatures, physical shape of flow passage, and process nitrogen flow rate, the fluid mechanics code is used to answer the following questions.
  - Is the surface temperature affected by flow rate?
  - Does separation occur at the top of the nose cap?
  - What will the projected deposition rate be along the nose cap surface?
  - What is the best temperature profile between the start of the nose cap and the end of the precooler?
  - Will additional MTS and  $\text{CH}_4$  have to be injected into the gas stream to assure constant deposition around the substrate surface?
7. Rework the thermal model to meet the flow and deposition requirements.
8. Recalculate flow and deposition parameters to verify that changes in the thermal model are adequate.
9. From parametric changes in flow and thermal models, determine sensitivity of coating process to controlled parameters.
10. Send model layout with process control specifications to instrumentation en-

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\* Various techniques to achieve this include coatings, scoring (roughness), polishing, and build-up in sections of different materials.

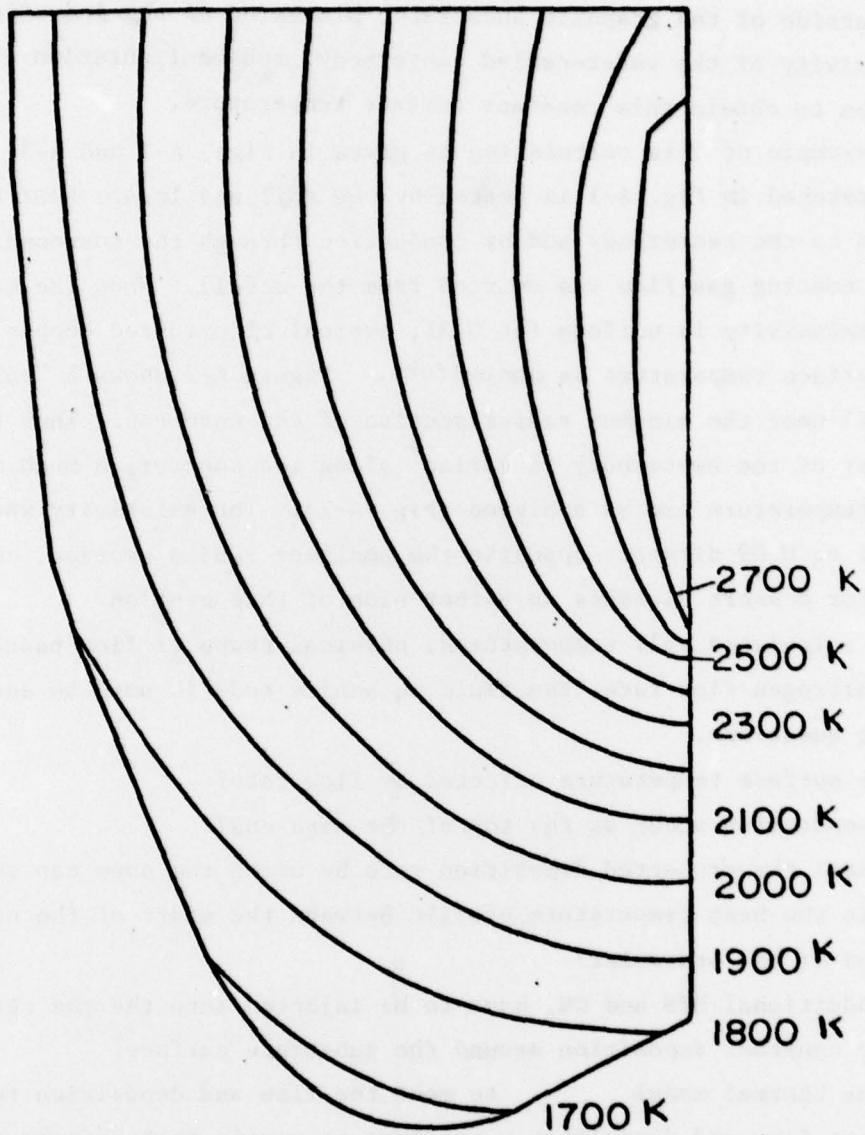


Fig. A-2. Isotherms with uniform centerbody emissivity.

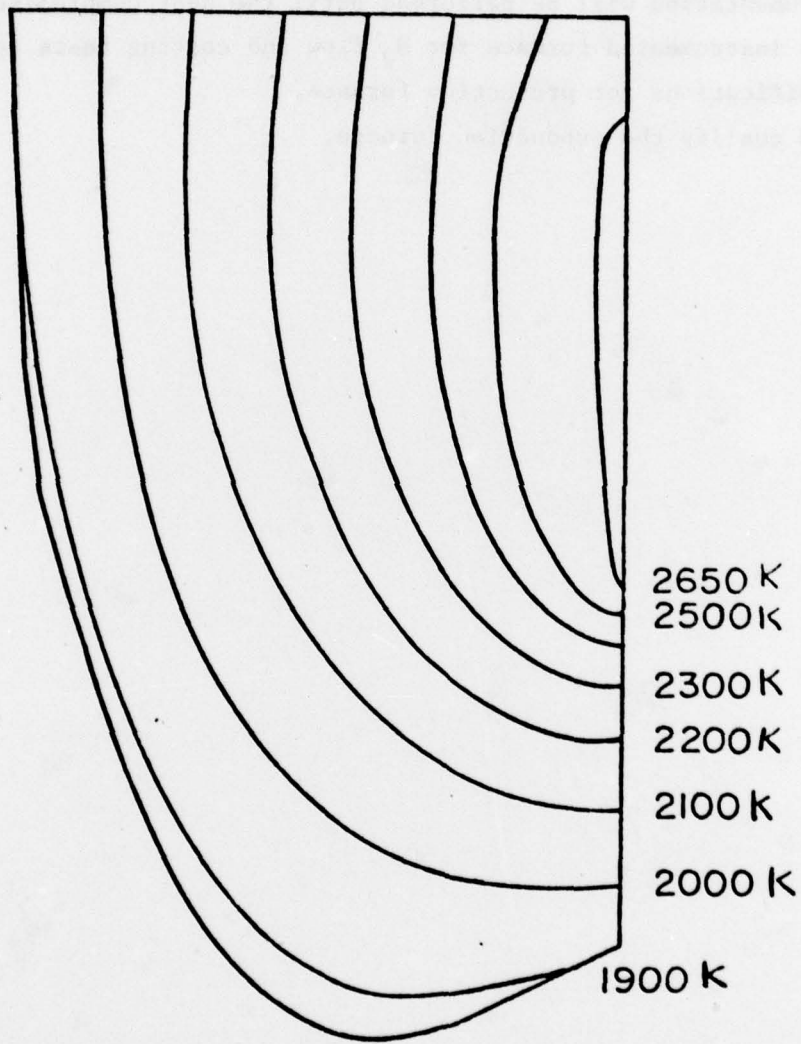


Fig. A-3. Isotherms with variable centerbody emissivity.

gineering to determine where instrumentation penetrations are required, as well as external requirements.

11. Send layout to design engineering to get detailed layout for implementing in the actual furnace configuration.
12. Refine the thermal/flow/kinetics model, based on engineering drawings, to verify that the furnace will work. An iteration with design engineering and instrumentation will be performed until the best compromise is reached.
13. Build the instrumented furnace for  $N_2$  flow and coating tests to specify final modifications for production furnace.
14. Build and qualify the production furnace.

5000K  
4000K  
3000K  
2500K  
2000K  
1500K

Fig. 2-1. Isotherms with vertical centerline.