HIGH TEMPERATURE ELECTRICALLY CONDUCTING CERAMIC HEATING ELEMENT AND CONTROL SYSTEM

By Carl R. Halbach and Russell J. Page

JUNE 1975

Prepared under Contract No. NAS8-29769 by

ADVANCED RESEARCH AND TECHNOLOGY (ARTCOR)

Costa Mesa, California 92626

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

The technology of an old art (circa 1900 by Dr. Nernst) has been advanced relative to the use of stabilized zirconia as an electrical conductor. Improvements have been made in both electrode technology and ceramic conductor quality to increase significantly the lifetime and thermal cycling capability of electrically conducting ceramic heater elements. In addition, these elements have been operated in vacuum, inert and reducing environments as well as oxidizing atmospheres adding to the versatility of the conducting ceramic as an ohmic heater.

Using stabilized zirconia conducting ceramic heater elements, a furnace has been fabricated and demonstrated to have excellent thermal response and cycling capability. The furnace was used to melt platinum-20% rhodium alloy (melting point 1904°C) with an isothermal ceramic heating element having a nominal working cavity size of 2.5 cm diameter by 10.0 cm long. The furnace was operated to 1940°C with the isothermal ceramic heating element.

The same furnace structure was fitted with a pair of main heater elements to provide axial gradient temperature control over a working cavity length of 17.8 cm. Inside diameter of the heater was 1.77 cm for an overall L/D of 10 to 1. A shorter 7.6 cm long secondary heater element using a platinum-10% rhodium alloy winding was used. The primary heater element was of stabilized zirconia ceramic and was placed on top of the secondary heater. This combination was used to demonstrate typical axial gradients with maximum temperatures of from 2050 to 2100°C during a run in which high purity alumina was melted. An axial temperature gradient of 250°C per cm was demonstrated based on heater element wall temperatures.

Transient response of the low mass ceramic heaters was found to be excellent. Heat up response depends on the limits set for the applied power. In cool down response tests, for example, in which the isothermal heater was commanded to a 100°C lower setting, 99% response occurred in 25 seconds.

Faster responses were noted for the smaller axial gradient heater. Using an industrial type solid state power controller, with both current feedback and temperature feedback modes, thermocouple sensed cavity temperature error was found to be less than ±1°C.

While the conducting ceramic heater must be operated with alternating current, it can be readily adapted to DC power sources. A typical power control adaptation is discussed relative to a DC powered conducting ceramic furnace for a sounding rocket flight application.

INTRODUCTION

Past manned space flight experiments have verified that there is a great potential in processing materials in space. Near-term opportunities to further demonstrate feasibility are contingent on experimental investigations being considered for sounding rocket flights and the Apollo-Soyuz mission. The next major opportunity to process materials in manned space flight await the 1980's with the Shuttle/Spacelab. The forthcoming NASA Sounding Rocket Program allows the earliest opportunity to explore the low acceleration environment available in space to achieve, in automated facilities, promising processing conditions for periods of several minutes that cannot be obtained on earth. At this time, however, not only are the requirements for the experiments not known, but even specific principle investigators themselves are not identified. The needs of investigators are known in general to involve the solidification of pure materials, alloys, composites, glasses and electronic materials under certain gaseous environments with certain needs for access, positioning and manipulation of the sample, scheduling, etc. This broad range of potential requirements dictates that the facility be general-purpose and adapt easily to specific needs. In general, the requirements of the facility are that it must:

(1) Automatically take the sample quickly through the desired process phases such as heat-up, establishing a thermal gradient, temperature soak and cool down, etc.;

(2) Be efficient with regard to power required so as to either maximize the sample size or to minimize the heat loading in the balance of the system;

(3) Perform in a rugged environment;

(4) Permit accurate data gathering automatically;

(5) Be light in weight and compact, fitting within the envelope constraints of the sounding rocket;

(6) Be cost effective. That is, be low in initial and refurbishment cost relative to the reliability needed;

(7) Have a long life so that it may be reused and readapted quickly for subsequent experimenters;

(8) Be modular in construction so that various experiment formats can be interchangeably pursued such as isothermal to gradient or special, etc.;

(9) Be adaptable to incorporation of special features of sample positioning, devices, access for mechanical sample manipulation, optical window systems, cooling, etc.

NASA(ref. 1) has developed a highly versatile - early availability furnace with temperature capability to 1150°C. Used on Skylab (and planned for the Apollo-Soyuz mission) the M518 Multipurpose Furnace has proved it advantageous to utilize the zero gravity of space flight to advance the art of processing certain materials. Logically, the operating temperature limit needs to be extended to accommodate higher melting temperature glass and crystal growing materials experiments and processing.

There are many basic types of furnaces and numerous variations of each which might at first be considered for this application. It is clear that in the case of the sounding rocket, that under practical conditions the electric furnace, operating from batteries, provides the greatest control of temperature and thus provides an opportunity to do materials sciences research in greater depth. Electric resistance furnaces provide relatively high efficiency of heating as well as control and cleanliness.

In manufacturing processes where oxidizing or, for that matter, reducing atmospheres and high temperature are required, the furnace materials exposed to the working cavity must chemically and physically withstand these conditions. Where careful temperature distribution control and freedom from charge contamination are required, a resistance furnace using a heating element of conducting ceramic in the cavity is particularly attractive. The muffle furnace (heating element in a protective external inert atmosphere chamber) has been used to satisfy those requirements to some extent. With aluminum oxide muffle material, the long lifetime temperature is limited to about 1800°C and there are no other practical muffle materials with higher temperature capability. There is possibly beryllia whose use is limited to a completely dry atmosphere. Zirconia and thoria have often been suggested as muffles but they are ionic conductors. Oxygen would pass through the walls by ionic diffusion and attack the heating elements. While hydrogen can be introduced on the heater element side to consume the oxygen, the subsequently occurring gradient in stoichiometry through the ceramic wall becomes a failure mode. Short-circuiting of the heater element would occur should they come in contact with the ceramic muffle.

The highest temperature capability requirement is that of the heater resistance elements. Table I summarizes the state-of-the-art of electric heater materials for resistance furnaces, comparing the more conventional materials with zirconia and thoria (used as conductors in this case).

TABLE I.

COMPARISON OF HEATER ELEMENTS
FOR RESISTANCE FURNACES

Material	Temperature °C	
	Working	Melting
Thoria	2500	3265
Stabilized Zirconia	2200	2725
Iridium	2080	2454
Platinum-Rhodium	1720	1902
Molybdenum Discilicide	1700	*
Silicon Carbide	1710	*
Kanthal A-1	1380	1512

*Silicon dioxide (cubic) melts at 1712°C

Industrial furnaces constructed in the last decade have used heater elements of materials molybdenum discilicide, silicon carbide or (Fe, Cr, Al, Co) alloys for oxidizing service. The first two of these high temperature electrical conductors are suitable for service at high temperature in air. These form a protective glassy-phase coating of primarily silicon dioxide which softens at about 1625°C. While these materials have been used at temperatures as high as 1725°C as air heater elements, they are more realistically rated from 1525 to 1625°C for long lifetime and clean air. The Kanthal A-1, of lower temperature capability, is dependent also on forming a protective oxide coating. Life is dependent on the performance of the coating and its removal due to various causes (i.e., over-temperature melting and sluff-off, reduction, etc.). Small furnaces for scientific or medical purposes have been constructed using platinum-rhodium elements. Crucibles have been constructed of iridium, but not without some chemical interaction problems. While iridium has a high temperature capability in air, it does have a severe oxidation rate problem and, hence, a short life. The dependence on the noble metals has been expensive in time and money to the scientific community.

Practical heaters, to date, have been limited to 1700°C in an oxidizing environment. It is believed that a whole class of manufacturing techniques in oxidizing environments would be denied above this temperature - where fine temperature control spatially and time-wise is required - if it were not for the conducting ceramic element.

The ceramic element furnace utilizes an electric resistance heater element made of stabilized zirconium oxide ceramic. This technology was first demonstrated in the late nineteenth century in the form of a light emitting device called a Nernst glower. These devices are still being used today, predominately as infrared sources for IR spectrophotometers. In addition to stabilized zirconium oxide, thorium oxide is also used for these electric resistance devices. Both are capable of operation in oxidizing environments to temperatures of 2200°C and higher.

Several investigators (ref. 2, 3, 4) have used zirconia and thoria for heating elements in oxidizing environment furnaces. Improved high temperature insulation (Zircar, a Union Carbide tradename for zirconia fiber insulation) has made it possible to fabricate a relatively compact - light-weight high temperature furnace with excellent thermal performance. Energy requirements are low because of the low thermal conductivity of Zircar and transient response is fast because of the low capacitance of the Zircar insulation.

The objectives of this study program include improvement in the conducting ceramic quality for greater lifetime and thermal shock resistance, improvement of the electrode-to-ceramic design for greater temperature capability, and the design of a furnace to demonstrate high temperature - oxidizing environment - long lifetime feasibility of the conducting ceramic heater element concept. A further objective was delivery of a zirconia conductor furnace to NASA/MSFC. This furnace, designated the Model 40 zirconia conductor furnace, has heater element interchange capability in order that it can be used with isothermal as well as axial gradient heater elements.

LIST OF SYMBOLS

a	thermal diffusivity, m^2/s
A	area, m^2
E	elastic modulus, N/m^2
f	fracture parameter, defined by eqva. (6)
G	evaporation rate, kg/m^2-s
k	thermal conductivity, w/m-K
M	molecular weight
P_v	vapor pressure, N/m^2
q	heat flux rate, W
R	gas constant, J/kg-K
R_1	thermal stress resistance parameter, K
R_2	thermal stress resistance parameter, W/m
R_3	thermal stress resistance parameter, m^2-K/s
S	geometry parameter
T	temperature, K
X	thickness, m
α	coefficient of linear thermal expansion
$\dot{\delta}$	surface recession rate, m/s
ε_n	normal emissivity
μ	Poisson's ratio
ρ	density, kg/m^3
σ_f	fracture stress, N/m^2
ϕ_f	surface temperature change rate, K/s

CERAMIC ELEMENT FURNACE CONCEPT

The concept of the ceramic element furnace using stabilized zirconia centers around the electrical characteristics of the oxide ceramic heater element itself. Figure 1 presents heater element resistance data obtained on the Model 40 furnace. When cold, the heater element has a resistance measured in megohms and acts as an excellent insulator of electrical current flow. With increasing temperature, resistance is seen to decrease rapidly. During this initial heating phase called "preheating" a separate preheater furnace element is required. Once the ceramic heater element temperature exceeds about 600°C, resistance is reduced to 1000 ohms or less and the ceramic heater element will begin to conduct sufficient current to heat itself further. From a temperature of about 800°C, the ceramic heater element is self-sustaining and the preheater element can be turned off.

Figure 2 indicates a simplified arrangement of a preheater element and a ceramic heater element within an insulation system with a cavity space for conducting high temperature experiments. If the preheater were located in the same cavity as the ceramic heater, then cavity temperature would be limited to the capability of the preheater. The preheater is separated from the experiment cavity by an insulation barrier and yet is surrounded by insulation to minimize its power consumption. Referring to the temperature curves in figure 2, the dotted line indicates the temperature profile that would exist with only the preheater turned on and set for 1000°C.

By proper sizing of the insulation system, an experiment cavity temperature of say 800°C would occur with preheating, sufficient to allow ignition of the main heater. The solid curve indicates the temperature profile occurring with the preheater off and the ceramic heater holding maximum temperature. The insulation system is sized under this condition so that the preheater is not overheated by the high temperature within the experiment cavity.

Separate controls are provided for the preheater and the ceramic heater. While the preheater can be operated from either AC or DC electrical power, it is necessary to use AC power for the ceramic heater. This is the result of the oxide ceramic being a solid electrolyte and conducting electricity by the motion of negatively-charged oxygen ions O^{-2} through the oxide rather than electrons.

Noble metal electrodes interface the oxide ceramic on each end. With an electrical current flowing, electrons move in the metal wires to the electrodes. In the case of a direct current, electrons flow toward one electrode, the cathode. Four electrons combine with each oxygen molecule in the surrounding atmosphere causing the oxygen to enter the ceramic at the cathode and proceed through the ceramic as two individual oxygen ions. The reverse occurs at the opposite electrode (anode) where the two ions form an oxygen molecule as it passes back into the surrounding atmosphere while the released electrons proceed through the metallic circuit to complete the circuit.

Under the influence of direct current, a polarization effect occurs in which oxygen ions within the oxide ceramic are depleted near the cathode and

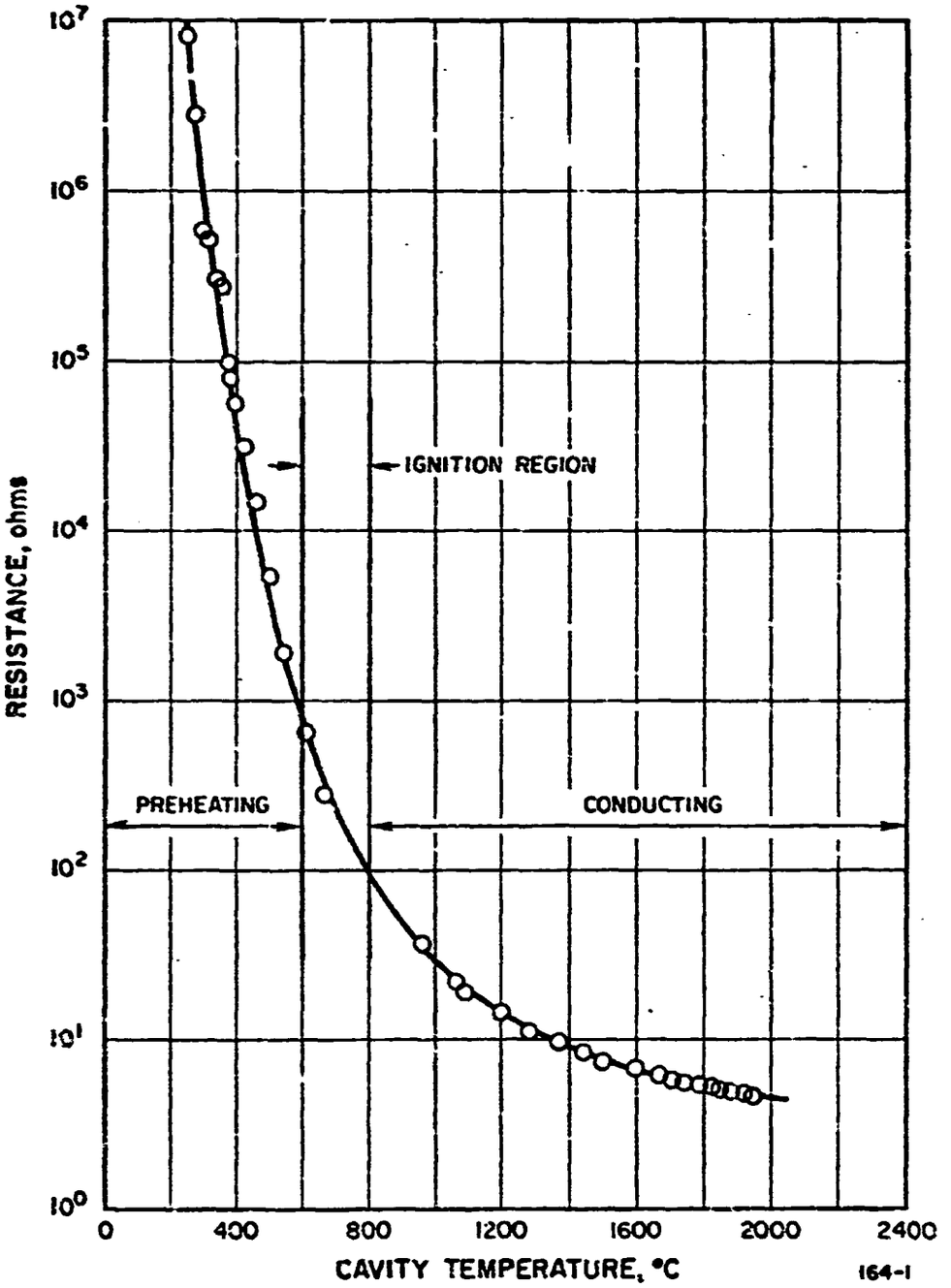

Figure 1.- Model 40 zirconia conductor furnace ceramic heater element resistance.

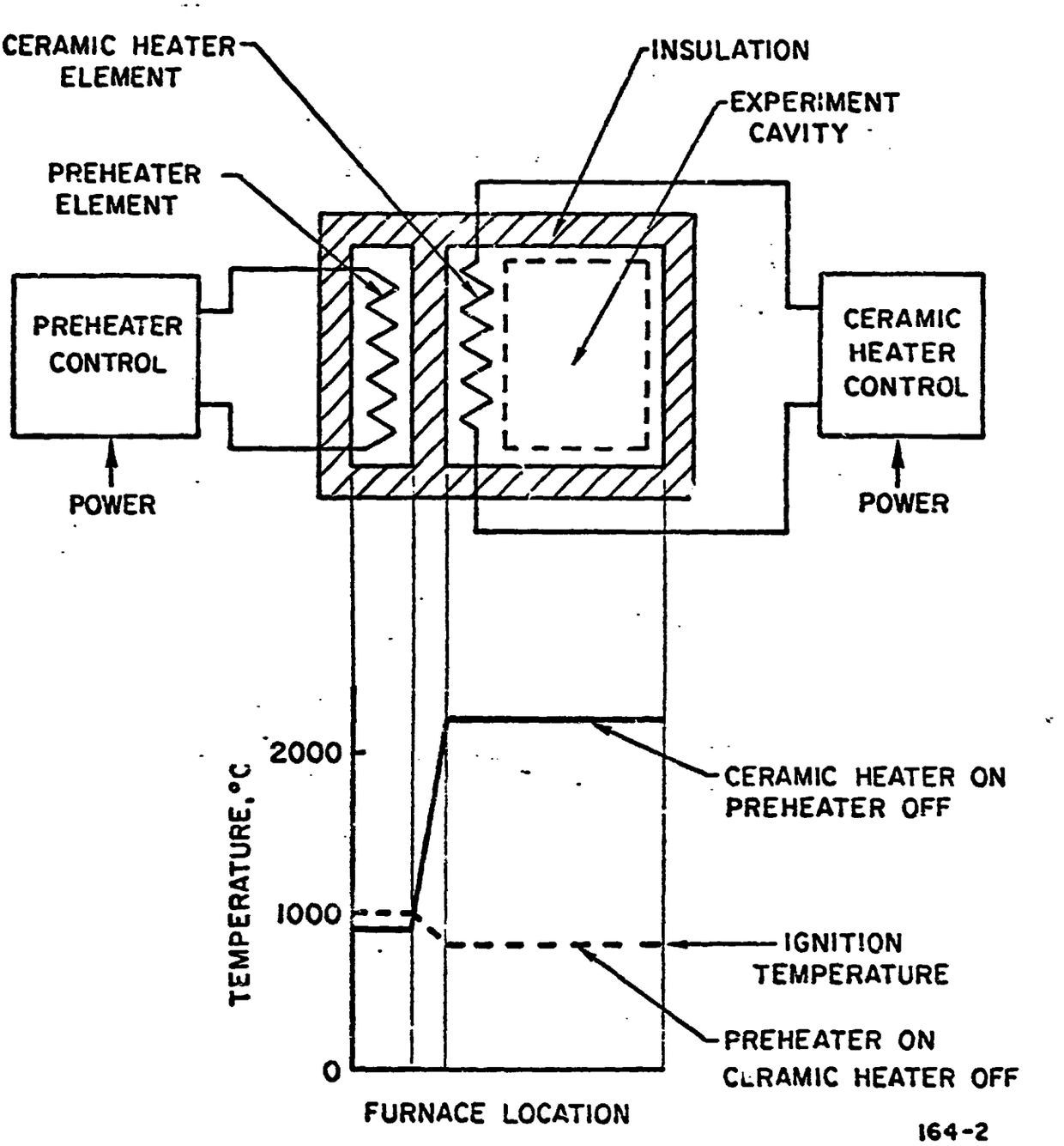

Figure 2.- Ceramic Element Furnace Concept.

accumulate near the anode. This is undesirable and can effect the electrical performance of the heater inducing a temperature gradient along the heater element and possibly a time varying performance characteristic. Without a surrounding atmosphere containing oxygen molecules (as with a vacuum or reducing environment), the effect is more pronounced as the oxide ceramic is shifted in its stoichiometry (reduced toward the metal constituent).

Under the influence of alternating current, the oxygen molecules in the oxide ceramic merely dither back and forth as the cathode and anode are rapidly interchanging. With AC, the oxide ceramic has sufficient stability against reduction to be used even in vacuum and reducing type environments.

FURNACE USER REQUIREMENTS

A furnace user survey was directed to a broad spectrum of potential users to solicit current design requirements. Since the temperature capability of the ceramic furnace is potentially very high, it was expected that the responses to the survey would be large. From a total of 216 corporate research and university institutions solicited, a response of 12% was achieved. The depth of response to the rather detailed questionnaire varied. Some desired to communicate further as experiment requirements become better known. Others had no requirement for those capabilities now existing while some thoughtfully offered thorough and completely detailed information followed in several cases by technical visits. The anticipated principal beneficiary technologies were expected to be those of glass, crystal growth and ceramics.

The results of the survey (see Table II) are considered valuable for design requirements. A few particularly attractive experiments where the ceramic furnace was a unique fit are Surveys 3 and 4 - glass making and oxide crystal growing. Orbital flights would be required for the latter because of the time involved while the former would adapt to the earlier scheduled sounding rocket experiment opportunities.

Maximum process temperatures to 2400°C are indicated by the survey as a requirement. Atmosphere requirements are broad including vacuum, inert, oxidizing and reducing. It is generally considered that the availability of a very high temperature oxidizing environment furnace would be beneficial in a variety of experiments, especially those involving the high melting point oxide materials.

Spatial uniformity in the furnace cavity of ±20°C or less is desired with two investigators suggesting ±0.5 and ±1°C, respectively, at temperatures of 2000°C and greater. Sample size requirements range from the order of 1 centimeter for sounding rocket flights to the order of 10 centimeters for Spacelab flights. Furnace cavity sizes to 15 cm diameter by 20 cm long are indicated for the Spacelab flights.

PROPERTIES OF THE CONDUCTING CERAMIC OXIDES

The physical characteristics of the conducting ceramic heater material zirconia, ZrO_2, are tabulated in Table III. Characteristics of another con-

TABLE II

FURNACE USER SURVEY

User Company/Institution	Title	Principal Investigator/ person to contact	Objective	Experimental Approach	General Category
1. General Dynamics Convair Division P.O. Box 80847 San Diego, CA 92138	Preparation of superconducting alloys in low gravity	Dr. W.H. Steurer/ Dr. S. Kaye (714) 277-8900 x 1956, 1302 Mail: 646-00	Produce samples of superconducting alloys with improved transition temp.	Melting & solidification of sting. Supported cylindrical samples prepared by powder metallurgy techniques. Sphere on melting of 1.2 to 1.5 cm diameter.	Materials melting and solidification
2. Jet Propulsion Lab California Institute of Technology 4800 Oak Grove Dr. Pasadena, CA 91103	* Acoustic positioning of materials at high temperature in zero gravity	Dr. Taylor Wang (213) 354-4110	Containerless positioning of materials by acoustics in zero g at high temperatures.	Method utilizes pressure produced by an acoustical standing wave excited with an enclosure.	Levitation subsystem components development.
3. Rockwell Intern'l 12214 Lakewood Blvd. Downey, CA 90241	Production of unique oxide glasses.	Mr. Ralph A. Happe (213) 594-2486 NASA MSFC NAS8-28991 (R. Nichols)	Produce samples of useful size of glasses formed from various oxide candidates.	Heat candidates to 200°C above their melting points to eliminate nucleation sites in a containerless process (free suspension) quick cooling to below the glass forming temperature.	Glass making - materials melting & solidification
4. Univ. of So. Calif. Electronics Science Lab University Park LosAngeles,CA 90007	Floating zone melting of oxides	Prof. Wm.R. Wilcox (213) 746-6203 (not currently sponsored)	Grow oxide electronic materials free of crucible contamination which is a serious problem with Czochralski growth.	Gravitational forces limit physical size with floating zone melting. Grow larger crystals in floating zone. No levitation required.	Crystal growth - floating zone.
5. Ill. Inst. of Techn. 10 W. 35th Street Chicago, Ill 60616	Space processing of chalcogenide glasses	W.B. Crandell or D.C. Larson (312) 225-9630 x 4741 NASA MSFC NAS8-30627 (R. Nichols)	Demonstrate feasibility of space mfg. of improved chalcogenide glass sys.	Move sample in closed clean furnace system from 1000°C to R.T. in partial pr ure atmosphere of As, S, Te, etc.	Glass making - materials melting & solidification
6. TRW Systems Group One Space Park Redondo Beach, CA 90278	Study on processing of immiscible materials	J.L. Reger (213) 535-0372 NASA MSFC NAS8-28267 (I.C. Yates) (205) 453-3036	Study methods of mixing materials exhibiting gap in the liquid state and demonstrate their feasibility by experimentation.	(1) Straight thermal heating above consulute temp. into single phase regime (2) heat to two-phase liquid regime & acoustically mix (3) heat & electromagnetically mix; i.e., all require some thermal source.	Immiscible materials - materials melting & solidification
7. Intersonics Inc. 450 East Ohio Chicago, Ill 60611	* Acoustic positioning of materials at high temp. in zero G.	Roy Wymark (213) 337-2205 NASA MSFC (R. Berge)	Demonstrate feasibility of acoustic positioning in a high temp. gas in zero g.	Use intense sound waves to compensate for severe viscous dampening of the medium.	Levitation subsystem component development.
8. Bendix Aerospace 3300 Plymouth Rd. Ann Arbor, MI 48107	Automated space processing payloads	Walter Crosmer, Proj.Eng. (313) 665-7766 x 397 NASA MSFC NAS8-30741 (J. Poe (205) 453-3424)	Processing various mat'ls independent of astronaut participation.	Automated general purpose furnace.	Various crystal growth/ glass making.

* N.S. - Not specified
N.P. - Not possible

N.A. - Not applicable
N.K. - Unknown

* Titles are those chosen by surveyors. The data shown is from the principal investigators of the facilities.

TABLE II

Key Material Name/Formula	Melting Point, °C (Max. Process Temp.)	Density gms/cc	Atmosphere Background (pressure)	Volatile Components of Melt	Gas Flow Rates cc/sec	Other Materials in hot zone	Water Cooled Parts in Cavity	High Conductive Supports into Cavity
1.a)V-based alloys V, Si, Ga	1900°C (2100°C)	5.0	Ar	Al	5 (Ar)	ceramic stings	No	No
b)Nb-based alloys Nb, Al, Ge	2400°C (2400°C)	7.2		Ga	5 (Ar)			
2. General glass experiments to certain metals.	300° to 2000°C	(N.S.)	Inert, O_2, possibly reducing.	(N.K.)	Zero	No	(N.S.)	(N.K.)
3.a)$50La_2O_3$ - $50Nb_2O_5$	---	---	Air or O_2 or N_2	You name it	None unless part of later levitation scheme	Glass wire support prior to melting	No	No
b)$25La_2O_3$ - $75Nb_2O_5$	---	---						
c)$25La_2O_3$ - $75Ta_2O_5$	---	---						
d)$40La_2O_3$ - $40Al_2O_3$ - 20CaO	---	---						
e)$85Nb_2O_3$ - 15CaO	1540 (1740)	4.4						
f)$90Ta_2O_5$ - 10CaO	1875 (2075)	8.7						
g)$80Ga_2O_3$ - 20CaO	1700 (1900)	---						
h)Mullite ($80Al_2O_3$ - $20SiO_2$)	1920 (2120)	3.4						
i)La_2O_3	2210 (2410)	6.7						
4. Oxides in general	500 - 2200 (2200)	Var.	Oxidizing but not critical for most samples	A few oxides may be a little volatile but most will not.	Zero	None	No	Yes. Need to rotate one or both rod ends. (Penetration required)
5. a)Arsenic trisulfide As_2S_3	~300°C	3.43	Ar/or He + S + etc.	As, S	Zero	Possibly carbon or tungsten	Yes	Yes
b)Chalcogenide glass Si-As-Te-Sb	~600°C	3.5	(1 - 5 atm)	As, Te, Sb	Zero	"	Yes	Yes
6. Binary metal elements	≥2000°C	(N.S.)	Inert (1 atmos)	Mat'ls processed encapsulated hence element not exposed to components.	Zero	Capsule thermocouple and holder	No	No
7. Materials are general	300 to 2000°C	(N.S.)	Inert O_2 possibly reducing	(N.S.)	Zero	No	(N.S.)	No
8. Various crystal and glass materials. (No specific candidates proposed.)	1300 - 1850°C range considered.	Var.	Ar, O_2 (1 atmos) Vacuum	Various	Zero	Crucibles, (Pt, Al_2O_3) Thermocouples	No	No

TABLE II

Maximum Furnace Wall Temperature Req'd °C	Spacial Uniformity	Gradient	Trace Contaminants Undesireable in Material Process	Sample Size		Hot Zone Size		
				Sounding Rocket	Space/ Lab	Minimums Cited	Sounding Rocket	Space/ Lab
1. a) ≥2200°C b) ≥2500°C	± 20°C	Isothermal	(N.S.)	0.8 cm OD x 2.1 cm long or 1.03 cm^3	(N.S.)	3 cm ID x 8 cm long	3 cm ID x 8 cm long	4 cm ID x 12 cm long
2. ~2000°C	(N.S.)	Isothermal	(N.K.)	(N.S.)	(N.S.)	(N.S.)	10.2 x 11.4 x 12.7 cm	(N.S.)
3. b) ≥2200°C	±20°C	Isothermal or zone with transport mechanism	(N.S.)	1.2 cm spherical samples	10 cm spherical samples (goal)	(N.S.)	4 cm ID x 12 cm	15 cm ID x 20 cm
4. 2200°C	Known to (±10°C) but controlled time-wise (±1°C)	Gradient furnace required. (100°C/cm or more)	Usually not critical. For lasers, transition metals may be harmful.	(N.A.) see remarks	2.5 cm OD x 10 cm long rod	2.0 cm	(N.A. because of time of 24 hrs required)	3.5 cm x 2.5 cm high but not critical
5. a) 1000°C	±2°C	None	O_2, N_2, C, H_2 (Deleterious to infrared transmission)	1 cm x 1 cm x 0.2 cm		3.8 cm OD x 10.2 cm	3.8 cm OD x 10.2 cm	5.1 cm OD x 15.2 cm
b) 1000°C	±2°C	(zone furnace required)		"		"	"	"
6. ≥2000°C	±0.5°C	<0.1°C/cm	Containerless (N.A.)	3 mm ID x 0.5 cm long	(N.S.)	0.5 cm ID x 1 cm	0.5 cm ID x 1 cm long	3.0 cm x 10 cm long
7. ~2000°K	(N.S.)	Isothermal	(N.K.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)
8. 1900°C	±5°C	100°C/cm	Volatile components of melt contained in cartridge.	Not considered.	10 cm	5 in. ID (12.7 cm)	Not considered.	12.7 cm ID x 20.3 cm long

TABLE II

Special Requirements	Furnace Instrumentation Desired				Temperature Cycle (°C) (Time - Secs)	
	Temperature	Pressures	Partial Pressures	Other	Preheat (Gnd)	Melting
1. (1) Because of short time available, water quench required to get solidification (2) check valve maintaining ΔP = 0.5 atm. between cavity and payload ambient; pressure reducer from argon supply.	Infrared sensor looking at sample (best accuracy available).	Regulator between cavity and payload ambient.	None	Pressure regulator from argon.	550°C (0 - 70) <80°C (0 - 70)	600 - 1900 (70 - 180) 100 - 2400 (70 - 300)
2. Three (3) accoustic driver ports, 2.5 cm (diam); two (2) service ports specified 2.5 cm (diam). Wall surfaces should be smooth.	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)
3. Containerless process is essential. Measurement of temperature of a transparent body is difficult. Hence, wall temp. measurement important.	(None except temperature of ceramic walls)	(N.S.)	(N.S.)	(N.S.)	h) 1325°C (0 - 70)	h) 1325 - 1920 (70 - 300)
4. Need heater surrounding molten zone. May also need heat shields to lower temperature gradients in solid. Will need to independently move at least rod. Rotation of rods desireable but not essential.	Yes	Not critical but not vacuum.	P_{O_2}, inert gases	(N.S.)	Amb. to melting point (0 - 1 hr)	(N.A.)
5. 1) Unloading capability required in glove box conditions because of toxic gases. 2) See levitation. 3) Min. time on condition is 5 min.	Thermocouples 1000°C ±20°C (continuous) "	1 - 2 atm. (continuous) "	P_{O_2} <10^{-25} S Te (continuous)	Optical window, temperature and pressure recording	160 - 300° (0 - 60) 250 - 600° (0 - 60)	160 - 300° (0 - 60) 250 - 600° (0 - 60)
6. (N.S.)	±5°C	(N.A.)	(N.A.)	Acceleration and temperature versus time.	1000°C (Pre-launch soak 2 hrs)	1000°C at launch
7. (N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)
8. Single end access for cartridges. Optical viewing port on axis.	±5°C	10%	(N.S.)	(N.S.)	Various	Various

TABLE II

Temperature Cycle (°C) (con't) (Time - Secs)					Levitation Technique	Visibility	Remarks
Liquid Phase	Heater Off	Solidif.	Cooling	Minimum Time			
1. 1900 - 2000 (180 - 350) 2400 - 2100 (300 - 420)	2100 (300) 2400 (350)	2000 (350) 2100 (420)	Balance	400 sec.	Not depended upon initially.	Only for infrared sensor.	Compatibility of aluminum with ZrO_2 important. Al very reactive. Al or Ga vapor may leave fine deposit on cavity surfaces.
2. (N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	Acoustic	One viewport specified.	The power level to run furnace and acoustic drivers must be matched to specific sounding rocket capacity. Figures given are desired without regard to this factor.
3.					Free-floating (if g level can be kept below 10^{-4} g but levitation would be used if available.	(N.S.)	(a) Rapid cooling to below glass forming temperature = $2/3T_{MP}$ of glass and may mean removal from furnace criteria. (b) Fluxes may be added to lower melting point. (c) 200°C superheat over melting required to eliminate nucleation sites. (d) This case carefully calculated as representative of rest (see text). (e) Larger furnaces it is believed will require transport mechanism.
h) (1920 - 2120) (300 - 318)	h) 2120 (318)	h) 1189 (332)	h) <1189 (balance)	(N.S.)			
4. Crystal growth phase typical (1 - 25th/hr)	Approx. 25 hr point	(N.A.)	M.P. to Amb. (25-30th hr)	24 hrs	None required Floating zone tech. at zero g adequate.	Need to review zone optically.	The surface tension will be sufficient at zero g to eliminate need for levitation. The time requirement prevents consideration for sounding rocket.
5. a) 300° (60 - 300°)	300° (300)	300° - R.T. (300 - 420)		5 min. on cond.	Must have positioning head operating inside furnace environment.	Optical quality viewing ports for movie camera.	Containerless melting essential.
b) 600° (60 - 300°)	600° (300)	600° - R.T. (300 - 420)		"			
6. 1000° (0 - 180)	1000 - monotectic temp. 180	Monotectic temperature 360		360	None required	No	
7. (N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	(N.S.)	
8. Various	Various	Various	Various	32 hrs typical	(N.S.)	(N.S.)	Study results directed towards Shuttle flights.

ducting ceramic material thoria, ThO_2, and the noble metal platinum are also shown for comparative reference.

Thoria has the highest melting point of any oxide and is, for that reason, of considerable interest for high temperature applications. Its melting point is listed in the range of 3200-3600K by various sources; the most probable figure is 3540K according to reference 5. It is very inert in its chemical properties. Thoria is stable under most conditions. It requires no phase stabilizers and like stabilized zirconia, is an electrical conductor at elevated temperatures. Thoria does have some disadvantages, however. Its creep strength is less than one third that of zirconia. This becomes a critical design factor relative to flight hardware. Also its resistivity is appreciably higher than zirconia. As a result, the ignition temperature is excessively high complicating the design of the preheater. Because of these factors, thoria was rejected as the heater element material for this project.

Zirconia was selected for the main heater element material for both the isothermal and the axial gradient furnace systems because its better mechanical properties give the main heater element increased strength and improved reliability.

The discussion which follows relates to those properties specifically involved in the design of a high temperature furnace utilizing conducting ceramics for electric resistance heater elements. For more detailed properties information, references 5 through 8 are recommended.

In pure ZrO_2 a reversible monoclinic-to-tetragonal transformation at atmospheric pressure occurs at about 1150°C. The monoclinic-to-tetragonal inversion is accompanied by a 3.2 percent length reduction on heating and an associated length increase (at about 1000°C) on cooling. Rapid cooling expansion occurs within a 10°C temperature interval and this would fragment a part made of pure ZrO_2. To permit its use in solid parts at high temperature, ZrO_2 must be stabilized by adding cubic oxides with cations of similar radius to Zr^{4+}. Such mixtures convert ZrO_2 irreversibly to the cubic form when heated. Many stabilizing oxides are available (ref. 7). CaO-stabilized and Y_2O_3-stabilized forms are quite common and will be discussed as examples.

Zirconia can be fully stabilized by the addition (typically) of 5 weight percent (10.4 mole percent) CaO or of 15 weight percent (8.8 mole percent) Y_2O_3. Purity has an important effect on some of the thermophysical properties of zirconia. There are many impurities which can be found accompanying zirconia in its natural state or unintentionally added in processing in addition to those deliberately added for stabilizing crystalline structure, enhancing electrical conductivity, etc. For instance, comparing the two important stabilizers Y_2O_3 and CaO on the basis of full stabilization, zirconia stabilized with the former is more refractory than the latter, having a melting point approximately 100°C higher. Of the various impurities, Al_2O_3 has a strong influence on electrical conductivity, but acts as a poison in percentages around 2%, an undesirable feature in conducting ceramics.

Figure 3 compares the electrical resistivity of typical high temperature heater element materials. There are two distinct groups of curves, divided

TABLE III

PHYSICAL, THERMAL AND MECHANICAL
PROPERTIES OF HIGH TEMPERATURE MATERIALS

Property	Thoria ThO_2 (For reference)	Zirconia ZrO_2	Platinum (For reference)
Additives	None	10 mole % Y_2O_3	None
Melting point, K	3540	2988	2045
Specific gravity	9.86	5.60	21.45
Specific heat, J/kg K			
@ 288K	235	460	133
1200K	304	670	158
2000K	331	858	180
Thermal conductivity W/m K			
373K	11.3	1.76	70.3
1200K	3.0	1.88	90.4
1600K	2.5	2.00	108.8
2000K	2.4	-	-
Thermal diffusivity, m^2/s			
1200K	9.8×10^{-7}	4.9×10^{-7}	2.6×10^{-5}
2000K	7.0×10^{-7}	-	-
Thermal linear expansion, % (298-1800K)	1.54	1.42	1.68
Electrical resistivity, ρ, Ω-m			
298K	-	-	1.05×10^{-7}
1200K	4 x 10	2.5×10^{-1}	3.95×10^{-7}
1600K	1	3×10^{-2}	4.95×10^{-7}
2000K	4×10^{-2}	9×10^{-3}	-
Total normal emissivity, ϵ_n			
1200K	0.29	0.4	0.12
1600K	0.27	0.4	0.17
2000K	0.30 - 0.55	0.5	-
Modulus of elasticity, $KN/m^2 \times 10^{-7}$			
298K	14.6	(15.5)*	17.1
1200K	12.4	(13.2)*	-
Estimated working stress (creep) 10,000 hrs @ 1800K, kN/m^2	300	1000	Nil
Sublimation recession at 1800K, kg/m^2s	1.7×10^{-8}	10^{-8}	5×10^{-7}

* 8.8 mole %

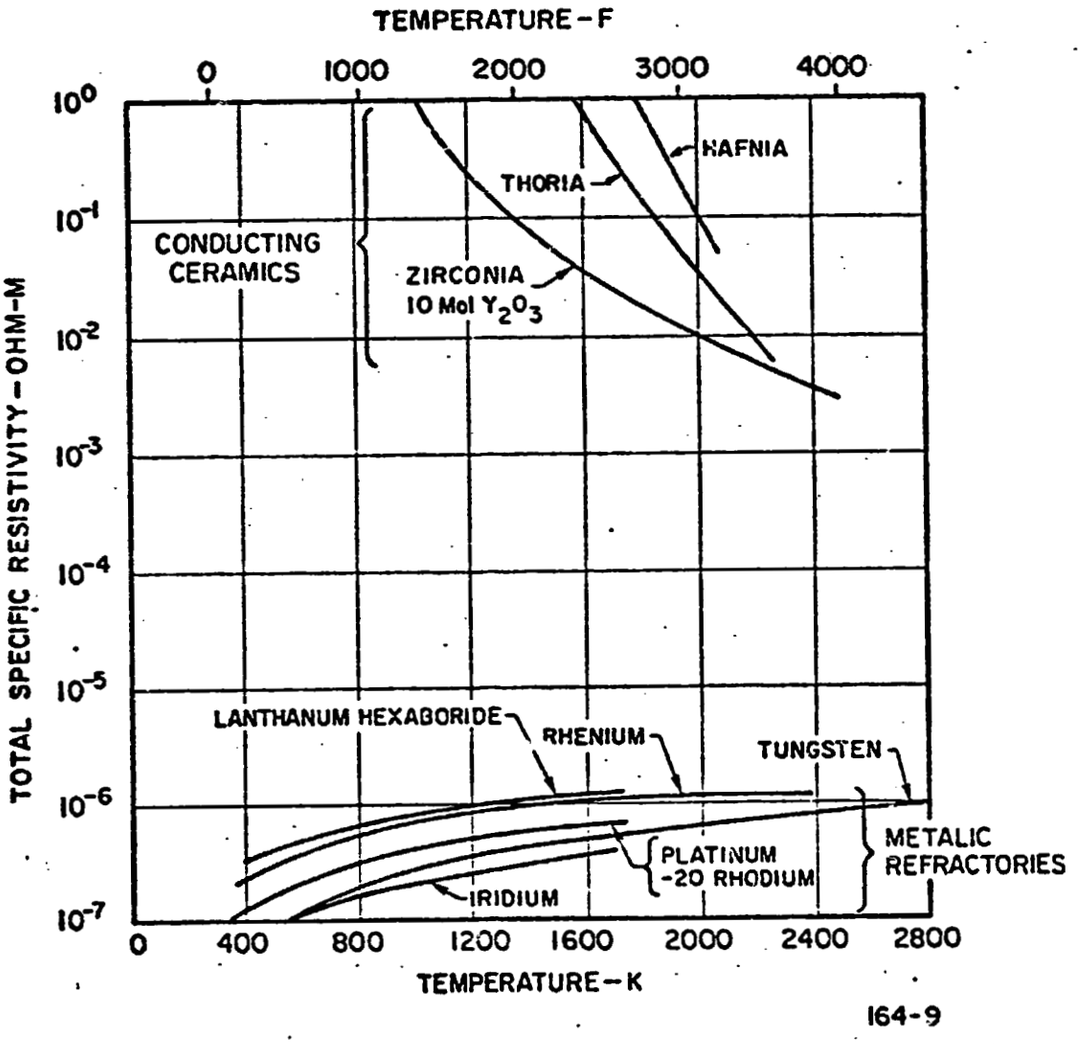

Figure 3.- Comparison of electric resistance of metallic and ceramic refractories.

essentially into the metals and the conducting ceramics. Rhenium is shown for reference only; it is not oxidation resistant. As a practical matter, zirconia and thoria do not conduct electrically below their ignition temperatures - 1000K for zirconia and 1600K for thoria. At high temperatures they have orders of magnitude higher resistance than the metals and hence are low current devices. This is a marked advantage in spacecraft design where wire size (weight) is thus smaller and I^2R losses are less. Note, however, that the resistance characteristics of these ceramics are negative with respect to increasing temperature. The hotter they are, the more they can conduct under a constant voltage. Either a series resistor (ballast) must be used to regulate the power consumption of a conducting ceramic heater element, an inefficient method, or a current limiting controller must be used.

The linear thermal expansion of several materials is shown in figure 4. The expansion characteristics of ZrO_2 and platinum are closely comparable. Therefore, platinum metal can be used with the stabilized zirconia ceramic heater element for forming an electrode interfacing the flow of electrons with oxygen ions.

The electrodes are designed to operate well below the element maximum temperature to prevent melting of the platinum. The temperature gradient from electrode to the maximum temperature zone in the ceramic element is high, resulting in a very short distance (e.g., a few millimeters) to the hot zone. Proper electrode design is a vital key to long life and is discussed in the furnace design section.

The process of sublimation, or evaporation, of material from a surface can be an important factor in determining the service life of a device operating at high temperature. The surface recession rate is greatest in a vacuum; it can be suppressed by several orders of magnitude by adding an inert atmosphere.

The surface recession rate due to sublimation is determined from:

$$\dot{\delta} = \frac{G}{\rho} \tag{1}$$

where $\dot{\delta}$ is the surface recession rate in m/s, G is the mass loss rate in kg/m^2s, and ρ is the density. G may be determined from experimental data, or may be found from the vapor pressure:

$$G = \frac{P_v}{\sqrt{2\pi RT/M}} \tag{2}$$

The recession rates for ZrO_2, ThO_2, Pt, Ir and Re in a vacuum are given in figure 5.

It is important to note that the sublimation rates are unacceptable for long life for all of these materials under vacuum conditions in the temperature range of interest. The suppression of sublimation by an inerting atmosphere has long been used. Langmuir used this fact in the invention and development of the gas filled tungsten-filament lamp. To ZrO_2, an oxidizing atmosphere is such an inert suppressant.

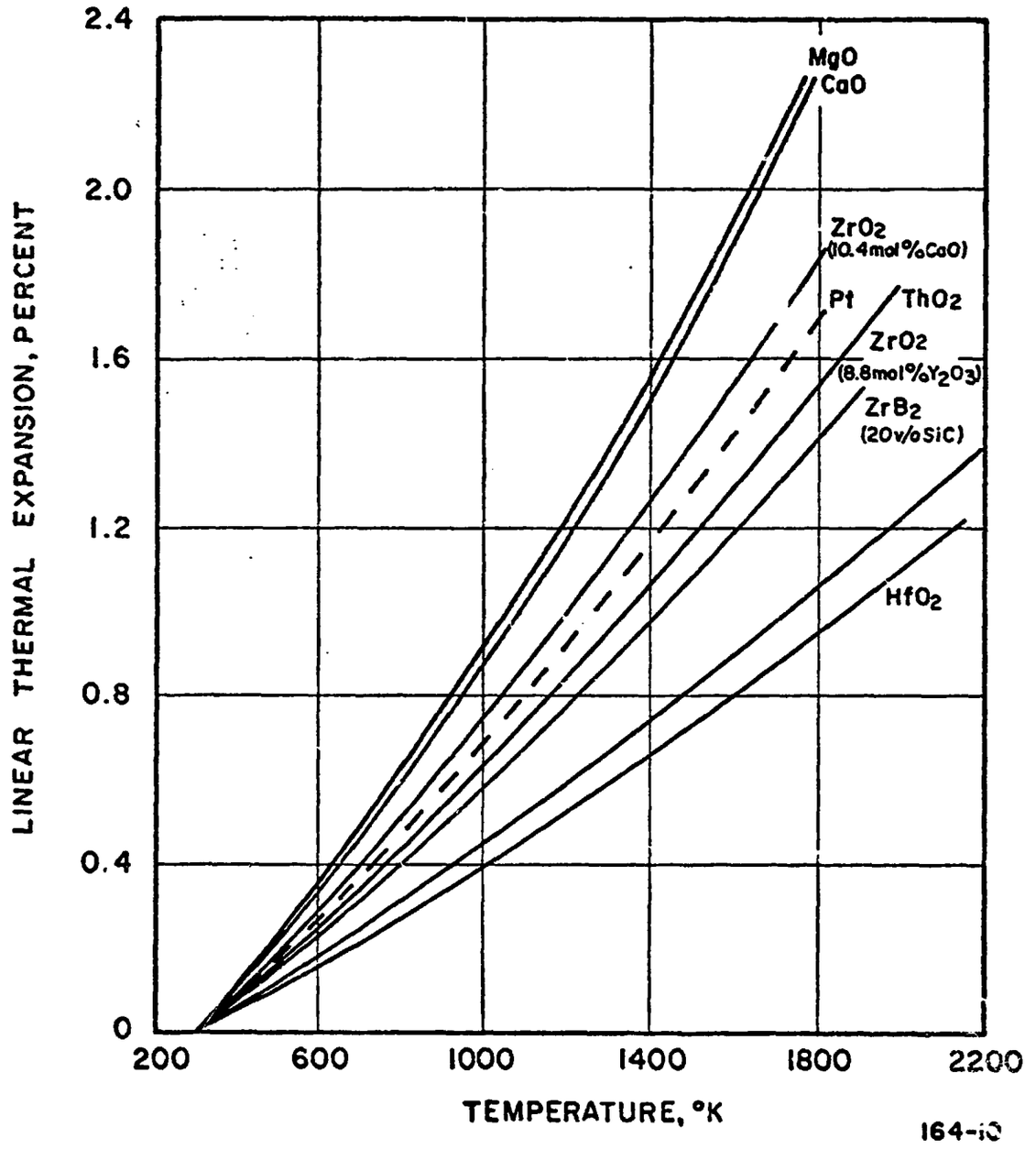

Figure 4.- Linear thermal expansion of zirconia compared with platinum.

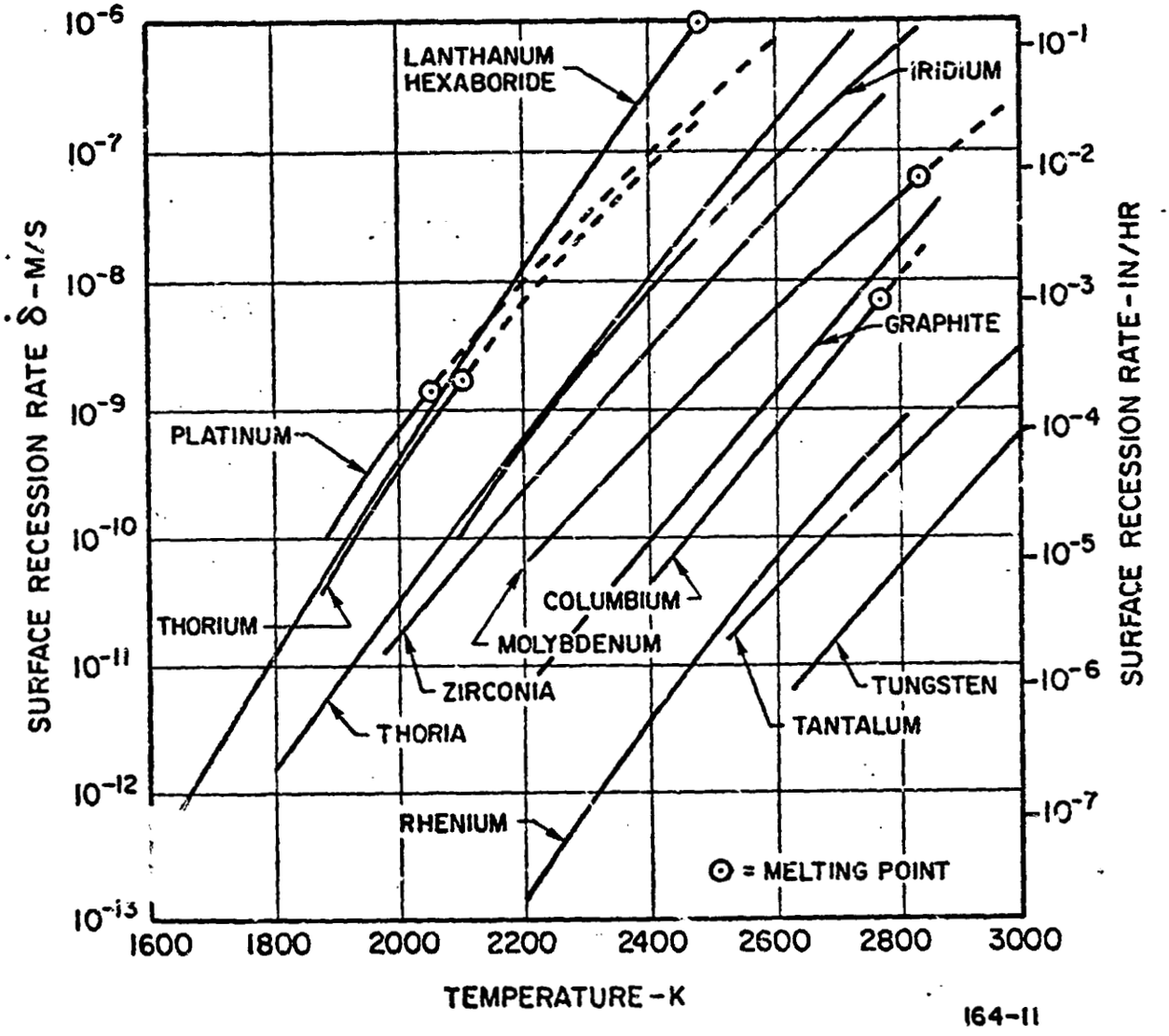

Figure 5.- Sublimation of zirconia compared to other materials in a vacuum.

Prolonged high temperature tests have indicated that the surface recession rate for stabilized zirconia heater element material is less than 10^{-13} meters per second at 2200K with oxidizing and inert gas atmospheres. Sublimation of the ceramic heater element is not considered a problem.

Creep, the deformation of a material with time under compressive or tensile loading due to pressure or restrained differential thermal expansion, is an important consideration in the design of electrothermal devices that must have long service lives with many parts operating at high temperature. Creep can cause two effects which cannot be compensated for and must be accommodated in the design. First, the parts may change shape to the extent that surfaces of a different electrical potential may touch causing short-circuiting. Second, they may creep to failure. The design must be suitable to withstand these effects during the furnace lifetime.

An extensive survey of available creep data for ZrO_2 and ThO_2 was conducted under Contract NAS1-10353 (ref.8). Unfortunately, few data are available on low stress, long-term creep at temperatures above 1600K. No tensile creep data are available at all. From the available data, it is apparent that furnaces employing ceramics must be carefully designed so that parts operating at temperatures in excess of about 1300K must be in compression or, if in tension, must have a stress well below 10^5 N/m^2 (15 psi).

If a body is uniformly heated, thermal stresses do not occur provided the body is homogeneous, isotropic and unrestrained. Thermal stresses arise from temperature gradients, differential thermal expansion and variations in material properties in the body of interest. The susceptibility of ceramic materials to thermal stresses has long been recognized and studied. It is an important consideration with regard to their potential for use in furnaces where high heat flux rates and temperature gradients might exist in some imposed circumstances.

Kingery (ref. 9) shows the various analytical descriptions which have been developed for the conditions to initiate fracture of a brittle material considering simple shapes in a variety of thermal stressing situations. Based upon these analyses, no single parameter or test value is a suitable index to rate a material's resistance for all conditions of thermal stressing. The material properties which affect thermal stress resistance are elastic strength, coefficient of thermal expansion, Poisson's ratio, and in some cases, thermal conductivity, diffusivity or emissivity.

Analyses for different conditions result in the following three parameters that can be used to rate the thermal stress resistance of material under conditions where plastic strain is insignificant:

$$R_1 = \frac{\sigma_f (1 - \mu)}{E\alpha} \tag{3}$$

$$R_2 = \frac{\sigma_f k (1 - \mu)}{E\alpha} \tag{4}$$

$$R_3 = \frac{\sigma_f a (1 - \mu)}{E\alpha} \tag{5}$$

where σ_f is either the tensile or shear fracture stress of the material, whichever is significant to the problem, E is Young's modulus, α is the coefficient of linear thermal expansion, k is the thermal conductivity, a is the thermal diffusivity, and μ is Poisson's ratio. For ceramics, the most serious thermal stresses are tensile. Since the compressive strength is generally four to eight times the tensile strength, failure from compressive stresses is relatively unimportant. Shear strengths for ceramics are always greater than or equal to the tensile strengths.

Conceptually, the critical condition for fracture f is defined by the product

$$f = R \times S \tag{6}$$

where R is the appropriate material parameter (R_1, R_2, or R_3), and S is a corresponding parameter dependent only on specimen geometry and size. R_1 can apply when fracture results from an extreme thermal shock, in which case f is the instantaneous surface temperature change ΔT_f of an object, initially at one temperature and then suddenly heated or cooled, a situation which is generally referred to as thermal shock. R_2 can apply under conditions of a steady-state heat flux q_{max} that will cause a sufficient temperature gradient to induce fracture. R_3 can apply to the minimum constant rate of surface temperature change ϕ_f that will cause fracture. The three corresponding equations are

$$\Delta T_f = R_1 S \tag{7}$$

$$q_{max} = R_2 S \tag{8}$$

$$\phi_f = R_3 S \tag{9}$$

In summary, it should be emphasized that the use of these factors would be exact only to a homogeneous isotropic body whose physical properties are substantially independent of temperature. These relations do not cover all possible conditions but are representative of the factors comprising thermal stress resistance.

The size and shape of a ceramic part greatly influences its resistance to thermal stresses. In particular, for moderate rates of temperature change, the thermal stress resistance of a part is inversely proportional to specimen dimensions. For very high rates of change, this size effect is only important for small dimensions. In general, shapes having sharp corners or edges are to be avoided, as are parts having both thick and thin sections together.

For complex shapes or materials which are subject to plastic flow, experimental measurements are the only reliable method for measuring thermal stress resistance of the specific system.

A useful parameter for comparing steady-state heat flux capacity parameter R_2 as in equation (8) is

$$\frac{q_{max} X}{A} \tag{10}$$

where q_{max} is the maximum heat flux, X the thickness normal to flow, and A is the heat transfer surface area. This is shown compared in Table IV for the contemporary as well as the new materials.

Table IV summarizes the various considerations relative to the choice of material for an electric resistance heater element. For high temperature with an oxidizing atmosphere, the ceramics thoria and zirconia are best. From creep strength considerations, zirconia becomes the prime candidate.

Another property, not easily specified in absolute value, is that of stability relative to conducting ceramic heating elements. The ceramic furnace concept was "born too soon." This fact has been the principal barrier to its acceptance. Its premature introduction at the turn of the century caused barriers to acceptance which haunt the scientific user community today. The barriers often cited are an electrothermal instability caused by its negative resistance coefficient with temperature resulting in low life and poor cyclic capability. This has been brought about by the improperly prepared ceramic elements and electrode-to-ceramic interfaces.

Heating elements composed of materials which exhibit the negative electric resistivity characteristics can be subject to this electrothermal instability phenomena. This requires careful application of the manner in which the elements are used. One well-known instability phenomenon of this type, when connected directly across a constant potential source, is the tendency of such elements to allow "unlimited" current to flow through the conductor until it fails. This condition is always encountered in the applications of, for example, fluorescent lamps, arc discharge devices, and oxide ceramics in infrared radiation sources. A common and simple solution is to use a ballast resistor - a resistance element, usually metallic, with positive temperature coefficient of resistivity - in series with the negative resistance element to produce a net positive resistance. Another solution is to employ a constant-current type of power source. Thus, adequate solutions for that type of "one-dimensional" instability have long been available and in use.

There is another type of instability, a phenomenon of spatial (circumferential) current distribution instability, frequently referred to as channeling (or striation) which can occur only in conductors with negative resistance characteristics. Basically, it occurs because any perturbation to a symmetrical distribution of current in such a conductor, from whatever cause, has a tendency to feed on itself and to grow larger, ultimately creating a channel of intense current and excessively high temperature <u>if not otherwise controlled</u>.

It appears that little attention has been given to this kind of stability control there being little treatment of this in the open literature. The reasons for this are: (1) the quality (life) of ceramic heaters was poor when their use in this manner was first discovered and the incentive to solve the problem was low, and (2) no high speed computers were available with which to solve the highly non-linear equations in the geometries of interest.

In small-sized conductors such as Nernst glowers, this channeling phenomenon normally produces no noticeable effect apparently because of the high thermal gradients which develop in such small-sized elements conducting

TABLE IV

PHYSICAL, CHEMICAL AND ELECTRICAL PROPERTIES OF OXIDATION RESISTANT RESISTOR (HEATER) MATERIALS OF DIRECT RESISTANCE FURNACES.

Material	Chemical composition (Trade name)	Melting temperature K (°F)	Maximum working temperature K (°F)	Electrical resistivity Ω - m		Surface power flux loading W/cm² (W/in².)	Thermal stress loading parameter W/cm (W/in)	Workability after initial fabrication	Vacuum sublimation rate at working temperature		Oxidation rate at working temperature Kg/m²s	Remarks chemical reaction, etc.
				Room temp.	At max. working temp.				Mass flux Kg/m²s	Surface recession m/s		
Thoria	ThO_2	3540 (5910)	~2500 (~4040)	Non-conductor	Unknown[a]	264[b] (1700) @2775K	~12.2[b] (31) @2775K	None	4×10^{-4}	4×10^{-8}	None (Stable in oxygen)	Little known. Reacts with C at high temperature.
Zirconia	ZrO_2; 10 mole % Y_2O_3 (yttria stabilized zirconia)	2998 (4935)	~2500 (~4040)	Non-conductor	3.98×10^{-3}	93[c] (600) 2200K	7.1[c] (18.1) 2200K	None	6×10^{-5}	10^{-8}	None (Stable in oxygen)	Reacts with H_2 at T_s >2475K; w/NH_3 at T_s >2275, C/N_2 at T_s >2275; C/C at high temp. to form carbides & eutectics.
Iridium	Ir	2727 (4450)	2350 (3770)	4.8×10^{-8}	6×10^{-7}[d]	N/A	N/A	Warm	9×10^{-9}	4×10^{-9}	3×10^{-2}	Most corrosion resistant metal known, but is readily attacked by molten Cu, Al, Zn, and Mg.
Platinum-rhodium	80 Pt; 20 Rh	2175 (3455)	1990 (3120)	2.1×10^{-7}	5.6×10^{-7}	N/A	N/A	Cold	N/A	N/A	8×10^{-5}	Superior heat alloy of platinum series.
Platinum	Pt	2047 (3225)	1775 (2735)	9.9×10^{-8}	5.8×10^{-7}	N/A	N/A	Cold	1.6×10^{-7}	7.5×10^{-12}	10^{-4}	Excellent oxidation resistance.
Molybdenum disilicide (Ref 12)	90 $MoSi_2$; 10 metallic and ceramic additions (Kanthal "Super")	e	1970 (3085)	3.0×10^{-7}	4.0×10^{-6}	1.1 (70) @1925K (Ref 13)	N/A	Hot (~1775K)	f	f	N/A	A pore-free ceramet with additives of glass with high silica content. Forms coat of protective SiO_2. Must be kept from firebrick. Mullite OK.
Silicon carbide (Ref 14)	94.4 SiC, 5.6 SiO_2; 0.3C 0.2 Al; 0.6 Fe; 0.3 Si; 0.6 (CaO + MgO) (Globar, Silcit)	g	1980 (3100)	1.8×10^{-3}	1.0×10^{-3}	2.3 (15) @1865K (Ref 13)	N/A	None	N/A	N/A	h	Tends to age and change resistance by factor of five (5). SiO_2 coating on surface.
Iron, chromium, aluminum, cobalt alloys (Ref 15)	22Cr; 5.75Al; 0.5 Co; balance Fe (Kanthal A-1)	1785 (2755)	1650 (2510)	1.45×10^{-6}	1.50×10^{-6}	1.25 (8) @1620K (Ref 13)	N/A	Hot	N/A	N/A	N/A	Depends on oxide coating formed for oxidation resistance.

NOTES:

a No measurements known at 2500K. At T = 2000K; $\rho = 4 \times 10^{-}$ Ω-m as compared to $\rho = 9 \times 10^{-3}$ Ω-m for zirconia stabilized with 10 mole % Y_2O_3.

b Preliminary AEROR data on small rods, for short duration tests, order of few hours.

c Preliminary AEROR data on small rods, for modest duration tests, <1000 hours.

d Extrapolated. No measurements available over 1600K. At T = 1600K; $\rho = 3.7 \times 10^{-7}$ Ω-m.

e SiO_2 (cubic) melts at 1985K.

f Not recommended for vacuum use, ex: T_{max} = 1375K for 10^{-5} Torr.

g In pure form, sublimes at 2275K; decomposes at 2485K; melts at ~2875K.

h Reference 13 gives a life of 3000 hr. @ 1865K (2908F) at 2.3W/cm².

the heat away from the channel rapidly and limiting its growth. Because of this lack of an understanding of the problem, the larger oxide ceramic heating elements in the past have not generally been built successfully in spite of the fact that they are needed for a variety of research devices. Those that were found to have been built indiscriminately in the past in larger sizes usually developed a channeling instability condition and quickly failed.

It has been shown analytically and it has been verified experimentally that by means of thermal radiative sharing between various elements of a cavity, instability is no longer a problem as long as certain stability criteria are met. In the furnace design, it is necessary to provide guard heaters and effective high temperature thermal insulation.

ISOTHERMAL FURNACE

Mechanical Design

Furnace Design.- The completed Model 40 zirconia conductor furnace assembly is shown in figures 6 and 7. Figure 8 identifies the Model 40 isothermal furnace components and shows the general component arrangement. The furnace assembly has overall dimensions of 23.5 cm (9.25 inches) diameter by 47.0 cm (18.5 inches) high not including the extensions due to the air inlet tubulation and the electrical conduit connector. The furnace assembly weight is 13.1 Kgm (28.9 pounds).

The furnace main structural components consist of the following:

(1) A stainless steel housing

(2) An aluminum base

(3) A flat aluminum bottom cover

(4) A nickel plated steel safety cage

These components are shown in figure 9.

Preheater power connections, preheater thermocouples and the main heater upper electrode connection are brought out to an electrode terminal plate assembly on the top of the furnace. The main heater lower electrode terminal is brought out through the housing and is located adjacent to the lower terminal heat sink. Figure 10 shows the terminal plate, the two terminal heat sinks and a conduit-terminal strip connector to which external electrical connections are made. The electrical schematic is shown in figure 11. External electrical connections are made to the appropriate terminal per the schematic. The figure 11 terminal strips correspond to those shown on the conduit connector terminal strip panel in figure 10.

A separate air blower is connected to the air blower inlet to provide cooling of two terminal heat sinks to which the main heater electrodes are

P164-1

Figure 6.- Model 40 zirconia conductor furnace.

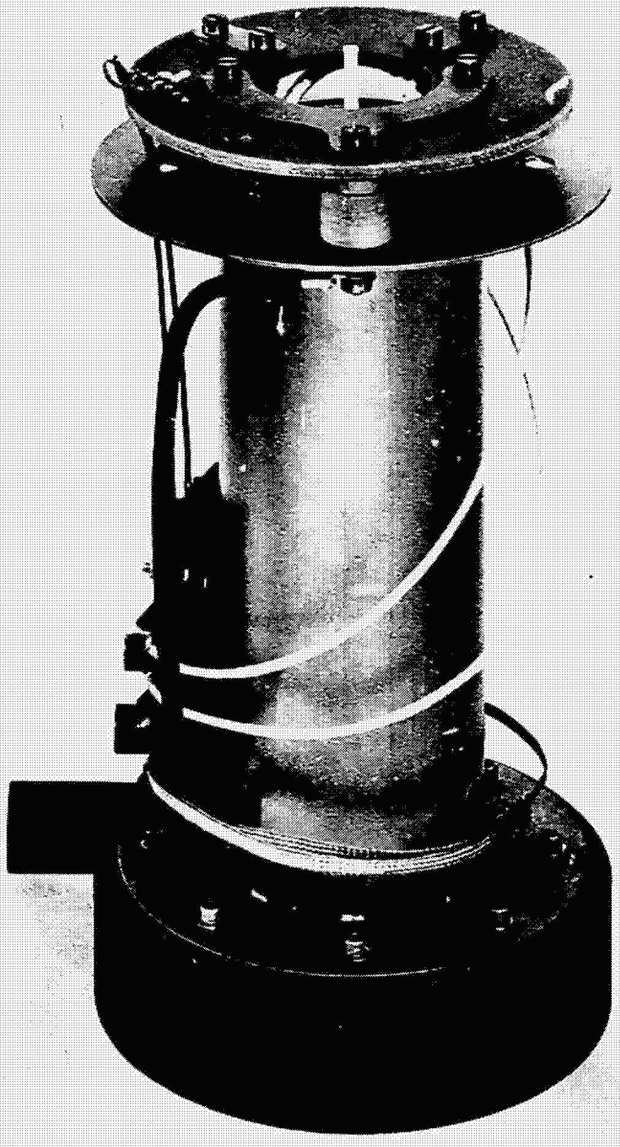

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Figure 7.- Model 40 zirconia conductor furnace with safety cage removed.

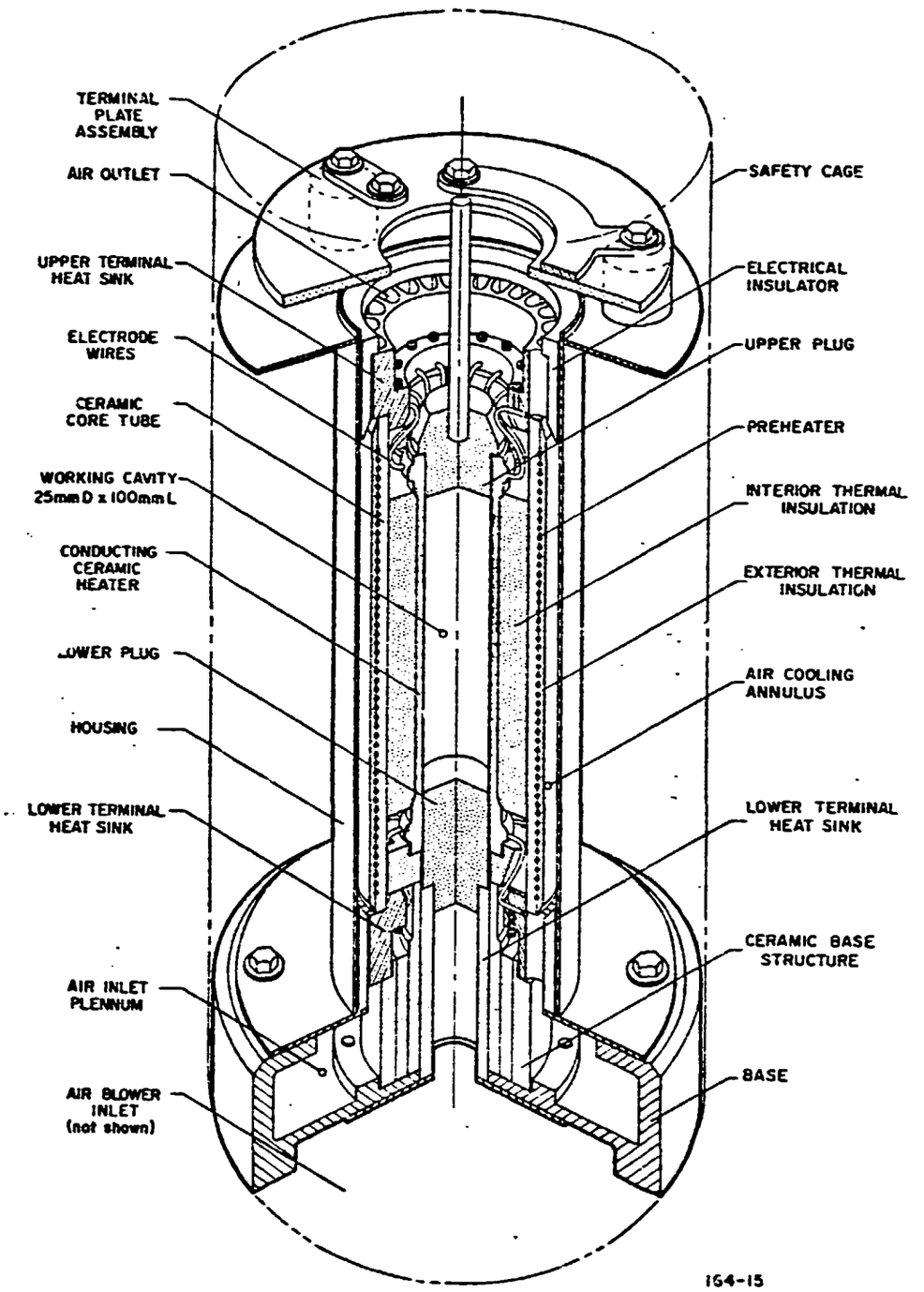

Figure 8.- Model 40 isothermal zirconia conductor furnace components identification.

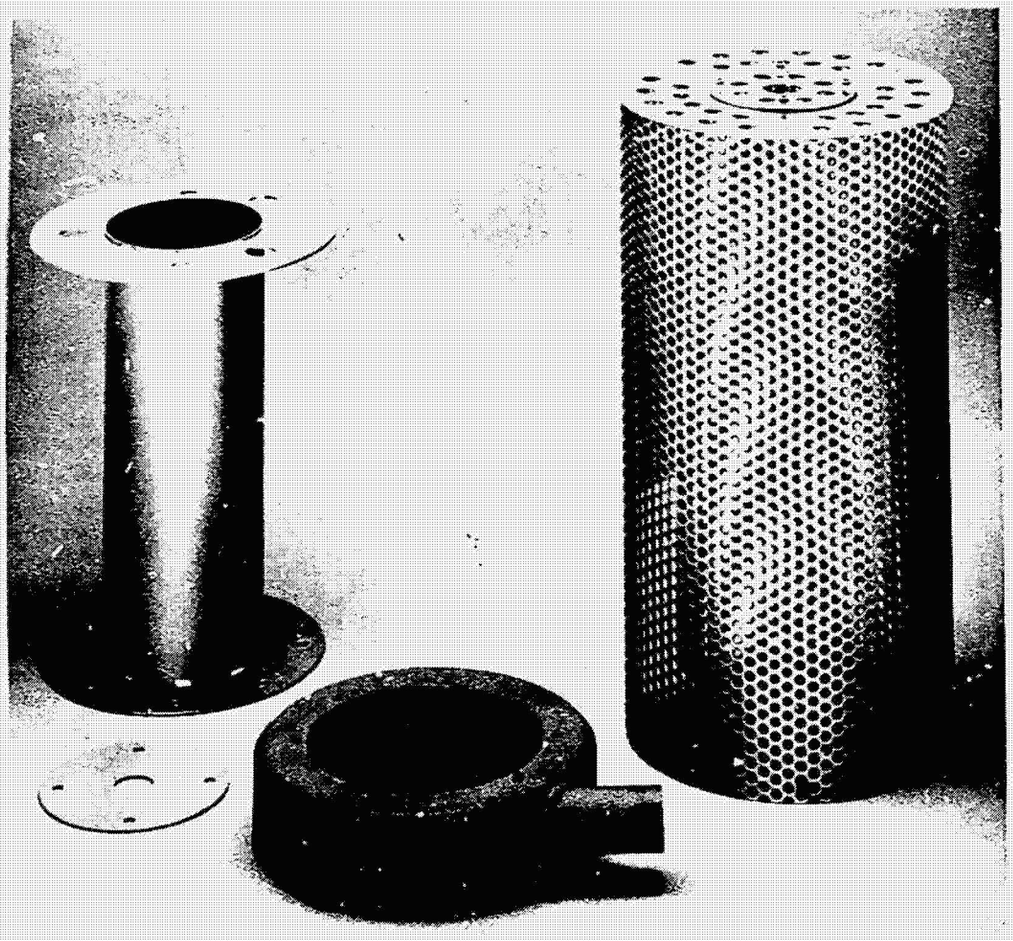

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Figure 9.- Model 40 zirconia conductor furnace structural component.

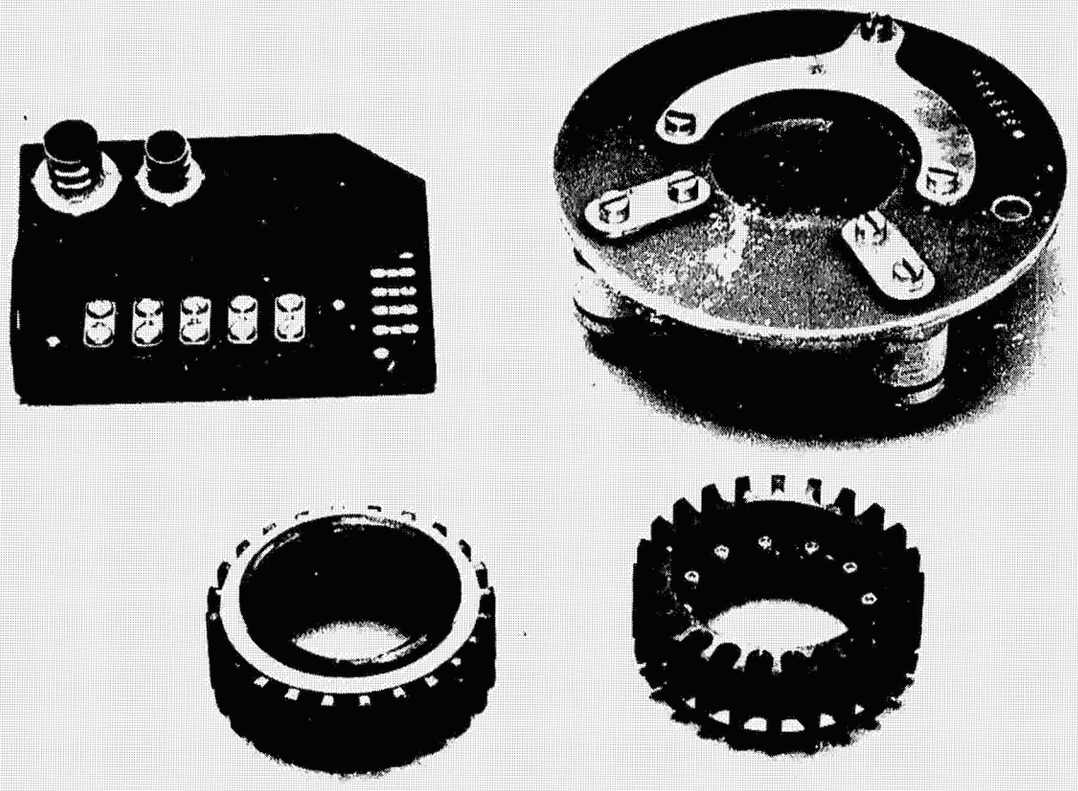

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Figure 10.-Model 40 zirconia conductor furnace electric terminal components.

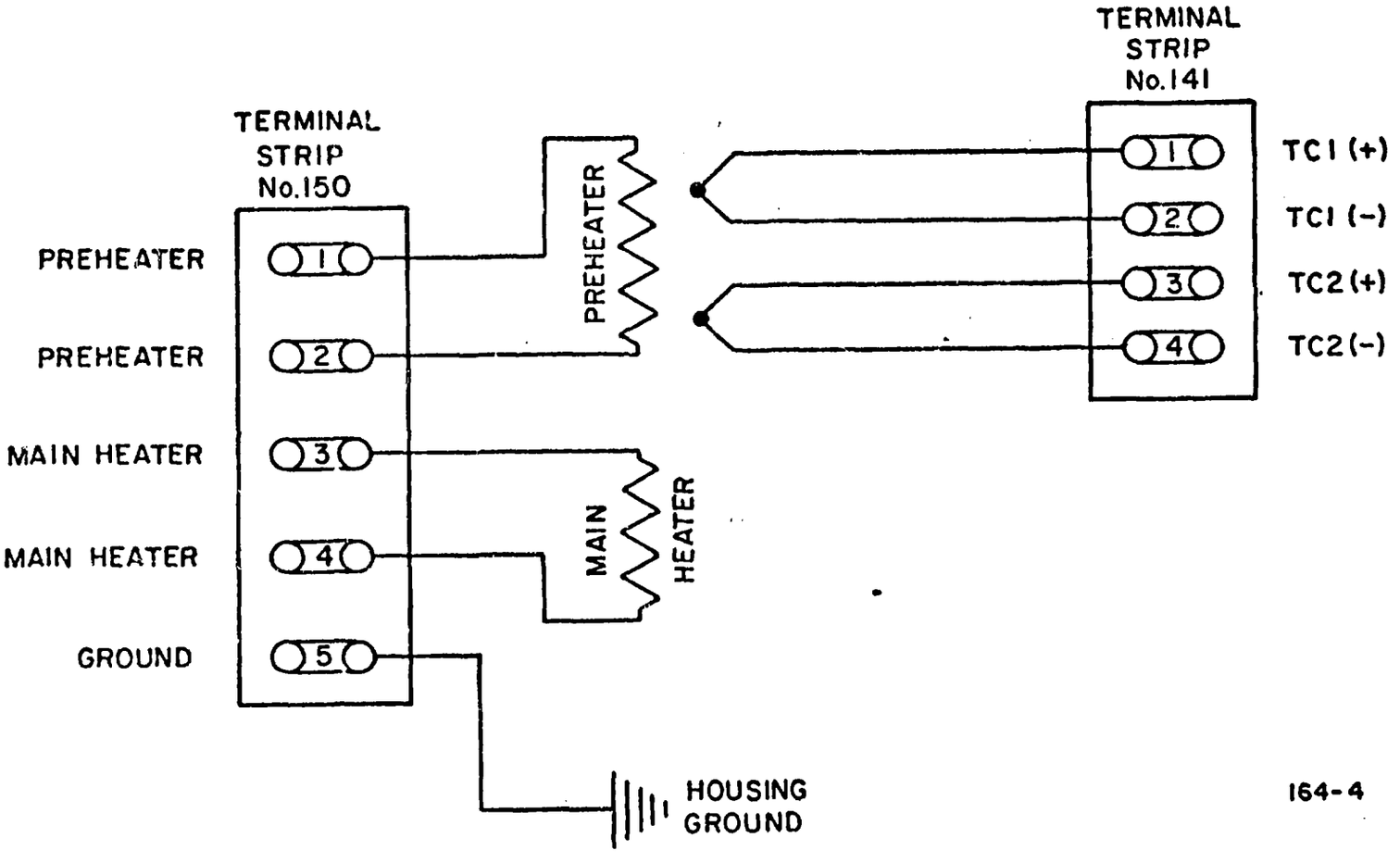


Figure 11.- Isothermal furnace electrical schematic.

attached. The heat sinks rest on either end of an alumina tube which serves as the core for the preheater. The preheater assembly consists of nichrome alloy wire wound on the core tube and covered with thermal insulation. This insulation forms an annular passage with the furnace housing to duct air flow from one terminal heat sink to the other and over the exterior thermal insulation of the preheater.

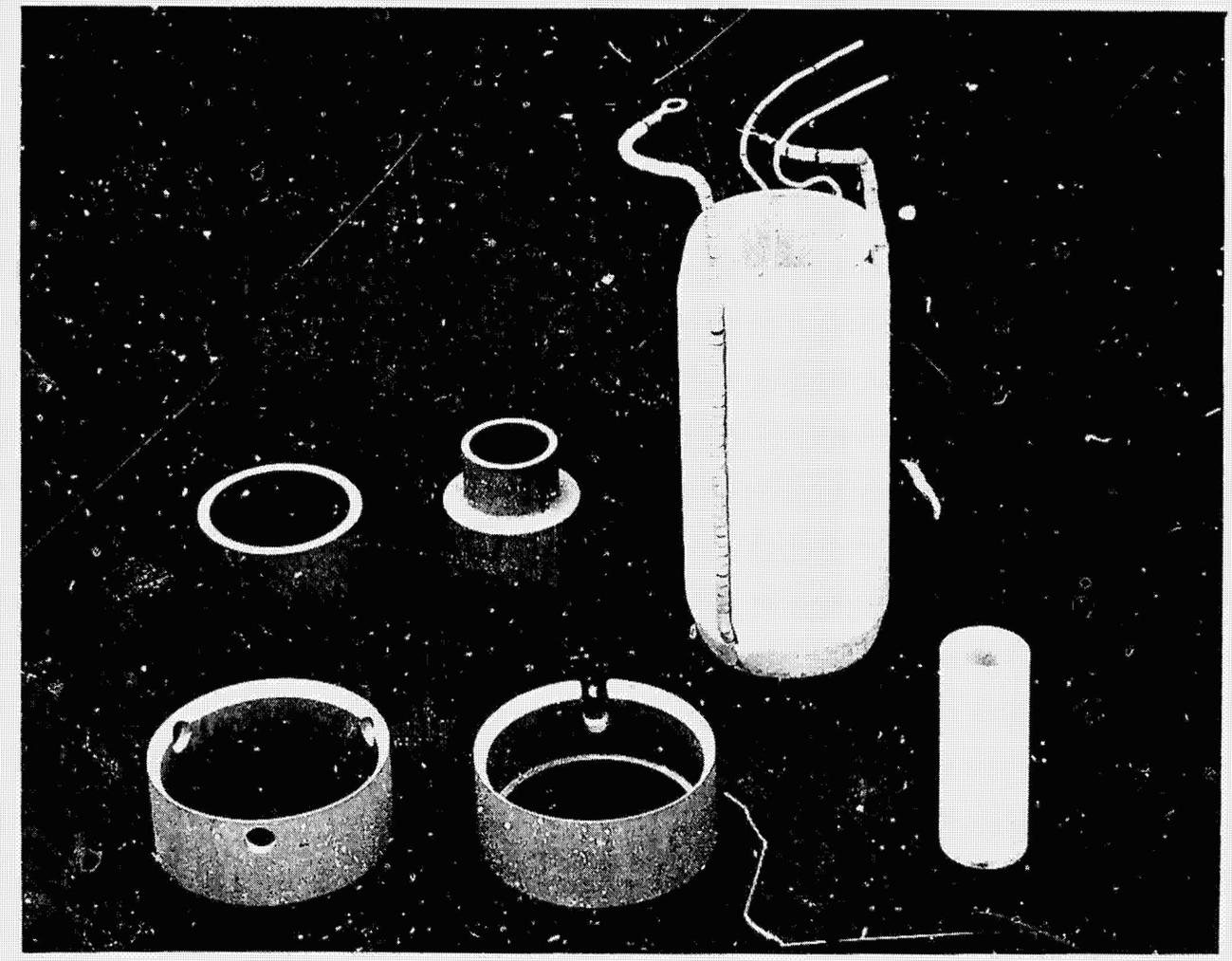
The, preheater, shown in figure 12, is a relatively rugged structural component comprised of an alumina core tube with a 7.62 cm (3.0 inches) bore and a 0.635 cm (1/4 inch) wall. Heavy 0.13 cm (.051 inch) diameter nichrome wire is wound in a spiral groove in the core tube. One groove is skipped 1/3 of the way down from the top end. Two (2) type K (Chromel-Alumel) 20 gauge thermocouple junctions are placed in this groove and brought out to a barrier terminal strip. One of the thermocouples is used to provide a feedback signal for a preheater power controller. The second thermocouple serves as a spare and can be used as an input to a preheater temperature indicator in cases where the controller used has no temperature indicator.

Also shown in figure 12 are various ceramic parts. The large diameter rings, made from LAVA A electrically insulate the terminal heat sinks from the furnace housing. Shown directly above these parts are ceramic base structure parts, also of LAVA A, upon which the preheater - main heater assembly rests. The longer-smaller diameter hollow cylinder is the lower plug support made of alumina.

A conducting ceramic (main) heater assembly is located within the core tube separated from the core by an internal insulation system made of Zircar high temperature insulation. This is the main heater as distinguished from the preheater and is shown in figure 13. The main heater rests on a slotted Zircar block as shown in figure 13. Slots are provided to pass the 12 electrode lead wires downward to the screw terminals in the terminal heat sink. Not shown are Zircar block wedges which surround the main heater element over the hot zone to form the interior thermal insulation system (see figure 7).

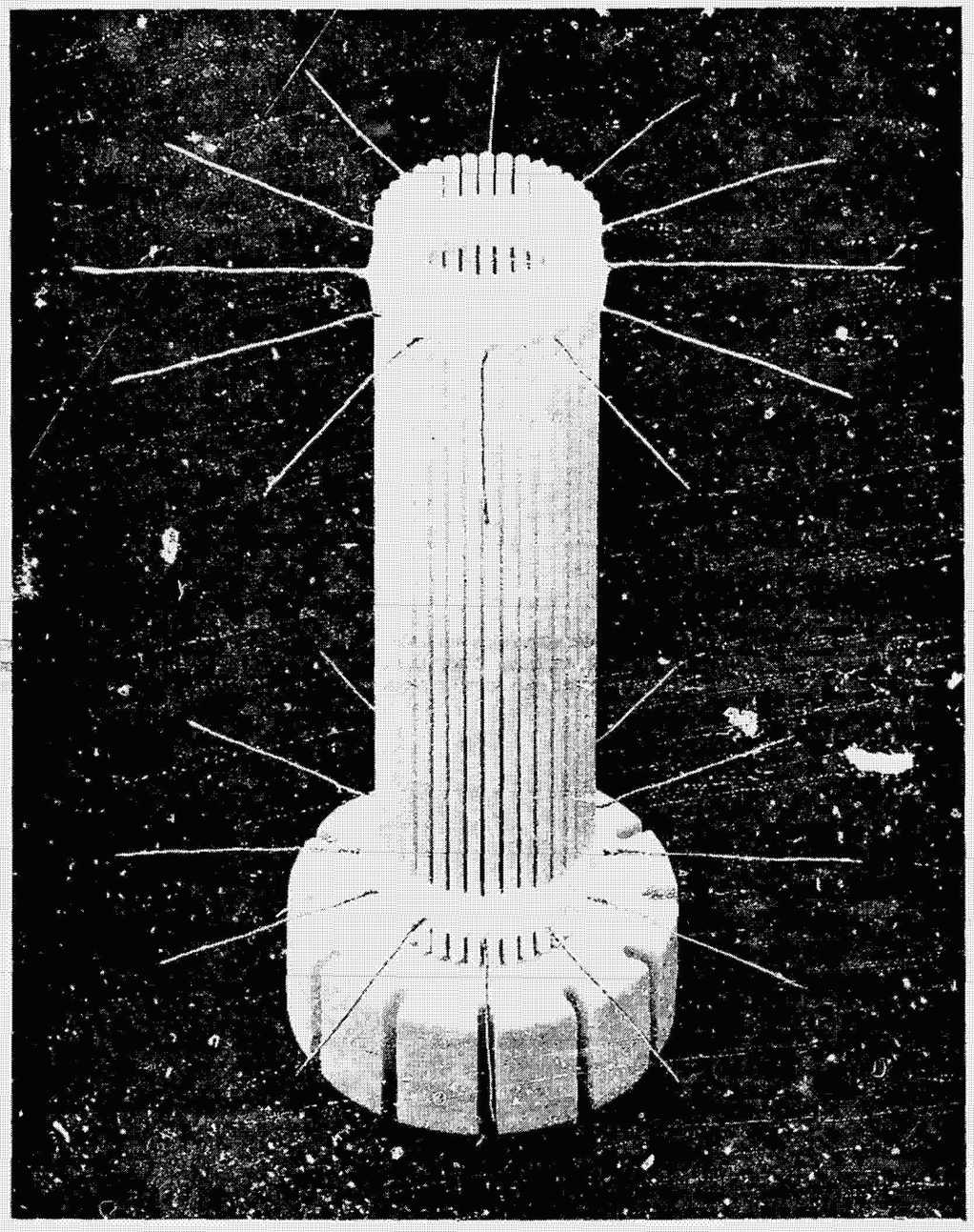
The conducting ceramic used for the main heater is stabilized zirconia. The zirconia is polymorphic and is stabilized in the cubic phase to achieve a thermo-mechanically stable material. The main heater element is cylindrical in shape having a nominal O.D. of 4.3 cm (1.69 inches) and a nominal overall length of 15.5 cm (6.1 inches). The inside diameter of the element varies depending upon the configuration from about 3.0 to 3.5 cm. Platinum electrode wires are cemented to each end of the cylindrical heater close to the heater ends. A heated typical length (between electrodes) is 13.2 cm (5.2 inches). As seen in figure 13, the main heater element is built up from extruded tubes. This structural arrangement has significant thermal-mechanical advantage over a solid cylinder being less susceptible to thermal expansion failure effects.

Nominally, a working cavity size of 2.5 cm diameter by 10.0 cm long is provided within the main heater. Plugs of Zircar insulation are used at both ends of the main heater cavity to prevent large radiation and drafting losses from the furnace working cavity. Both insulation plugs are removable for access to the furnace cavity. Figure 14 is a photograph looking through the furnace cavity with the end plugs removed. The Zircar block wedges are



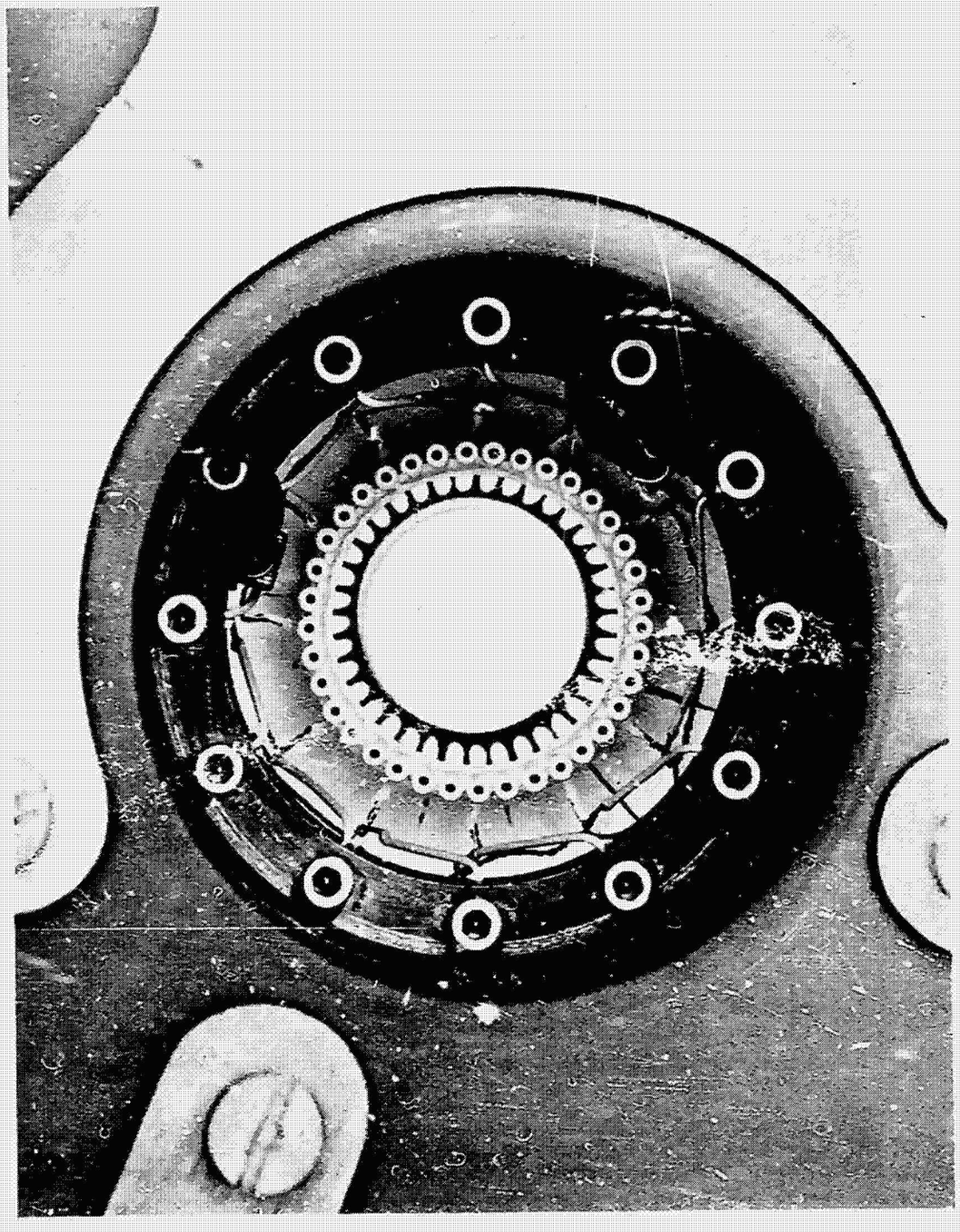
P164-5

Figure 12.- Model 40 zirconia conductor furnace preheater and ceramic parts.



P164-6

Figure 13.- Model 40 zirconia conductor furnace main heater element and support block.



P164-7

Figure 14.- Model 40 zirconia conductor furnace as viewed looking down into cavity with plugs removed.

visible in this view.

Cooling Package Design.- The cooling package is compact and consists of a cabinet mounted blower, air filter, power switch, indicator light and fuse and a separate flexible duct. The cooling package is contained in an enclosure separate from the furnace. Cooling air is ducted to the furnace assembly by a 0.91 meter (36 inches) flexible conduit 5.08 cm (2 inches) in diameter. Cooling is provided by a squirrel cage blower rated at 80 CFM of free air. When connected to the furnace, the unit delivers approximately 29 CFM at a static furnace inlet pressure of 0.6 inches of H_2O. Inlet air is filtered to prevent contamination of the furnace.

Thermal Design

Thermal considerations are most important in the design of the conducting ceramic furnace. The interfacing of materials resistant to oxidation at high temperature is a critical design consideration as well as the insulation system to efficiently contain temperatures to 2200°C.

Figure 15 schematically presents the isothermal furnace to assist in visualizing various thermal considerations. The heater element electrodes present the most critical thermal design area. The electrode ceramic-to-platinum junction temperature must be maintained between the acceptable limits of 1000°C to 1500°C during operation. If the junction temperature is cooler, ceramic element resistance increases and adequate electrical current conduction becomes a problem. When junction temperature is too high, increased electrode metal evaporation can seriously shorten heater element life and, of course, if the junction temperature is excessively high, melting of the electrode metal will cause catastrophic failure.

In order to protect the electrode regions from the intense cavity temperatures, cavity end insulation plugs are projected into the hot zone region nominally 1.4 cm beyond the electrodes. In effect, a 10 cm long working cavity is available to a diameter of 2.5 cm. The 2.5 cm diameter is arbitrary and provides a reasonable stand-off distance from the main heater element wall.

Some stand-off distance between the furnace cavity inside wall surface and the furnace load is required to allow the heater element to distribute any local high heat flux regions to adjacent surfaces to prevent spatial current distribution instabilities. In the case of a load which follows the heater element wall temperature closely as with slow power transients and which re-radiates effectively, the stand-off distance can be short. In the other extreme, as for example, for a relatively cold load thrust into the furnace cavity, more stand-off distance is required.

Electrode cooling is accomplished primarily by radiation of the ceramic heater structure in the vicinity of the electrode to adjacent cooler structures. Some convective cooling may occur as well which is difficult to determine analytically. However, the convective cooling contribution is sufficiently small to be of little consequence in the overall electrode configuration design. Secondary cooling is provided by thermal conduction though the electrode lead wires to the upper and lower terminal sinks. The sinks are

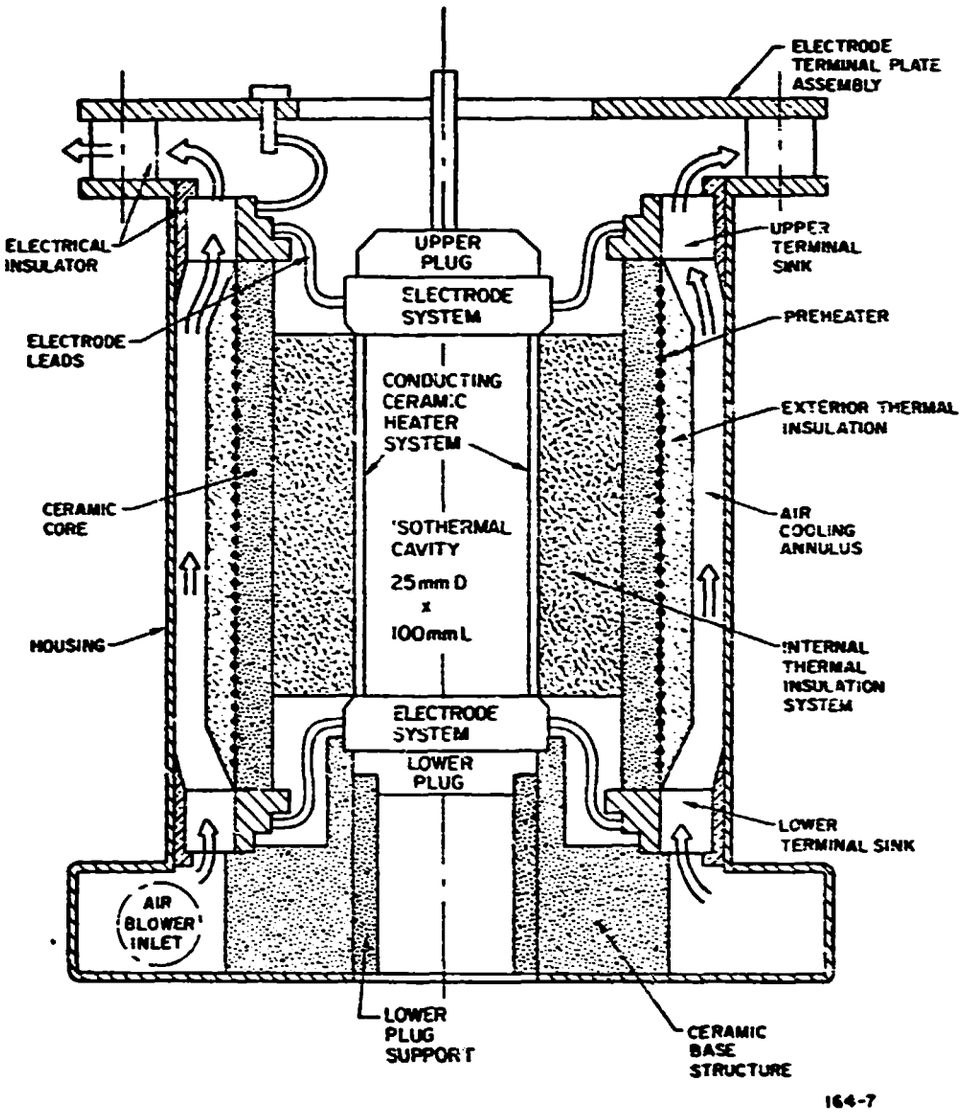


Figure 15.- Isothermal furnace schematic.

finned metallic rings cooled by air flow supplied by the air blower. The air flow is about 29 CFM and removes approximately 0.5 Kw from the electrode sinks to maintain a system thermal balance at full temperature.

Figure 15 indicates the relationship of the various insulation systems. There are basically three systems, the internal insulation system between the ceramic heater and preheater, the exterior system between the preheater and air cooling annulus, and the upper and lower plugs. The plugs control direct axial thermal losses from the furnace cavity. The internal and external insulation systems control radial losses and are arranged to provide proper thermal coupling between the preheater and ceramic heater. The relationship between these insulation systems and the two heaters was explained in the Ceramic Element Furnace Concept Section (see figure 2).

Figure 16 shows more accurately the temperature distribution resulting from a typical heater-insulation arrangement. The curves shown are calculated design radial temperature distributions for the internal cavity condition of 2200°C (2475K) and for a starting preheater temperature of 975°C with cooling air flow. Note that only a slight overtemperature of about 200°C is required on the preheater to achieve an ignition condition (dashed curve). Note also that with full cavity temperature and the preheater power off (solid curve) the preheater temperature is only slightly increased at its inside surface.

Additional discussion on the thermal aspects of the isothermal heater element relative to temperature capability is given in the Thermal Design Section under Axial Gradient Furnace.

AXIAL GRADIENT FURNACE

Mechanical Design

The axial gradient furnace utilizes the same housing, base, and preheater and has the same external physical dimensions as the isothermal furnace. A schematic of the axial gradient furnace depicting a two-zone heater element arrangement is shown in figure 17. In place of the single conducting ceramic heater element zone of the isothermal furnace, the axial gradient furnace has two separately controlled heater element zones. The overall working cavity length between the faces of the lower support and upper insulation plug is 17.8 cm (7.0 inches).

The preheater function is the same as in the isothermal furnace to provide ignition for the ceramic heater element. The same type K (Chromel-Alumel) thermocouples are provided for preheater temperature control and their leads are terminated at a barrier strip as shown in the electrical schematic, figure 18.

The main heater is comprised of two separate heating elements. The higher temperature primary element is made of stabilized zirconia and is 17.7 mm (0.697 inch) ID and 22.6 mm (0.390 inch) OD by 127 mm (5.0 inches) long. Figure 19 shows the primary element construction to be similar to the element in the

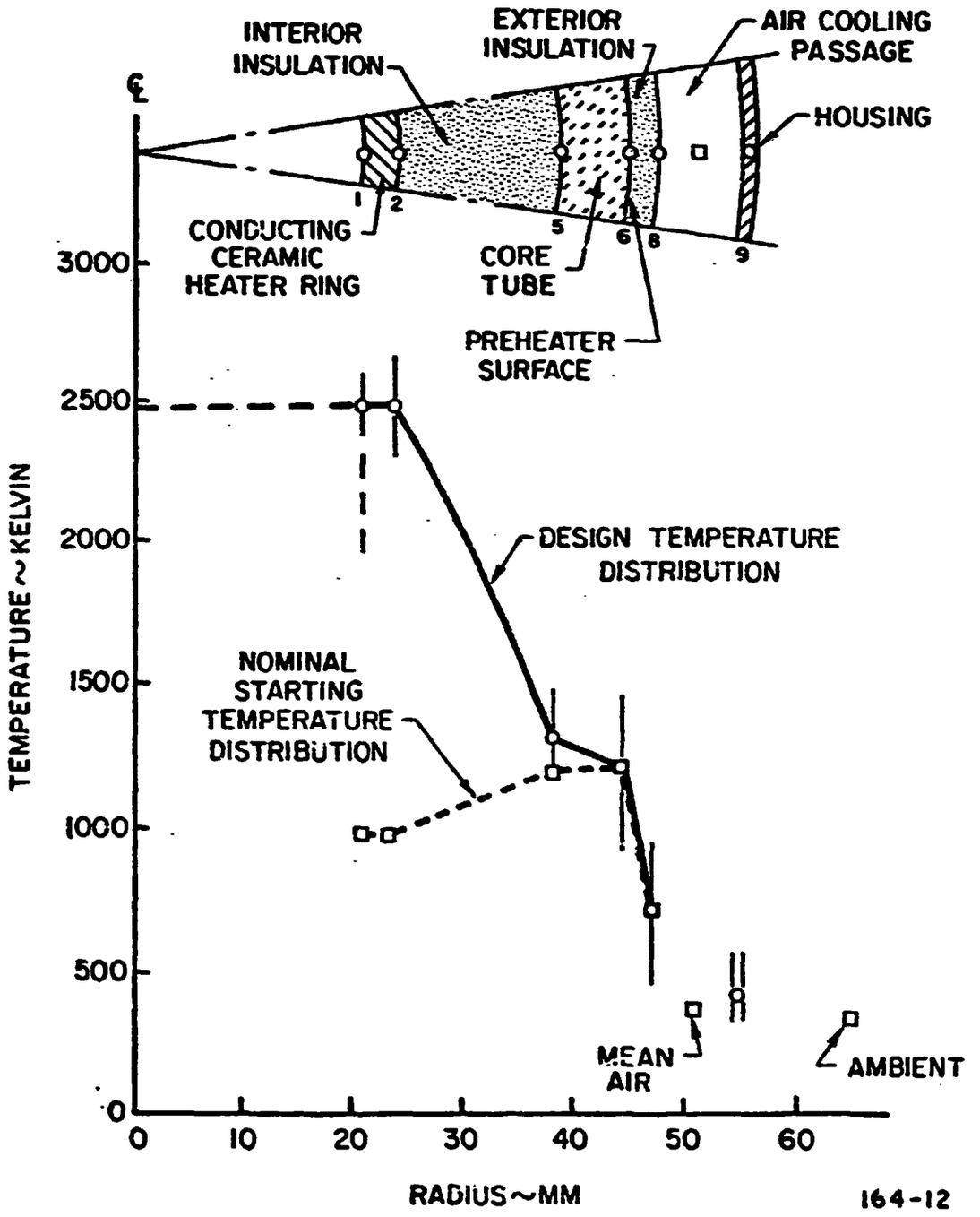


Figure 16.- Model 40 zirconia conductor furnace design radial temperature distribution.

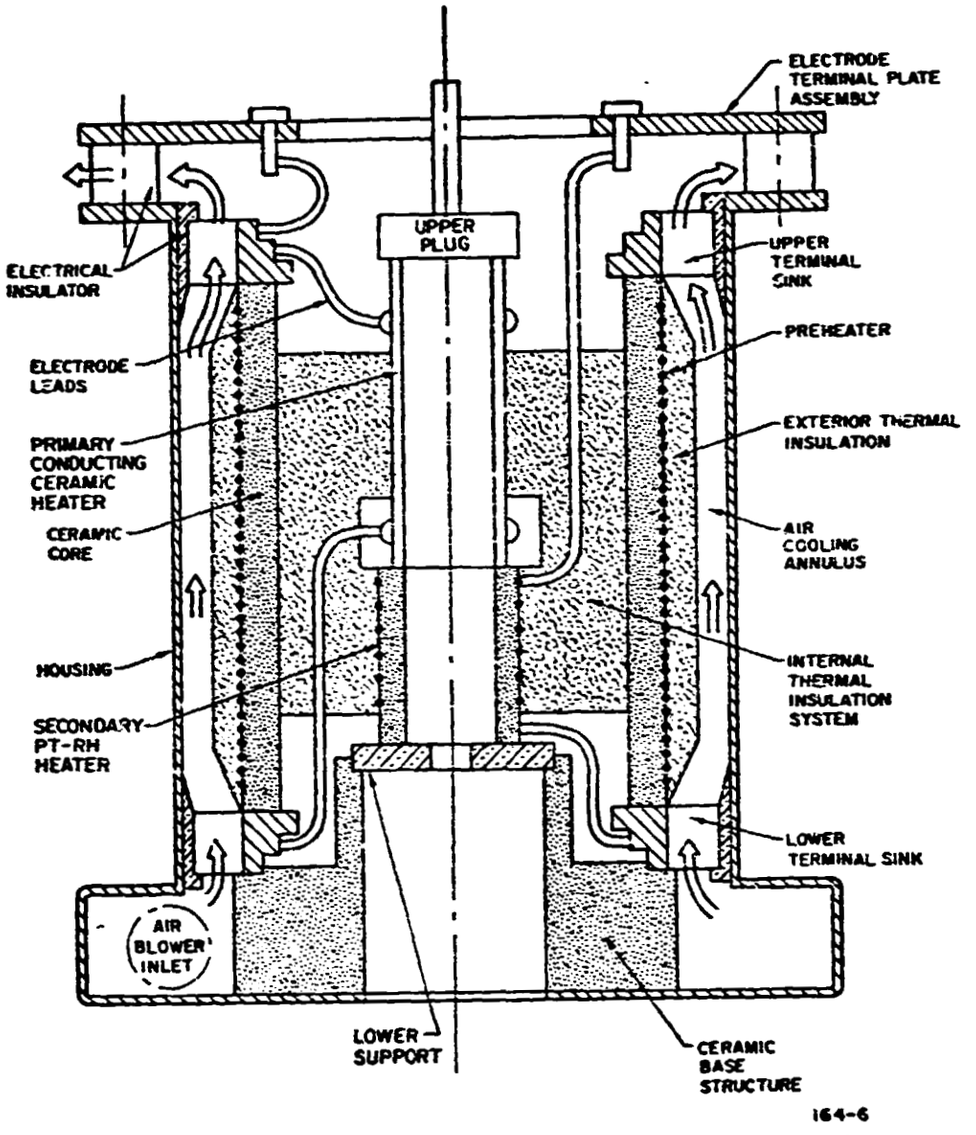


Figure 17.- Axial gradient furnace schematic.

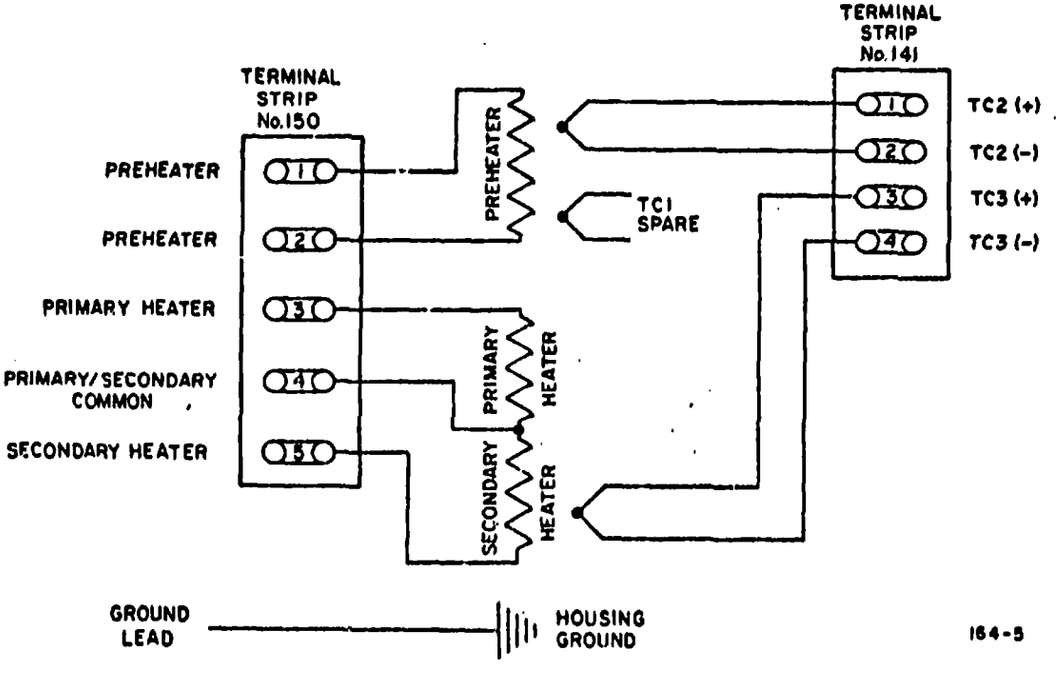


Figure 18.- Axial gradient furnace electrical schematic.

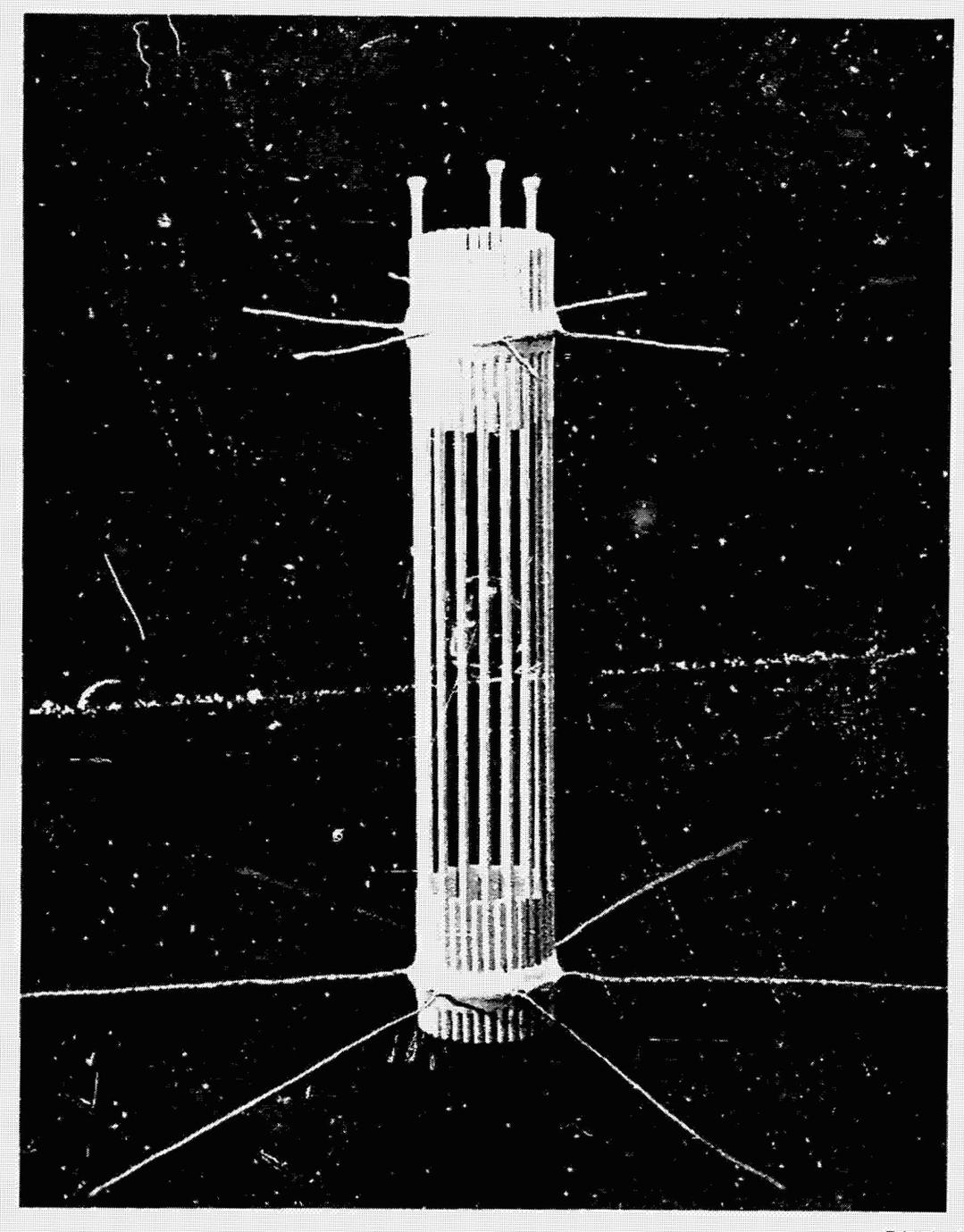


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Figure 19.- Axial gradient furnace ceramic heater element.

isothermal furnace. Tubular elements are spaced in this case by alternating shorter tubes on each end in the electrode region. Such construction offers good visibility as evidenced by the dime suspended within the element. The primary element electrode wires attach to identical upper and lower terminal sinks as used in the isothermal furnace.

The lower temperature secondary element is made from platinum-10% rhodium alloy wire 0.508 mm (0.020 inch) diameter wrapped on an alumina core tube. The core tube has an ID of 19.05 mm (0.75 inch), an OD of 28.58 mm (1.125 inches) and is 76.2 mm (3.0 inches) long. The secondary element's lower lead wire is terminated at the lower terminal sink making it common with the primary element lower lead wire. The secondary element's upper lead wire passes upward through the insulation and is brought out on a separate terminal on the terminal plate assembly.

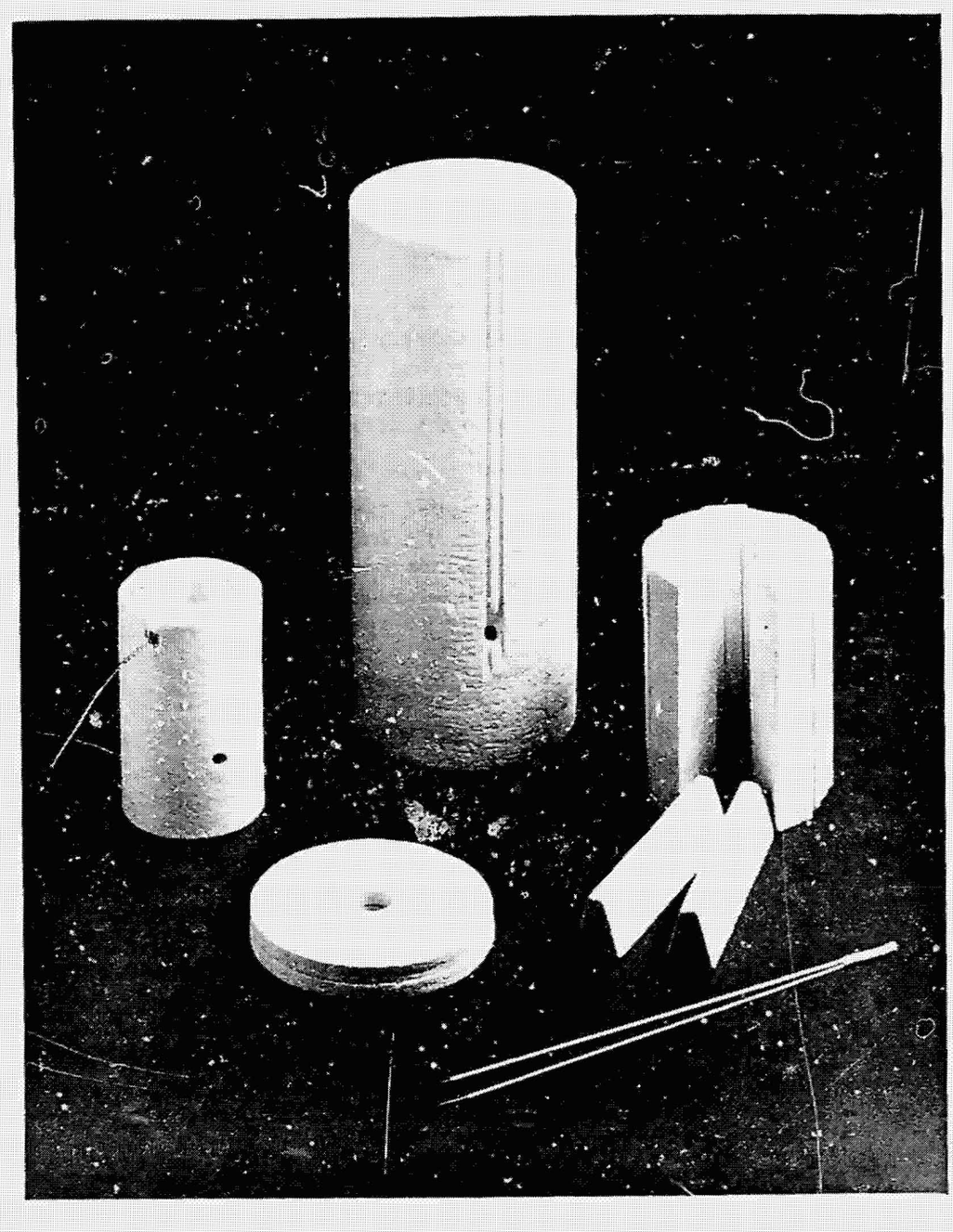
Components which make up the axial gradient furnace main heater in addition to the primary element (figure 19) are shown in figure 20. The secondary element is seen wrapped with Zircar felt insulation. Zircar block insulation wedges are shown which surround the primary element. All of these fit inside the large cylinder made of alumina-silica insulation. A platinum-13% rhodium thermocouple is fitted to the outside grooves in the large cylindrical insulation body, through the hole in the Zircar felt surrounding the secondary element, with the junction located in a hole in the secondary element core tube. This thermocouple provides a feedback signal for control of the secondary element.

Thermal Design

The thermal design considerations discussed relative to the isothermal furnace apply to the axial gradient furnace as well. Preheater temperature with the main heater under power and the preheater unpowered are lower because of the smaller diameter of the main heater.

A significant thermal difference exists between the conducting ceramic element of the isothermal furnace (figure 13) relative to that of the gradient furnace (figure 19). The mechanical difference is obvious in that half of the tubes forming the gradient furnace primary element are full length and, therefore, active conducting elements, the other half being discontinuous and acting as spacers on each end. Recall that the platinum-to-ceramic electrode interfacing region must operate at a temperature below the melting point of platinum (preferably 1500°C or less). In the case of the heater element construction shown in figure 13, the conducting portion of the element (between electrodes) would tend to be equally high in temperature over the full conduction length except for the effect of the Zircar block insulation.

The Zircar block insulation adjacent to the conducting length of the isothermal furnace heating element is made shorter than the conducting length thereby reducing heat losses over a central portion of the element. The electrode region operates cooler because of increased thermal losses to the preheater core tube wall. Unfortunately an adverse compensating effect exists in that current is the same throughout the conducting length, yet, in the cooler regions near the electrodes, resistivity is higher and the local



P164-9

Figure 20.- Axial gradient furnace components.

ohmic (I^2R) heating is greater. Some temperature difference is achieved with the electrode region operating cooler than the central working cavity region. Thus, cavity temperatures to 2200°C are possible with electrode region temperatures limited to of the order of 1750°C.

Another technique used to achieve an even cooler relative temperature in the electrode region is depicted in figure 7. Here the conducting ceramic heater element has tapering incorporated. The section adjacent to the electrodes is of increased cross section thereby reducing local resistance relative to the central portion based on the same temperature. Using foreshortened interior thermal insulation as before, the electrode region temperatures can be reduced to about 1500°C for working cavity temperatures to 2200°C.

Still another technique is shown in figure 19 and is used with the axial gradient furnace heater element. The shorter spacer tubes are non-conducting tubes. Using foreshortened interior thermal insulation just covering the open portion of the heater element, the electrode region is allowed to cool effectively permitting a large temperature difference between the working cavity and the electrode region.

In the discussion above, it is assumed that internal end plug insulators are used to prevent direct radiation of the working cavity space onto the electrode regions. Open end operation would increase thermal loading of the electrodes and necessitate reducing the maximum operating temperature. In general, the upper electrode suffers the greatest thermal loading in such a situation with convective drafting providing some cooling of the lower electrode while adding to the upper electrode thermal load.

It is because of the drafting effects that the lower temperature secondary main heater section is located below the primary heater section. The Pt-Rh alloy secondary heater is rated for temperatures to 1500°C while the primary heater is rated for 1000 to 2200°C. The preheater can be used to hold a background (guard) temperature to control the transition between the top of the secondary heater element and the bottom end of the conducting region of the primary element, that is, in the lower non-conducting region of the primary heater. Between the three heater elements (preheater, secondary and primary) considerable flexibility of temperature and gradient control are provided by the Model 40 gradient furnace.

FURNACE POWER CONTROL

General Consideration

Separate controls are required for the various heater elements, the preheater and the ceramic or main heater in the case of the isothermal furnace, the preheater and the primary and secondary main heaters in the case of the axial gradient furnace. While the preheater and the secondary Pt-Rh alloy heater can be operated from either AC or DC electrical power, it is highly desirable to use AC power for the ceramic heater. This is the result of the oxide ceramic being a solid electrolyte and conducting electricity by the motion of

negatively-charged ions (O^{-2}) through the oxide rather than electrons.

Noble metal electrodes interface the oxide ceramic on each end. With an electrical current flowing, electrons move in the metal wires to the electrodes. In the case of a direct current, electrons flow toward one electrode, the cathode. Four electrons combine with each oxygen molecule in the surrounding atmosphere causing the oxygen to enter the ceramic at the cathode and proceed through the ceramic as two individual oxygen ions. The reverse occurs at the opposite electrode (anode) where the two ions form an oxygen molecule as it passes back into the surrounding atmosphere while the released electrons proceed through the metallic conductor to complete the circuit.

Under the influence of direct current, a polarization effect occurs in which oxygen ions within the oxide ceramic are depleted near the cathode and accumulate near the anode. This is undesirable and can effect the electrical performance of the heater inducing a temperature gradient along the heater element and possibly a time varying performance characteristic. Without a surrounding atmosphere containing oxygen molecules (as with a vacuum or reducing environment), the effect is more pronounced as the oxide ceramic is shifted in its stoichiometry (reduced toward the metal constituent).

Under the influence of alternating current, the oxygen molecules in the oxide ceramic merely dither back and forth as the cathode and anode are rapidly interchanging. With AC, the oxide ceramic has sufficient stability against reduction to be used even in vacuum and reducing type environments as well as in oxidizing environments.

Using the Model 40 isothermal furnace as an example, a schematic of a typical control system is shown in figure 21. The preheater is designed to achieve ignition temperatures with a nominal voltage of 120 volts. Unless a current limiting controller is used, the preheater should be connected to a 120 volt AC supply to prevent excessive current flow. The main (ceramic) heater is also operated with a 120 volt AC supply, however, a 240 volt AC supply can be used to provide higher voltage to improve ignition response.

Figure 21 shows that the preheater is operated closed loop using a thermocouple feedback signal. The controller includes a temperature indicator for monitoring the preheater temperature. Power for the air blower is provided by the main power switch so that cooling air flow is assured prior to operation of the furnace.

The ceramic heater is also normally operated closed loop although it can be operated on a power schedule having previously established a power versus temperature calibration. Closed loop control is recommended, however, to prevent accidental over-temperature of the furnace. Feedback signals which can be used include the following:

1. Ceramic heater element resistance
2. Ceramic heater element current
3. Thermocouple output
4. Pyrometer output

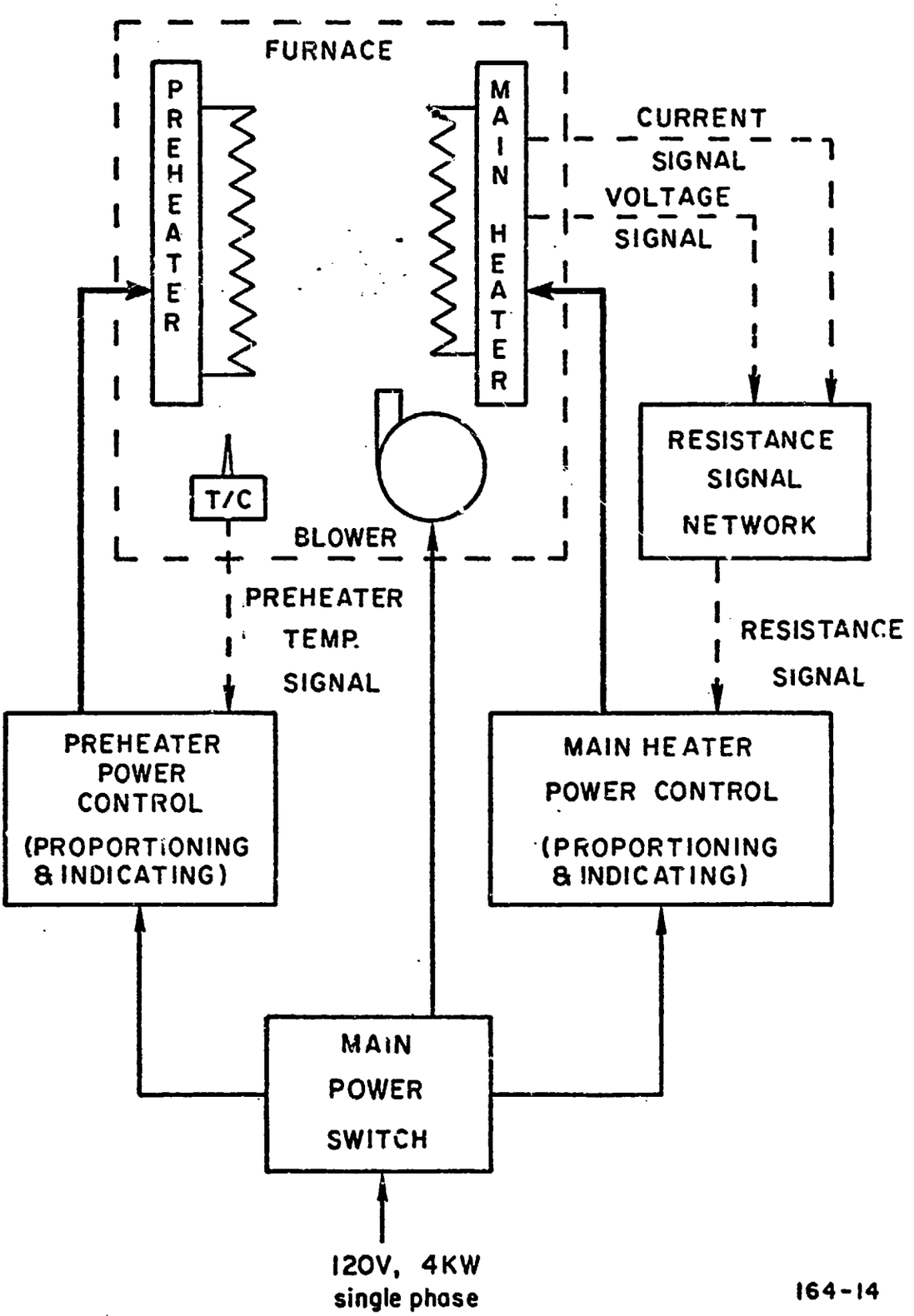


Figure 21.- Isothermal furnace control schematic.

Resistance of the ceramic heater derived from a voltage and current signal offers the most effective means of furnace cavity temperature control, especially at very high temperatures where thermocouples become inadequate (incapable of withstanding the very high temperatures and/or suffering from oxidation). Recalling the heater element resistance versus temperature characteristic given in figure 1, the ceramic heater resistance provides an accurate signal for cavity temperature control. A resistance signal network which ratios the heater element voltage and current is used to derive a resistance signal which is used to command the ceramic heater power controller. Data are presented in the Experimental Performance Section to indicate how well ceramic heater resistance represents cavity temperature even with varying thermal loading conditions. Thermal loading condition as used here is meant to include those effects on furnace power consumption associated with the penetrations into the cavity relative to an unloaded-completely sealed furnace cavity. That is, the user modifies the power vs temperature characteristic with his particular experimental setup.

Voltage of the ceramic heater element is relatively constant over the operating temperature range of the heater in the case of a fixed thermal load situation. Therefore, heater element current also can be used as an effective feedback signal. This simplifies the control feedback signal adaptation considerably over the first method of using a computed heater element resistance. A calibration of the current versus cavity temperature is required for the particular experimental setup. Where thermal loading of the cavity is unchanged from the calibration value, the current feedback signal can be more accurate than the resistance feedback signal in not having to compute a parameter from two measured parameters (voltage and current).

Thermocouples appropriate to the furnace cavity atmosphere are required if this method is to be used. In an oxidizing atmosphere, Pt-6Rh/Pt-30Rh is recommended for long service to 1750°C and short service to 1800°C. Iridium-Rhodium can be used for higher temperatures, however, this thermocouple suffers from oxidation of the iridium and is not recommended for long exposure times. It should be mentioned that while the ceramic heater element concept has proven applicability to atmospheres other than oxidizing ones, the Model 40 furnace used to demonstrate the ceramic heater element concept was used only with an air environment. With a suitable environmental enclosure, other atmosphere environments can be used. Tests on sample heater elements were conducted in a variety of atmospheres for the purpose of qualifying the furnace concept in these atmospheres.

A pyrometer having a suitable output signal to adapt to a process controller can be used to control cavity temperature. In this case there are no restrictions on temperature or kind of atmosphere within the cavity. With a pyrometer, however, an unobstructed view of the cavity must be provided. In the event the view is obstructed, even partially as by smoke for example, a false signal - lower than actual conditions - will result and possibly cause burn-out of the furnace due to over-temperaturing. The best protection in this situation is to use a current limit setting based on a pre-calibration for the particular thermal load condition to back up the pyrometer feedback loop.

For laboratory operation, the furnace can be operated manually using auto transformers (Variacs) for voltage control. Figure 22 shows such a setup for the isothermal furnace operating from a 120 volt AC supply. The Variacs are wired for 0-140 volt operation for greater flexibility. The preheater resis-

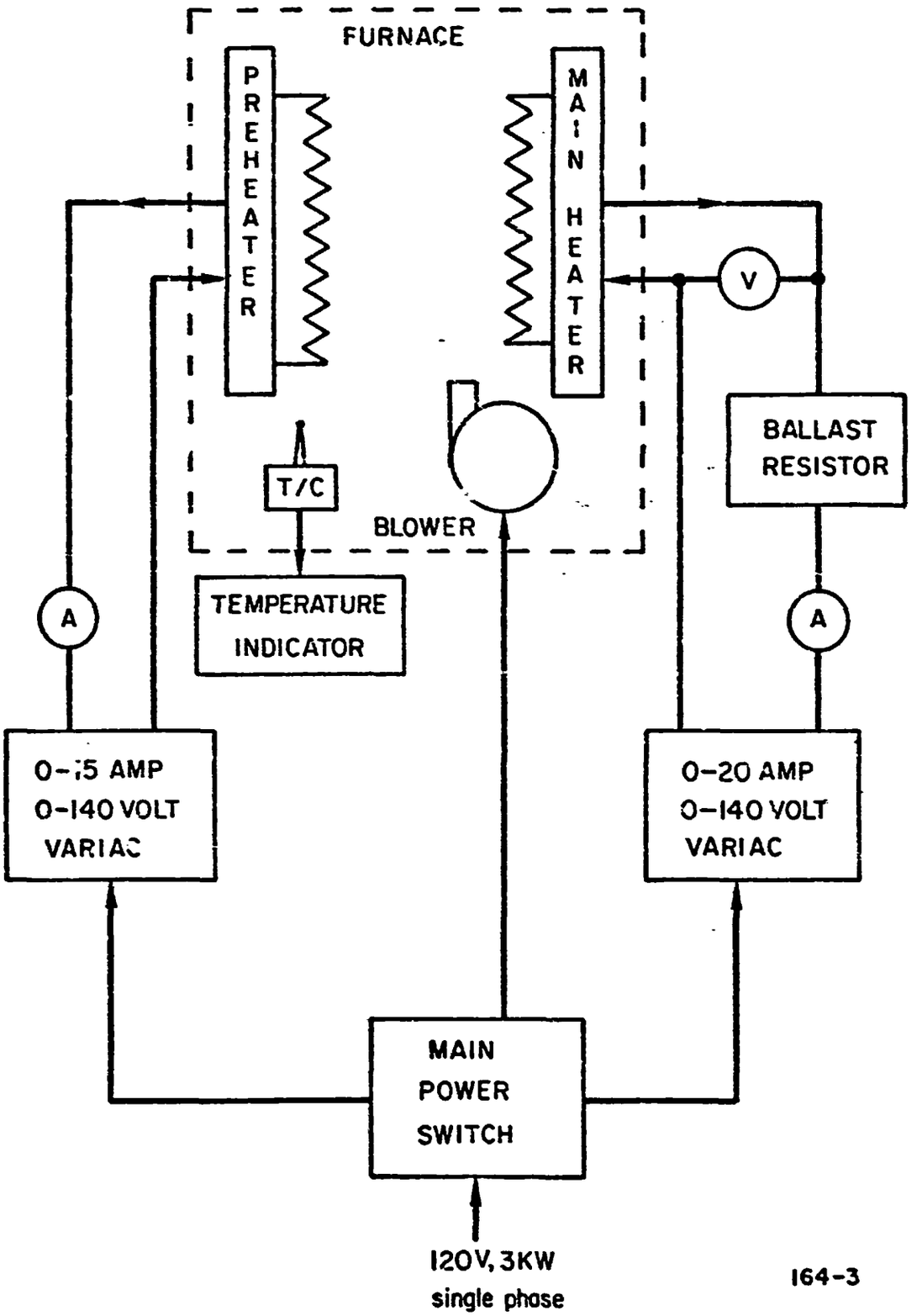


Figure 22.- Isothermal furnace manual control setup.

tance is nearly constant over its temperature operating range and only one parameter (current or voltage) need be monitored. The preheater thermocouple is put on a temperature indicator to show core temperature.

Both current and voltage instrumentation are provided for the ceramic main heater. Because of the "negative resistance" characteristic of the conducting ceramic heater, a ballast resistor must be used in series with the ceramic heater to provide power stability. The ballast resistor should have a resistance of approximately 20% of the desired operating point resistance of the ceramic heater. To prevent too rapid a power increase, the ballast resistance should be larger at lower temperatures and reduced accordingly as the ceramic heater resistance decreases (cavity temperature increasing).

Solid-state (SCR) type controllers are most convenient for closed loop operation. Either the phase angle firing or zero-crossing firing types can be used. The zero-crossing firing type of controller has the advantage of less electro-magnetic interference where EMI is a problem but has the disadvantage of not providing current limiting to reduce start up power surges or being able to be used in a current feedback mode. For most applications, the phase angle firing type of control is acceptable and has greater versatility. EMI is controlled by shielding all power leads with metallic conduit and using the protective (shield) cage over the furnace housing as shown in figure 6.

Space Flight Considerations

The control aspects of adapting the conducting ceramic heater element furnace for space flight applications was studied during this program. Eventual use of this furnace concept for materials processing within the Shuttle/Spacelab programs is possible. Most likely alternating current power would be available and power control philosophy would be much like that discussed in the previous section.

Adaptation of the ceramic furnace to sounding rocket flights requires a different power control philosophy in that power is battery supplied and is therefore DC. In addition to power adaptation, the study includes an evaluation of instrumentation conditioning equipment compatibility with the vehicle electronic recording and flight telemetry system which requires a 0 to 5 volt DC input. The Black Brant VC sounding rocket was used as a reference vehicle for the study.

A baseline system was defined relative to the isothermal ceramic furnace concept having as a groundrule the requirement that the ceramic heater element be brought to the desired operating temperature prior to rocket launch. That is, the preheater is to be operated from a ground controller to achieve ceramic heater ignition and the ceramic heater is to be powered up to the operating condition also from ground control. This is necessary because of the relatively short zero-gravity time (5 to 7 minutes) in the sounding rocket flight.

Table V presents a controller and instrumentation preliminary specification for a ceramic furnace requiring a maximum of 2500 watts of power from a 28 volt DC battery supply. For cost effectiveness, control system accuracy is set reasonably loose from a furnace control point-of-view while being tight relative

TABLE V

Preliminary Specifications for

MAIN POWER CONDITIONING, CONTROL AND

TELEMETRY SIGNAL CONDITIONING

Controller Input Voltage	24 - 32 VDC
Controller Input Signal Operating Range	0-20 millivolts
Control Accuracy	± 100μ volts
Control Stability	± 20μ volts per 10 minutes
Control Proportional Band	0.1 to 5.0 millivolts
Main Heater Power	
Voltage	80-120 VAC *
Power	2500 watts maximum average *
Frequency	10 KHz
Duty Cycle	0-100%
Secondary Switching	Zero Crossing
Efficiency	≥88%
Operating Temperature Range	
DC/AC Inverter Transformer	0-50°C
Electronics	0-70°C
Interfaces	
Input Signals Required	
Temperature Programmer	
a) 10 line binary	
b) 12 line BCD	
c) Two discrete programmed temperatures by switch closures or logic levels	
Output Signals	
Analog Telemetry	
Channels	29 plus calibration and frame sync.
Signal Range	0-5 VDC
Coupling	Differential
Sampling Rate	1 Frame/Sec. minimum
Required Accuracy	1% of FS or better

* The controller output sees a furnace with a resistive load which may drop to 2.0 ohms and require an average power of 2500 watts maximum (average voltage of 70.7 volt and average current of 35.4 amps, for example). To provide a slight reserve in this case, the inverter transformer would be wired for an 80 volt output resulting in a 78% duty cycle with a peak current of 40 amps. For higher furnace load resistance, the transformer would be wound for a correspondingly higher voltage not expected to exceed 120 volts. Peak current would be correspondingly lower.

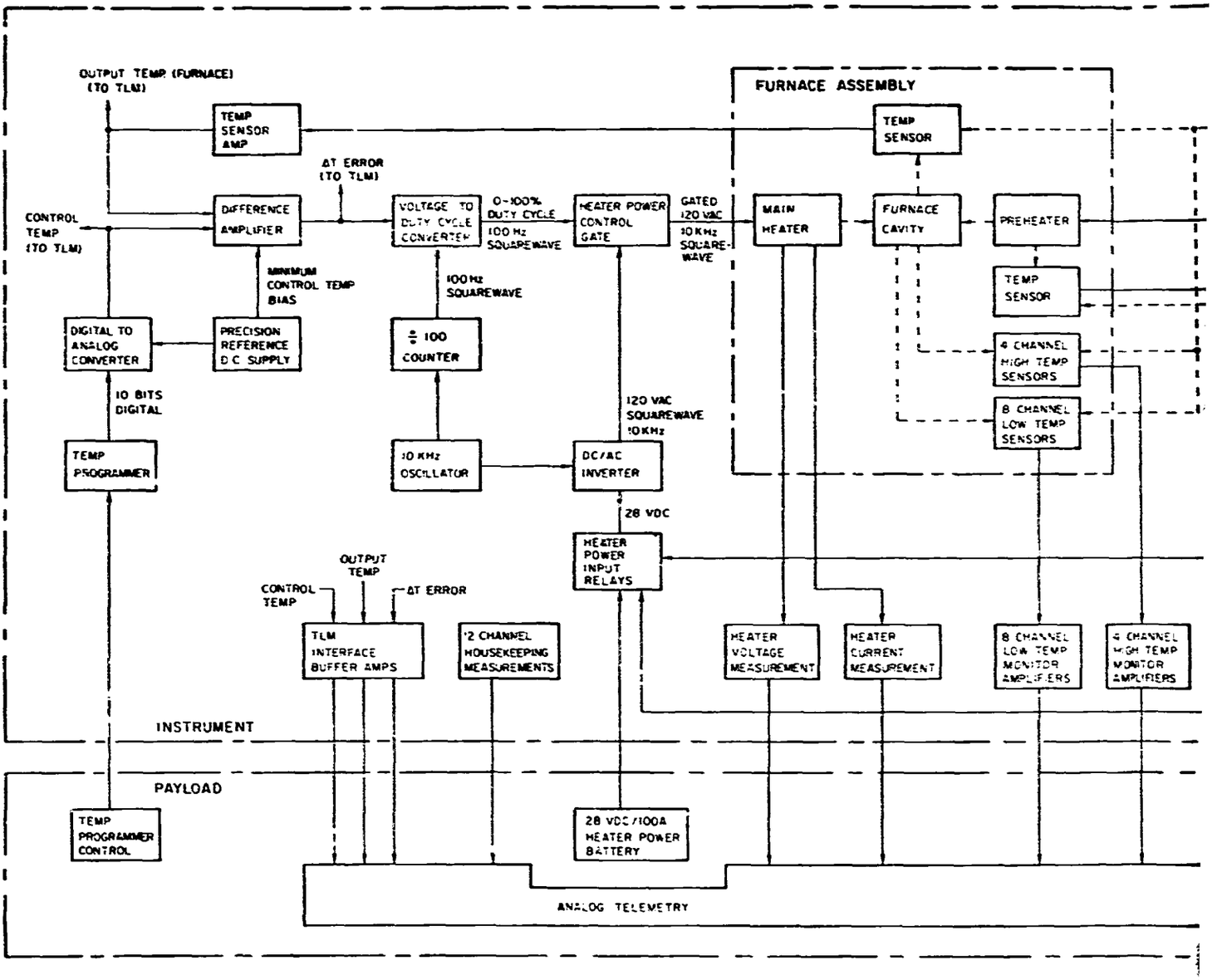
to typical experimenter's requirements. Furnace and control parameter monitoring has been included which is sufficiently broad to give a high degree of yield to any required diagnostics which might be called upon in the event a first or early flight suffers abnormal problems. Temperature programming capability is included to add flexibility to the furnace usefulness and also to allow the use of furnace heater thermal capacitance to bridge any transient power gaps which might arise. The baseline system is described as follows.

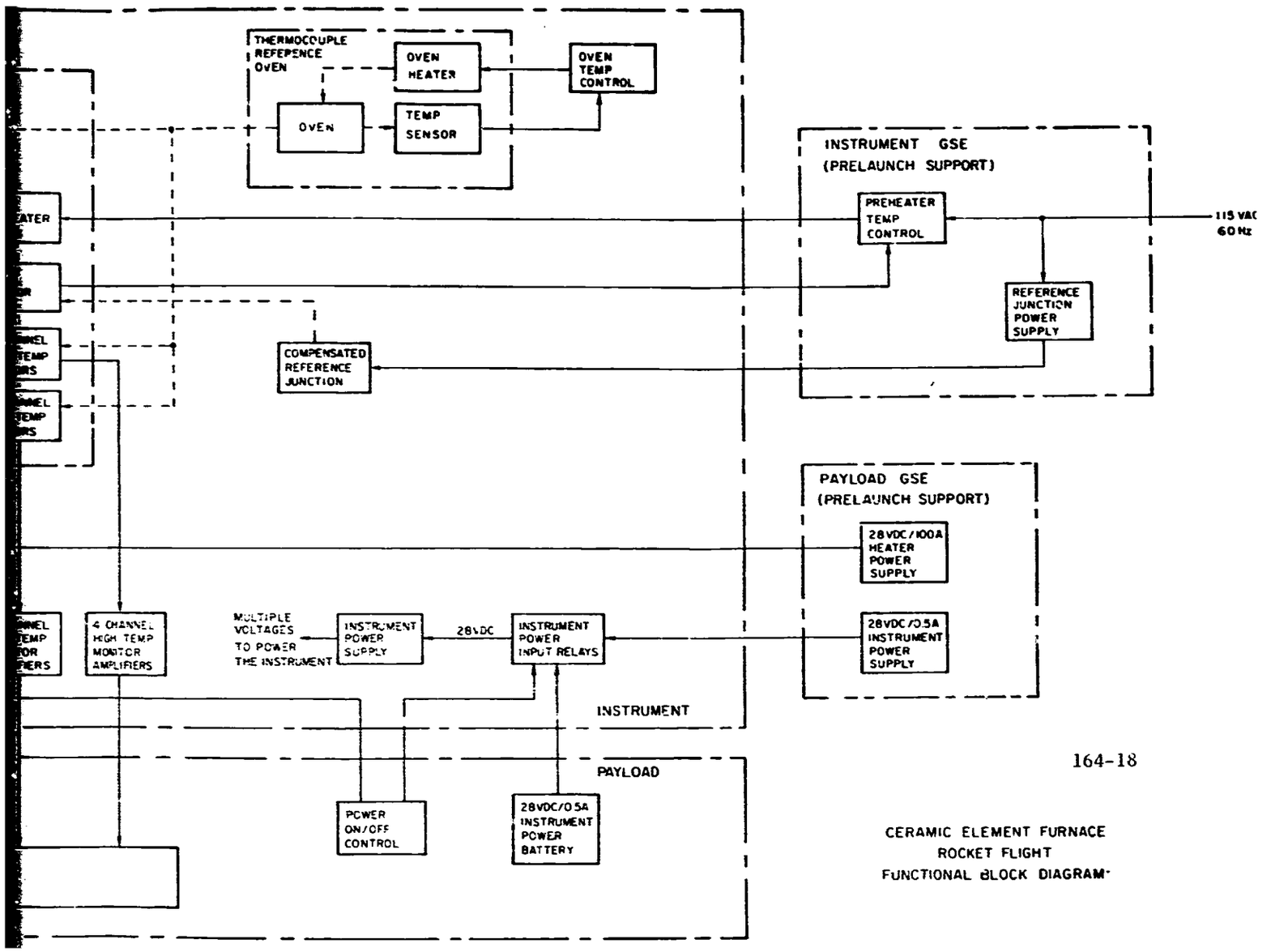
The functions of the electronics subsystem are to convert the nominal 28 VDC mains power for instrument power and for heater power, to maintain the furnace set temperature by controlling heater power, to accept switch closures or digital signals, to set the furnace temperature, to monitor furnace and electrical signals and condition them for telemetry, and to provide GSE functions. Figure 23 is a block diagram of the furnace system study baseline design. The elements are described as follows:

Furnace Controller.- Referring to the block diagram, the baseline design employs a thermocouple to sense the furnace temperature. Ceramic element resistance, derived from voltage and current measurements, can be used as an alternate feedback signal and is recommended. For the baseline design, the thermocouple type can be optimized for the specific experiment. For very high temperature operation (to 2200°C), iridium-rhodium thermocouples or tungsten-rhenium thermocouples sheathed against the oxidizing environment would be applicable. A platinum-rhodium thermocouple standing back from the furnace cavity can also be used by correlating its response to the cavity temperature, although this approach would have a slower response. Response of the resistance feedback signal is essentially instantaneous.

The thermocouple signal varies from zero to 20 millivolts full scale for the range of furnace temperatures and is amplified by an operational amplifier (temperature sensor amplifier) having a maximum offset drift of 14 microvolts, corresponding to approximately 1.5°C, in the environmental temperature range of 0-70°C. In the "difference amplifier" the thermocouple signal is compared to a DC voltage derived from the digital "temperature programmer" through the "digital to analog converter." A fixed offset derived from the "precision reference DC supply" is used to compensate the 100°C thermocouple reference over temperature either to 0°C or to any desired lower bound for the furnace temperature setting. The resulting voltage difference, or error signal, which is proportional to the difference between the furnace sensor temperature and the set temperature, is used to modulate the duty cycle of a 100 Hz square wave in the "voltage to duty cycle converter." This square wave will be high for zero to 100% of each cycle, proportional to the error signal, and is a low power analog indicating the proportion of time for which power is applied to the furnace.

The actual high power furnace drive is provided by the "DC/AC inverter" shown in the block diagram. The furnace power is assumed to be about 120 volts RMS AC due to the characteristics of the ceramic heating element, thus requiring transformer coupling from the nominal 28 volt battery supply. A minimum transformer weight can be achieved with ferrite cores operated at high frequency, in the 5 to 15 KHz range. Also, in order to improve power handling efficiency, raw power from the battery mains is utilized for the inverter input. The baseline inverter design employs a ferrite transformer driven at 10 KHz by the oscillator shown in the block diagram. The transformer could be eliminated if a higher voltage battery could be provided on the sounding rocket.





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CERAMIC ELEMENT FURNACE
ROCKET FLIGHT
FUNCTIONAL BLOCK DIAGRAM

FOLLOUT FRAME 2

The 120 volt square wave inverter output is gated to the furnace heating element by Triac gates in the "heater power control gates" which are driven by the "voltage to duty cycle converter." The high level of the variable duty cycle 100 Hz square wave enables the high power Triac gates to fire. Thus, during each 10 millisecond interval, an integral number from zero to 100, of 10 KHz-120 volt square wave cycles is applied to the ceramic heater. This power is switched only at zero crossings of the high power waveform.

Not shown in figure 23 is a current limiter loop which derives a signal from a current transformer in the heater element measurement circuit. This serves to limit current to a preset value and protect the controller high power components.

The furnace temperature can be set by several methods as follows:

1. For normal ground operation, thumbwheels in the GSE can be set to a decimal digital number from zero to 999. This number is generated as a BCD code and transmitted to the instrument through the umbilical. In the instrument the BCD number is converted to a binary code in the "temperature programmer" and then converted to a DC voltage by the "digital to analog converter." The DC voltage is then applied to the difference amplifier as previously discussed.

2. A BCD or straight binary code can be input to the instrument temperature programmer by the payload programmer control. This setting may be pre-programmed or set by radio command.

3. Two or more switch closures or logic lines may be used to set two or more discrete temperatures. These inputs also may be pre-programmed or commanded.

In order that the experimenter can turn the furnace/controller power off, a 28 VDC relay coil power interface is provided which is closed by the experimenter's sequencing timer. Thus, the furnace can be pre-programmed off prior to reentry and after completion of the heating phases of the experiment.

The following five controller parameters should be monitored and telemetered:

1. Temperature sensor amplifier output

2. Control temperature (set temperature)

3. Error signal (ΔT error)

4. Ceramic heater voltage

5. Ceramic heater current

Preheater Control.- The preheater element is a platinum resistance heater with temperature sensed by a thermocouple. This preheater can be controlled

by a commercial thermal controller in the instrument GSE located in the blockhouse or on the pad. In order to avoid a long run of thermocouple wire, the thermocouple can be terminated in an electronic temperature "compensated reference junction" in the instrument. Copper wire can be used to transmit the signal to the controller. Power for the electronic reference can be provided through the umbilical in order that the preheater may operate independently of the flight power source.

Temperature Measurement.- The instrument may contain up to fourteen thermocouples, one in the main heater loop, one in the preheater loop, and twelve used for monitoring temperature of the furnace assembly. The reference temperature for all but the preheater is provided by the "thermocouple reference oven." An alternative approach to providing the reference would be individual electronic references. The cost of this approach would be higher. If the signals can be multiplexed, a single synchronously multiplexed electronic reference for the twelve monitors would be cost effective. In this case a separate electronic reference for the main heater controller would be preferred. The offset drift of the amplifiers used for the temperature monitor thermocouples corresponds to less than 7°C measurement error over the 0 to 70°C environmental temperature range.

Housekeeping Measurements.- Monitor circuits and telemetry inferfaces should be provided for verifying instrument performance and for routine ground diagnostics. These housekeeping functions would include the following in addition to furnace controller functions and furnace assembly temperatures:

1. Reference Oven Temp.-Temp.
2. Precision DC Supply - Voltage
3. 28V Heater Power - Voltage
4. 28V Instrument Power - Voltage
5. Control Temp. Bias - Voltage
6. 10 KHz Oscillator - Frequency
7. Inverter Transformer - Temp.
8. 120 VAC square wave - Voltage
9. Instrument Electronics - Temp.
10. +5V Supply - Voltage
11. +15V Supply - Voltage
12. -15V Supply - Voltage

All of these signals should be converted to 0-5VDC signals and sent to payload telemetry.

Instrument Power Supply.- The inverter for this supply has three secondary

windings, one each for the +5 volt regulated logic supply and the +15 volt and the -15 volt regulated analog electronics supplies. Additionally, unregulated +15 and -15 volts are supplied to the oven heater and -20 volts unregulated to the analog switches. The supply input is current limited.

Telemetry.- The 29 functions to be telemetered are individually hard wired to the vehicle telemetry. Analog FM-FM telemetry with a full-scale accuracy of 1% is assumed with no multiplexer located in the experiment package. An accuracy of 0.5% of full-scale is desirable for the temperature monitors but is not required. Locating a multiplexer in the instrument would be desirable.

Present State of Development.- All of the electronic approaches discussed utilize current commercial practice; however, their implementation does require careful application of good engineering practice. For example, in order to meet the furnace control accuracy requirement, an amplifier thermal offset stability of 1.4 microvolts per °C is required. Three commercial IC operational amplifiers have been found which exceed the requirement. Two are specified at 0.2 microvolt/°C. The only area of development is that of the high power inverter, and even here similar supplies have been manufactured commercially. Overall, the electronic techniques discussed are highly developed and need only be adapted to these particular circuits.

Providing a separate high power battery for the furnace inverter would help reduce electromagnetic interference. A higher voltage battery could be provided and could contain the same number of cells, hence, not be any larger in volume or weight. The main power lines would carry a much lower current making the electronics design easier and it would eliminate a heavy transformer.

Weight and size estimates.- The study control system depicted in figure 23 for a 2500 watt ceramic heater power was evaluated in sufficient detail to define the number of components required. These in turn were projected into typical packaging configurations to estimate the weight and size of a typical sounding rocket flight controller. Including the transformer but not the 28 volt battery, the 2500 watt isothermal furnace controller with instrumentation is estimated to have a weight of 9.1 Kgm (20 pounds) and a volume of 6600 cubic cm (400 cubic inches). The isothermal furnace would have a weight of about 15.9 Kgm (35 pounds) and a volume of 11500 cubic cm (700 cubic inches).

Estimates were also made for the case of an axial gradient furnace having three separate heater element zones requiring in flight control. These were sized for 900 watts each of maximum power. The same furnace housing and preheater subassembly are used with changes only in the main heater configuration. Furnace weight and size are basically unchanged. A three-900 watt loop controller, however, will weigh approximately 16.3 Kgm (36 pounds) and occupy 9000 cubic cm (550 cubic inches).

EXPERIMENTAL PERFORMANCE

Ceramic heater element tests were conducted on sample elements as well as on the completed furnace elements (figures 13 and 19). Electrode interfacing is well modeled with the single elements and results obtained are representative of the multiple tube assemblies themselves.

Two isothermal furnace elements were tested, one being the figure 13 element. The other had a cavity diameter of 2.6 cm and length of 7.0 cm. Two ceramic elements for the axial gradient furnace were fabricated. The one shown in figure 19 has an alternate tubular arrangement of long and short tubes providing a cage like structure. The second was similar in size to the figure 19 element but with all tubes of the same length giving it the appearance of the figure 13 element. This element was not tested extensively. The following sections discuss the experimental results of the various element tests and furnace assembly tests.

Sample Heater Elements

The sample heater elements are made from tubes of stabilized zirconia having an OD of about 2.9 mm and an ID of about 1.3 mm used to fabricate the isothermal furnace heater element and tubes of about 1.6 mm OD by about 0.6 mm ID used for the axial gradient furnace elements.

The tubular elements are formed by extrusion. Electrodes are attached in the same manner as those on the furnace heater element assemblies. Braided platinum wire is used for the electrodes and attached with stabilized zirconia cement. The cement is applied as a paste using a suitable binder and then fired to sinter the cement in place over the platinum electrode wires.

The stabilized zirconia is made at ARTCOR by a proprietary process and results in a conducting ceramic element which can withstand extreme thermal shock and many heat-up cycles. These materials are routinely evaluated as sample conductors in cyclic tests to verify their integrity. Cycling tests to very high temperatures result in earlier electrode failures rather than ceramic failure. Therefore, more meaningful evaluation of the ceramic cycling capability is obtained at a nominal lower temperature of about 1600°C. Relative to a furnace condition, the sample heater elements are operated free-standing, without insulation, resulting in more severe wall temperature gradients. The increased wall temperature gradient is the result of increased thermal losses (radiation and convection) from the surface of the free-standing element. In a furnace application, the per unit area losses are reduced considerably by the surrounding insulation.

Failures experienced are generally associated with electrode wire failure by depletion of the wire material due to vaporization and oxidation. Therefore, the temperature of the ceramic tube adjacent to the electrode interfacing region is most critical. In general, with a ceramic tube surface temperature of 1600°C adjacent to the electrode region, a lifetime of about 4000 hours can be expected for steady-state conditions. Dropping to 1500°C, the lifetime is extended beyond 6000 hours. At 1700°C, lifetime is reduced to about 2000 hours. With hourly cycles included, the lifetime is reduced approximately 1500 hours for each of the above conditions. It is not likely that the furnace would be used in such a severe cycling condition and lifetimes approaching the steady-state values are to be expected. The above values are those corresponding to operation with an air environment.

Accelerated steady-state life tests conducted with air at a ceramic surface temperature of 1940°C results in electrode failure in about 500 to 600 hours.

Tests are being conducted under an Air Force contract (reference 10) to evaluate the conducting ceramic heater elements in various atmospheres. In one of these tests an identical element to that above was operated in argon for 3000 hours at a surface temperature of 1940°C without failure. In the absence of oxidation of the platinum electrode, lifetime appears to be extended a factor of 5 or more. Oxidation and not vaporization is, therefore, the life determining mechanism of the platinum electrodes relative to operation in air.

Another conclusion from the argon tests is that the conducting ceramic heater elements function well in an inert environment with AC. 60 Hz AC is used in testing the ceramic heater elements. Another test series as part of the reference 10 program included operation of the ceramic elements in a reducing environment.

Also as part of the reference 10 study, a sample heater element was placed in a quartz environmental chamber and was ignited in air. After two days, the chamber was purged with nitrogen (high purity grade) for two days and then exposed to dry hydrogen (high purity grade). After 31.5 hours on H_2, the heater was unintentionally shut off. During an attempt to re-ignite the heater in an air environment with a flame preheater, the heater element cracked. While the flame preheater exposed the element to severe thermal shocks and gradients, the failure was considered to be due to changes in the oxide stoichiometry (sub oxide on hydrogen reverting toward stoichiometry in air).

A second heater element was subsequently tested and operated 812 hours on hydrogen. Using nitrogen purges between gas changes, the same heater was cycled five (5) times to carbon dioxide and back to hydrogen. Finally the element was exposed to air and secured after 1324 hours of which 956 hours were on hydrogen. Temperature was held at a moderate 1525°C during the test with calibration excursions made from 1325 to 1625°C. 1525°C is a typical electrode condition where lifetime criteria of the heater elements is associated with electrode failure conditions.

Of importance here is that the stabilized zirconia heater elements can sustain cycling between reducing and oxidizing atmospheres. Stoichiometry does appear to change. That is, X in ZrO_x becomes less than 2.0 while on hydrogen as observed by the gray appearance of the first element after it was secured under hydrogen. It, therefore, appears that the zirconia conductor furnace will have a more universal position in being able to be used with oxidizing, inert, and reducing environments.

A sample ceramic heater element was operated on DC to verify the type of problem associated with this kind of power supply. Upon igniting the sample heater element with DC power, a severe circumferential temperature gradient of 200 to 300°C developed in a few minutes. Under AC, the circumferential temperature difference would be no more than 10°C. An axial gradient of 150°C developed as well which would have been nominally about 15°C under AC. The cathode end corresponded to the lower temperature in this situation. These tests were conducted in air. In a reducing atmosphere, catastrophic effects would be expected with DC. This test on DC was with a free-standing - uninsulated heater element. In a furnace cavity, thermal loading would be reduced and so too would be the tendency to generate a maldistribution of temperature. The important factor is that such a tendency exists and is greatly amplified with DC power relative to AC power.

A sample heater element test was conducted to demonstrate the effect of contouring of the element on its surface temperature. Tapering of the ceramic element to achieve a lower electrode region temperature relative to the cavity zone temperature is mentioned in the thermal design discussion in the Axial Gradient Furnace Section. The sample element had a reduced cross section corresponding to an outside diameter of 2.27 mm over a portion of its length. Near the electrodes, the outside diameter was 2.84 mm while the inside diameter of the element was 1.27 mm.

Figure 24 indicates the arrangement of the contouring being closer to the lower electrode than the upper electrode. Temperature profile measurements are shown and indicate that greater than 200°C temperature difference is achieved at about 2200°C with a reduction in diameter of 20 percent. Greater temperature difference would occur with a greater reduction in diameter relative to the diameter near the electrodes.

To better indicate the consequence of contouring of ceramic conductor elements, figure 25 is presented showing how temperature varies with current for a family of element sizes (OD). Experimental data for an element of 2.8 mm OD (0.11 inches) is compared to the calculated values and shows excellent agreement. The figure 25 data are for a free-standing element without thermal insulation in free vertical convection subject only to radiant and convective heat losses to an ambient environment. The dashed line example shows how an element could run at 2202°C (2475K) yet have electrodes which are subjected to only 1927°C (2200K). With the addition of insulation over the higher temperature region, the temperature differential can be further enhanced as discussed in the Thermal Design Sections.

Isothermal Furnace

The smaller of the two isothermal furnace ceramic elements (2.6 cm ID by 7.0 cm long) was used as a model to evaluate the effect of changes in thermal loading on the use of the element electrical resistance as a temperature signal. The model furnace includes a preheater and the cavity was closed at both ends with Zircar insulation plugs. To simulate a large change of thermal loading, the preheater acting like a thermal guard zone when the ceramic heater is conducting, was varied in power.

The model furnace was operated with manual power controls (autotransformers) in two modes. In the first mode the preheater was under power to maintain a preheater temperature of 1200K. In the second mode, the preheater was left off. The corresponding conducting ceramic (main) heater element resistance is shown in figure 26 over a typical range of temperature. Different symbols are used for the preheater on and preheater off cases and show no significant difference in the main heater resistance.

To indicate the main heater power change for the two modes, for example, consider the data points near about 1760K. With the preheater on holding 1200K, the main heater used 236 watts while with the power off (at 868K), the main heater used 325 watts. All of the data points fall on the curve fit within the instrumentation accuracy of the test setup (±1%) and indicate that the ceramic heater element resistance provides an accurate temperature signal suitable for fine control of the furnace.

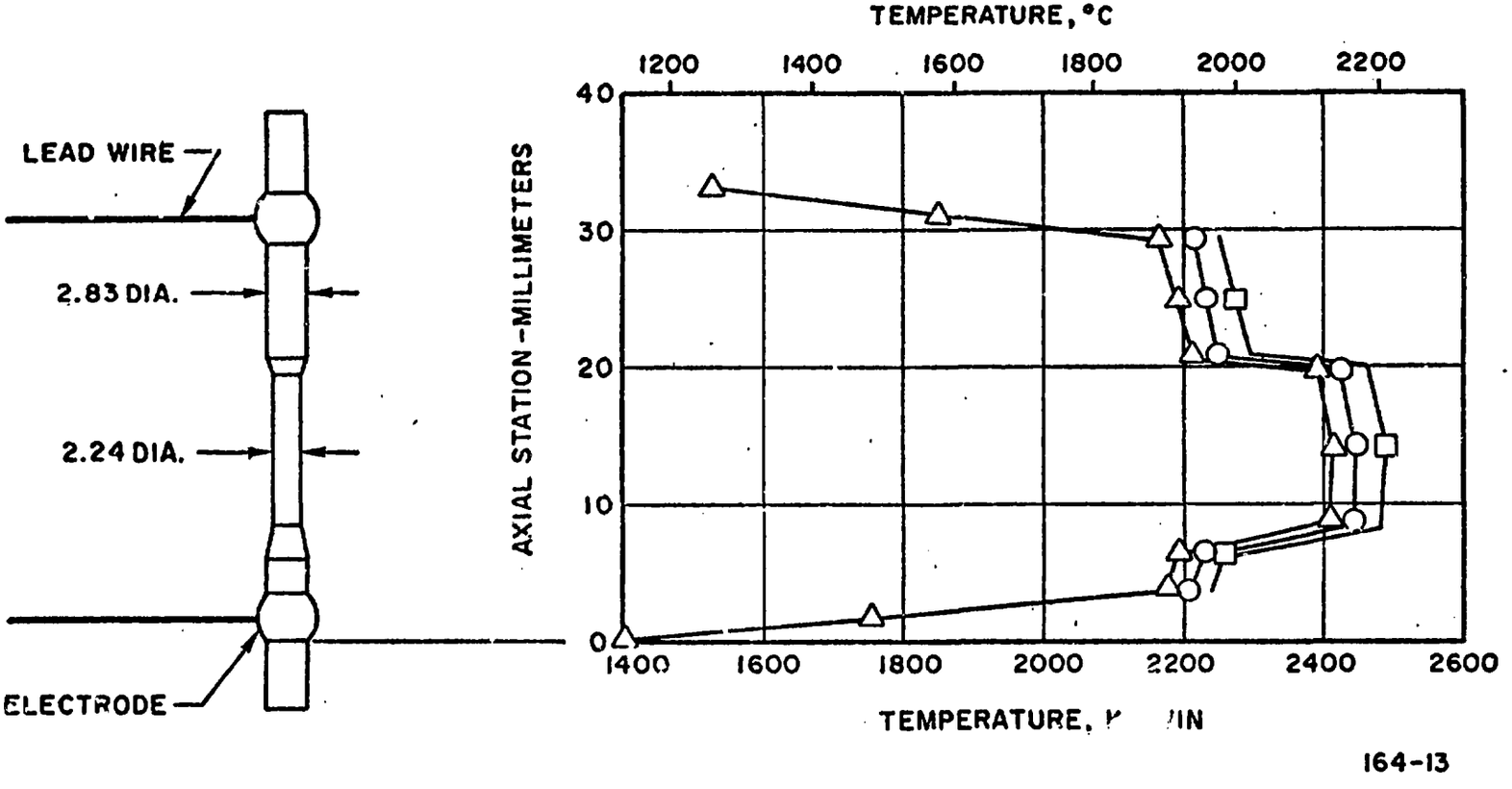

Figure 24.- Effect of contouring on ceramic element surface temperature.

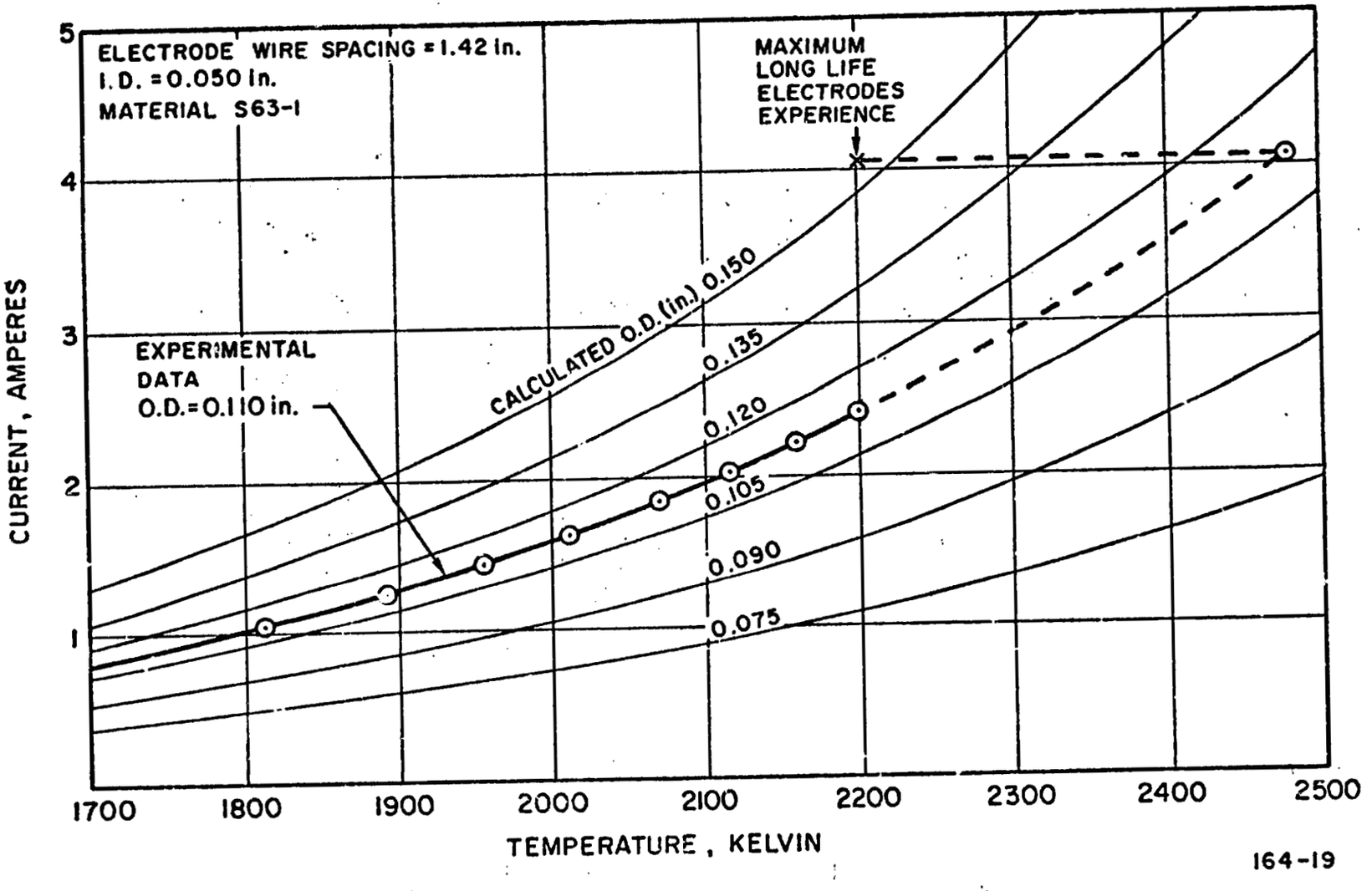

Figure 25.- Effect of ceramic conductor size on the current-temperature relationship.

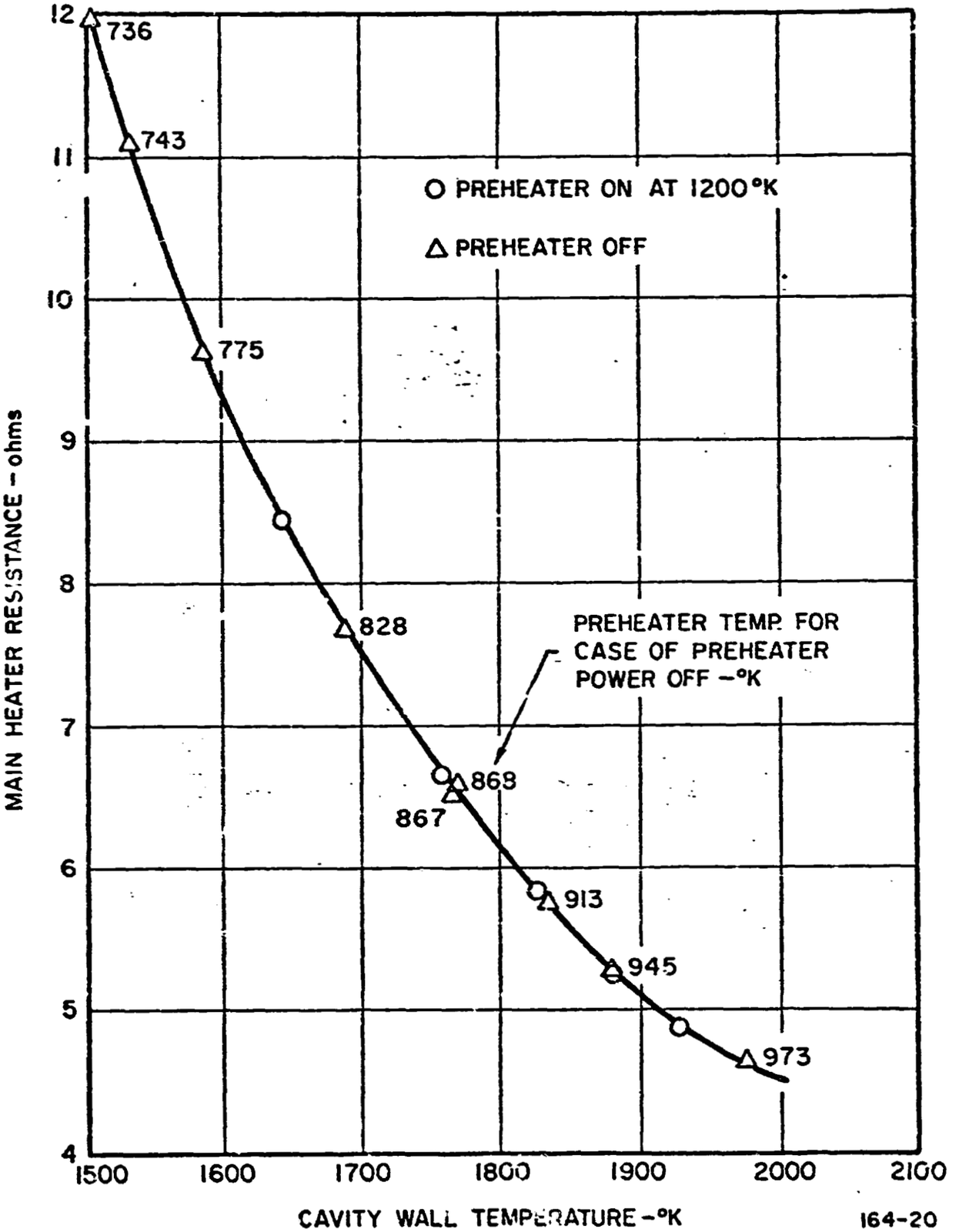

Figure 26.- Isothermal furnace element resistance characteristic.

The following discussion describes the data obtained from tests of the Model 40 furnace (figure 6). A single Research Inc. Thermac Model 625 power controller was used. Both the preheater and main heater were operated with the controller, one at a time, using an autotransformer for the unit not on the controller. In this way, control stability was checked on both heaters. On the preheater, the controller was operated in the temperature feedback mode using the built in type K thermocouple. The main heater was operated in both the current feedback mode in some tests and in the temperature feedback mode in other tests. Temperature was sensed by a Pt-6Rh/Pt-30Rh thermocouple placed in the main heater cavity. This thermocouple was good for measurements just over 1800°C. In some instances the Pt-6Rh leg was melted (1825°C) serving as a calibration point. Other platinum-rhodium alloys were used in this manner to establish higher temperature calibration points.

Table VI summarizes the highlights of the Model 40 furnace test and indicates the platinum-rhodium melt run points taken.

TABLE VI

MODEL 40 FURNACE TEST SUMMARY

Total Time Main Heater Conducting	75.9 hrs
Total Time at 1800°C or Greater	12.3 hrs
Maximum Cavity Temperature Achieved	1940°C
Platinum-Rhodium Alloy Melt Runs Made	
Pt-6Rh (1825°C)	2 runs
Pt-10Rh (1851°C)	1 run
Pt-13Rh (1867°C)	2 runs
Pt-20Rh (1904°C)	2 runs

Figure 27 presents the electrical characteristic of the preheater and the resulting steady-state temperature. The main heater cavity temperature which results at various steady-state preheater conditions is indicated in Figure 28. Figure 29 shows how the main heater resistance varies with main heater cavity temperature. An ignition region is indicated where main heater resistance is from 100 to 1000 ohms. Having preheated the main heater to 600°C or more, the main heater will begin to conduct electricity and heat itself with nominal applied voltages (less than 150 volts). This self-heating condition is referred to as ignition of the main heater element. Ignition is possible at cavity temperatures below 600°C with correspondingly higher applied voltages; however, it is recommended that the 600°C condition be reached to avoid having to apply unnecessarily high voltages to the main heater element.

In terms of the 600°C main heater ignition condition, one can trace back through figure 28 to figure 27 and find that a minimum preheater voltage of 109 volts is required for normal ignition conditions. At 109 volts, the time

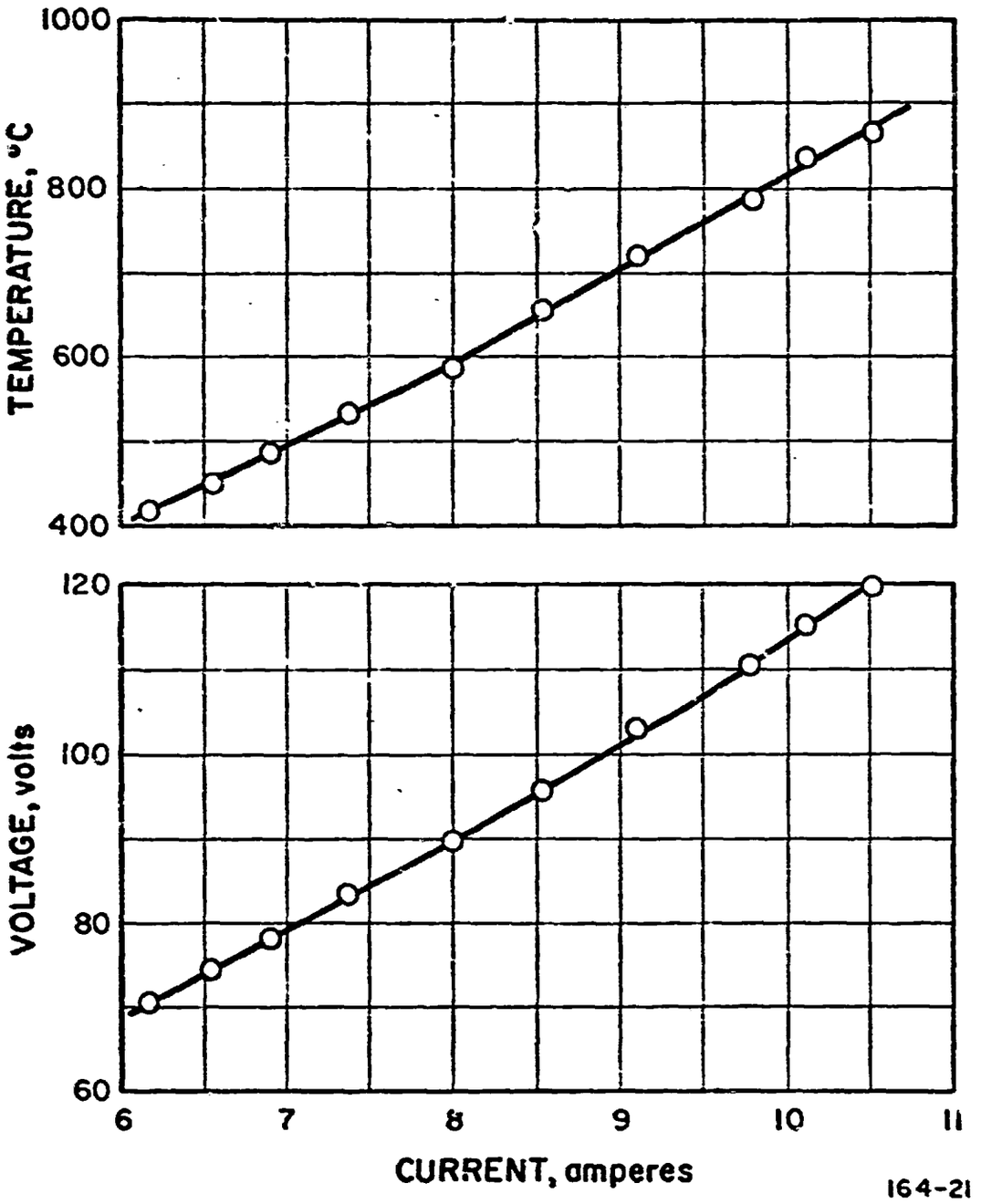

Figure 27.- Model 40 zirconia conductor furnace preheater steady state performance.

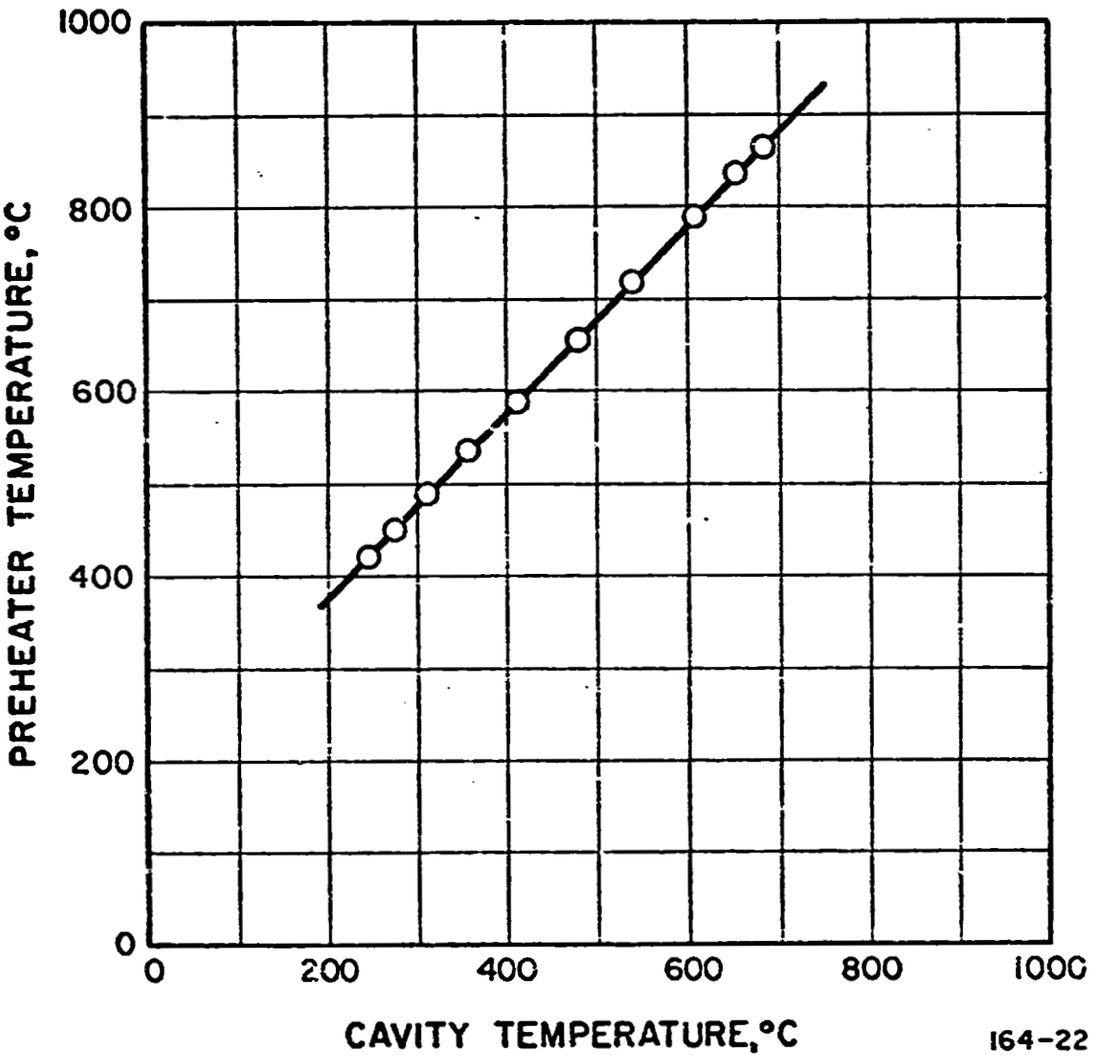

Figure 28.- Model 40 zirconia conductor furnace cavity temperature for preheating only.

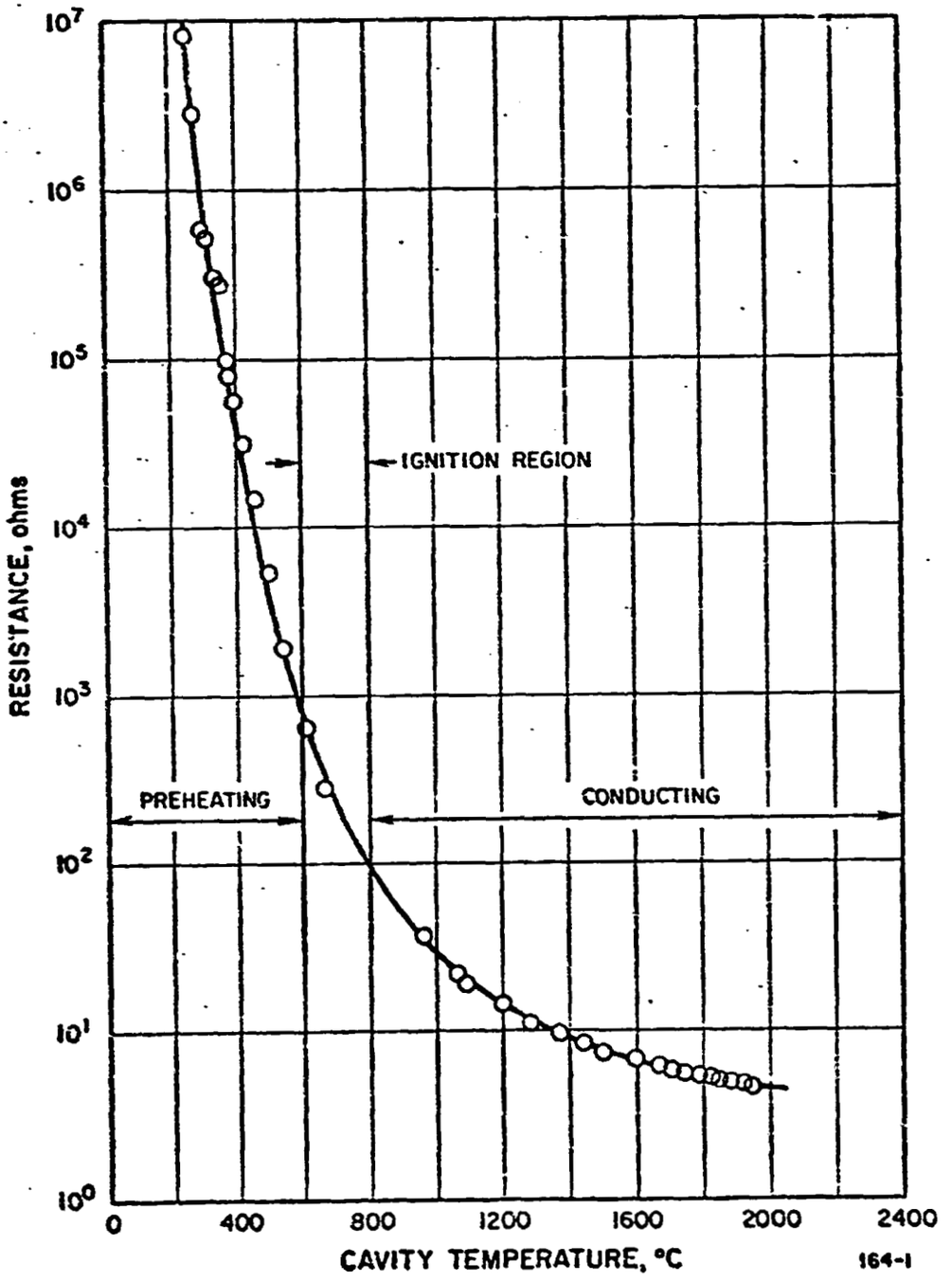

Figure 29.- Model 40 zirconia conductor furnace ceramic heater element resistance.

to reach, say a 1000 ohms main heater resistance, would be long. Figure 30 indicates the time required to reach normal ignition conditions with higher preheater voltages. For example, the 1000 ohm condition is reached in about 30 minutes with a preheater voltage of about 123 volts.

Figures 31 and 32 present main heater electrical characteristics and the corresponding main heater cavity temperature. In addition, the effect of various preheater conditions for current below 10 amperes is shown where the preheater was powered simultaneously with the main heater to hold the preheater power at 500 and 700°C. Above a main heater current of 10 amperes, the preheater power was off. Some resulting preheater temperatures are indicated in the above 10 amperes range as a result of heat conducting radially outward from the main heater cavity to the preheater core. Data were obtained for a main heater cavity temperature to 1940°C (2213K). Performance of the Model 40 furnace is projected (by extrapolation of the test data) at a 2200°C (2475K) condition to be 18.2 amperes at 77.0 volts corresponding to 1400 watts of power.

Figure 33 presents an enlargement of the high temperature end of the main heater resistance curve of figure 29. Included are data for the two preheater temperature conditions of 500 and 700°C. Holding the preheater at these two different temperatures represents effectively a different thermal loading on the main heater element. That is, the main heater power requirement is affected by an external factor, in this case the preheater temperature, rather than just having a normal trend of power versus cavity temperature. The figure 33 data indicate that main heater resistance is affected little if any by nominal changes in external thermal loading. Thus a main heater resistance signal would provide an ideal control feedback signal for applications where thermal loading might change as noted relative to figure 26 as well. In figure 32, between 4 and 7 amperes, the effect of thermal load change on cavity temperature caused by the two preheater conditions is apparent. For applications where the thermal load characteristic is not changing except with cavity temperature, current feedback provides a simpler control signal than does resistance feedback.

Transient response was observed in the 1600 to 1800°C range for step changes in power. Using a Variac (auto-transformer) with a suitable ballast resistor in series with the main heater, the current was stepped between 11.65 and 13.15 amperes corresponding to 1728 and 1838°C steady-state conditions as shown in figure 34. Stepping up in power (heating up) resulted in a time constant (63.2% response) of 75 seconds. Stepping down in power (cooling off) resulted in a time constant of 95 seconds. These responses are for a bare Pt-6Rh/Pt-30Rh thermocouple located in the center of the furnace cavity. Heater element response is necessarily faster assuming some lag occurs in the thermocouple response.

Using the Research Inc. Thermac 625 power controller, similar transients were observed, in this case between 1630 and 1730°C. In the current feedback control mode, responses similar to those above occur as the step would be between discrete current settings. In the temperature feedback mode with the cavity located thermocouple providing a feedback signal, faster responses are realized since the controller can operate outside of power levels corresponding to the steady-state power levels for 1630 and 1730°C. For example, in a cool-down step from 1730 to 1630°C, the controller goes to zero power until 1630°C is approached closely (depending upon the controller gain settings).

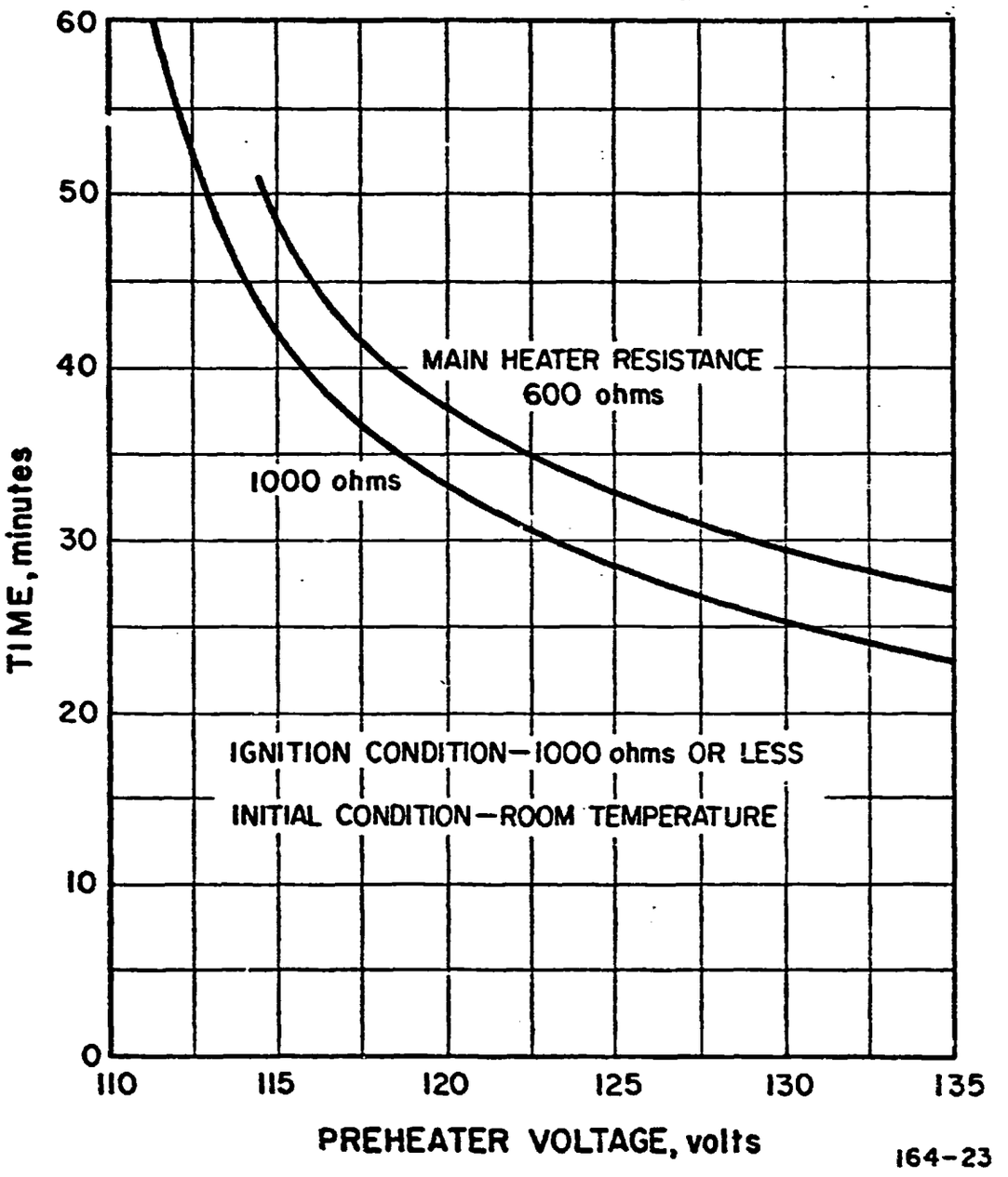

Figure 30.- Model 40 zirconia conductor furnace time to reach main heater ignition condition.

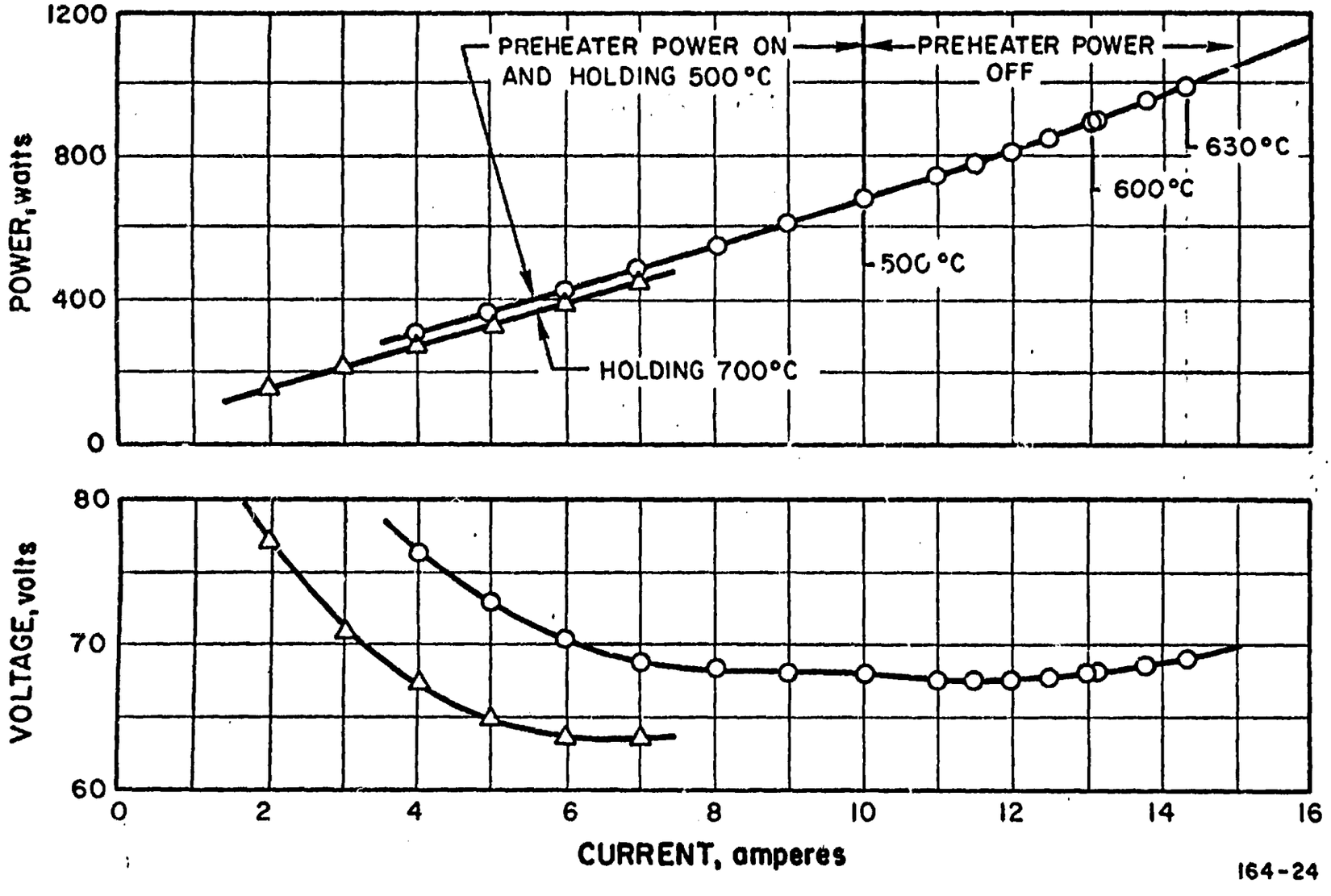

Figure 31.- Model 40 zirconia conductor furnace main heater characteristics.

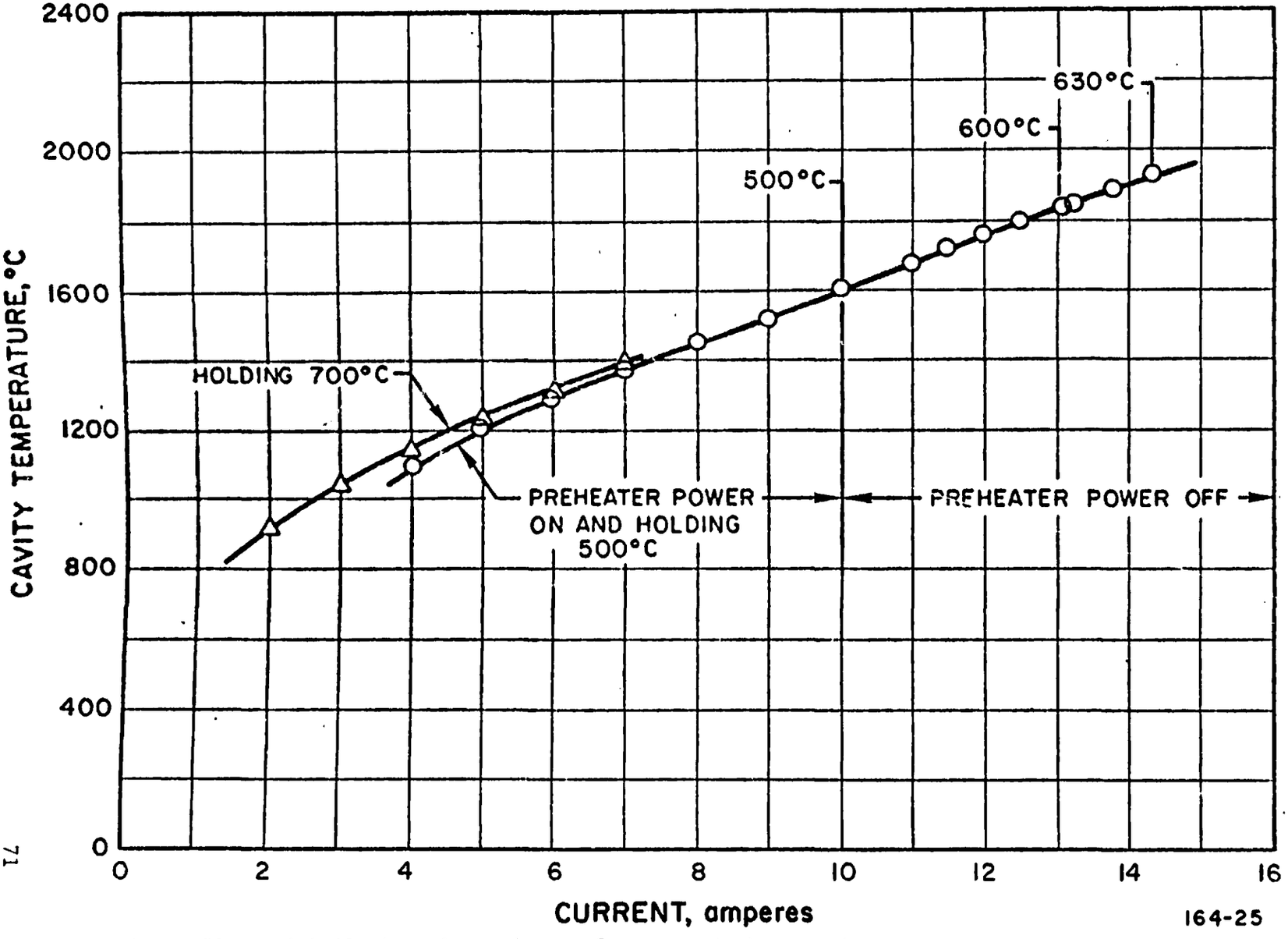

Figure 32.- Model 40 zirconia conductor furnace main heater characteristics.

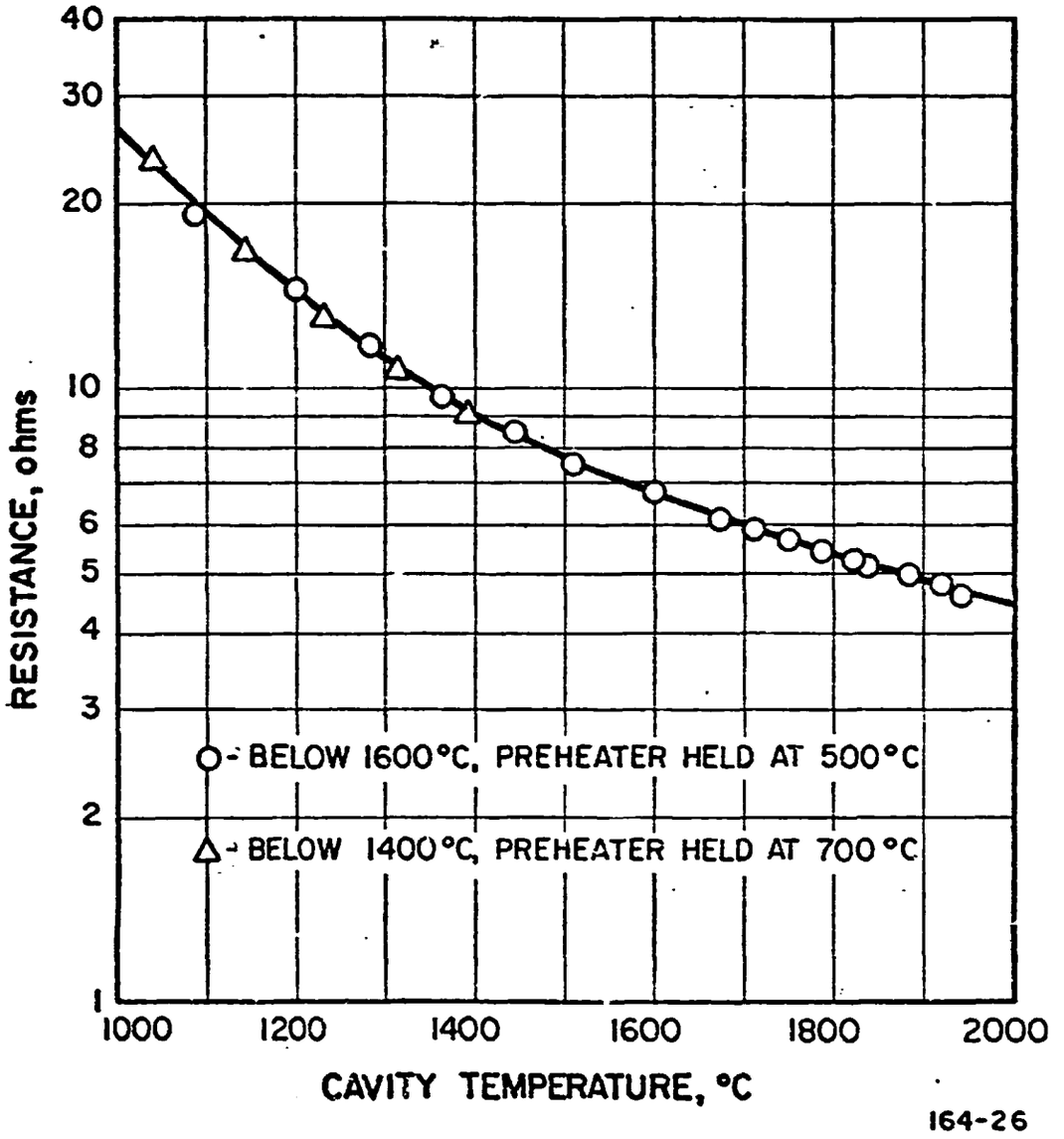

Figure 33.- Model 40 zirconia conductor furnace main heater element resistance.

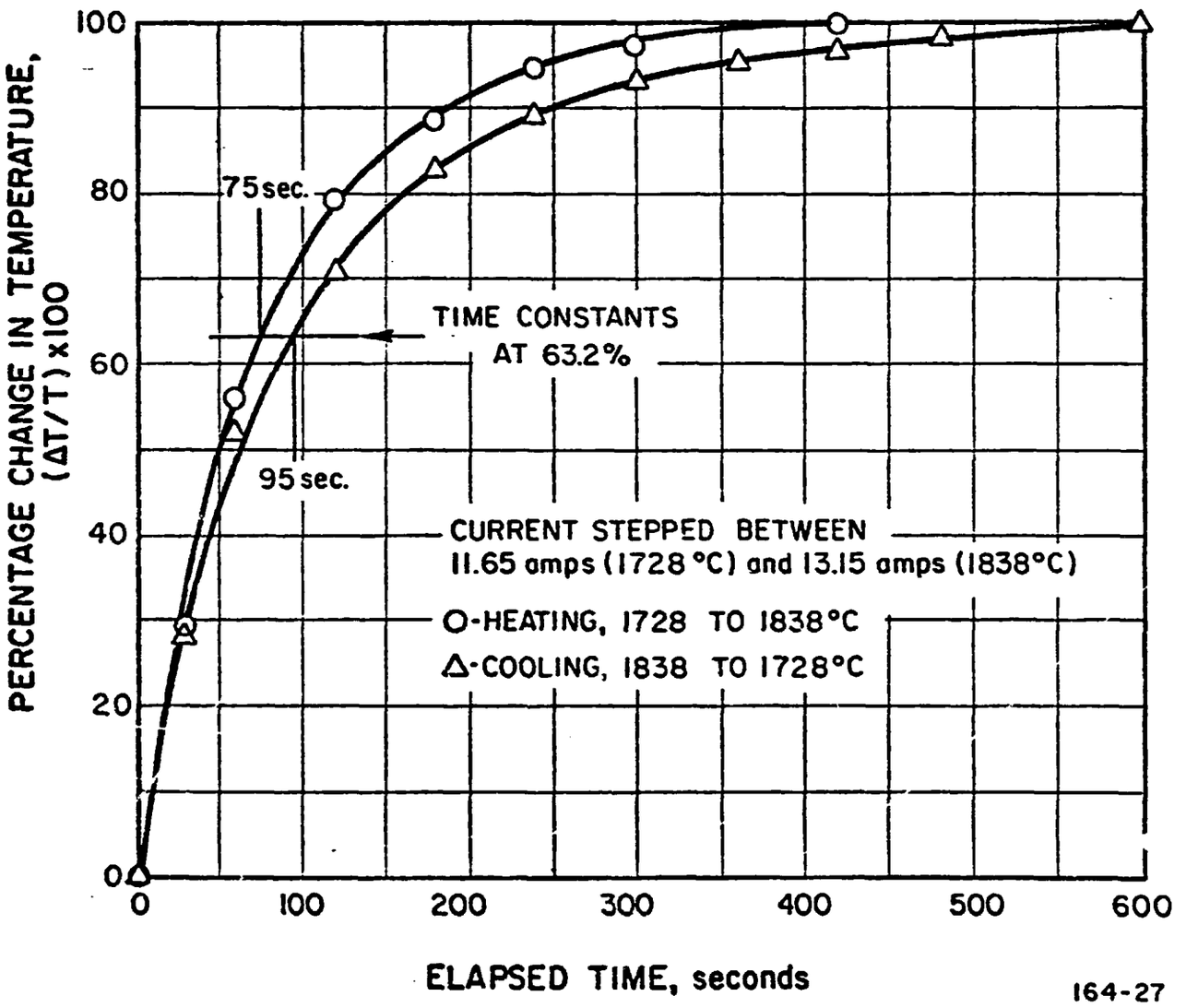

Figure 34.- Model 40 zirconia conductor furnace transient response.

These more severe transients were run also to verify that the main heater element could handle some reasonably severe thermal shocks. Typical gain and reset settings were used to provide a narrow proportional band (corresponding to less than 10°C). In order to prevent over-ranging the heater element during increasing temperature steps, the controller current limiter was used and adjusted for 15 amperes (corresponding to about 2000°C). The following response to step changes in the controller set points of 1630 and 1730°C were observed. During the cool-down from 1730 to 1630°C the cavity-mounted thermocouple reached 1640°C in 18 seconds, had undershoot to 1626°C at 22 seconds and reached 1629°C in 25 seconds. During a heat-up from 1630 to 1730°C, the thermocouple reached 1720°C in 32 seconds, reached 1725°C in 120 seconds, and reached 1729°C in 240 seconds. Control in both current feedback and thermocouple feedback modes was found to be excellent with fluctuations in the thermocouple-sensed cavity temperature being much less than ±1°C.

The above transient test results corresponding to step changes in temperature of 110 and 100°C are indicative of larger step changes in temperature (response) and current (driving force). These indicate that the conducting ceramic furnace can change temperature rapidly relative to the time frame of the sounding rocket experiment flights depending on the "acceleration" power available.

Power controller accuracy experienced with the Thermac 625 driving the Model 40 furnace was excellent. Controlling the preheater using the preheater type K thermocouple for a feedback signal, the controller held ±0.2°C at 975°C. Using gain adjustments only, the temperature droop was of the order of 10°C. Applying reset to the control loop reduced the droop to about 0.1°C.

Controlling the ceramic main heater at 1700°C, the controller operating closed loop on a bare Pt-6Rh/Pt-30kh thermocouple junction in the center of the heater cavity held ±0.25°C. Controlling the main heater with current feedback, the heater cavity was held to ±0.5°C. Using gain and reset adjustments, droop could be reduced to a fraction of a degree centigrade.

Axial Gradient Furnace

Of primary importance in the tests of the axial gradient furnace was documentation of the temperature gradient along the furnace cavity axis. This was done using a thermocouple of Pt-6Rh/Pt-30Rh and an alumina radiation target viewed by a Micro-Optical pyrometer. The thermocouple consisted of a bare junction of .05 mm diameter wires supported by a 3 mm (1/8 inch) OD double bore high purity 99.8% alumina tube. Near the junction, a bead of the same tube 7.5 mm long was used as a pyrometer target. The bead, separated from the support tube and having only contract resistance with the support tube, responded better to the furnace cavity temperature than would have the end of the support tube.

In addition, for operation above 1800°C, a similar support tube was used to hold two separate 3 mm long beads. These were held on top of the support tube by a U-shaped loop of .025 mm diameter 60Ir-40Rh alloy wire. The upper most bead appeared to have excellent response to cavity temperature changes.

These support tubes were inserted into the furnace cavity through a hole in the lower support of the furnace and, in the case of pyrometer sightings, were viewed through a 3 mm diameter hole in the upper insulation plug. The data thus reported is in terms of the measured temperatures which are not necessarily the same as the heater element temperatures, they being higher by some differential amount allowing for radiation and conduction errors of the thermocouple and support tube. For large penetrations into the furnace, good agreement was generally found between the thermocouple and the sighting on the single supported bead below the thermocouple junction suggesting that any error in measured temperature values was small (less than 1%).

Toward the bottom end of the working cavity, support tube conduction errors were observed to become significant and measured temperatures in this region would be low relative to the secondary heater element wall temperature. This was evident since the pyrometer sightings on the alumina bead yielded temperatures significantly higher than the thermocouple signalled temperatures. For example, at an axial location 2 cm up from the bottom of the furnace cavity with the secondary heater at 1020°C, the pyrometer reading indicated 1090°C while the thermocouple indicated 700°C. Undoubtedly the thermocouple leads which pass through the alumina bead have a cooling effect on the bead. The bead in turn was responding to radiation being received from the primary (ceramic) heater which was at a nominal temperature of 1475°C. For this reason, the double beaded support without a thermocouple is believed to represent more accurately the true cavity temperature.

Figure 35 presents some typical temperature gradient data. The lower two curves correspond to thermocouple data, the middle curve to the bead adjacent to the thermocouple, and the upper two curves are from the pyrometer readings on the double beaded support tube. The bottom curve corresponds to preheater power only with the preheater core at 800°C. Relative to the isothermal furnace (see figure 28) insufficient heating of the primary heater has occurred for an ignition condition.

The second curve from the bottom shows how ignition temperatures are achieved by adding the secondary heater (preheater at 820°C and secondary heater at 925°C). For the remaining curves, all three heaters are being powered to hold nominal temperatures as indicated. The primary heater temperature was not measured and can be taken to be at least the maximum observed temperature on each curve since element temperature does not vary significantly over the active length of the conducting ceramic element. Most likely, the true element temperature is slightly higher, perhaps 1950°C in the case of the upper-most curve of figure 35.

The maximum temperature gradient reflected in the upper most curve of figure 35 is no more than about 100°C per cm. However, this is the gradient response registered by the pyrometer target of 3 mm diameter in a 17.7 mm cavity. The target naturally responds significantly to radiation fluxes relative to a considerable distance in both directions along the furnace cavity axis. Consider for example the upper most curve of figure 35 and an axial station of 9 cm. This station is about midway between the top most end of the secondary heater estimated to have a wall temperature of no more than 1300°C and the bottom end of the high temperature zone of the primary heater estimated to have a wall temperature of not less than 1900°C. The axial distance between these two stations is 2.36 cm (0.93 inch) and the corresponding

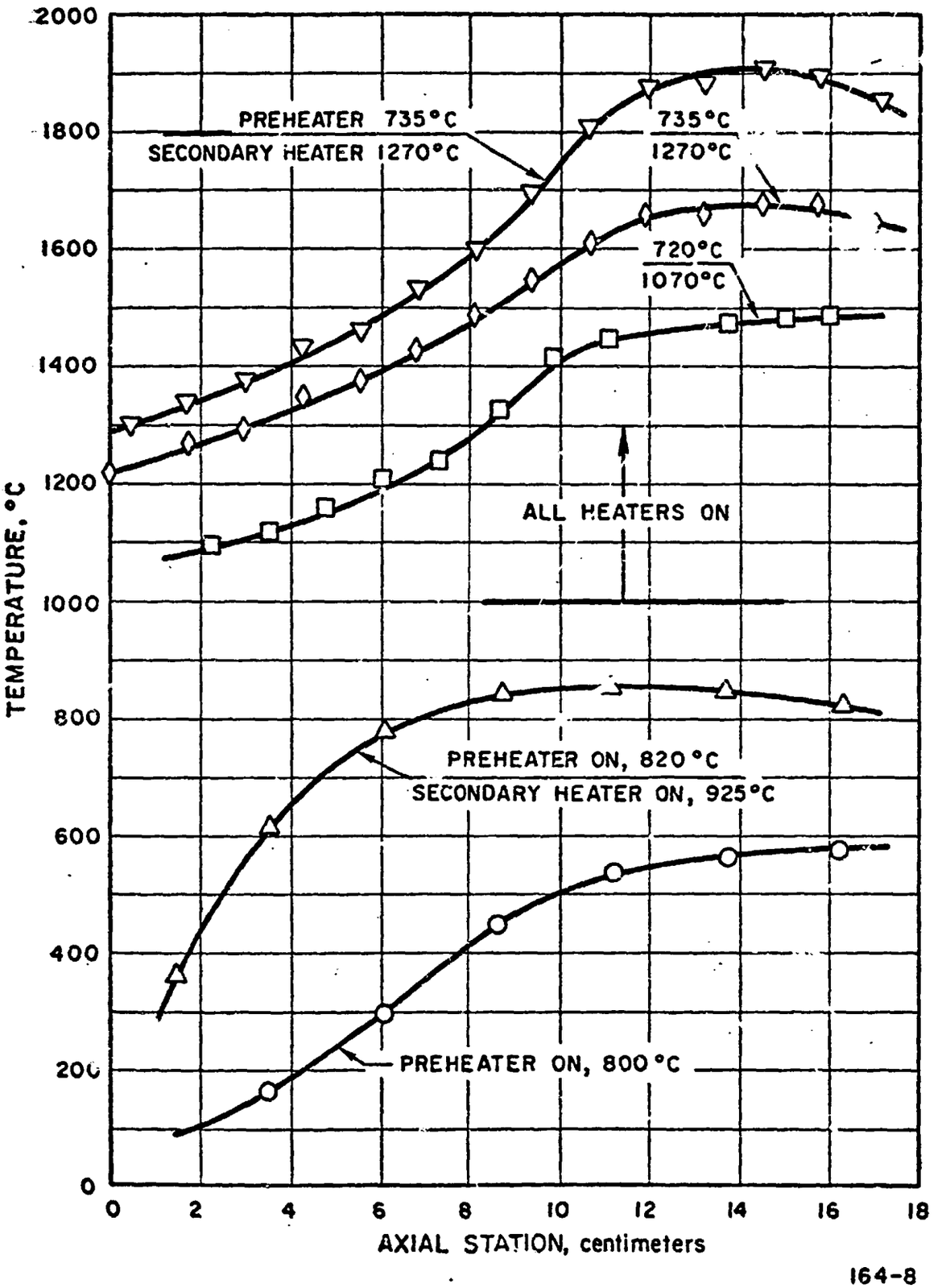

Figure 35.- Temperature gradients for the axial gradient furnace.

gradient in terms of furnace wall temperature is at least 250°C per cm. With higher primary element temperatures and lower secondary element temperatures the axial gradient in terms of wall temperature can be increased further.

To demonstrate a higher temperature, the primary heater element was increased incrementally (about 25°C per increment) while observing the top bead of the double beaded support tube set at an axial location of 14 cm. The 60Ir-40Rh wire was observed to evaporate and oxidize to near nothing as 2000°C was reached. At a pyrometer reading of 2020°C (±5°C), the 99.8% alumina bead was observed to melt. Temperature by pyrometer reading was increased to 2027°C at which time the alumina support tube began to sag. To avoid having the alumina tube touch the primary heater the furnace power was reduced. It is estimated that the primary heater element temperature reached from 2050 to 2100°C when the melting was observed.

Electrical characteristics for the primary heater are shown in figure 36. The power and temperature curves have trends similar to those for the isothermal furnace (figure 31). The voltage curve, however, is different and is increasing relative to near constant voltage for the isothermal furnace ceramic heater over the same temperature range. While not shown on the figure 36 curve, voltage continues to decrease with decreasing current down to ignition conditions (about 1000°C). For example, with a preheater temperature of 800°C, a secondary heater temperature of 1200°C, the primary heater operates at 55 volts at 1000°C and 91 volts at 1200°C. Figure 37 presents the primary heater resistance for these conditions as well as another set of preheater-secondary heater conditions. The voltage is sensitive to both the preheater and secondary heater temperatures. Thus, primary heater voltage as well as current can be used as a temperature signal for other conditions (preheater and secondary heater, for example) constant.

Transient response of the axial gradient furnace ceramic heater was observed to be considerably faster than that for the isothermal ceramic heater (see the discussion relative to figure 34). This is to be expected because of the smaller size of the axial gradient element. While the gap type construction of the axial gradient element over the isothermal element exposes some of the internal insulation which would tend to slow thermal response, this factor appears to be compensated by the reduced mass of the conducting element (every other conductor is eliminated in the high temperature zone). Temperature changes of several hundred degrees centigrade per minute are possible with small driving forces (power perturbations) with the axial gradient heater element shown in figure 19.

CHEMICAL INTERACTIONS

Chemical interactions of the furnace materials and materials to be processed within the furnace are difficult to specify without actual experimental data. In general, the processed materials do not directly contact the furnace materials but are transferred to the furnace parts as vapors or as fine particulates. The three most susceptible furnace materials include the stabilized zirconia heater element, the zirconia insulation and the platinum electrodes. These are located near the process materials in regions of high temperature and would be most susceptible to possible chemical reactions. Reactions must be related to the furnace cavity atmosphere as well as operating temperature.

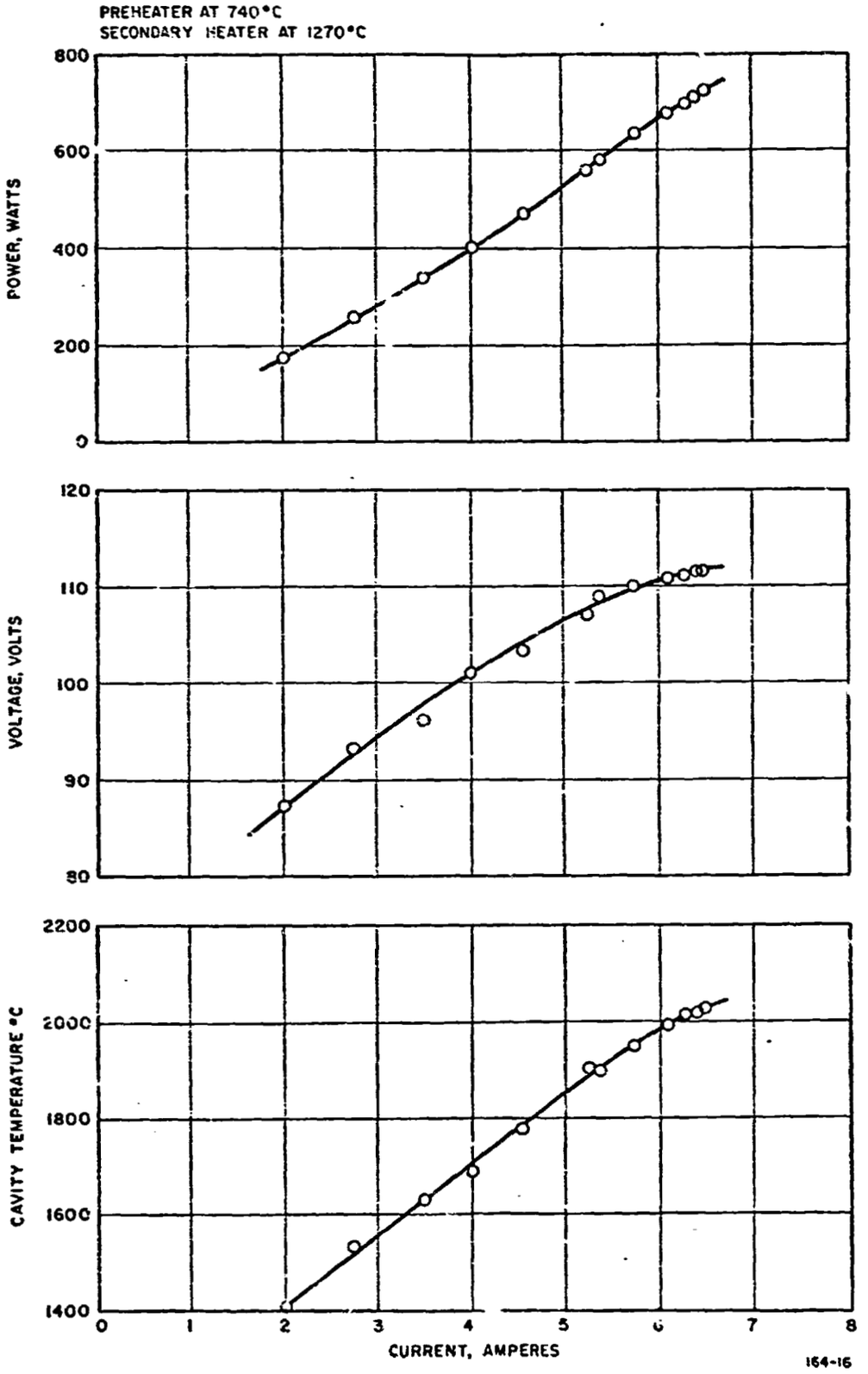

Figure 36.- Electrical characteristics of the axial gradient furnace ceramic heater.

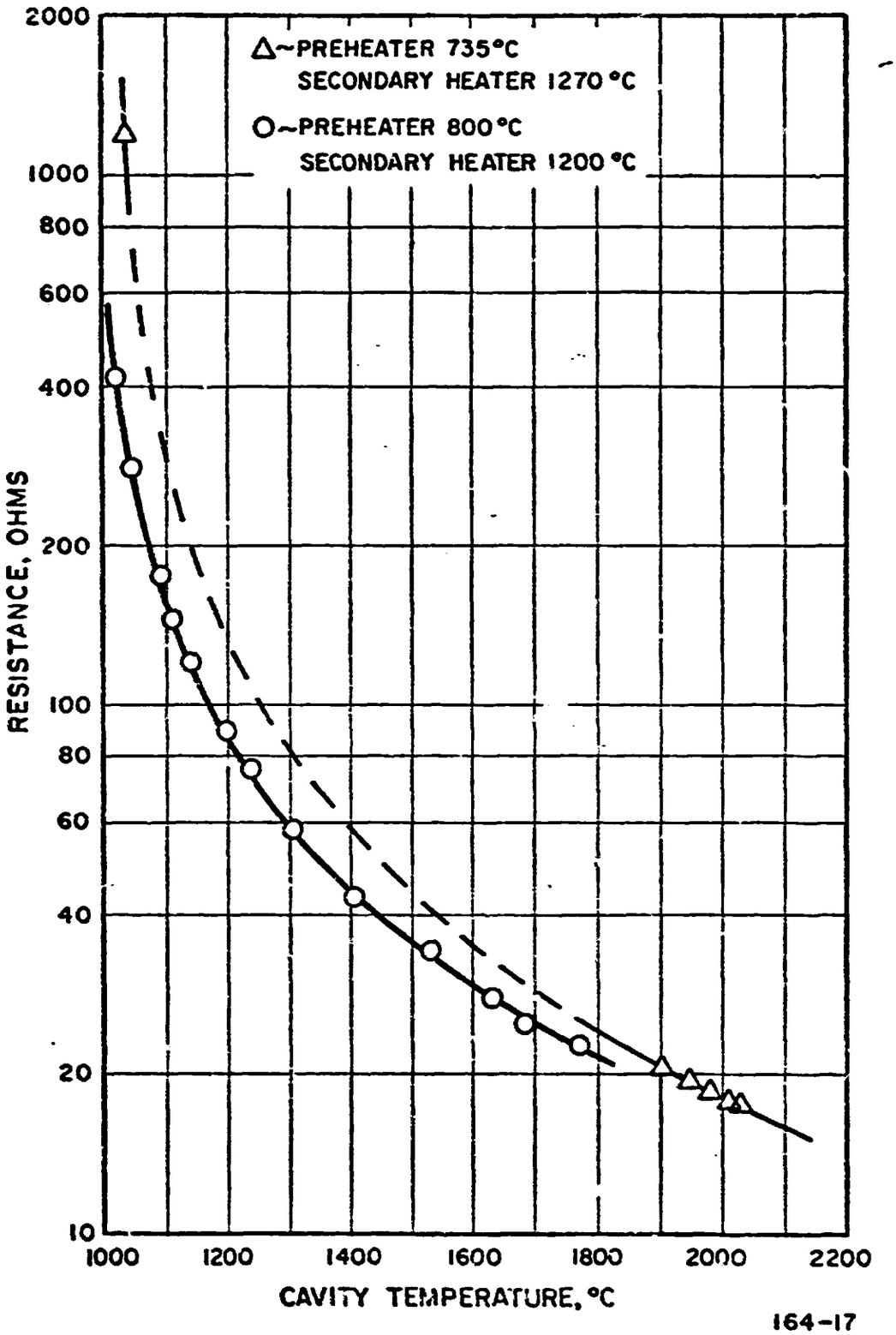

Figure 37.- Axial gradient furnace ceramic heater element resistance.

While the ceramic heater element within the furnace cavity is at the highest temperature, the surrounding insulation and adjacent electrodes being cooler, potential contaminating or reacting vapors tend to be driven away toward the cooler locations within the furnace. Extraneous vapors being emitted by the process find their way to the cooler regions at temperatures where condensation (or solidification) can occur. Particulates which may dust off a process material body or form as the result of a commingling of different vapors may interact directly with even the hotest portions of the ceramic heater element.

Direct mechanical contact as the result of faulty placement of process material, or failure in support fixtures, or from splattering of a boiling material, for example, are to be avoided. These would represent abnormal abuses to the furnace heater element and would be difficult to design against.

Both materials to be processed and fixtures such as crucibles, seed holders, pull rods, instrumentation, etc. are to be regarded as potential contaminants of the furnace heater elements. Compounds from these materials such as oxides in the case of an oxidizing atmosphere, must also be considered. Thus chemical interaction phenomenon are complicated with many aspects. The furnace user should evaluate related published data in choosing fixtures and process materials and, if necessary, is encouraged to run samples of the appropriate materials to determine their suitability with the furnace materials.

The following presents a brief discussion of some specific results of having exposed ZrO_2 and ThO_2 to typical materials to be used as furnace fixtures.

Reference 11 indicates that both ZrO_2 and ThO_2 can be used in vacuum for extended periods to at least 2300°C. Short time (a few minutes) stability problems arise for the following materials combinations:

1. ZrO_2 with W or C at 1600°C.
2. ZrO_2 with BeO at 1900°C.
3. ZrO_2 with MgO at 2000°C.
4. ZrO_2 with Mo or ThO_2 at 2200°C.
5. ThO_2 with Mo at 1900°C.
6. ThO_2 with C at 2000°C.
7. ThO_2 with BeO at 2100°C.
8. ThO_2 with W and MgO at 2200°C.

Further data relative to in vacuum performance are given in reference 12 which indicate that neither Mo nor W react with ZrO_2 at 1500°C after 500 hours exposure. With Ta at the same conditions, a slight reaction was noted.

Reference 13 describes a crystal puller furnace used to grow NbO, Ti_2O_3 and Al_2O_3, for example, having melting points of approximately 1945, 2000 and 2050°C, respectively. It is mentioned that dense stabilized ZrO_2 can be used

as a suitable pedestal material for supporting crucibles of Ir, Mo, W or Ta. An argon atmosphere was used for the tests described. Regarding interactions, reference 13 mentions that crucibles of W and Ta can be used with Mo supports, however, at 2000°C, Ir reacts with Mo. Relative to ZrO_2, reactions were noted with NbO and also V_2O_3 where ZrO_2 crucibles were destroyed with these melts.

Some reaction data between pertinent oxides in oxidizing atmospheres is given in reference 14. In materials compatibility tests the following were noted:

1. ZrO_2 + ThO_2 did not react to form a compound at 2000°C - 10 minutes.
2. ZrO_2 + Al_2O_3 form a eutectic just above 1900°C.
3. ZrO_2 + BeO appear to react significantly at about 1950°C.
4. ThO_2 + Al_2O_3 form a eutectic melting at about 1900°C.
5. ThO_2 + MgO appear to form a eutectic above 2000°C.

The combination of ZrO_2 in contact with ThO_2 is the basis for the electrode to element transition in the Geller (references 3 and 14) furnace and also in the Rothwell (reference 4) furnace. Rothwell used CaO stabilized ZrO_2 in contact with Y_2O_3 doped ThO_2. Geller used Y_2O_3 stabilized ZrO_2 with variously doped thorias. Davenport (reference 2) used zinc oxide as an electrode interface with stabilized zirconia, however, temperatures are limited to 1500°C at the interface because of volatility of the zinc oxide.

Reference 15 presents tables of possible crucible materials. Of the high temperature oxides listed, alumina, magnesia, and thoria are suggested for working temperatures to 1900, 2600 and 2800°C, respectively, and are rated fair in thermal shock resistance. Zirconia and beryllia are rated for 2300°C with zirconia rated good and beryllia rated very good in thermal shock resistance. These ratings are without regard to chemical reaction and are based on capability of the respective materials themselves.

Zirconia and thoria and ceramic oxides which are used as stabilizers for zirconia are evaluated relative to platinum and platinum-rhodium alloy with an air atmosphere in reference 10. This study, while evaluating the noble metals as electrode materials for the conducting ceramic oxides, also equate to the use of the noble metals as crucibles for the oxides tested.

In the reference 10 tests, ceramic oxide powder samples were placed in dense-high purity alumina crucibles. Pieces of thin ribbon wire of platinum and of 90 platinum-10 rhodium were placed in the powder as well. Two runs were made to expose these materials to high temperature. The first run was to 1600°C for one hour and the second run with new samples was to 1700°C for one-half hour. The second run was made to verify observations made from the first run, some of which indicated very slight reactions.

Results of these tests are presented in Table VII using a numerical grading system of 0 to 5 where 0 indicates no reaction and 5 indicates a prominent reaction. In all cases, any reaction is based on observed coloration of the

TABLE VII

NOBLE METAL VERSUS CERAMIC OXIDE
INTERACTION TEST RESULTS

Ceramic Oxide	Exposure at 1600°C - 60 Minutes)		Exposure at 1700°C - 30 Minutes	
	Pt	Pt-10Rh	Pt	Pt-10Rh
La_2O_3	5	5	-	-
CeO_2	3	3	-	-
Y_2O_3	0	1	0	1
ZrO_2	0	2	0	2
Sc_2O_3	0	1	0	0
ThO_2	1	1	1	2
MgO	0	4	0	4

Graded on the basis of 0 to 5. 0 for no coloration, 5 for most prominent coloration occuring with La_2O_3. All colorations observed were yellowish except with La_2O_3 which appeared brownish-gray.

oxides where they made contact with the noble metal strips. All ceramic oxide powders used were white with the exception of the CeO_2 powder which was very light yellow. The furnace atmosphere was air and no changes in color of the bulk of the oxides was noted. It must be mentioned that any compounds that might have formed and were white in color would have gone unnoticed for the oxides except perhaps for the La_2O_3 and CeO_2 and MgO with Pt-10 Rh. The blanks in Table VII indicate that the first test with La_2O_3 and CeO_2 was conclusive and not repeated at 1700°C.

The conclusions to be made from these results relative to noble metals are as follows:

1. La_2O_3 and CeO_2 are expected to present near-term problems with either Pt or Pt-Rh alloys.

2. Y_2O_3, ZrO_2, Sc_2O_3 and MgO appear suitable with Pt. Some reaction is indicated with Y_2O_3 and ZrO_2 with Pt-Rh alloy. A greater reaction of MgO to Pt-Rh alloys is indicated.

3. ZrO_2 is compatible with Pt but not Pt-Rh alloy. And finally,

4. A slight reaction problem is to be expected with ThO_2 with both Pt and Pt-Rh alloy.

It is also worthy of mention that no apparent reaction occurred with the oxides tested and the Al_2O_3 crucible with the exception of MgO. Some wetting of the crucible by the MgO was noted.

CONCLUSIONS

Stabilized zirconium dioxide ceramic has been used to fabricate furnace heater elements capable of high temperature operation to 2200°C with oxidizing atmospheres. While this technology is an old art, improvements in the ceramic oxide quality and in the platinum to ceramic electrode interfacing have resulted in a reliable furnace heater element capable of many thermal cycles and rapid changes in temperature. The unique geometry of the conducting ceramic heater elements (parallel tubes) in part contributed to the success of the demonstrated furnaces.

In addition to being compatible at high temperature with oxidizing environments, the stabilized zirconia conducting ceramic heater elements were shown to be compatible with inert and hydrogen reducing environments. Operation with hydrogen was demonstrated by changing from oxidizing to reducing back to oxidizing environments several times while the furnace heater element remained hot. The conducting ceramic heater element is, therefore, universal having been operated in vacuum, inert and reducing environments as well as in oxidizing environments.

Both an isothermal type ceramic oxide heater element having a working cavity size of 2.5 cm by 10.0 cm and an axial gradient heater element having an inside diameter of 1.77 cm and overall length to diameter ratio of 10 to 1 were demonstrated. Temperatures to 1940°C were demonstrated with the isothermal element and from 2050 to 2100°C with the axial gradient element. In the case of the axial gradient element, high purity alumina was melted at 2020°C. The temperatures demonstrated do not reflect upper bounds of the furnace elements, rather they are limits relative to instrumentation used in the various experimental runs made. None of the heater elements tested were damaged or destroyed.

The axial gradient furnace had a two-zone main heater configuration comprised of a platinum-10% rhodium heater rated for 1500°C and a conducting cerámic heater rated for 2200°C. An axial temperature gradient of 250°C per centimeter was demonstrated based on heater element wall temperatures.

Thermocouple sensed cavity temperatures indicated control error with a solid state type industrial controller to be less than ±1°C. This accuracy was demonstrated using both current feedback and temperature feedback modes of control. Transient response of the conducting ceramic heaters was found to be excellent; for example, the isothermal heater commanded to a 100°C lower temperature reached a 99% response condition in 25 seconds.

The conducting ceramic, being an ionic conductor, must be powered with alternating current. Direct current causes changes in the ceramic oxide stoichiometry (particularly in a reducing environment) adversely affecting its performance relative to lifetime and uniformity of temperature.

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