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DEVELOPMENT OF A HIGH TEMPERATURE HEATER USING AN YTTRIA STABILIZED ZIRCONIA CORED BRICK MATRIX

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Ames Research Center
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FOREWORD

This work was performed for the NASA/Ames Research Center by FluiDyne Engineering Corporation under Contract NAS2-2691. The contract description is: "Refitting the Ames Pilot Heaters," FluiDyne Job 0456. Mr. Frank Pfyl of NASA/Ames Research Center was the contract technical monitor.

Valuable contributions in the testing of ceramic materials and in the evaluation of the test results were rendered by Dr. Jerry Plunkett, consultant to FluiDyne, and by Major L. Fehrenbacher and Dr. Robert Ruh of the Air Force Materials Laboratory.
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NOMENCLATURE

\( A \) Cross-section area of bed
\( A^* \) Sonic Throat area of nozzle
\( C \) Coefficient in sonic throat mass flow equation (Figure 7)
\( C_p \) Specific heat of air
\( D \) Diameter of holes in cored brick
\( E \) Modulus of elasticity
\( L \) Bed height (10.5 feet)
\( P \) Pressure
\( \bar{P} \) Means pressure in heater bed = \( \frac{P_L + P_0}{2} \)
\( P_L \) Pressure at top of bed \( (z = L) \)
\( P_0 \) Nozzle stagnation pressure
\( \Delta P \) Pressure difference
\( R \) Gas constant
\( R_a \) = Equivalent tube O.D./I.D. = 1.05 (holecenter to center opening) / hole diameter
\( R_1 \) = \( \frac{R_a^4 (\ln R_a - 3/4) + R_a^2 - 1/4}{R_a^2 - 1} \)
\( R_3 \) = \( \frac{R_a^2 R_1}{R_a^2 - 1} \)
\( S \) Tensile Stress
\( S_t \) Tensile Strength
\( T \) Temperature
\( \bar{T} \) Average bed temperature = \( \frac{1}{L} \int_0^L Tdz \)
\( T_L \) Temperature at top of bed \( (z = L) \)
\( T_0 \) Nozzle stagnation temperature
\( T_{ave} \) Average temperature in web or in equivalent tube wall
\( T_{surf} \) Surface temperature in hole of cored brick or equivalent tube
\[ \Delta T = T_{ave} - T_{surf} \]

\[ W \]
Weight per unit volume of bed = cermic density \( x \) \((1 - \sigma)\)

\[ \text{YRE} \]
Yttria - rare earth oxide mixture

\[ a \]
Thermal diffusivity

\[ f \]
Friction factor

\[ g \]
\[ 32.2 \text{ lb}_m \text{ ft/lb}_f \text{ sec}^2 \]

\[ k_s \]
Thermal conductivity of cermic

\[ m \]
Mass flow rate

\[ p \]
Porosity of ceramic

\[ u \]
Velocity

\[ z \]
Distance from bottom of bed

\[ \alpha \]
Coefficient of expansion

\[ \theta \]
Time

\[ \nu \]
Poisson's ratio

\[ \sigma \]
Porosity of bed = area of holes divided by \( A \)

Subscript \( o \) refers to zero ceramic porosity \((p = 0)\) or to stagnation conditions
SUMMARY

The Ames pilot heater is a ceramic regenerative heater that provides high temperature air for aerodynamic and combustion experiments. This report describes development of this heater to provide a heat storage bed with temperature capability of about 4600°R. A bed was designed and installed having cored brick elements of yttria-stabilized zirconia. The bed dimensions are 14 inches in diameter by 10 feet high. The thermal stress limitations of the bed were studied and maximum air flow rates based upon these limits were established. A combustion reheat system was designed and installed to provide the necessary control over the bed temperature distribution. The revised heater system was successfully operated at a maximum bed temperature of 4600°R. The successful operation demonstrated that yttria-stabilized zirconia cored brick can satisfy the high temperature-long duration requirement for storage heater applications.
1.0 INTRODUCTION

The heater that is the subject of this report was one of the earliest ceramic storage heaters used for aerodynamic testing. The purpose of the work reported herein was to modify the heater in order to achieve both the highest feasible air temperature and the minimum contamination of the air by dust from the ceramic materials. A maximum air temperature of 4400°F was estimated to be a reasonable goal. This would provide simulation of free flight at hypersonic Mach numbers in the 7 to 8 range.

The improvements were to be gained through selection and development of materials and through design of the entire ceramic structure; heater bed and insulation. The most severe operational conditions exist in the heater bed; therefore, its materials and construction details establish the upper limits of temperature and mass flow and exert the major influence on dust production.

In early configurations, ceramic pebbles were used for the heat storage bed. At that time (before 1964) spherical pebbles were the only shapes used in wind tunnel storage heaters that operated at temperatures above 2250°F (approximate metal temperature limit). Among the various heaters, experience has been gained with alumina at temperatures to 3500°F and with zirconia at temperatures to 4400°F. There was also more limited experience with magnesia.

These heaters operated with varying degrees of success. The most serious difficulties were dust in the heated air (with both alumina and zirconia) and deformation of the zirconia pebbles. The air from the heaters carried dust particles from the ceramics which damaged model surfaces and could influence combustion processes. High air flow rates aggravated the problem such that dusting sometimes imposed operating limits. Zirconia beds were found to "clump" or compact through deformation of the pebbles at high temperatures.
The basic problem with zirconia materials was destabilization of the crystalline structure, causing loss of strength and, sometimes, severe structural deterioration of the material. Destabilization contributed to dusting and to high temperature deformation, and usually was the determining factor in fixing the useful life of the material. Successful application of zirconia required that the destructive destabilization process be avoided, and, therefore, considerable attention was given to this problem.

Heater studies by the Air Force (Reference 1) indicated that bricks of the "cored brick" shape (Drawing 7003-008C) would have advantages over spherical pebbles in producing less dust, in permitting higher temperature operation with less creep, and in allowing higher flow rates. The cored brick shape eliminates the point-to-point contact of pebbles which was considered a significant cause of dust production, and further, the rubbing of cored bricks against the sidewall insulation was judged to be less damaging than that by pebbles. Elimination of the point contact loading would also reduce deformation by high temperature creep, enabling a bed to be operated at higher temperature. Finally, the flow resistance through the cored brick holes is much less than that through a bed of small spheres. This would increase the flow rate at which the bed would lift (the flotation limit).

On the other hand, the cored brick shape introduced new problems regarding thermal stress failure, which is one of the major limitations of ceramic materials. Thermal stresses caused by heating or cooling an object generally tend to increase with the object's size. The cored bricks would be larger than the pebbles used in the heater beds (3/8 to 1 inch diameter). Operational experience with pebble beds has indicated that thermal stress failure is not a serious problem provided the pebbles are of high quality and excessive cooling rates are avoided (determined by test; see, for example, Reference 2). It was judged that the thermal stress levels in the cored brick could be kept within tolerable limits through design of the brick and through control of the heating and cooling rates.
The Air Force program mentioned earlier (Reference 1) was continued in the direction of the development of a heater system using zirconia cored brick (References 3, 4, and 5). This Air Force program and the work described here were carried on in parallel by FluI DynE Engineering Corporation. The two programs were coordinated in the area of ceramic materials. The Air Force work had defined a pilot heater that was somewhat larger than the present heater; vessel I.D. = 36 inches versus 30 inches and bed length = 16 feet versus 10 feet. These dimensions were close enough to allow the same bed diameter (14 inches), the same cored brick configuration, and some identical insulation shapes. Information on materials development and tests was exchanged, and the results were applied to both heater designs.

The first decision was whether to use magnesia or zirconia. The limitation on magnesia is its high vapor pressure and consequently, its weight loss at high temperature. From special tests and published data, it was estimated that the maximum useful temperature limit for magnesia was approximately 3500°F. Consequently, zirconia was selected.

The remainder of the materials development work was directed toward zirconia. Based upon the Air Force work, it was decided to use a dense zirconia for the heater bed as opposed to the lower density material typically used for insulation shapes. The distinction is discussed in References 3 and 4, and is reviewed later in this report. The dense zirconia would provide the highest temperature capability, increased heat capacity, and the lowest dust level.

Another decision involved the selection of the stabilizer for the zirconia. Commercial zirconia materials used calcia and/or magnesia. These materials were found to have service temperature limits significantly below 4400°F, primarily because of the destabilization phenomenon mentioned earlier. Yttria-stabilized zirconia proved suitable and was chosen for the high temperature locations in spite of its high cost. Lower cost materials were selected for the lower temperature regions of the heater. These included alumina cored brick for the lower one third of the bed, alumina insulation, and fibrous insulation materials.
The work also involved redesign of the reheat system for the heater. Burners were developed that provided the wide ranges of temperature and mass flows needed for control of the bed temperature distributions. Further, the burners could remain in the heater during high pressure operation.
2.0 PILOT HEATER DESCRIPTION

The pilot heater consists of a vessel containing ceramics which make up the heat storage matrix and vessel insulation. A sketch of the heater is shown in Figure 1, and the assembly details are shown in Drawing 0456-026B. A cored brick configuration, Drawing 7003-008, of high density alumina and yttria-zirconia is employed in the 14 inch by 10 foot long matrix or bed. Support for the bed is provided by a stainless steel grate located in the bottom of the heater. The heater is thermally charged, using two water-cooled burners mounted on the vessel upper head. These burners penetrate the ceramic dome assembly, and combustion gases flow downward through the matrix and are exhausted to atmosphere. The burners are designed to remain in place when the heater is pressurized. The vessel is protected from high temperatures of the matrix by sidewall and dome insulation and is cooled by free convection and radiation from the outer surface. The upper dome insulation is a built-up refractory brick configuration which is supported by an uncooled shelf. In the lower head, thermal protection and sidewall insulation support are provided by an alumina castable insulation.

The lower five feet of matrix, the insulation, and vessel are instrumented with thermocouples. Optical pyrometer penetrations are provided in the upper head and in the sidewall 8-1/2 feet above the grate for viewing the top and side of the matrix, respectively. The pyrometer and matrix temperature instrumentation provide operational information regarding the matrix temperature profile. The insulation and gel temperature instrumentation are intended to provide information which would indicate impending overheating.

After the heat storage matrix is thermally charged to the desired temperature profile, cold blowdown air from high pressure storage is brought into the bottom of the heater, flows upward through the matrix, is heated, and is discharged to the test facility through a nozzle located
The final heater configuration and materials utilized are shown on Drawing 0456-026B and summarized below:

**Dimensions**

- Matrix Length (cold) 10.5 feet
- Matrix Diameter (cold) 14.0 inches
- Sidewall Insulation Thickness 8.0 inches
- Hexagon Cored Brick Flat-to-Flat Dimension 3.78 inches
<table>
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<tr>
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<td>5 to 12 inches</td>
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**Matrix Materials**
- Lower 4 Feet: Alumina (3.83 g/cc)
- Upper 6.5 Feet: 10.4 w/o Yttria-Zirconia (5.4 g/cc)

**Insulation Materials**
- **Hot Face Tongue and Groove Brick**
  - Lower 4 Feet: Alumina (190 lbs/ft³)
  - Middle 1.5 Feet: Fully-Stabilized Calcia-Zirconia (260 lbs/ft³)
  - Conical Dome and upper 5-ft of bed liner: 9-1/2 w/o Yttria-Zirconia (260 lbs/ft³)
- **Backup Insulation - Arch Brick**
  - Lower 3 Feet: Alumina (190 lbs/ft³)
  - Middle 2.5 Feet: Fully-Stabilized Calcia-Zirconia (260 lbs/ft³)
  - Upper Vessel and Dome Backup: Fully-Stabilized Calcia-Zirconia (170 lbs/ft³)

**Miscellaneous Insulation Materials**
- Lower Vessel Head: Alumina Castable
- Between Arch Brick and Vessel: Fiberfrax Bulk Fiber (wool)
- Dome Shelf: Fiberfrax Paper
- Upper Vessel Head: Fiberfrax Castable
3.0 THERMAL STRESS DESIGN CONSIDERATIONS

Thermal stress failure is the major limitation in the use of ceramic materials in heat exchangers because ceramic materials are brittle. Therefore, locally high stress levels are not relieved through plastic deformation (except at temperatures near the service limits). Thermal stress considerations were dominant factors in much of the heater design and in the establishment of operating procedures. These design considerations were particularly important to the bed. The discussion will, therefore, be directed primarily to the bed, and comments regarding the insulation will be made where appropriate.

Thermal stresses arise from restraint of thermal expansion and/or contraction. The restraint may be external to the brick or it may be internal (within the brick). Consider a brick that is part of the bed or the insulation. If the brick is not free to expand when heated from room temperature, the external restraint of the neighboring bricks will cause thermal stresses. Excessive stresses from this source are avoided by providing space for the thermal expansion. Accommodation of overall thermal expansion does not constitute a serious problem in design or operation.

On the other hand, thermal stresses arising from internal restraint cannot be controlled by simply providing space for expansion. These stresses depend only upon the temperature distribution within the brick and its physical properties (primarily, thermal expansion coefficient, modulus of elasticity, and Poisson's ratio). In general, these stresses decrease as the size of the brick is decreased. The use of small bricks to minimize stresses is very effective and has been applied for many years to the design of insulation assemblies. In this case, there is a backlog of experience regarding brick sizes and shapes, and excessive thermal stress conditions can usually be avoided. The situation with respect to the cored bricks is different. Here, experience is very limited, and the
highest feasible performance was desired. Development of procedures for designing cored brick beds within thermal stress limitations was begun only recently (References 1 and 3). The operation of the present heater and the Air Force pilot heater (Reference 4) have provided further understanding, but a complete design procedure that would provide the confidence normally associated with heat exchanger construction is not yet available.

Design of the bed in terms of thermal stress limitations requires consideration of: (1) thermal stress failure modes, (2) material selection as related to failure modes, and (3) magnitude of the stresses. Each will be discussed in turn, and, as noted above, it is necessary to consider only stresses resulting from internal restraint.

3.1 Thermal Stress Failure Modes

Ceramic materials are much weaker in tension than in compression. Both tensile and compressive stresses exist in a brick thermally stressed by internal restraint, i.e., with stresses arising only from the temperature distribution. Thus, most thermal stress failures are tensile failures. Shear failures sometimes develop, especially in areas of high compressive stress.

A brick exposed to thermal stresses greater than its strength can fail in several ways. A crack (or cracks) can develop, but not propagate through the brick, leading to reduced overall strength. Cracks can propagate through the brick, fracturing it into one or more pieces. Cracks can propagate and intersect to spall the surface, but not necessarily fracture the brick. The readiness with which cracks propagate differs greatly among ceramic materials. Most ceramics can be compared either to glass at room temperature, where cracks extend swiftly and usually lead to fracture, or to concrete, where cracks meander and branch, causing loss of strength and usually leading to spalling.
Consideration of these two modes of failure has led to two thermal stress resistance criteria (Reference 6). The first is to avoid the initiation of cracks by keeping the stress levels below the material strength. The second is to allow thermal stresses greater than the strength, thus permitting cracks to be initiated but to minimize the resulting damage to the brick, such as loss of strength or loss of material through spalling.

The first criterion is the one commonly seen in the thermal stress literature. The procedure is to calculate the temperature field in the body, then calculate the stress field, then compare stress and strength. The results have been applied successfully to those materials in which cracks propagate readily to produce fracture. For ceramics these are typically the low porosity, fine grain materials that have high strength. As discussed later, this kind of material was chosen for the bed and, therefore, this criterion was applied.

The second criterion applies to those materials in which cracks meander and branch, typically the coarse grain ceramics having a pore volume of roughly 20% or more. These materials can be exposed to conditions where the tensile stress at the surface exceeds the strength, but without fracture or spalling providing the conditions are not too severe. The only effect then is the development of cracks. Cracks do weaken the brick, but not necessarily to a level where it is no longer serviceable. This situation is common in insulation brickwork where the thermal stresses cannot be kept below the strength. This design approach has been used for many years and relies heavily on test data, such as from the ASTM panel spalling test, and experience. Experience was the major guide in the present design of the insulation. Essentially, the approach is to make the individual bricks as small as possible without compromising the structural integrity of the assembly and without excessive cost (which increases with the number of bricks).
3.2 **Material Selection as Related to Failure Modes**

The material selection will determine which mode of thermal stress failure is dominant: complete fracture or damage caused by cracking (without fracture). The choice is between a fine grain material of low porosity and a coarse grain material of higher porosity. In this context, low porosity means a total pore volume of not more than about 10% (therefore, 90% or more of the theoretical density), and high porosity is roughly 20 to 30% pore volume. The term, density, will not be used in the absolute sense— to compare different materials— but only as a measure of the porosity of a given material, i.e., how close the material is to its theoretical density (zero porosity). A low porosity (high density) material offers performance advantages, specifically, the highest service temperature, highest mass flow capability, and lowest dust production. The disadvantages are cost and susceptibility to thermal fracture. These factors are discussed in the following paragraphs.

The maximum temperature of the bed is ultimately limited by deformation of the bricks through creep. Creep resistance increases as the pore volume is reduced, which thereby increases the service temperature.

The mass flow rate capability of the bed (for a given run time) is limited by flotation, the amount of energy stored, and the rate at which the stored energy can be removed to heat the air. Flotation is frequently the most restrictive limitation for pebble beds but with cored brick, thermal stress limitations are usually the most restrictive. The amount of stored energy increases as the material porosity is reduced, both directly and because the higher creep resistance allows a greater depth of bed to be heated to the maximum temperature.

Extraction of the energy at high rates requires that the product of heat transfer coefficient and heat transfer surface area be large. Large values are achieved in cored brick by use of small, closely spaced holes. The effect on surface area per cubic foot of bed is given below. Values for spherical pebbles are included for comparison.
<table>
<thead>
<tr>
<th>Hole or Sphere Diameter inches</th>
<th>Web Thickness inches</th>
<th>Surface Area ft²/ft³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spheres</td>
<td></td>
<td></td>
</tr>
<tr>
<td>.38</td>
<td></td>
<td>122</td>
</tr>
<tr>
<td>.5</td>
<td></td>
<td>86</td>
</tr>
<tr>
<td>1.0</td>
<td></td>
<td>43</td>
</tr>
<tr>
<td>Cored Brick</td>
<td></td>
<td></td>
</tr>
<tr>
<td>.25</td>
<td>.25</td>
<td>44</td>
</tr>
<tr>
<td>.20</td>
<td>.10</td>
<td>97</td>
</tr>
<tr>
<td>.194</td>
<td>.085</td>
<td>108</td>
</tr>
</tbody>
</table>

The fine grain versus coarse grain choice enters into the web thickness selection because coarse grains cannot be used to fabricate thin webs. Coarse grain materials have been limited to a minimum web thickness of about 0.25 inch, providing a surface area equivalent to one inch spheres. The hole and web sizes chosen were .194 and .085, respectively, giving a surface area between those for 3/8 and 1/2 inch spheres.

The heat extraction rate is also dependent upon the resistance to heat conduction in the web. Thin webs of high conductivity give up their heat more readily. Both factors are improved by use of a low porosity (high density) material.

Dust is produced in the bed by the rubbing of adjacent surfaces. Dense, fine grain materials have smooth, hard surfaces with tightly bonded grains. These produce less dust than the rough textured, coarse grain materials.

As described above, the failure modes are different between the low and high density materials. Overstressing the dense (low porosity) bricks will result in fractures. The seriousness of such fractures depends upon the number and their orientation. Fracturing usually relieves thermal stress levels; therefore, the fractured bricks may not progressively fracture into smaller parts upon repeated exposure to temperature.
conditions. Fractures in horizontal planes are considered less damaging than those inclined to the vertical. Vertically fractured pieces may produce pieces which could fall downward and become wedged in the bed, enhancing the conditions for additional fracture during repeated cycling. Basically, some fracture in a bed of dense bricks is tolerable, provided it relieves a local condition and does not lead to progressive deterioration. The fracture behavior can only be determined from an operating bed.

Exposure to the same conditions will also crack the bricks in a bed of the lower density, coarse grain material. The cracks will reduce the strength and may cause spalling. Progressive deterioration may occur because of the loss of strength and because the cracks will tend to propagate upon repeated temperature cycling.

At this time, there is not sufficient information to allow comparison of the two kinds of materials in terms of allowable bed flow rate and bed life except to state that the dense materials have the greatest potential. This potential for high performance was the reason for selection of dense ceramics for the bed.

3.3 Thermal Stresses in the Bed

This section describes the problems of predicting the thermal stress levels in the bed and of relating these stresses to the bed design and operating conditions. The intent will be to predict the maximum tensile stresses, then estimate the tensile strength (or modulus of rupture) of the material, and apply the criterion: stress less than strength. In this analysis, stresses due to the weight of the bed are negligible and are ignored. Thermal stresses that would arise from "pinching" of the bricks due to restraint of overall expansion are ignored. It is assumed that adequate expansion space has been provided. Only thermal stresses from internal restraint within each cored brick will be discussed.
A brief qualitative summary of thermal stresses of this type will be useful. Cooling of all surfaces of a block produces tensile stresses in the surface and compressive stresses in the interior. Failure is initiated by surface cracks that usually enter at 90°. Heating has the reverse effect. Rapid heating may cause tensile failures in the interior or shear failures at the surface, usually entering the surface at about 45°.

The thermal stress level is strongly dependent on the nonlinearity of the temperature distribution within the brick. If the temperature distribution through the brick is linear in Cartesian coordinates and if the properties are constant (independent of temperature), no thermal stresses will develop (Reference 7, p. 271 and Reference 8, p. 403). However, no simple relationship exists between the temperature distribution and the stress levels for "brick" shaped bodies, as opposed to shapes such as plates, spheres, tubes, etc. (The simple equation that applies to tubes will be used later.) Calculations for "brick" shapes require numerical solutions by computer on a case-by-case basis. Insofar as the authors are aware, no such calculations have been made for three-dimensional temperature and stress distributions.

A simpler case of a one-dimensional temperature distribution and a three-dimensional stress field led to the following conclusions (Ref. 9):

1. Temperature distributions having negative second derivatives produce tensile stresses on the surface of the block and compressive stresses near the center of the block.

2. Temperature distributions having positive second derivatives produce the reverse effect: compressive stresses on the surface and tensile stresses inside of the block.

3. Thermal stresses are roughly proportional to the deviation of the temperature distribution from linearity.
4. Thermal stresses can be reduced significantly by changes in size and/or shape. Reducing overall size is a common approach to reduction of stresses. But if only one or two dimensions can be reduced, the orientation with respect to the temperature distribution is important. For example, the stresses can be reduced by decreasing the dimension of the block in the direction along which the temperature varies and/or by decreasing the dimensions of the block which lie perpendicular to the temperature variation (i.e., reducing the cross-sectional area by reducing both dimensions of which it is comprised.

With this background, we will now examine the temperature distribution in the heater to qualitatively assess the thermal stress conditions. An idealized sketch of the temperature distribution is shown in Figure 1. Measured distributions are discussed in Section 7.0. Not shown in this figure is the radial distribution of temperature. The cored bricks in contact with the insulation are cooler than those in the center of the bed. This distribution is caused by heat loss through the insulation and can be quite significant, 300° to 400°R from the center to the edge of the bed.

Thus, individual bricks have a vertical temperature distribution and a horizontal temperature distribution even without the added effect of gas flow through the bed. During reheat, the bricks are heated at the surface of the holes, and during blowdown they are cooled. These flows produce temperature distributions within the webs between the holes. The thermal stresses are, of course, determined by the entire temperature distribution within the brick. For discussion purposes, however, it is convenient to separate these into two kinds of stresses: "body stresses" caused by the vertical and horizontal temperature distributions acting alone and "web stresses" caused by the temperature distribution in the web acting alone. The separation is convenient because the temperature distribution in the webs (and, therefore, the web stresses) is dependent upon the heater flow conditions over which the operator has control, while the horizontal distribution is controlled by the insulation design. Thus, only the vertical temperature distribution is controlled through the reheat process.
The vertical temperature distribution has, for the most part, a negative second derivative which will cause body stresses that are tensile at the surface. In the ideal case of a linear ramp and linear plateau, the linear portions would produce stresses only because the material properties are temperature dependent. The maximum second derivatives would be produced at the cold air entrance to the bed and at the "intersection" of the ramp and plateau. This suggests that the maximum stresses caused by the vertical temperature distribution will be at these locations also, but a detailed analysis that included the effects of temperature dependent properties and creep would be needed to establish stress levels with certainty.

The radial temperature distribution within the bed also has a negative second derivative. Therefore, both the vertical and radial distributions tend to produce tensile stresses in the surface of the bricks. Calculations of the stress field would require a substantial programming and digital computer effort. This was not done. It was hoped that the heater operation would provide information in this area, recognizing that interpretation would be difficult because of the presence of the web stresses.

Web stresses caused by the reheat gas flow are much lower than those caused by the blowdown air flow with the exception of the top foot or so of matrix, because the rate at which the bed is heated is much lower than the rate at which the bed is cooled. Also, the air flow produces tensile stresses at the surface of the holes, whereas the reheat flow produces tensile stresses inside the webs. The former condition is more critical. Consequently, air flow conditions that would produce excessive web stresses must be avoided. The analysis used to establish the air flow limitations is described in Section 3.4.

The web tensile stress is proportional to the rate at which the web is cooled, which, in turn, is proportional to the air flow rate. A bed designed to give maximum performance will have a large fraction of its
cored bricks operated at the design stress level. Therefore, excessive flow rates could cause wide scale web cracking throughout the bed. Further, this would happen during the blowdown process when structural failure of the bed could result in broken pieces being blown out of the heater. Consequently, operational safety and bed life were judged to be more dependent upon the web stresses than upon other conditions, such as body stresses and creep.

Many of the bricks in the bed will be cooled through a temperature range of several hundred degrees during a tunnel run. (It is interesting to note, in comparison, that the temperature difference within the webs is only about 40°F at the maximum conditions.) Nonuniform cooling can produce large temperature variations within a brick and therefore, large "body" stresses. Uniform flow through the holes is needed, which requires that the bricks be held in alignment. This was achieved through the use of tongue and groove keys.

The top and the bottom of the bed deserve mention. At the bottom the cold air causes rapid cooling and tensile stresses. The grate structure and the metal grate plates provide support and also receive the frontal blast of the air. The alumina bricks at the grate are never operated at high temperature, but always in a range where the thermal conductivity and strength are high. The actual condition of the bricks near the grate will not be known until the bed is disassembled sometime in the future. But the test program did not reveal any problems with the alumina.

The top of the bed receives the hot combustion gases from the burners. Failure from too rapid heating is definitely possible. For this reason, the upper layers of the bed are thin (1/2 to 1 inch) cored bricks (called buffer layers) which develop lower stresses than the long bricks. Burner flow rates and temperatures are controlled to limit rapid temperature changes and, therefore, limit thermal stresses during the reheat process.
3.4 **Web Stress Analysis**

The web stresses caused by the air flow were analyzed on the basis of an idealized model (Reference 1, Appendix H). The stresses that would be caused by the vertical and radial temperature distributions were ignored (i.e., the body stresses). This is equivalent to assuming that the radial temperature across the bed is uniform and that the vertical temperature distribution does not contribute to the stresses. Remembering that a linear temperature distribution existing across a material with temperature independent properties does not create stresses, makes the latter assumption fairly reasonable, except at the ramp-plateau junction.

Under these assumptions, a single cored brick becomes equivalent to a nest of tubes, each with a circular hole and a hexagonal outer surface. The hexagonal tube was further simplified to an equivalent round tube having the same cross-section area in the tube wall. The hole pattern used is one with a hole diameter of 0.194 inch and a web thickness of 0.085 inch. The equivalent circular diameters are 0.194 I.D. and 0.293 O.D. For the circular tube, the elastic stress at the surface is (plane strain with tube ends not restrained, References 7 and 8):

$$ S = \frac{\alpha F}{1-\nu} \left( T_{ave} - T_{surf} \right) $$

(1)

where $S$ is the surface stress, $T_{ave}$ is the average temperature of the tube wall, and $T_{surf}$ is the temperature of the surface in question (inner or outer). In this application, the inner wall stress is desired, and $T_{ave}$ is also equal to the average temperature through the web of the cored brick.

The relationship of the $\Delta T$ of Equation (1) to the hole pattern dimensions and the heater operating conditions is presented in Reference 1.
(Appendix H). Equating the heat transferred from the ceramic (i.e., from the equivalent circular tube) to the air gives the following equations:

\[ T_{ave} - T_{surf} = \frac{R_3}{8} \frac{D^2}{k_s} \frac{C_p}{A} \frac{m}{dz} \]  

\[ T_{ave} - T_{surf} = -\frac{R_1}{8} \frac{D^2}{\rho} \frac{dT}{dV} \]  

\[ R_1 \text{ and } R_3 \text{ are functions of the equivalent tube O.D./I.D. (} R_a = 1.51 \text{) and are equal to .22 and .38, respectively, for the geometry used. The temperature on the right hand side is the temperature at any location within the web. Examination of computer solutions for the temperature distribution through the bed has shown that this is a good approximation for the ramp portion of the bed temperature distribution, except near the cold air inlet.}

Equations (1), (2), and (3) provide the desired relations among the ceramic properties, the tube inner and outer diameters (or cored brick hole diameter and web thickness), and the bed operating conditions: mass flow per unit area, slope of the temperature ramp, and temperature rate of change. The equations can be applied to any vertical location in the bed using material properties corresponding to the local temperature. By setting the stress in Equation (1) equal to the tensile strength, the maximum allowable temperature difference is obtained. Equation (2) will then give the maximum allowable flow rate and temperature ramp, and Equation (3) will give the corresponding time rate of change of temperature.

This procedure was applied in Reference 1 to alumina and calcia-stabilized zirconia through use of property data from the literature. In order to apply literature data to design, it is necessary to account for
the strong dependence of strength and modulus of elasticity on material density. The relations used are (Reference 1, p. 96):

\[
S_t = S_{to} e^{-7p}
\]

\[
E = E_o e^{-4p}
\]

where \( p \) is the porosity and the subscript zero denotes zero porosity. With these relations, the corresponding result from Equation (1) is:

\[
\Delta T = T_{ave} - T_{surf} = (T_{ave} - T_{surf}) o e^{-3p}
\]

A plot of the temperature difference versus material temperature, using Equation (1), indicates that the lowest temperature differences occur in the range, 1000° to 3000°R, for both alumina and calcia-stabilized zirconia (Reference 1, Figure 7c). The porosity levels of interest in the present program are approximately 5% for alumina and 10% for zirconia. Corresponding minimum values of \( \Delta T \) from Reference 1 are 40°R for alumina and 60°R for zirconia.

From Equation (2), the allowable mass flow is proportional to \( K_s \Delta T/C_p \). With an increase in temperature, the conductivity of alumina decreases rapidly (for temperatures to about 3000°R), the specific heat of air increases, and, therefore, \( K_s \Delta T/C_p \) decreases. Hence, the critical temperature for alumina is the highest value which occurs at the alumina-zirconia interface in the bed.

The thermal conductivity of zirconia increases slightly with temperature (Reference 1, Figure 3c). Using the property values of Reference 1, the parameter \( K_s \Delta T/C_p \) varies only 12% in the temperature range, 1000° to 3000°R and rises for temperatures above and below this range. The
actual minimum occurs at 2500°R, but this is not too significant because of the uncertainty in the properties. The mass flow limit from Equation (2) can be determined by using zirconia properties within the temperature range, 1000° to 3000°R, or the minimum temperature, if it is above 3000°R. This requirement is satisfied by using the temperature at the alumina-zirconia interface.

Comparison of air flow rates passing through alumina and zirconia cored bricks, as given by Equation (2), shows that zirconia will have the lowest allowable flow rate. Consequently, estimates of the web stress limited flow rates have been based upon the zirconia portion of the bed and specifically, by use of properties at the interface temperature. As noted above, the property values combine to give \( k \Delta T C_p \) values that are nearly independent of temperature in the range, 1000° to 3000°R. The minimum values, together with the chosen hole pattern dimensions, give the following results from Equations (2) and (3) for zirconia of 10% porosity:

\[
\frac{\dot{m}}{A} \frac{dT}{dz} = 5800 \text{ lb} \cdot \text{°R} \text{ sec ft}^3
\]

\[
\frac{dT}{d\theta} = 50° \text{ R/sec}.
\]

The web thickness at the outer surface of the hexagonal bricks is the same as that between the holes (Drawing 7003-008C). However, the outer web will be cooled from only one side when adjacent bricks are in contact. This will increase the local stresses. An approximate correction for this effect was made by increasing the wall thickness of the equivalent tube. The larger value was calculated by using the true ratio of open area to total area of the hexagonal brick, which is 0.40 (the repeating hole pattern gives .44). The equivalent tube with this ratio of open to total area has an O.D./I.D. of 1.58. This increases the value of \( R_3 \) used in Equation (2) from 0.38 to 0.47, which then gives:
\[ \frac{\dot{m}}{A} \frac{dT}{dz} = 5800 \text{ lb °R/sec ft}^3 \]

\[ \frac{dT}{d\theta} = 50^\circ\text{R/sec}. \]

The web thickness at the outer surface of the hexagonal bricks is the same as that between the holes (Drawing 7003-008C). However, the outer web will be cooled from only one side when adjacent bricks are in contact. This will increase the local stresses. An approximate correction for this effect was made by increasing the wall thickness of the equivalent tube. The larger value was calculated by using the true ratio of open area to total area of the hexagonal brick, which is 0.40 (the repeating hole pattern gives 0.44). The equivalent tube with this ratio of open to total area has an O.D./I.D. of 1.58. This increases the value of \( R_3 \) used in Equation (2) from 0.38 to 0.47, which then gives:

\[ \frac{\dot{m}}{A} \frac{dT}{dz} \text{ fracture limit} = 4700 \text{ lb °R/sec ft}^3 \]

\( R_1 \) increases from 0.22 to 0.29, giving

\[ \frac{dT}{d\theta} \text{ fracture limit} = 38^\circ\text{R/sec}. \]

These results provide a basis for determining operational limits for the heater. It is emphasized that they are based on property data from the literature for calcia-zirconia and upon an elastic stress analysis that considered only the stresses produced by the web temperature distribution during blowdown, i.e., that does not include the "body" stresses. In applying these results, a factor of safety is introduced to account for the various uncertainties. Tests were also made to confirm these results. Based upon those tests (Section 3.5), operational limits of one-half the above values were selected.
Since the completion of the work described in this report, another reference (Reference 10) dealing with a similar thermal stress problem has come to the authors' attention and provides additional useful information. Reference 10 describes a thermal stress analysis of a gas-cooled nuclear reactor. The reactor elements studied were tubes with circular holes and a hexagonal exterior. The additional stress caused by the hexagonal shape, as compared with the equivalent circular tube (as used above), was calculated. The maximum tensile stress occurred on the inner surface and at the thinnest part of the tube wall. The stress was 40% larger than that given by Equation (1) for the dimensions of interest here. Application of this result reduces the numerical values given above to 3600 lb °R/sec ft³ and 36°R/sec.

The relationship between mass flow and the vertical temperature gradient (dT/dz) within the bed indicates that a linear vertical temperature distribution will allow maximum performance. This distribution is controlled through design of the insulation around the bed (i.e., control of heat losses) and through control of the reheat gas temperature and mass flow rate. The effect on the insulation design was to require less thermal resistance at the low temperature end of the bed (see Reference 1 for discussion).

3.5 Web Stress Tests

As discussed in the text, the high temperature material chosen for the matrix was yttria-stabilized zirconia. The results described above were based upon published properties of calcia-zirconia. This difference, combined with the dependence of ceramic properties upon manufacturing methods, required that additional information be developed. One approach could have been to measure the high temperature properties and then use the method of Section 3.4 or even a more complete analysis. It was decided instead to perform subscale tests and to use Equations (1) through
(3) to scale the results to the full-scale size. Tests were made (Reference 4) in two steps. Both steps involved heating and cooling a small bed of cored bricks (or pieces cut from cored bricks) in a way similar to the large heater operation. Heating was relatively slow, but cooling was fast and at controlled rates, with the web temperatures measured with platinum/rhodium thermocouples.

The first step employed a bed 1-1/2 inches in diameter by 6 inches long. Specimens were heated to 3200°F and then cooled by blowing air. Runs were made in groups of five with the cooling rate increase successively at roughly 5°F/sec intervals. The specimens fractured early in the test because of the large radial and axial temperature variations (body stresses). But the onset of web cracking was clearly evident as the cooling rate was increased. The results gave a wide range for the three yttria-zirconia materials tested: 20°F/sec, 50°F/sec, and 65°F/sec.

The second test employed a bed 5 inches in diameter by 6 feet long. The bed contained alumina at the cooler end and the remainder was zirconia (three kinds, side-by-side). This test was made at a cooling rate of 25°F/sec. The hole pattern was nearly identical to the .194 x .085 used in the heater. These results were qualitatively similar to those obtained with the smaller set-up. Again, the bricks fractured because of the large radial temperature variations, but there was very little evidence of web cracks. The conclusion was that 25°F/sec represented a reasonable design value, but that the radial and vertical temperature effects could be damaging. It was recognized that these temperature variations would be less severe in a larger heater and that some fracturing could occur without impairing the usefulness of the bed.

Thus, the decision was made to base the bed thermal stress limited performance upon a cooling rate of 25°F/sec and a corresponding value $\frac{m}{A} \frac{dT}{dz} = 2350 °F/sec$. Performance curves based upon this limit are presented later.
4.0 CERAMICS EVALUATION AND SELECTION

Alumina and magnesia were the first materials considered. The lower temperature portions of the heater would be alumina, and the higher temperature portions, upper half of the heater, would be magnesia. Magnesia has the disadvantage of a high vapor pressure, and its characteristics had not been previously investigated under conditions that simulated the heater environment. Therefore, small scale tests were performed to measure the weight loss characteristics in a flowing hot gas. It was determined that the loss rates were too large, and consequently, the alternate material choice, zirconia, was selected. The magnesia tests are described in Section 4.1.

As mentioned in Section 1.0, a concurrent heater development program was being carried on by the Air Force (References 1, 2, 4, and 5). This work had led to the selection of zirconia for development as the high temperature material. Tests of various zirconia compositions were planned (under the Air Force program) which would provide material selection and design information. It was concluded, therefore, that a part of the zirconia materials tests be performed under the NASA program. The entire test program and results are described in References 3 and 4, and are briefly reviewed in Section 4.2 of this report.

Alumina materials for the matrix and insulation design were available. Manufacturing techniques for the cored brick fabrication were known, and actual experience with dense alumina cored brick in operating heaters existed.

4.1 Magnesia

A small test facility (Figure 2) which simulates a full-scale storage heater was used to perform the magnesia tests to investigate weight loss
characteristics. Magnesia samples were obtained from several suppliers and were tested in a flowing combustion atmosphere (pure oxygen and propane). Two separate tests were performed, one with the specimens held at 4660°F for 25 hours, and the other with the specimens at 4160°F for 60 hours. The combustion gas flow rate approaching the sample was 430 lbs/hr-ft², which corresponded to the maximum anticipated burner reheat flow for the pilot heater.

Photographs of the specimens before and after the 4160°F test are shown in Figure 3. The weight loss rate during this test was 4 mgm/cm²-hr, which corresponds to a surface recession rate of 1/2 mil per hour. This loss rate is too high. It would cause severe loss of material at the top of the bed and also lead to a serious dust problem. The vaporized magnesia would condense in the lower part of the bed. Magnesia condensate found after the 4160°F test is shown in Figure 4. This material has a very weak, friable structure and, therefore, would be dislodged by the air during heater blowdown.

The results of these tests compare favorably with the measurements reported in Reference 11 and shown in Figure 5. The data suggest that 3500°F is a reasonable upper limit for magnesia when exposed to a flowing gas environment where the vapor is continuously removed. Consequently, no further work was done with magnesia.

4.2 Zirconia

Zirconia (Zirconium dioxide) is a highly refractory material with a melting temperature of about 5300°F. It is polymorphic and undergoes a reversible crystalline phase change (monoclinic-tetragonal) at about 2400°F. The phase change is accompanied by a destructive volume change. This volume change reverses between heating and cooling and occurs over a relatively narrow temperature range. Repeated cycling through this
temperature range results in complete fragmentation of the material. Zirconia is made a useful refractory by suppressing the phase inversion (and volume change) through addition of a second oxide, referred to as the stabilizer. The stabilizer, when taken into solid solution, "stabilizes" zirconia in the cubic crystalline phase. The most common stabilizing materials are calcia, magnesia, yttria, yttria-rare earth oxide mixtures (YRE) and combinations of these. The stabilizers are added in quantities to convert all or part of the zirconia to the cubic phase, hence, the terms, "fully-stabilized" and "partially-stabilized" zirconia.

The level of stabilization is measured by the amount of monoclinic phase that exists at room temperature. A fully-stabilized material will have, by definition, no monoclinic content. However, the term is commonly used to describe materials that do, in fact, have a few percent of monoclinic phase. Partially-stabilized zirconias will typically have 15% to 30% monoclinic phase.

Experience has shown that the monoclinic content will tend to increase when zirconia is cycled through the monoclinic-tetragonal phase inversion. This process is called destabilization. As the monoclinic content increases, the effect of the volumetric change caused by the phase changes increases, which leads to a loss of strength and frequently renders the material unusable.

Destabilization will also occur if the stabilizer itself is removed from the zirconia. This has been found to happen with calcia and magnesia-stabilized zirconia when exposed to high temperature for long periods. (References 3, 4 and 5).

The major factors to be considered in selecting and specifying a particular zirconia material for a particular application may be summarized as follows:
1. Volume Porosity. Affects the mode of failure, as discussed in Section 3.2. Affects thermal conductivity, which decreases as porosity increases.

2. Partially-Stabilized or Fully-Stabilized. Partially-stabilized has lower, overall thermal expansion, but destabilizes.

3. Monoclinic Content. May be specified to assure zero or near-zero value for fully-stabilized material.

4. Kind of Stabilizer. Calcia and magnesia are inexpensive, but give lower service temperatures than yttria and yttria-rare earth (YRE) mixtures.

5. Amount of Stabilizer. Usually selected by manufacturer to produce either partially- or fully-stabilized material. Was included in YRE-zirconia specification because of cost and concern that an excess might reduce the creep resistance.

6. Impurities. Commercial materials typically 2%. Impurities that cause glassy phases detrimental at high temperature and therefore, restricted to limits established by current manufacturing methods and cost (see specifications).

7. Firing Temperature. Usually selected by manufacturer, current limits in range, Cone 33 to 35 (3630° to 3700°F), which is below maximum heater temperature. May be specified.

Much of the information needed to make these choices came from special tests made under this program and the concurrent Air Force program. These tests and the results have been described earlier in References 3, 4, and 5. A brief review is given in the next section.
4.2.1 Tests of Zirconia

Tests were made primarily to identify compositions that would not destabilize because of temperature cycling and because of loss of stabilizer at high temperature. In each case, the composition and phase structure of the specimens were measured before and after the tests.

Phase changes caused by temperature cycling were determined by heating and cooling specimens through the temperature range, 1000° to 2500°R, for a total of 150 cycles. Tests were made in an air atmosphere and in a combustion-fired kiln, in case there was an effect of gas composition. No such effect was found.

The specimens tested included calcia-, magnesia-, yttria-, and YRE-stabilized zirconias. All partially-stabilized materials destabilized, i.e., showed an increase in monoclinic content. The fully-stabilized material did not. Some of the "fully-stabilized" specimens contained up to 5% monoclinic phase before the test, and the content did not increase (Reference 3). This suggested that, although zero monoclinic content is desired, some small amount may be tolerable. Later experience with YRE-stabilized zirconia in the Air Force heater indicated that monoclinic levels even less than 3% can lead to destructive destabilization (References 4 and 5).

Preliminary tests at about 4200°R had shown that calcia and magnesia were lost from the material to the gas stream (Reference 3). The target temperature for the material was 4660°R. Therefore, a test was made with various specimens (as above) held at this temperature in a combustion gas stream for 240 hours. The calcia- and magnesia-stabilized materials did destabilize because of loss of the stabilizing agent. The yttria- and YRE-stabilized materials did not destabilize and did not lose their stabilizing agents. It was concluded that of this group of stabilizers, only the yttria and YRE materials were suitable for the high temperature portions of the heater. YRE was selected instead of yttria because of its lower cost.
4.2.2 Zirconia Specification

On the basis of the above test results and other information available at that time, maximum interface temperatures were established for the high temperature ceramics as follows: calcia-zirconia in contact with YRE-zirconia, 3960°F; alumina in contact with calcia-zirconia, 3260°F. An initial selection of materials was made on this basis. Each zirconia composition was to be fully stabilized. The YRE stabilizer level was specified to be 9-1/2% on the basis of the tests discussed in Section 4.2.1. This value was later increased to 10.4% for the cored bricks, but was left at 9-1/2% for the insulation. Fabrication difficulties with the calcia-zirconia cored brick resulted in its being dropped, so that the final bed contains only alumina and YRE-stabilized zirconia. The only advantage of the calcia-zirconia was cost, and in the small amount needed for the bed, this factor was not significant.

Specifications were prepared for those materials that were new, in terms of a commercial product, or where a simpler description was not sufficient. These specifications and corresponding quality control procedures are presented under specifications. All materials used are identified in the Refractory Assembly Drawing 0456-026B.

4.3 Cored Brick Procurement

Due to the development nature of the ceramics, liaison between possible material suppliers was carried on continuously to provide some assurance that the final materials and shapes selected could be produced economically in pilot heater quantities with uniform product consistency. The specifications and quality control procedures were developed in conjunction with manufacturers to assure that the requirements were within their current fabrication abilities. However, fabrication problems were encountered, and the specifications were modified accordingly. These changes and the fabrication experiences are discussed below.
As noted earlier, the first bed design consisted of three materials: alumina in the lower temperature region, fully-stabilized calcia-zirconia in the mid-temperature range, and 9-1/2 w/o YRE-zirconia in the high temperature region. Coors Porcelain Company and Zirconium Corporation of America (Zircoa) had supplied the test specimens for the materials evaluation and were selected to fabricate the cored bricks. Coors fabricated the alumina bricks. The calcia and YRE-zirconia requirements were to be supplied equally by Coors and Zircoa, i.e., half the calcia-zirconia and half the YRE-zirconia were to be supplied by each. This dual-supplier procurement was undertaken to provide a competitive market and to provide a safeguard should one supplier be unable to produce a satisfactory cored brick.

The two manufacturers used different fabrication methods. Zircoa attempted to use the die pressing technique, where a metal die is filled with powder and pressure is applied from two ends. This method did not prove satisfactory. The basic difficulty was the height of the brick (minimum of 4 inches). Die pressing probably could be used for a brick height of 1 to 2 inches.

Coors used the isopressing method. This did prove successful, and all of the cored bricks were subsequently manufactured by Coors. However, an initial lot of 9-1/2 w/o YRE-zirconia bricks were found to have monoclinic contents in the 5% range. To reduce the monoclinic level the YRE content was increased to 10.4 w/o. The production lot was successfully produced with a monoclinic content of less than 1%. Coors had difficulty producing a satisfactory calcia-zirconia cored brick, and, therefore, YRE-zirconia bricks were substituted in their place.

4.4 Insulation Materials

The insulation design and materials selection were based on several considerations: 1) provide thermal protection for the vessel
wall and maintain the vessel wall at near constant temperature to avoid creating thermal stresses, maximum vessel temperature - 960°C; 2) provide a uniform heat loss along the length of the bed, which tends to produce a linear temperature profile during bed heating; 3) provide materials with thermal stress damage resistance and with relatively tight surface texture to minimize the dust contamination; and 4) provide materials that are compatible with the bed materials and with adjacent insulation, so that reactions between materials are avoided.

The insulation is a built-up refractory brick structure consisting of two brick layers and a thin section of fiber insulation near the vessel wall (Drawing 0456-026B). The total insulation thickness is approximately 8 inches. Tongue and groove bricks are used for the hot liner, and standard arch bricks are used for the backup insulation.

An additional consideration given to the selection of the insulation was the possible effects of rapid pressurization and depressurization of porous insulating brick. Special pressurization-depressurization tests performed on full-scale bricks (Reference 4) gave assurance that damage would not occur. These tests were made at room temperature with pressurization and depressurization rates up to 1200 psi/second at a maximum pressure of 3500 psi.

Specific materials were selected on the basis of the test results and past experience. The bed liner materials selected were alumina in the lower temperature portion of the heater, fully-stabilized calcia-zirconia in the mid-temperature range, and 9-1/2 w/o YRE-zirconia in the high temperature region of the heater. The backup insulation follows the same pattern, except in the high temperature regions where fully-stabilized calcia-zirconia is used between the YRE-zirconia bed liner and vessel wall. The insulation specifications for the alumina and calcia-stabilized zirconia are defined on the Refractory Assembly Drawing 0456-026B. The YRE-zirconia insulation specification, 7000-108C, is enclosed in the Specifications Section.
All of the zirconia insulation materials were fabricated by Zircoa. The quality control procedures outlined in Specification 7000-120B were followed. In addition, sample test cubes fabricated from the same material batches as the insulation bricks were subjected to 15 cycles between 560° and 2660°F as a check on the permanence of stability. The results showed that all of the zirconia bricks installed in the heater contained less than 1% monoclinic phase, and there were no indications of destabilization.

The alumina bed liner and backup arch bricks were supplied by the Norton Company and Harbison-Walker, respectively.
5.0 MATRIX DESIGN AND PERFORMANCE

5.1 Cored Brick Dimensions

The cored brick dimensions are shown in Drawing 7003-008C. As described in Section 3.2, small holes and thin webs are needed to permit rapid extraction of the stored energy (requiring large surface area and short heat conduction paths). Thin webs are needed to reduce the web thermal stresses. On the other hand, there is a lower limit on the hole diameter because of the need to maintain hole alignment. Fabrication tolerances also restrict the selection of dimensions.

These considerations were studied for both the Air Force pilot heater (Reference 4) and for the present heater. As noted in Section 1.0, it was decided to use the same brick geometry in both heaters. The final selection of hole diameter and web spacing provides high heat transfer performance, low pressure drop, and is within current limits of fabrication technology. The fabrication in alumina poses no significant problems. However, in zirconia, the combination of shape, low ceramic porosity, purity, and low monoclinic content combine to require careful fabrication procedures.

The hexagon flat-to-flat and length dimensions were selected in part on the basis of later using this same brick shape in larger heaters and upon an early estimate of the cost-size relationship.

The bricks were fabricated by isostatic pressing. Efforts at fabrication by die-pressing (die cavity with pressure from two ends only) were not successful. Consequently, the fabrication cost savings inherent in the relatively rapid die-pressing process were not available. Costs of isostatic pressing were significantly dependent upon the number of bricks, especially because the tongue and groove keys had to be machined to their final shapes. Therefore, there was a desire to use large bricks in an effort
to reduce cost. The cost savings, of course, would be most significant with large heaters. The overall brick dimensions are as large as could be produced and still maintain the desired tolerances.

The overall brick dimensions are larger than needed in a heater of this size. Bricks of about two inches across and two inches long should be very satisfactory. Smaller bricks would have the important advantage of reduced body thermal stresses.

Keying of bricks is important to preserve hole alignment and thereby prevent nonuniform flow through the holes, which would cause nonuniform cooling. Nonuniform cooling could create large temperature variations within a brick, because the individual bricks cool through a temperature range of several hundred degrees during a run. This could cause high "body stresses" as described in Section 3.3. The key configuration shown in Drawing 7003-008C was selected as a compromise in order to simplify machining. An improved configuration has been used more recently with the bricks for the NASA/Ames 3.5 foot hypersonic tunnel heater. The key has radial tongues and grooves that extend to three of the points of the hexagon. This configuration provides better rotational alignment.

5.2 Air Flow Performance

This section describes limitations imposed on the air flow rate by flotation of the bed and by thermal stresses; the latter are found to be the most restrictive. Limits for the heater pressurization rate and for the steady flow rate are developed. The most critical air flow control requirements exist during pressurization. During this period, the maximum flow rates occur (exceeding the eventual steady values) and cause the highest thermal stresses associated with cooling of the bed. A flow control method using the measured pressure drop across the bed is described.
5.2.1 Flotation

The bed is held in place by gravity. There is also friction between the bed and the adjacent insulation. These frictional forces are of uncertain magnitude and, therefore, they are ignored in the following analysis, which tends to make the results conservative.

The bed or a portion of the bed will be lifted when the force due to air pressure drop exceeds the weight to be lifted. The flotation criterion is, therefore, that for all values of \( z \),

\[
\Delta P \frac{L}{z} \leq W A (L - z)
\]

A special, but significant case is that of study air flow with the bed at a uniform temperature from top to bottom. Under these flow conditions, the entire bed will tend to lift (i.e., \( z = 0 \) in above criterion) if the air flow \( \Delta P \) is excessive. This limit on \( \Delta P \) in this case, is the bed weight divided by the bed area (1.07 ft\(^2\)). The specified densities of the alumina and zirconia are 3.83 g/cc and 5.40 g/cc, respectively. From Drawing 7003-008C, the volume of the cored brick consists of 40% holes and 60% ceramic material. Therefore, the bed weight per unit volume (\( W \)) is 143 lb/ft\(^3\) for the alumina portion and 202 lb/ft\(^3\) for the zirconia portion. From the bed dimensions, the alumina weight is 610 lbs. and the zirconia weight is 1390 lbs., total 2000 lbs. The corresponding flotation pressure drop is 13.0 psi. This value must never be exceeded, but, furthermore, the normal operating conditions are much more restrictive. The effects of the vertical temperature distribution in the bed and the transient flow during heater pressurization must be considered.

Under steady flow conditions, the vertical temperature variation causes the velocity and local pressure gradient to increase as the air
moves upward. In this case, the uppermost layer of the bed will tend to lift first. The flotation criterion in this case reduces to:

\[
\frac{dP}{dz} \leq \frac{W}{L} \quad (W = 202 \text{ lb/ft}^3)
\]

\[
= 1.40 \text{ psi/ft.} \quad (4)
\]

It was feasible to measure only the pressure difference across the entire bed. Therefore, it is necessary to relate the pressure gradient at the top of the bed to the bed \( \Delta P \). The equation for frictional pressure drop in a tube,

\[
\frac{dP}{dz} = \frac{f}{D} \frac{\rho u^2}{2g}
\]

is first rewritten in terms of bed design parameters, assuming uniform flow through all of the holes in the cored brick matrix.

\[
\frac{dP}{dz} = \frac{fRT m^2}{2gPD} \left( \frac{m}{A} \right) \frac{1}{\sigma^2}
\]

Integrating for steady flow and assuming that the local temperature difference between the air and ceramic is negligible, gives:

\[
\frac{\Delta P_{\text{bed}}}{L} = \frac{P_L}{\bar{P}} \frac{\bar{T}}{T_L} \left| \frac{dP}{dz} \right|_L^{-L} \quad (5a)
\]

\[
= \frac{fRT m^2}{2gPD} \left( \frac{m}{A} \right) \frac{1}{\sigma^2} \quad (5b)
\]

where:

\[
\bar{T} = \frac{1}{L} \int_0^L T \, dz \quad \text{and} \quad \bar{P} = \frac{P_L + P}{2} \quad \text{at} \quad z = 0
\]
Under steady flow conditions, the flotation value of $\Delta P_{\text{bed}}$ is generally much less than the heater pressure levels, and, therefore, it can be assumed that:

$$\bar{P} = P_L = P_{z=0} = P_0$$

where: $P_0$ is the nozzle stagnation pressure. This assumption is valid for the operating conditions of interest and was used in developing flow rate limitations.

Combining Equations 4 and 5a gives:

$$\Delta P_{\text{flotation}} = 14.6 \frac{T}{T_L} \text{ (psi)}$$

for the bed $\Delta P$ at which the top of the bed will lift under steady flow conditions.

Examination of data from the shakedown runs (discussed in Section 7.3) indicated that the ratio $\frac{T}{T_L}$ varied over the range, 0.6 to 0.7. Thus, the bed flotation pressure drop is in the range, 8.8 to 10.2 psi. It will be seen later that much lower values are imposed by thermal stress limitations. (If this were not so, these limits would apply, and it would only be necessary to select a suitable safety factor for the flotation $\Delta P$.)

The steady flow results do not apply to the heater pressurization process. Calculations were made by digital computer to examine restrictions on pressurization. Both flotation limits and thermal stress limits were imposed. For pressurization at the maximum allowable rate, the thermal stress limits restricted all except the very early part of pressurization (below pressures on the order of 50 psi).
During pressurization, air is stored in the heater and, therefore, the inflow exceeds the outflow. Hence, the mass flow entering the bed exceeds that leaving the bed. This tends to increase the pressure gradient at the bottom of the bed, relative to that at the top, in opposition to the effect of the vertical temperature distribution. At the beginning of pressurization, this effect can be large enough to cause the entire bed to be lifted if the \( \Delta P \) is excessive. In this case, the limit \( \Delta P_{\text{bed}} = 13.0 \) psi (calculated above) would apply. However, in practice, the heater is not pressurized at the maximum allowable rates, and consequently, pressure differences of this magnitude are not developed. The basis for control of the pressurization rate is described in Section 5.2.2.

5.2.2 Thermal Stress

The thermal stress limitations were based upon the web stress analysis described in Sections 3.4 and 3.5. The test results of Section 3.5 indicated that yttria-zirconia cored bricks having a density of 5.4 g/cc (10% porosity) would survive web stresses corresponding to \( \frac{m}{A} \frac{dT}{dz} = 2350 \) lb °R/sec ft\(^3\). The literature data for calcia-zirconia suggested that this was equivalent to a factor of safety of two on web stress. (Based on the equivalent circular tube analysis, the work of Reference 10 suggests that the factor of safety is 1.4.)

The above value of 2350 lb °R/sec ft\(^2\) places limits on flow rates that are directly applicable to steady flow conditions in the heater. As noted in Section 3.4, the critical temperature range for zirconia, in terms of thermal stress resistance, is the range 1000 to 3000 °R. Therefore, for any bed temperature distribution (such as those shown in Figures 18 and 22 through 26), the maximum temperature slope \( \frac{dT}{dz} \) that exists within this temperature range should be used with the equation \( \frac{m}{A} \frac{dT}{dz} = 2350 \) to determine the maximum allowable flow rate.
Determination of comparable air flow limits that apply during the pressurization process is more complicated because, during pressurization the flow rate decreases from the bottom to the top of the bed. If the temperature ramp is linear, (as in Figure 18), the maximum web stress in the zirconia occurs at the alumina-zirconia interface. Therefore, a number of computer calculations of the pressurization process were made with various temperature distributions of the form shown in Figure 18. In each case, the flow rate at the interface was set equal to the maximum allowed by \( \frac{\Delta T}{A} \frac{dT}{dz} = 2350 \). To pressurize in finite time, this flow had to exceed the final steady flow rate; 10% excess yielded short pressurization times and, therefore, was used. The time interval to pressurize from 1/10 the final pressure to the final pressure was calculated. (The method was not accurate at low heater pressure levels; therefore, the calculation was started at 1/10 the final pressure.)

The calculated results showed that the pressure drop across the bed decreased with time until the final steady conditions were reached. This appeared to be an impractical control requirement and unnecessary because the corresponding pressurization time intervals were short. Additional calculations were made with the pressure drop across the bed held constant at the steady flow value. In these cases, the bed flow rates were still highest at the bottom of the bed, but they did not reach the levels of the earlier calculations. The time required to reach steady pressure was increased by only a few seconds. Therefore, this method of pressurizing was selected, i.e., pressurization at a constant value of bed \( \Delta P \), equal to the steady run value.

The web stress limit equation used here is plotted in Figure 6. The plot shows the steady flow rate values and assumes that, during pressurization, the flow rates in the lower part of the bed exceed the steady value by as much as 10%, as discussed above.
The relation between flow rate and bed pressure drop was given earlier as Equation (5b). The pressure drop can be related to throat size and stagnation conditions by combining Equation (5b) with the equation for flow through a choked throat (plotted in Figure 7),

\[
m = C \frac{P_o A^*}{\sqrt{T_o}}
\]

gives:

\[
\Delta P_{\text{bed}} = \frac{C^2}{2} \frac{f R L}{gD} \frac{T}{\sigma^2 A^2} (P_o A^*)^2 \left( \frac{T}{T_o} \right) ^{T_c R / T_o}.
\]

The friction factor was estimated to be 0.05. This value accounted for the size and slight roughness of the holes and included a small increment for the effect of misalignment between bricks. The equation becomes

\[
\Delta P (\text{psi}) = 0.00198 \frac{P_o (\text{psia})}{(A^* \text{in}^2)^2} \left( \frac{T_c R}{T_o} \right) \frac{T^o}{T_o}
\]

and is plotted in Figure 8. The stagnation temperature is assumed equal to the ceramic temperature at the top of the bed \( T_L \). Thus, the ratio \( T/T_o \) varies from about 0.6 to 0.7, as mentioned in Section 5.2.1.

The air stagnation will be lower than the temperature at the top of the bed. Approximate calculations for temperature distributions of the type shown in Figure 18 gave air temperatures approximately equal to the matrix temperature at the ramp-plateau junction, for the initial flow period of about 10 seconds duration. As flow continues, the air temperature will drop. The assumption of \( T_o = T_L \) made above is sufficiently accurate for establishing the \( \Delta P_{\text{bed}} \) values for heater pressurization, but it is not accurate enough for the air \( T_o \) determination.
5.2.3  Operating Procedure

A procedure for using the above results is outlined here. The purpose is to determine the stagnation pressure and bed pressure drop, as limited by web thermal stresses, for a given throat size and initial bed temperature distribution.

1. Determine $dT/dz \left[ \right]_{\text{max}}$ from the bed temperature distribution. The maximum value is that for the yttria-zirconia in the $1000^\circ \text{C}$ to $3000^\circ \text{R}$ temperature range.

2. Read the steady mass flow rate from Figure 6 for a safety factor of two or larger (as desired).

3. Calculate the nozzle stagnation pressure (= heater pressure) from the nozzle equation or use Figure 7.

4. Read the bed pressure drop from Figure 8.

The heater is to be pressurized at the constant value of bed $\Delta P$ obtained from Step 4. Exceeding this $\Delta P$ by a factor of 2 or 3 during the early part of the pressurization will not cause excessive stresses or flotation.

The relation between pressure drop and mass flow depends upon the accuracy of the friction factor and therefore, requires experimental verification. The shakedown runs verified that the prediction was satisfactory for most of the runs and is discussed later. Logging of pressure drop data may provide a useful record to indicate the onset of bed deterioration.
6.0 SYSTEM MODIFICATIONS AND OPERATING HARDWARE

6.1 Vessel

To improve the utility of the existing vessel, provide for the installation of a new ceramic configuration, and provide improved operating characteristics, several modifications to the existing vessel were made. These modifications include:

a. rearrangement of the combustion gas exhaust outlet and high pressure inlet to the vessel;

b. installation of a grate system to support the matrix;

c. installation of a spacer flange and shelf between the upper vessel head and cylinder;

d. installation of two viewports to provide a means to measure the matrix temperature optically;

e. installation of a water crack-ceramic sleeve assembly in the heater outlet for thermal protection of the upper vessel head.

The lower vessel head was modified by moving the combustion gas outlet-high pressure air inlet from the side of the vessel to the lower head. This allowed the matrix design to utilize the full length of the vessel, Drawing 0456-026B. The side outlet was capped and a new outlet elbow installed at the bottom of the vessel; the new outlet is shown on Drawing 0456-901. A stainless liner was installed to protect the elbow and welds from the thermal stresses which arise from introducing cold, blowdown air into the piping, which has been heated during the flow of combustion gases.
A stainless steel grate system was installed in the lower vessel head to provide support for the entire ceramic bed and provide proper flow entrance conditions to the bed, Drawing 0456-901. A 1-inch thick plate, perforated with the cored brick hole pattern, was placed between the cored brick bed and support structure to provide a thermal mass to reduce the thermal shock to the cored brick during blowdown. This plate also incorporates a keying feature which locks the cored brick columns to the plate and assures hole alignment. The grate temperature is limited to 1460°F for structural reasons. To provide a safeguard in the event of overheating, an air cooling system and grate temperature instrumentation are incorporated.

The spacer flange and shelf assembly (Drawing 0456-037A) is located between the vessel head and cylindrical section and provides a shelf or support for the built-up refractory dome structure. The spacer flange also provides additional distance between the burners and the top of the bed to reduce the effects of direct flame impingement on the bed. The shelf temperature is limited to 1660°F. Provisions for temperature instrumentation for the shelf and the dome refractory were incorporated in the design. The details of the spacer flange and shelf design are shown on Drawing 0456-005B and -604A.

Two viewports of similar design are installed, one on the vessel upper head and one on the cylindrical section of the vessel (at the 8-1/2 foot bed level), to permit optical temperature measurements of the top and side of the bed, respectively. The design details are shown on Drawings 0456-008 and -034R. A cold air purge system is incorporated into both viewports to prevent "fogging" of the glass windows during the heating operation and for thermal protection during heater blowdown. The purge system details are shown on Drawing 0456-402A.

The opening in the upper vessel head, which provides the hot air outlet, must be protected from heating loads during the heater blowdown.
and reheat cycles. The design approach adopted consisted of a water jacket with a ceramic sleeve insert. The ceramic insert is a thin wall (approximately 1/4 inch), yttria-zirconia cylinder and is supported by a ledge incorporated in the water jacket. The primary requirement of this outlet design was that in the event of ceramic sleeve failure, the backup water jacket would provide sufficient protection for the upper vessel head and outlet penetration. The yttria-zirconia sleeve was fabricated, utilizing a castable fabrication technique by Zircoa. Experience in existing storage heaters with similar ceramics fabricated by Zircoa, utilizing this fabrication process, has shown the ceramic to have high thermal stress damage resistance as discussed in Section 3.1. The material does not fail by shattering into small pieces, but rather, it develops interlocking cracks which leave the basic shape structurally intact and continue to provide the necessary thermal protection. This characteristic was again confirmed during the initial operation of the Ames heater. The installation of the water jacket and sleeve is shown on Drawing 0456-037A.

6.2 Reheat System

The reheat system provides a controllable source of thermal energy to the heater matrix by supplying metered fuel and oxidant to the burners, where the oxidant and fuel are mixed and combusted before entering the matrix. The system was designed to have a high flow rate capability to minimize the reheat time required to establish the desired temperature distribution in the bed and a low flow rate capability to prevent thermal shock damage (cracking) of the cored brick during initial heating from a cold bed. The reheat system consists of two burners which mount on the upper vessel head and a control system to regulate the burner reactant flow rates.

Two premix type burners (Drawing 0456-001D) with water-cooled zirconium copper combustion chambers mount to the upper vessel head 120° apart. The burners are designed to remain in place during the high
pressure, high temperature heater blowdown operation. The fuel and oxidant are brought together and mixed within the burner prior to combustion, which results in cleaner combustion. Combustion occurs in the water-cooled zirconium copper burner tube and thus protects the ceramics from unburned gas. To reduce the build-up of condensation on the water-cooled burner tube and combustion chamber surfaces, a thin coating of zirconium oxide was applied. This feature, combined with adjustment of the burner cooling water flow rate, significantly reduces the formation of condensate during the heating operation.

The burner control system is composed of valves, regulators, flow meters, and pressure and temperature instrumentation. Drawing 0456-011F schematically illustrates the control system. The salient feature of the control system is the coupling between the natural gas and oxidant systems, which allows the operator to adjust the oxidant supply with the fuel supply following in a proportion as to hold the fuel-oxidant ratio nearly constant. This feature allows the total mass flow through the burner(s) to be changed without danger of slipping into a reducing mixture, which could damage the ceramics, or exceeding the flashback or blowoff limits of the burner.

The following table illustrates the design maximum and minimum flow rate capability of the burners.

<table>
<thead>
<tr>
<th></th>
<th>BURNER #1</th>
<th></th>
<th>BURNER #2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>max. lbs/hr</td>
<td>min. lbs/hr</td>
<td>max. lbs/hr</td>
</tr>
<tr>
<td>Total burner flow</td>
<td>250</td>
<td>20</td>
<td>250</td>
</tr>
<tr>
<td>Oxygen flow</td>
<td>240</td>
<td>5</td>
<td>240</td>
</tr>
<tr>
<td>Air flow</td>
<td>100</td>
<td>10</td>
<td>100</td>
</tr>
<tr>
<td>Natural gas flow</td>
<td>30</td>
<td>1</td>
<td>30</td>
</tr>
<tr>
<td>Heating rate, BTU/hr</td>
<td>(0.6 \times 10^6)</td>
<td>(0.02 \times 10^6)</td>
<td>(0.6 \times 10^6)</td>
</tr>
</tbody>
</table>

The reheat system used during the heater shakedown runs (Section 7) incorporated a method by which part of the combustion gases could...
flow out of the heater dome region through the nozzle without entering the bed, thereby "bypassing" the bed. This method was used during the initial bed heating from room temperature and during the final heating steps to achieve the bed temperature profiles of 3960°C and above. Initiating combustion in a cold heater is a critical operation from the standpoint of thermal shock to the top of the heater matrix. As originally designed, the minimum combustion product temperature was approximately 2960°C. To reduce the possibility of matrix damage, the top of the bed was initially heated for several hours by passing the combustion gases directly out through the nozzle rather than through the matrix. The second case where bypass was used was during heating conditions above 3960°C. In this case, the combustion gases lose a substantial amount of energy before entering the matrix through radiation to the cooled burners, nozzle, and viewport. In order to compensate for this radiation loss, the burner flow rate was set in excess of the required flow rate through the bed. The excess flow was removed by bypassing it through the nozzle.

After a few hundred hours of burner operation with this burner system, corrosion and pitting of the zirconium-copper burner tube and combustion chamber became apparent. The gold plating and zirconium oxide coating flaked off and exposed the base metal. It was concluded that frequent inspection and repair of the burners would probably be necessary to assure structural integrity. A burner redesign and fabrication, incorporating a stainless steel burner tube and combustion chamber, were undertaken by NASA/Ames to alleviate the pitting and corrosion problem. The new burners have identical performance characteristics as the old and have been successfully operated for 3000 hours without evidence of corrosion. In addition, a "secondary" air feature of the new burners allows temperature dilution of the combustion gas, such that heating the bed using the bypass method is not necessary during initial bed heating. It has also been demonstrated with the new burners that the high temperature ramp profile can be achieved without utilizing bypass. This is accomplished by making frequent changes (approximately every 5 to 30 minutes) in the burner flow rate and fuel oxidant ratio settings.
The burners have two operating characteristics -- flashback and blowoff -- which place limits on the operating envelope. These limits are shown in Figure 9. Flashback describes the phenomenon whereby the flame moves upstream of the combustion chamber into the mixing portion of the burner. This phenomenon limits burner operation in the low mass flow and high fuel/oxidant region. A flashback condition cannot be tolerated in the burner for more than a very short time (on the order of a few seconds). Consequently, a Chromel/Alumel thermocouple has been placed in the mixing chamber to sense a flashback as soon as it occurs.

Blowoff describes the phenomenon whereby the flame leaves its normal position in the combustion chamber and either is extinguished or becomes stabilized at some undesired location downstream of the burner exit. This phenomenon limits burner operation in the low fuel/oxidant region of the operating envelope illustrated in Figure 9 (i.e., it places a limit on how lean the burner can be operated). Blowoff can be sensed only when the heater ceramics are below 1960°R. When blowoff occurs below this temperature, the flame will be extinguished. At temperatures above 1960°R blowoff will just stabilize the flame at some point downstream of the burner, and combustion will continue. There are no viewports on the heater that allow viewing the burner exits; therefore, blowoff at high temperatures cannot be detected. The operation of the burners is, therefore, restricted to areas within the operating envelope.

6.3 Heater Temperature Instrumentation

The temperature instrumentation system for the pilot heater serves two important functions: it provides operational information regarding the matrix temperature profile and heater component temperature information which would indicate impending overheating.

The matrix temperature instrumentation consists of thermocouples in the lower portion of the heater and optical pyrometer equipment to measure
temperatures of the bed top and the edge of the bed at the 8-1/2 foot level. The bed thermocouples are located at approximately 1 foot intervals up to the 5 foot level. At each instrumented bed level, two bare wire thermocouples were installed at different radial locations in the bed. These thermocouples are cemented into the cored brick and cannot be replaced without removal of the heater ceramics. The thermocouple type and location of installation were designed such that the service temperature limit of the thermocouple wire would not be exceeded. The installation details are shown on Drawing 0456-027A.

During the reheat process to establish a specific bed temperature profile, the burner flow rate and/or fuel oxidant ratios are changed in small increments to effect changes in temperature at the top of the bed. These increments are small to avoid thermal shock and avoid developing large temperature gradients which could damage the cored brick. In order to make burner setting changes with confidence and achieve the desired temperature profile in a timely manner, an accurate indication of the bed top temperature is necessary. A characteristic of the temperature measurements made at the top of a cored brick bed is that the web temperature will be somewhat less than the temperature observed in the cored brick hole. This effect is due to heat loss by radiation from the webs to the dome region of the heater. The temperature observed in the holes is a more accurate indication of bed temperature and should be used along with the proper pyrometer corrections.

The side viewport pyrometer temperature measurement is used primarily to determine depth of the "plateau" portion of the bed temperature profile. The location of this temperature measurement is at the approximate location of the maximum "plateau" depth anticipated. This measurement in conjunction with the other bed temperature measurements can be used to determine the final burner setting time and indicates when the desired bed temperature profile is achieved.
The side viewport pyrometer measures the temperature at the edge or side of the matrix. Temperature readings of this nature tend to be low because of losses in the long viewing hole through the insulation and because the pyrometer is sensing the temperature of the bed edge which is in contact with the insulation, which has a cooler mean temperature. Absolute accuracy of the side viewport temperature reading is not necessary to determine the depth of penetration of the temperature plateau. Experience during heater operation has shown that the side viewport temperature readings rise rapidly with time after initiation of the burner setting to establish the temperature profile plateau. Within a short period of time, the rate of temperature rise drops off sharply, indicating that the temperature plateau formation is complete down to the side viewport bed level. It is necessary that a time-temperature record be kept during the temperature plateau formation to fully utilize the side viewport temperature measurement.

Thermocouple instrumentation was provided for the temperature measurement of critical metal structures, such as the grate, shelf, and vessel walls. The location of these thermocouples is shown on Drawing 0455-027A.

Vessel insulation thermocouples are located at approximately the 7 and 9 foot levels above the grate and in the dome region. Thermocouples were not installed in the insulation in the lower temperature portions of the heater because experience has shown that they are not essential if adequate vessel instrumentation is provided.

6.4 Installation of Heater Internals

The heater system installation began with the insertion of the lower end pipe liners and welding the grate support ring to the bottom of the vessel. Proper alignment with vessel centerline and rotation for grate cooling and thermocouple penetrations were the primary concerns. After checking the grate cooling system for leaks, verifying the flow distribution, and checking
the thermocouple instrumentation, the castable alumina was poured around the grate to form a flat structural support for the ceramic insulating materials. Figure 10 illustrates the grate system installation prior to pouring the castable alumina.

The ceramic installation began with the installation and orientation of the perforated grate plate. The orientation of grate plate sets the orientation of the matrix with respect to the vessel. The orientation of the vessel side wall thermocouple penetrations with respect to the matrix was specified for ease of installation and thermocouple maximum life.

The installation of the ceramics begins with the installation of the first cored brick course which is keyed to the grate plate. The vessel insulation is then installed around the bed out to within 1/2 inch of the vessel wall. The remaining 1/2 inch was filled with fiberfrax installation. The thermal expansion requirements were maintained by installing low silica content cardboard spacers. The thermal expansion requirements are shown in Figure 11. A photograph of the cardboard spacer installation between the bed liner brick and arch brick is shown in Figure 12.

The thermocouple instrumentation was installed as the bed, and insulation build-up progressed. The heater bed and side wall insulation thermocouples were bare wire, chromel/alumel or platinum (6-30 Rh) type. The bare wires were protected with double bore, ceramic tubing between the vessel penetration and the thermocouple junction. The bed thermocouple wires were cemented in grooves cut into the cored brick side wall. The shelf and dome insulation thermocouples were the sheathed, insulated type and were used in these areas for ease of installation. The details of the instrumentation installation are illustrated on Drawing 0456-027A and a typical installation shown in Figure 13.

The spacer flange containing the refractory dome support shelf was installed on the vessel and the inner surfaces thermally protected with two
1/8-inch thick layers of fiberfrax paper. The refractory dome was then installed on the shelf as shown in Figure 14. The thermocouple assemblies on the dome backside were also installed and are shown on the above figure. At this point, a jig was utilized to complete the insulation installation. The jig assembly duplicated the internal surface of the upper head, and a large dowel system was utilized to provide exact location and angles for the two burner and viewport penetrations. Figures 15 and 16 illustrate the jig system and the completed structural build-up, respectively. The inner surface of the upper portion of the vessel head, in the outlet region, was protected with a 1/2-inch layer of fiberfrax castable.
7.0 PILOT HEATER OPERATION

Heater operation involves two sets of procedures: the reheat procedures used to establish the desired bed temperature profile, and the air blowdown procedures. The relationship between bed temperature profile and air flow rate, as limited by thermal stresses, was discussed in Section 5.2.2. The corresponding air blowdown procedures were described in Section 5.2.3. Reheat procedures were also developed and were presented in detail in the Operations Manual. A brief review of these procedures is presented in Section 7.1.

A run program consisting of 20 blowdown runs and 450 hours of heater operation was carried out. This program tested the ceramic materials and the overall system. The program and its results are discussed in Sections 7.2 and 7.3.

7.1 Reheat System Procedures

Operation of the reheat system in three modes is required. These are heating from a cold bed, heating between runs and standby operations. A series of burner heating tables were prepared for a range of bed temperature conditions. These tables specify in step by step form the desired burner reactant flow rates, fuel-oxidant ratio and approximate time required to achieve the heater condition for each step. The step changes in the burner setting were designed to produce slow increases in bed temperature and thus minimize thermal stresses. A representative heating table is shown in Figure 17, and a series of ideal temperature profiles generated by the use of these tables is shown in Figure 18.

One of the most critical periods is the interval following burner ignition when the bed is cold. The bricks in the upper part of the bed can be heated too rapidly, causing large thermal stresses. During the run program described in Section 7.3, the combustion gases were passed out the nozzle...
rather than through the bed, when heating from room temperature. After this run program was completed, the burners were modified to provide secondary air flow, and consequently lower reheat gas temperatures. This permitted elimination of the bypass method (see Section 6.2).

After the initial heating period, burner flow conditions are changed in small steps, and in a particular order, because of the following limitations: (1) burner blowoff, (2) burner flashback, (3) refractory damage due to thermal stress, (4) production of fuel-rich products. As shown in Figure 17, the burner flow rate adjustments involve a change in total mass flow (requiring a proportional change in all of the reactant flow rates) and/or a change in the fuel/oxidant ratio (ratio of fuel to air plus oxygen), which changes the temperature. The order for the adjustment between steps is indicated by brackets placed around the specific conditions to be changed in each substep.

The next to the last step in each burner table is used to form a nearly linear temperature distribution from the top to the bottom of the bed. The slope is equal to the slope of the temperature ramp. Formation of this "linear" distribution requires a longer time interval than the earlier steps, and steady state conditions are approached (thereby useful for standby conditions, as noted below).

The last step in the tables is used to form the temperature "plateau" in the upper portion of the matrix. Both burners are used to provide the high flow rates needed. The time interval for maintaining these flow conditions will be determined by the depth of plateau desired. During this period, the "linear" distribution referred to above is moved down in the bed to form the ramp. The bed temperature instrumentation and the side viewport pyrometer are used to determine when the desired conditions are reached. Although the side viewport pyrometer views the edge of the bed, where the influence of the insulation on the matrix temperature is quite high and large radiation losses occur, an indication of the temperature plateau depth is available. Figure 13 presents side viewport temperature data taken during the heater run program.
As indicated in the figure, the temperature first increases rapidly, then approaches a steady state condition. Forming the plateau requires approximately 20 to 30 minutes at the final heating step.

There are limitations to the depth of plateau that can be safely established. The zirconia-alumina interface should not be allowed to exceed 3260°R (see Section 4.2.2) and the grate should not exceed 1460°R. If the grate should approach the allowable maximum temperature, with the interface temperature at a safe level, the grate cooling system can be used. However, it is noted that no difficulties with grate overheating were encountered during the run program, in fact, the lower part of the bed was cooler than expected.

The same burner tables are used for heating from room temperature and for heating between runs. For the latter case, the tables are entered at a steady state bed temperature step approximately 200°R above the bed top temperature after the run. The burner flow rates are preset and combustion initiated at this step.

A storage heater, being support equipment to a test facility, often must be held in a state of readiness awaiting delays on other portions of the facility and during nonoperating work shifts. Such standby periods will vary from a few hours to several days. The temperature level at which the ceramics are held depends on how long the heater will be maintained in a standby condition.

Standby operation can be divided into two general categories; short and long term. For short term standby, such as delays between runs, the heater should be maintained at the temperature condition given by the next to the last step in the burner tables, i.e., the step that produces the linear steady state temperature distribution, as described above. This heating step requires only one burner.

If the standby period will be many hours, such as overnight and weekend operation, heating at the next to the last step of the 3260°R table is
recommended. This condition requires only one burner, no oxygen, and will produce a nearly linear distribution from 3260°R at the top of the bed to about 800°R at the grate. The alumina-zirconia interface temperature will be about 1700°R, which will place all of the zirconia in the cubic phase portion of the phase diagram (based upon 10.4 w/o yttria-rare earth stabilizer and unpublished phase diagram work by Dr. Robert Ruh). This should extend the life of the zirconia by avoiding temperature conditions that may cause phase changes.

7.2 Preliminary High Temperature Operation

The first operation of the heater consisted of heating to a temperature of 4460°R at the top of the bed, and holding this condition for 45 hours. During this time, no air was blown through the heater. The purpose was to extend the firing that the bricks had been given by the manufacturer. The manufacturer had sintered the bricks by firing them at 3630°R. This is significantly below the intended maximum service temperature.

Further, bricks that were similar, except for stabilizer content, had destabilized with severe loss of strength in the Air Force pilot heater (Reference 4). The Air Force bricks contained 9-1/4 w/o YRE stabilizer and the monoclinic content ranged from nondetectable (less than 1%) to 5%. These bricks were successfully "refired" in the heater, resulting in recovery of strength and reduction in monoclinic content to less than 1%, and allowing their continued use.

The Ames bricks contained 10.4 w/o YRE and had less than 1% monoclinic phase "as manufactured." Therefore, the probability of a similar destabilization was very low. But, additional firing, especially at a temperature above the original sintering temperature, could only improve the bricks, giving a more uniform structure and reducing the already low monoclinic phase content.

Measurements made by Coors Porcelain Company on selected bricks (of the 9-1/4 w/o YRE composition) demonstrated that heating at 3560°R for
a period of 75 hours would reduce the monoclinic content. Progressively shorter time periods were needed at higher temperatures.

The bed was heated to a steady state temperature distribution in 45 hours. The top was at 4460°R and the 3460°R level was 4 feet below the surface. Thus, 2-1/2 feet of zirconia were below 3460°R. The intent was to hold for 75 hours, but after 45 hours, an accumulation of water in the air outlet piping was detected and the system was shutdown. This proved to be condensate that collected because of a plugged drain. The condition was corrected and did not recur.

This operation provided a thorough shakedown of the burner system. No problems of significance were encountered and all equipment functioned satisfactorily. An inspection was made by removing the burners and the air outlet nozzle. Corrosion of the burner barrels was found, but it was not serious enough to prevent their use for the run program.

No bricks were removed, but they were examined visually by viewing through the burner and nozzle ports. The cored bricks and the insulation bricks appeared to be in excellent condition, with no visible cracks. It was decided to proceed with the run program.

7.3 Heater Run Program

The purpose of the run program was to evaluate the performance of various components, the performance of the entire heater system, and the operating procedures. The heater system is a high pressure, high temperature system utilizing critical water cooled components and brittle ceramic materials. The consequences of errors in operation could, therefore, be quite serious. This being the case, the shakedown program and procedures were formulated to develop information in a progressive fashion, while avoiding hazardous operation. A flexible heater run program was established to determine:
1. The behavior of the reheat system.
2. The behavior of the blowdown system.
3. The behavior of the ceramic shapes within the heater.
4. The behavior of the ceramic materials within the heater.

The program consisted of 20 runs at temperatures between 2960°R and 4660°R. The stagnation pressure range was 50 to 500 psi. The run schedule and flow conditions are shown in Figure 20. The program was completed in 10 calendar days, including a cool down period and component inspection after Run 12. Every phase of the operation ran smoothly and no major malfunctions or component failures occurred.

7.3.1 Temperature Data

The initial bed heating from room temperature is shown in Figure 21. Representative heater temperature profiles are shown in Figures 22 through 26. As illustrated in these figures, the lower two feet of the bed remained cooler than desired throughout the test program. The heat loss through the alumina insulation in this region is too large. This characteristic tends to increase the slope of the axial temperature gradient in the alumina which increases thermal stress levels. To eliminate this problem, it was recommended that the lower portion of the vessel be wrapped with insulation.

The top of the bed temperature was measured through the dome viewport, utilizing an optical pyrometer. These temperatures were very close to those predicted as being steady state bed temperatures during burner operation. During the period of the last six runs of the program, the viewport glass became "clouded" with a white haze, which caused low pyrometer readings. A redesign of the purge air system has improved this condition.

This pyrometer was also used to measure the temperature at the top of the bed after each blowdown run was completed. This measurement was made several minutes after the run (but on no definite schedule) in preparation
for initiating the burner operation. Without the burner operating the hot surfaces in the dome region (including the top of the bed) lose heat to the cooled burners and nozzle. This causes a temperature depression, but the depression is shallow because of the low thermal conductivity of zirconia. Hence, these particular measurements were not useful in establishing the post run temperature profile.

The temperature at the edge of the bed at the 8-1/2 foot level was monitored utilizing the side viewport and an optical pyrometer. The temperature readings (raw data) at this location are below the actual temperatures because of radiation losses from the viewport opening. Nevertheless, the temperature readings are useful in indicating the depth of penetration of the temperature plateau as previously discussed in Section 7.1.

The shelf and spacer flange temperatures were closely monitored during the high temperature heating periods. The maximum temperature attained on the shelf was approximately 1360°F and was controlled by blowing air directly on the outside surface of the flange. The shelf maximum design temperature is 1660°F.

During the 20 heating and cooling cycles of the run program, the bed and insulation thermocouples remained operative with the exception of several insulation thermocouples which became erratic, probably because of thermal expansion movement of the insulation.

7.3.2 Pressure Data

The time to pressurize, as shown on Figure 20, generally decreased throughout the program as operational experience was gained. Rapid pressurization minimizes the bed heat losses and maximizes the steady state run time at near constant outlet temperature.
Measurements of pressure drop across the bed are compared to calculated values in Figure 27. The agreement is good, except for the runs made at pressures above 400 psi. For these runs (Runs 7, 8, 11 and 12) the measured values are considerably less than the calculated values. At this point, with the limited run data available, the reason for this difference is not known.

7.3.3 Post Run Inspection

After completion of the run program, inspections were performed to establish the condition of the equipment and ceramics.

The burner barrels were examined for possible damage; it was found that both barrels were pitted and corroded. The barrels were copper, covered with gold plating and Rokide Z. Corrosion was eliminated by changing the barrel material to stainless steel. Redesign and fabrication was done by Ames, and subsequent operation for a duration of 3000 hours has not caused any corrosion.

The backside, water cooled choke nozzle was removed and inspection made of the hot flow surface. This surface has a thin alumina coating. The nozzle was in excellent condition with only slight flaking of a portion of the alumina coating in the inlet and throat areas. The ceramic sleeve upstream of the nozzle inlet was cracked in several places, but no pieces had fallen out. The condition was satisfactory, but a need for frequent inspection was indicated.

The upper vessel head and spacer flange were removed to allow direct access to the bed and insulation ceramics. Approximately 30-inches of cored brick and bed liner insulation were removed from the heater for inspection. The dome insulation hot face was inspected in place without disassembly. Photographs of the heater during the inspection are shown in Figures 28 through 31.
Specific inspection information was sought in regards to the following:

a. Bed thermal expansion allowances.
b. Cored brick hole plugging.
c. Thermal shock cracking in the upper portion of the matrix.
d. Ceramic material destabilization.
e. Web thermal stress cracking.
f. Body thermal stress cracking.
g. Insulation destabilization and cracking.

Prior to the removal of any cored brick, the general appearance of the top of the bed was checked to determine if the allowances for thermal expansion, provided during the initial installation, were adequate. Experience has shown that inadequate thermal expansion allowances cause the cored brick hexagon shape and holes to become distorted. In addition, the bed liner insulation brick would contain deep scratches and/or cracks caused when the bed expands against the bed liner. Inspection showed that sufficient allowances were made. No deformation of the bed liner or cored bricks was found.

An inspection for the possibility of hole plugging, from dust generated from sidewall or dome insulation, was also made. All cored brick holes were found open. Close inspection of the bed top for contamination due to dome dust, chips from the burner coating, or water was made and little or no contamination was found.

The 1-inch buffer layer bricks were intact with the exception of two which were fractured in half. Approximately 10% of these bricks contained cracks which penetrated less than half way across the brick. Most of these bricks had a highly glazed surface, and all were reinstalled.

The 12 to 15-inches of cored bricks just below the buffer layer, 3 to 6-inch lengths, were also found in good condition. Most of these bricks contained tight, hairline cracks, both axial and transverse in direction. None of these bricks were fractured and all were reinstalled.
The cored bricks 15 to 30-inches from the top exhibited more cracks and many were open, as compared to the tight, hairline type. These bricks were 10 to 12-inches in length, and although cracked, could be handled and were reinstalled, as none were actually fractured.

An attempt was made to remove one column down to the alumina-zirconia interface. This attempt was terminated approximately 60-inches from the bed top, or about 1-foot above the interface, because the brick condition became progressively worse the deeper the penetration. The last 15-inches of bricks were each fractured into 3 or 4 lengths. These smaller lengths were often again fractured into pieces.

With approximately 30-inches of cored brick removed from the heater, a close inspection was performed to establish the type of cracking and to investigate the possibility of zirconia destabilization. The characteristics of a destabilized brick are blotchy color changes and a chalky material texture. None of the bricks removed had these characteristics.

Several cored bricks from each level and from different radial location were dye checked to inspect for web cracking. Blowdown cycle thermal stress web cracking is characterized by cracks radiating into the webs from the holes. This type of cracking was not found in any of the inspected bricks.

Almost all of the cracks tended to extend across the bricks, entering the surface at about 90°, with cracks transverse and parallel to the brick axis. It is most probable that these cracks were caused by "body stresses" as discussed in Section 3.3.

The transverse cracks tended to break the bricks into shorter lengths. A large majority of the cored bricks contained transverse cracks at 2 to 3-inch intervals. These cracks were generally not open and not completely through the brick. Since this characteristic was noted on so
many bricks, it is apparent that future bricks for this heater, in the dense material, should be of shorter length; 2 to 3-inches is indicated.

Axial cracks along the brick flats and corners were found at all bed levels below the buffer layer and within the 30-inches removed. In the upper 12 to 15-inches, the axial cracks were tight and of the hairline type. Below the 15-inch bed level, some bricks contained open cracks which may, in time, cause the bricks to fracture. Several bricks had axial cracks that would tend to fracture off a corner along the complete length of the brick. Such pieces could become wedged between brick columns and cause further damage.

In general, the use of shorter bricks should reduce both axial and transverse cracking. To permit further investigation, bricks of 1 to 3-inch lengths (total length 3 feet) were used to replace the long bricks from the column which was removed to approximately the 60-inch depth. All other bricks were reinstalled.

The bed liner insulation was inspected to a depth of approximately 30-inches and found to be in excellent condition. Several bricks had open cracks on the hot face. Most of these bricks were located in the upper 10-inches and generally opposite from the two burners. The reason for cracking is, therefore, suspected as being either from thermal shock or inadequate allowance for hot face expansion in the brick design.

The dome hot face bricks were inspected in place, without dismantling the structure. The bricks were in excellent condition, with only slight rounding of several corners being apparent. The alignment of all penetrations was good. All of the insulation was suitable for continued service.
8.0 CONCLUSIONS

The following are the important conclusions that can be made from these storage heater tests.

1. Inspection of the cored brick elements and insulation bricks after 450 hours of testing revealed no zirconia destabilization in any sections of the heater.

2. Yttria-stabilized zirconia fabricated to have less than 1% monoclinic content is a suitable heat storage and insulation material for temperatures to at least 4660°F.

3. Although many cracks in the cored bricks were found during the inspection, there was no catastrophic brick failures, and the bed condition was suitable for continued operation. The insulation condition was excellent.

4. Thermal stress cracks of the cored brick are believed caused primarily by the large size of the bricks, especially relative to the bed size. The crack patterns indicate that the brick length should not have exceeded three inches. Bricks with overall dimensions of two to three inches would be suitable and should eliminate most of the cracks and fractures.

5. The thermal stress limits for a bed of low porosity (dense) ceramic impose restrictions on the air flow rate and on the vertical temperature gradient in the bed. The bed length of 10 feet requires large vertical temperature gradients, which can readily lead to excessive thermal stresses. Therefore, 10 feet is too short for a bed of this type.

6. Operation of the reheat and blowdown systems was satisfactory, and no mechanical or system failures occurred.
REFERENCES


FIGURES
FIGURE 1. BASIC HEATER CONFIGURATION
FIGURE 2. CERAMIC MATERIALS TEST APPARATUS
MAGNESIA CONDENSATION IN CORED BRICK LOCATED DOWNSTREAM OF TEST CONFIGURATION

MAGNESIA CONDENSATION IN PRESSURE DROP ELEMENT LOCATED BETWEEN TEST ELEMENTS AND CORED BRICK ABOVE

FIGURE 4. MAGNESIA TEST SAMPLE CONDENSATION
Weight Loss Characteristics in Vacuum of 10^-4 mm Hg

Weight Loss in Helium at 0.2 Atm.

FIGURE 5. MAGNESIA WEIGHT LOSS DATA
FIGURE 6. STEADY STATE MASS FLOW LIMITED BY THERMAL STRESS DURING PRESSURIZATION
\[ m = \frac{C_p A^*}{\sqrt{T_o}} \]

**FIGURE 7. STAGNATION PRESSURE VERSUS MASS FLOW**
FIGURE 8. PRESSURE DROP ACROSS BED DURING STEADY FLOW

\[ \Delta P = 0.00198 P_o A^2 \frac{T}{T_o} \]

- \( \overline{T}/T_o = 0.7 \)
- \( \overline{T}/T_o = 0.6 \)
FIGURE 9. PREDICTED BURNER PERFORMANCE (1 BURNER)
FIGURE 10. GRATE SYSTEM INSTALLATION
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<th>Axial Location (ft from grate)</th>
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<th>Gap #3 (inches)</th>
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*Spacers should be installed between every 4th or 5th brick in each course, with an accumulated spacing equal to Gap #3.

FIGURE 11. CERAMIC INSTALLATION THERMAL EXPANSION ALLOWANCES
FIGURE 12. CERAMIC INSTALLATION WITH BURNOUT THERMAL EXPANSION CARDBOARD
FIGURE 13. TYPICAL BED THERMOCOUPLE INSTALLATION
FIGURE 14. DOME HOTFACE INSTALLATION
FIGURE 16. COMPLETE REFRACTORY DOME INSTALLATION
<table>
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<th>No. of Burners</th>
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<th>( \dot{m}_{\text{fuel}} ) (lb/hr)</th>
<th>( \dot{m}_{O_2} ) (lb/hr)</th>
<th>( \dot{m}_{\text{total}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{bed}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{bypass}} ) (lb/hr)</th>
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**FIGURE 17. BURNER HEATING TABLE, 3960° R BED TEMPERATURE**

(Sheet 1 of 3)
<table>
<thead>
<tr>
<th>Step No.</th>
<th>No. of Burners</th>
<th>Time at Setting</th>
<th>$m_{air}$ (lb/hr)</th>
<th>$m_{fuel}$ (lb/hr)</th>
<th>$m_{O_2}$ (lb/hr)</th>
<th>$m_{total}$ (lb/hr)</th>
<th>$m_{bed}$ (lb/hr)</th>
<th>$m_{bypass}$ (lb/hr)</th>
<th>f/ox</th>
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**FIGURE 17. BURNER HEATING TABLE, 3960°F BED TEMPERATURE**

(Sheet 2 of 3)
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<tr>
<th>Step No.</th>
<th>No. of Burners</th>
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<th>( \dot{m}_{\text{air}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{fuel}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{O}_2} ) (lb/hr)</th>
<th>( \dot{m}_{\text{total}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{bed}} ) (lb/hr)</th>
<th>( \dot{m}_{\text{bypass}} ) (lb/hr)</th>
<th>( f/\text{ox} )</th>
<th>Steady State Bed Temp. (°F)</th>
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**FIGURE 17. BURNER HEATING TABLE, 3960° R BED TEMPERATURE**

(Sheet 3 of 3)
FIGURE 18. IDEAL TEMPERATURE DISTRIBUTIONS PRODUCED BY BURNER REHEAT TABLES
FIGURE 19. SIDE VIEWPORT PYROMETER CHARACTERISTICS
<table>
<thead>
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<th>Run No.</th>
<th>Run Date</th>
<th>$T_0$ °R</th>
<th>$P_0$ psia</th>
<th>m lbs/sec</th>
<th>$dT/dz$ °R/Ft</th>
<th>Time - Seconds Press.</th>
<th>Run</th>
<th>Depress.</th>
<th>$\Delta P$ run psi</th>
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<td>26</td>
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<td>290</td>
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<td>360</td>
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<td>26</td>
<td>.75-.62</td>
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</tr>
</tbody>
</table>

(1.25-Inch Diameter Heater Choke Nozzle)

* ΔP line valve to bypass U-tube left open, ΔP reading void
** ΔP recorder instrument interference - erratic readings - fixed after this run

FIGURE 20. SHAKEDOWN RUN SCHEDULE AND TEST CONDITION SUMMARY
FIGURE 21. VARIATION OF BED TEMPERATURE PROFILE WITH TIME STARTING WITH COLD BED
FIGURE 22. SHAKEDOWN RUN TEMPERATURE PROFILE, RUN 3
FIGURE 23. SHAKEDOWN RUN TEMPERATURE PROFILE, RUN 7
FIGURE 24. SHAKE-DOWN RUN TEMPERATURE PROFILE, RUN 10
FIGURE 25. SHAKE-DOWN RUN TEMPERATURE PROFILE, RUN 16
FIGURE 26. SHAKEDOWN RUN TEMPERATURE PROFILE, RUN 19
FIGURE 27. CALCULATED VERSUS MEASURED BED ΔP COMPARISON
Figure 28. Zirconia bed top after 20 run shakedown program
FIGURE 29. DOME INTERIOR SURFACE AFTER 20 RUN SHAKEDOWN PROGRAM
FIGURE 30. ZIRCONIA CORED BRICK BED REMOVAL FOR INSPECTION, AFTER 20 RUN SHAKEDOWN PROGRAM
FIGURE 31. TYPICAL CORED BRICK REMOVED FROM PILOT HEATER FOR INSPECTION
YTTRIA-RARE EARTH STABILIZED ZIRCONIA

1. Stabilizer to be 10.4 weight percent of yttria-rare earth oxides mixture with total of lanthanum, cerium, praeodymium, neodymium, promethium, samarium, and europium oxides not to exceed one weight percent. Minimum concentration of yttria to be 88 weight percent. Minimum concentration of yttria plus rare earth oxides to be 99.5 weight percent.

2. Maximum individual impurities (weight percent):
   - Titania 0.2
   - Ferric Oxide 0.2
   - Silica 0.2
   - Alumina 0.2
   - Calcia 0.4
   - Magnesia 0.4

3. Maximum total impurities not to exceed 1.2 weight percent (excluding hafnia).

4. Minimum bulk density of fired shapes to be 5.40 grams per cubic centimeter.

5. Shapes to be fired at Cone 33 (3170°F) or higher.

6. Monoclinic phase of fired shapes not to exceed 1.0 percent.
YTTRIUM–RARE EARTH OXIDES MIXTURE

1. The minimum $Y_2O_3$ concentration shall be 88 percent by weight.

2. The total percentage of lanthanum, cerium, praseodymium, neodymium, promethium, samarium, and europium oxides shall not exceed 1 percent by weight.

3. The minimum concentration of yttria and rare earth oxides shall be 99.5 percent by weight.

4. At least 90 percent of the oxide powder shall be composed of particles with average diameter less than 4 microns (Fisher sub-sieve sizer).

5. Weight loss of oxide powder shall not exceed 2 percent in air at 1400°F.
Yttria Stabilized Zirconia

1. Stabilizer to be 9.0 to 9.5 weight percent of yttria or yttria-rare earth oxides mixture with total of lanthanum, cerium, praseodymium, neodymium, promethium, samarium, and europium oxides not to exceed one weight percent. Minimum concentration of yttria plus rare earth oxides to be 99.5 weight percent.

2. Maximum individual impurities (weight percent):
   - Titania 0.2
   - Ferric Oxide 0.2
   - Silica 0.3
   - Alumina 0.4
   - Calcia 0.4
   - Magnesia 0.4

3. Maximum total impurities not to exceed 2.0 weight percent (excluding hafnia).

4. Bulk density of fired shapes to be in range 250 to 280 pounds per cubic foot.

5. Shapes to be fired at cone 35 (3250°F) or higher.

6. Fired shapes to be essentially free of monoclinic phase.
(1) Data forwarded to Fluidyne in "as recorded" form.
(2) Sample retained at Zircos.
(3) Data retained at Zircos.
(4) Sample and ordered shapes will be identified with:
   a. Drawing number (ordered shapes only)
   b. Composition number or type of material
   c. Press or cast batch number
   d. X-ray lead.
(5) Small, 1-inch cylinders will be fired with each kiln load. Two of these samples from each kiln load will be forwarded to Fluidyne and the remainder retained at Zircos for analysis.
(6) Firing records retained at Zircos.
(7) Tests performed at Fluidyne.
(8) Data reported to Fluidyne.
(9) Cored brick dimensions will be checked on every tenth block. Dimensions will be recorded.
(10) Insulation bricks will have random dimensions checked on every fifth block. Dimensions will be recorded.
(11) Cored brick density measurements will be performed on one full-size brick randomly selected from every fourth kiln load. Data will be recorded.
(12) Insulation brick density measurements will be performed on one full-size brick randomly selected from every fourth kiln load. Data will be recorded.
(13) Visual inspection criteria will be established by mutual agreement (Fluidyne and Zircos) within specifications, after initial blocks are fired.
(14) Certificate of Conformance, samples and all pertinent test data must be approved by Fluidyne before refractories are shipped.
(15) Tests will be performed on one sample fabricated from each batch of material.
(1) Coors Proprietary Data.

(2) Sample and ordered shapes will be identified with:
a. Drawing number (ordered shapes only)
b. Material
c. Grain batch
d. Kiln load.

(3) Small, 1-inch cylinders (or some convenient shape) will be fired with each kiln load. Two of these samples, from each kiln load, will be forwarded to Fluidyne and remainder retained at Coors for analysis.

(4) Firing records retained at Coors.

(5) Cored brick dimensions will be checked on every tenth block. Data will be recorded.

(6) Data forwarded to Fluidyne in "as recorded form."

(7) Cored brick density measurements will be performed on one brick randomly selected from each kiln load. Data will be recorded.

(8) Visual inspection criteria will be established by mutual agreement (Fluidyne and Coors) within specifications, after initial blocks are fired.

(9) Certificate of Conformance. Samples and all pertinent test data must be approved by Fluidyne before refractories are shipped.

(10) Tests will be performed on one sample fabricated from each batch of material.