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HIGH TEMPERATURE RANGE RECUPERATOR

Phase I, Materials Selection, Design Optimization, Evaluation and Thermal Testing

Final Report for the Period April 1977—May 1978

By
Dean V. Power

June 1978
Date Published

Work Performed Under Contract No. EY-77-C-07-1660

Terra Tek, Inc.
Salt Lake City, Utah

U. S. DEPARTMENT OF ENERGY

Division of Industrial Energy Conservation

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HIGH TEMPERATURE RANGE RECUPERATOR

PHASE I

MATERIALS SELECTION, DESIGN OPTIMIZATION, EVALUATION AND THERMAL TESTING

FINAL REPORT FOR THE
PERIOD APRIL 1977 - MAY 1978

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Date Published - June 1978

Prepared for the
Department of Energy
Division of Industrial Energy Conservation

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ABSTRACT

Initial efforts to develop, test, and evaluate counterflow recuperator designs are reported for the "High Temperature Range Recuperator" project conducted by Terra Tek, Inc., and Millcreek Glass Corporation and sponsored by the Department of Energy, Division of Industrial Energy Conservation, through the Program Opportunity Notice PON #BI-B6-1000 under contract No. EC-77-C-07-1660.

Potential materials to withstand glass furnace exhaust environments at temperatures up to 2800°F were evaluated on the bases of material properties, fabrication capability, and relative performance in the flue environment of a day tank glass furnace. Polycrystalline alumina (Vistal), reaction sintered silicon carbide (KT and NC 430) chemically vapor deposited silicon carbide (CVD) and sintered alpha silicon carbide proved most satisfactory in the material temperature range of 2300°F to 2800°F. Relatively pure alumina (AD 998 and AD 94), mullite and cordierite were most satisfactory in the material temperature range of 1700°F to 2300°F.

Recuperator designs were evaluated on the bases of cold air flow tests on laboratory models, fabricability, and calculated thermomechanical stress under expected operating conditons. Material strengths are shown to be greater than expected stresses by factors ranging from 2.6 for KT silicon carbide to 16 for cordierite. Recuperator test sections were fabricated from KT silicon carbide and subjected to thermal stress conditions in excess of twice the expected operating conditions with no deterioration or failure evident. A test section was subjected to the thermal shock of instant transfer between room temperature and a 2000°F furnace without damage.

Economic analysis based on calculated heat transfer indicates a recuperator system of this design and using currently available materials would have a payback period of 2.3 years.

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EXECUTIVE SUMMARY

This report presents the results of initial efforts of the "High Temperature Range Recuperator" project. Terra Tek, Inc., and its subcontractor Millcreek Glass Corporation, performed this work under the sponsorship of the Department of Energy, Division of Industrial Energy Conservation, in response to the Program Opportunity Notice PON #BI-B6-1000 under Contract No. EC-77-C-07-1660.

The overall purpose of this project is to develop, test, and evaluate designs for an industrial recuperator capable of a 70 percent heat recovery in the recuperator material temperature range of 1700°F to 2800°F for a glass furnace application as well as other industrial applications such as steel, aluminum, and cement. Maximum air recovery temperature is to be 2100°F. A payback period of 3 years is desirable.

Specific research objectives for Phase I were:

1) Design optimization of the continuous counterflow high temperature range recuperator.
2) Selection of the best recuperator materials and fabrication techniques through evaluation of material properties and performance in an expected environment
3) Construction and short-term thermal testing of laboratory test modules
4) Development of design criteria and a test plan for extended testing of prototype modules.

Potential material candidates were originally screened on the basis of meeting the minimum requirements of:

1) A compressive strength in excess of 2000 psi at temperatures above 1800°F
2) Thermal conductivity in excess of 0.5 BTU/hr-ft-°F
3) Potential product cost less than $50 per pound
4) Previous usage in high temperature applications
5) Potential tolerance for glass furnace atmospheres
6) Potential fabricability of the specific design in dimensions of one foot or greater.

On this basis 31 ceramic materials for modules were identified and designated for further evaluations and tests. Four joint seal materials were identified for testing on the bases of material flexibility and previous high temperature usage.

Material properties evaluations utilized available literature data. Properties considered were physical (grain size, density, porosity, gas permeability), mechanical (elasticity, strengths, fracture thoughness), and thermal (conductivity, expansion, cycling resistance, shock resistance, creep). Fabricability, chemical stability, and cost were also considered. Fracture toughness values were not generally available and these data were measured on prime module material candidates.

Material performance in furnace exhaust atmospheres was determined by inserting materials into the exhaust streams of operating day tank furnaces. Two exhaust atmospheres were tested: a soda-lime glass furnace exhaust and combustion product exhaust. Both exhausts resulted from natural gas and air combustion inside refractory chambers. Performance tests included both continuous furnace operation and cycling operation.

The evaluations identified several module and joint seal material candidates for future prototype tests. Module material candidates selected include four silicon carbides (trade name Sintered Alpha, KT, NC 430, and CVD) and two purities of alumina (Vistal and AD 998) for the highest range of 2300°F to 2800°F. For the temperature range of 1700°F to 2300°F, the candidates are cordierites, mullites, and lower purity (94 to 998 percent) aluminas. Joint seal candidates are 3M Ceramic Fiber and Saffile above 2300°F and Fiberfrax and Kaowool between 1700°F and 2300°F.

Physical design optimization was based on heat transfer requirements, fabricability, fouling considerations, thermal stress calculations and cold air flow tests. Depending upon the material, module strength capability exceeds thermal stress by factors ranging from 2.6 to 16.0.

Fabrication methods are unique with each candidate material. Present potential module manufacturer kiln limits vary between 0.5 feet and 3.0 feet for all primary candidates. Considerable investment may be required to achieve 3-foot diameters in Vistal. Anticipated industrial module size is 3 feet or less.

Six 8-inch test modules were fabricated from a fine-grained silicon carbide (KT) and tested under thermal operating conditions. Hot and cold gas streams were directed simultaneously through all flow channels. A temperature difference (ΔT) between the hot and cold gases of 1200°F (2.4 times expected operating ΔT) was achieved without causing any failure or visible deleterious effects on the test sections. Joint seals of Kaowool, Fiberfrax, and 3M Ceramic performed acceptably during these short-term tests.

One test module was subjected to the thermal shock of instant transfer between room temperature and a 2000°F furnace without sustaining damage.

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INTRODUCTION AND OBJECTIVES

Background of Recuperator Program

The Department of Energy, Division of Industrial Energy Conservation, sponsored this project under Contract No. EC-77-C-07-1660 in response to the Program Opportunity Notice PON #BI-B6-1000.[1] The purpose of this program is to conserve primary fuels by developing high temperature recuperators for industrial furnaces to recover and utilize waste energy. Terra Tek and Millcreek Glass Corporation performed the work in their Salt Lake City, Utah facilities.

While all thermal processes discharge heat to the environment in one form or another, there is great variation in the amount of the recoverable discharged energy. In general, direct heating furnaces, in which there is radiant and convective heat transfer from the combustion materials directly to the material being heated in an enclosed space, are obvious choices for thermal recovery because they are very inefficient. Efficiencies range from 5 percent for furnaces in forge operations to 25 to 35 percent for furnaces where some recuperation is practiced. Exhaust temperatures range from 1200°F for annealing furnaces to 2300°F to 2700°F for steel reheating and glass furnaces.[2] Direct heating processes using gas and oil in the United States industry consume on the order of $6x10^{15}$ BTU/year at an estimated annual cost of twelve billion dollars.[3] At least 75 percent of this energy is discharged to the atmosphere without direct utilization, resulting in an annual waste of nine billion dollars. The losses in coal-fired direct heating furnaces make the total waste figure even greater.

In a direct fired unrecuperated glass furnace, the energy loss is about 80 percent of the combustion energy.[4] The glass industry alone consumes approximately 0.4×10^{15} BTU of energy annually.[5,6] A 35 percent recovery of this energy (a 70 percent efficient recuperation unit applied to the exhaust after 20 percent of the energy has been imparted to the glass and 30 percent has been lost through means other than flue gas) would amount to 0.14×10^{15} BTU or an equivalent of 6.6×10^{4} barrels of oil per day. At $12.00 per barrel, this value represents $0.8 million per day in savings for the glass industry alone. This savings will result in a reduction in the U.S. balance of payments. A savings potential of this size is a strong incentive to develop new or improved methods to recover and utilize this waste energy.

In the past, efforts to recover the heat from high temperature industrial furnaces in a concentrated stream have proceeded in one of two directions. In many operations, the flue gas is either systematically diluted with ambient air or run in a cocurrent mode to reduce the material temperature, allowing the use of a conventional metallic shell and tube recuperator. Such recuperators are extremely wasteful because of the reduction in extractable energy. Although conventional metallic shell and tube systems have the advantage of easy cleaning and maintenance, they are expensive and have an efficiency of less than 50 percent.

A second type of recuperation practiced in industry consists of a checker system regenerator. The flue gas and combustion air are directed through two alternating chambers of refractory bricks. When optimum checker chamber temperature is reached, the flow direction is reversed permitting incoming combustion air to absorb heat previously given up by the exhaust.

The exhaust and combustion air are switched back and forth at 20 to 30 minute intervals. The advantage of the checker system is that the flue gas can often be used without dilution (enabling high quality recuperation). But the disadvantages are:

1) Thermal quality of the inlet air is cyclic, creating air to fuel ratio control problems and/or uneven heating cycles.
2) Systems are very large and consequently must be initially included in the design (i.e., they are not usually capable of retrofit).
3) Initial cost is high and their maintenance costs are significant.

Project Objectives

This project is aimed at developing a recuperator that overcomes these heat recovery problems in the following ways:

1) Selection of materials capable of heat recovery operations at high temperatures (1700°F to 2800°F) to enable high enthalphy heat recovery.
2) Design of a continuous counterflow system to eliminate the temperature fluctuation and related problems that result directly from cyclic systems such as checker works.
3) Use of a graduated material system so that inexpensive materials can be used where temperature and other conditions do not warrant the use of expensive ones. Checker systems usually incorporate this philosophy to advantage now but most continuous operational systems do not.
4) Development of a system composed of unitary modules to facilitate ease of assembly, disassembly, cleaning, partial replacement or

modification and to permit material graduation mentioned in 3) above.

The overall purpose of the project then is to develop, test, and evaluate designs for an industrial recuperator for a glass furnace as well as other possible industrial applications such as steel, aluminum, and cement.

Specific goals to be achieved ultimately for a glass furnace recuperator are:

1) Recover up to 70 percent of furnace exhaust heat to maximize conservation.
2) Recover some heat in excess of 2100°F to make use of high grade heat and excess energy not required for preheating combustion air.
3) Reduce recuperator construction, maintenance, and replacement costs through modular design.
4) Operate in and survive a glass furnace exhaust environment.

Recovery of heat at 2100°F represents a somewhat, heretofore, unused energy source and opens avenues for increased efficiency and utilization, such as cogeneration of electricity or shaft power.

The specific Phase I research objectives were:

1) Design optimization of the basic concept. Considerations included thermomechanical stress stability, dimensions and aspect ratios, joint design, seal materials, and heat transfer surface design.
2) Selection of the best materials for recuperator modules and joint seals based on material properties, material performance in the expected environment, and projected cost and fabrication limitations.
3) Construction and short-term thermal testing of laboratory test modules.

4) Development of design criteria and test plan for prototype and extended test modules.

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MODULE MATERIAL SELECTION

Selection of the best recuperator materials was a multistep process. Following identification of design and operating constraints, the required properties were delineated. A preliminary assessment of potential material candidates was then made based on general known properties to identify the preliminary candidates. These candidates were then evaluated for their precise properties as they relate to the specific design requirements and for their relative performance in the working atmosphere. The conclusions of these efforts are presented below and in the conclusion section under "Summary of Results and Material Evaluations". The following discussions provide details of the process.

Delineation of Required Properties

The environmental constraints are imposed by glass furnace gas effluents and high temperature combustion exhausts. In the issuing PON, the sponsor also stipulated some economic and operating requirements. Additional requirements are imposed by the basic design concept and counterflow operational mode. The general constraints used in selecting preliminary candidate materials were:

1) Compressive strength retention in excess of 2000 psi at temperatures above 1800°F
2) Thermal conductivity in excess of 0.5 BTU/hr-ft-°F
3) Potential manufacturing costs of less than $50 per pound of product
4) Previous usage in high temperature applications
5) Potential tolerance for glass furnace atmospheres
6) Fabricability in complex shapes of one-foot diameter or greater.

The following paragraphs discuss the material properties identified and used in the evaluations and note the relative importance of each property to the overall evaluation. A material's capability to perform in a given application depends upon both individual material properties and effects of their complex interactions. Generally, a minimum property value must be met but beyond that no single property can be used as a selection determinant. Rather, all materials meeting all minimum requirements remain viable candidates subject to performance tests, design analysis calculations, economic studies, and fabrication limitation evaluations.

In many cases data on an important property were not available in the literature because many candidates are new and developmental in nature[7-27]. In these cases alternative, related properties were used as an indication of probable relative values. (For example, there is an approximate correlation between flexure strength and tensile strength. A measurement of either provides an indication of the other value.)

Physical Properties

a) Grain Size

Deadweight loads, thermal induced stress, and vibratory loads are inherent in the application. Grain size and size distribution are factors in flaw propagation, fracture toughness, and material strengths. Structural weaknesses because of individual grain defects and bonding become the limiting material property as grain size approaches material thickness dimensions. It was recognized that in order to avoid bond and grain failures from resulting in module failure, a material thickness to grain size ratio of 20:1 was desirable as this limits the effect of a single grain failure to a 5 percent effective reduction in strength if the effect of the placement of the failure point is ignored.

b) Density

Density is a factor in stress calculations to some degree because generated compressive loads are expected in all recuperator configurations. More importantly, density is an economic factor because initial cost and firing energy are both related to the weight of a ceramic piece. No limit is set but the value enters into economic and stress calculations.

c) Porosity and Gas Permeability

Except for some unique and special cases, porosity often correlates to gas permeability. Gas permeability of the interface permits interpath leakage between hot and cold gases and is extremely detrimental to recuperator effectiveness. Because some leakage is expected to occur at joints, it is desirable to limit material permeabilities to 1.0 millidarcy or less. This value was selected, since it results in about 0.1 percent leakage at 2000°F for a pressure difference of 2 psi across a barrier of 0.02 feet where the recuperator gas interface area ratio is 5 ft^2 per 1000 ft^3 of cooling air per hour.

Mechanical Properties

a) Elasticity

As the measure of the strain produced by a given stress, elasticity is a key factor in all stress calculations including thermomechanical stress. Elasticity at room temperature is readily determined and available. At elevated temperatures the elastic range for most materials decreases considerably and the elastic limit becomes a key factor as creep and non-elastic deformations occur. The elastic limit as a function of temperature was not generally available in the literature for candidate materials. The scope of work for this effort did not permit extensive determination of elevated-temperature property values. This will be required in future efforts to check thermal stresses in optimized designs.

b) Strengths

Because a vertical stack configuration is a possibility, a self-support capability is needed, and compressive strengths of several thousand psi would be desirable for the load bearing part of a structure. Thermal stresses are always present and flexure and tensile strengths are a measure of failure resistance under thermally induced stresses. Low tensile strength can be designed around to some extent. Due to the brittle nature of most ceramics, tensile tests are difficult and costly.[28,29]

A critical measure of a material's ability to withstand crack propagation from small defects in a stressed state is fracture toughness. This is normally a difficult and expensive test for brittle materials. Terra Tek has recently developed a relatively simple and extremely reliable method of measuring the fracture toughness of brittle materials from room temperature to about 650°F.[30 32] These measurements were made on prime candidates where values were not available in the literature.

Thermal Properties

a) Thermal Conductivity

High conductivity is a principal element in heat transfer and cost effectiveness. Because of the wide range of coupled factors, a broad range of conductivities must be initially considered. Conductivity in the range of 0.5 BTU/hr/ft^2/ft/°F (about 1.0 W/m•K) or greater was considered as the cutoff between a conductor and an insulator.

b) Linear Thermal Expansion

Thermal expansion is the principal cause of stress in a recuperator because differences in temperature result in differential expansion and distortion. A low value is desirable, as it minimizes the design limitations particularly at module joints. An average expansion rate from room temperature to 2732°F of greater than 6×10^{-6}/°F was deemed unacceptable.

c) Thermal Cycling and Thermal Shock

These properties are not strictly thermal or mechanical but are a complex function of both. Cycling failure is a fatigue type failure resulting from repeated stressings at less than the one-time failure stress. Shock failure is a result of excessive stresses related to a single rapid temperature change. It is not always easy to differentiate afterwards between these types of failure. Because both cycling and shock are avoided whenever possible in continuous glass furnace operations, they are not imposed as primary constraints.

d) Thermal creep

Strongly dependent upon temperature, creep is the predominant failure mode as materials approach the melting point. Available data are very limited for candidate materials.

Fabricability

Three factors enter into this: cost of fabrication, tolerance control (which is most often a function of the forming method and material properties), and the inherent size limits because of firing furnace limits and material properties. A module dimension of 6 inches was used as a practical cutoff to minimize potential fouling. Tolerance control was secondary, because some small amount of machining on critical joint surfaces may be tolerated if overall costs are kept within bounds. Cost and fabrication are treated in detail in later sections.

Chemical Stability

This is a critical factor which is difficult to define except by actual tests in the proper environment because of the variability in potential exhausts. Available information such as known high temperature stability, inertness in an oxygen-rich high temperature atmosphere and good resistance to CO_2, CO, N_2, H_2O, SiO_2 and alkali attack at elevated temperatures was used for initial screening.

Properties Evaluations

Prior to performance tests on viable candidates it was necessary to survey a broad range of potential candidate materials. Through an iterative evaluation procedure the most viable candidates were identified.

Preliminary Assessment

Initial property evaluation efforts centered on a literature search, discussions with prominent ceramicists and metallurgists, and interactions with technical representatives of manufacturers of ceramics and glass. Potential candidates were identified in the five categories discussed below.

Metals This group contains such high temperature materials as platinum, iridium, molybdenum, niobium, osmium, rhenium, rhodium, tantalum, tungsten, ytterbium and several others. Some of these candidates can endure the environment but the cost factors are prohibitive in all cases when compared with ceramics.

Carbides One of the most highly touted candidates in this group, silicon carbide has high thermal conductivity and relatively low expansivity. It can be made strong and dense and, up to 2550°F, has shown good slag corrosion resistance.[11] Many processes and trade names confuse the choice as different binders will present their own chemical characteristics in a furnace environment.

Nitrides Only silicon nitride can be considered a real possibility. It has limitations similar to silicon carbide plus lower thermal conductivity. Boron nitride has good conductivity, but it is anisotropic, and forming usually causes the minimum value to occur in a direction perpendicular to the thin section.

Oxides This is the largest group of candidates. With a few exceptions, they are generally strong and relatively stable. Beryllia or beryllium oxide has the highest thermal conductivity. Aluminum oxide is used extensively in the glass industry because of its corrosion resistance, but has a lower thermal conductivity. The expansivity is also greater in general for oxides than nitrides and carbides. Other oxides such as MgO, $Mg \cdot Al_2O_3$ and ZrO_2 are candidates if sodium vapor limits the use of Al_2O_3; otherwise they have no advantage over Al_2O_3 and are less available. Also, ZrO_2 has a phase change which can be detrimental under certain thermal conditions.

Silicates This group includes mullites and cordierites. Although cordierites have a temperature threshold in the range of 2200°F, their extensive use in thin substrate form for automotive catalytic converters is a definite plus for considering them for at least the lower temperature modules. Mullite is weaker than aluminum oxide and comparable with cordierite. Mullite also has expansivities intermediate to these two materials. Cordierite may have the lowest expansivity of all candidates.

From these general categories, 31 module materials were identified and assessed as being able to meet the broad design criteria, using available fabrication techniques (Table I). The specific qualities were shown in this table only if they represented a potential problem or an unusual advantage or situation. The materials were separated into three categories: 1) Prime candidates, meaning very likely to meet design criteria, 2) secondary candidates, meaning probably able to meet design criteria, and 3) tertiary or questionable candidates for meeting design criteria. Generally, refractory grade materials have large grains and exhibit very low strengths compared with the fine grained, high quality materials. This is the principal reason for their low assessment. Nevertheless, the best were chosen for further evaluation on the basis of low cost considerations. Castables are highly variable and quality control is difficult because of the fabrication methods used. Nevertheless, their availability, cost, and ease of fabrication warranted their inclusion in the evaluation program.

From this assessment, 15 leading candidates were identified and material samples for testing were ordered. A sixteenth material (AD 998 alumina) was added to the program later.

TABLE I

MATERIALS ASSESSMENT SUMMARY

MATERIAL NO.	MATERIALS	SUPPLIER/ TRADE NAME	POSITIVE QUALITIES	NEGATIVE QUALITIES	REMARKS	TEST PRIORITY	PURCHASE ORDER	FURTHER EVALUATION
1 to 8	Silicon Carbide	-	High thermal conductivity, Low thermal stress, Low thermal expansivity.	Behavior questionable in condensing glass fume contact.	Porosity, if any, should partially close during use.	-	-	
1	SiC Dense, Sintered	Carborundum/ Sintered	No Free Si, Strong, Stable at high temperature Estimated to ultimately be least expensive SiC form.	Material is still in developmental stage.		1	Yes	Yes
2	Porous SiC Self bonded sintered,	Norton/ NC 400	Pure SiC. Possibly a cheap base for a CVD coated material.	Very porous and permeable.	Use as a base for CVD (see #7) Use as an untreated material.	1 3	Yes No	Yes No
3	Reaction bonded SiC	Refel/Refel Norton/NC-430, Carborundum/	Dense, nonporous	Contains some free silicon.	NC 420 is a reaction form of NC-400: Super KT similar to NC 430 and refel: Refel is a British product - first on market	1	Yes NC 430	Yes
4	Reacton Bonded SiC	Carborundum/ KT	Dense, nonporous	May contain some impurities.	Knowledge of exact processing for manufacture not available.	2	Yes	Yes
5	Si_3N_4 bonded SiC	Norton/CN-130, Carborundum/ Refrax 20	Readily available, inexpensive.	Refractory grade grain size, some porosity.	(see #6)	1	Yes Refrax 20	Yes
6	Si_2ON_2 bonded SiC	Norton/NC-163, Carborundum Refrax 50	Readily available, inexpensive.	Refractory grade grain size, some porosity.	Porosity - 15%, Evaluation shows no apparent advantage of Si_3N_4 over Si_2ON_2. Differences may show up in furnace test.	2	Yes Refrax 20	Yes
7	CVD coated SiC base	MTC/CVD	Dense coating of pure, impermeable SiC on an inexpensive core of SiC.	Requires 2 suppliers and 2 operations.	Behavior in furnace may show advantages of pure CVD (#8) (also see #2)	1	No, but may order when NC 400 is available (See #2)	No
8	Pure CVD SiC	MTC/CVD	Dense pure SiC, non-permeable.	Expensive, must be formed around a core usually carbon.	2nd order choice.	2	Yes	Yes
9	Oxide (Fe_2O_3) bonded SiC	Norton/NC 127		Iron may be reduced in furnace, iron should be avoided due to its coloring effects on glass.		3	No	No
10 to 11	Silicon Nitride		Used in turbine applications and other high temperature applications, Dense, pure.					
10	Si_3N_4 Hot Pressed	Norton/NC 132		Very expensive, limited size availability.		3	No	No
11	Reaction bonded Si_3N_4	Norton/NC 350 Garrett/RBN 101				1	Yes RBN101	Yes
12	Si_2ON_2	Norton/Sioxyn		Expensive in fine-grain form, cannot be made dense in coarse grains.	Inexpensive medium grain brick available for testing. Cutting will be required.	2	Yes	Yes
13	Sialon	Univ. of Utah/ Sialon		Not commercially available, character in doubt.	University of Utah makes small pieces, inadequate for testing.	3	No	No

TABLE I (Continued)

MATERIALS ASSESSMENT SUMMARY

MATERIAL NO.	MATERIALS	SUPPLIER/ TRADE NAME	POSITIVE QUALITIES	NEGATIVE QUALITIES	REMARKS	TEST PRIORITY	PURCHASE ORDER	FURTHER EVALUATION
14 to 17	Alumina, Al_2O_3		Good high temperature qualities, used in many glass operations.	Thermal expansion greater than SiC, resulting in greater stress.				
14	Polycrystalline Al_2O_3	Coors/Vistal	Dense, transparent to broad IR and visible radiation spectrum.	Expensive, usually limited to small shapes.	99.9% pure	1	Yes	Yes
15	94% Alumina	Coors/AD 94	Dense	Thermal stresses may be a problem.		1	Yes	Yes
16	85% Alumina	Coors/AD 85	Dense	Thermal stresses may be a problem.		1	Yes	Yes
17	Refractory	Norton/AH 194B		Not dense, refractory shapes only.	Fuze cast then reground and refired.	3	No	No
18 to 20	Mullite				Thermal expansion intermediate to SiC and Al_2O_3.			
18	Mullite	Coors/Mullite	Dense			1	Yes	Yes
19	Refractory	Norton/Mulnorite or KH-176A		Not completely dense, for refractory shapes, porosity 16%.	Fuze cast, reground and then refired.	3	No	No
20	Mullite Refractory	Coors/ -		Not dense, refractory shapes.	Differences between Norton & Coors processing are not known. Materials may be different in character.	3	No	No
21	Fuze Cast AZS	Corning/AZS	Dense, good in glass contact.	Must be shaped with diamond tools. Very expensive in formed shapes.		3	No	No
22	Cordierite	Coors/CD-1	Inexpensively fabricated, large existing market.	Maximum temperature limited, 2200°F-2550°F		1	Yes	Yes
23 to 25	Basic Refractories		Easily available in non-dense forms, inexpensive.	High C.O.E.				
23	M_gO		Successful checker material.	No one fabricating dense forms.		3	No	No
24	$M_gO \cdot Cr_2O_3$		Successful checker material.	No one fabricating dense forms.		3	No	No
25	$M_gO \cdot Al_2O_3$	Coors/Spinel	More resistant to alkalis than Al_2O_3.	Commercial Fabrication limited.		2	No	No
26	ZrO	Coors/Zirconia	May have good corrosion resistance.	High thermal expansion ($>M_qO$)	Better insulator than most materials.	2	Yes	Yes
27	CaO Bonded castable refractory		Possibly inexpensively fabricated.	Considerable porosity, thin membrane difficult to fabricate.	Readily available	2	Order later	Yes
28	Phosphate Bonded Castable Refractory		Possibly inexpensively fabricated.	Considerable porosity, thin membrane formation difficult.	Readily available.	2	Order later	Yes
29	Beryllia, BeO		High Conductivity	Potential health hazard.	Hard to obtain due to health hazard.	3	No	No
30	Platinum Pt.		Excellent material for this application.	Extremely expensive.		3	No	No
31	Boron Nitride			Anisotropic conductivity. Very difficult to fabricate. Oxidizes at about 1200°F.		3	No	No

Mechanical and Thermal Properties Evaluations

Many of the candidate materials are proprietary with a specific manufacturer. Furthermore, the fabrication process usually includes variables that give rise to property deviations as compared with the pure generic material made for laboratory tests. As a result, much of the available information is in the form of manufacturer specifications.

The following subparagraphs discuss the unique properties and considerations for the prime candidates. Each material is identified by its material number.

No. 1 Sintered Silicon Carbide. With about 0.3 percent free carbon and 0.5 percent free boron, silicon carbide of less than a micron in particle size can be sintered to a nonporous condition. General Electric's Beta and Carborundum's Sintered Alpha products are in the research and development stage.

No. 2 Recrystallized Silicon Carbide. This is fine grained silicon carbide formed and heated to sintering temperatures. Without free carbon and boron it will yield vapor transport, and crystals will grow and stick together without shrinkage. The result is a porous and permeable but rather strong SiC skeleton. Thermal conductivity is thereby decreased by about a factor of 2 from pure, dense SiC. The variation intended here involved using CVD to coat or form an impervious skin on this material, making it satisfactory for design criteria.

Nos. 3 & 4 Self Bonded Silicon Carbide. This silicon carbide is reaction sintered using silicon and carbon as admixtures that form silicon carbide in situ. These are much less porous than recrystallized SiC. The

Refel type uses silicon carbide with graphite into which is absorbed melted silicon much as a sponge absorbs water. Some free silicon fills the pores.

Nos. 5 & 6 Nitride and Oxynitride Bonded Silicon Carbide. Carborundum uses silicon carbide with silicon and reacts the powder compact at temperatures near the melting point of silicon with nitrogen to give Si_3N_4 as the bonding agent. Norton uses the heoxynitride in a similar fashion. These materials proved to be too weak to stand sample preparation in a 3/8-inch cross section.

Nos. 7 & 8 Chemically Vapor Deposited Silicon Carbide. This can be formed as a pure material on a removable substrate, usually carbon, or a permanent substrate such as self-bonded, sintered SiC. It was originally intended to do the latter using NC 400 as a base material (No. 7). Delivery of sufficient NC 400, however, could not be achieved and alternative samples of pure CVD (No. 8) were ordered from MTC in Dallas, Texas.

Nos. 10 & 11 Silicon Nitride. Hot pressing is possible, but tolerance control is so poor that "diamond truing" and/or grinding is usually necessary for any moderate tolerance control. This raises parts costs tremendously. The reaction bonding technique is a nonshrinking process and thus is capable of very tight tolerance control. Some problems with heat buildup and reaction completion in thick shapes may be difficult to overcome if wall thickness ever needs to exceed 0.75 inches. The lower costs, however, make the reaction bonded material (No. 11) the first choice.

No. 12 Sioxyn (Si_2ON_2). A moderately inexpensive plate of this material was obtained from Norton. It showed considerable weakness during cutting operations for furnace coupon sample preparation.

<u>Nos. 14, 15 & 16 Aluminum Oxide</u>. The three purities represented in these materials span the range of high quality alumina. The impurities in AD 85 and AD 94 are of considerable concern, because their reaction to furnace atmospheres may dictate overall effectiveness. Vistal is particularly attractive for high temperature uses, because of its high purity and translucence to the visible and infrared portions of the energy spectrum. AD 998 was subsequently added to the test program because of the poor performance of AD 85 and AD 94.

<u>No. 18 Mullite</u>. A mullite porcelain can be obtained as a dense sintered material. Because it has a lower expansivity, it has an advantage over alumina. Its mechanical strengths, however, are somewhat less than those of alumina.

<u>No. 22 Cordierite</u>. This is a readily available material in wide use now; hence, its value for testing for this application. Its temperature limitation of about 2200°F and reported reaction with glass furnace exhausts are major concerns.

<u>No. 26 Zirconia</u>. This material has proven high temperature capability, but is lacking in necessary conductivity and expansivity limits. Furthermore, slow acting but seriously damaging phase changes occur when ZrO_2 passes through a specific elevated temperature range. It was selected for further testing because a) its use as an insulator may prove valuable for the recuperator shell and b) it is being tried by other high temperature recuperator researchers.

Table II presents specific property values as available for the 15 candidates initially selected for further evaluation. Many differences were noted when generic values from the literature were compared with specific product

TABLE II

RECUPERATOR MODULE
MATERIALS PROPERTIES EVALUATION

	Units							
Material Number		1	2	3	4	5	6	8
Material		Sintered Alpha	NC-400	NC-430	KT	Refrax 20	Refrax 50	CVD
Supplier		Carborundum	Norton	Norton	Carborundum	Carborundum	Carborundum	MTC
Chemistry	--	SiC, Dense	SiC, Porous	SiC Bonded with SiC·Si	SiC Bonded with SiC·Si	SiC Bonded with Si_3N_4	SiC Bonded with Si_2ON_2	Chemically vapor deposited SiC
Color	--	Dark Grey	Dark Grey	Dark Grey	Dark Grey	Grey	Grey	Dark Grey
Grain Size	Micro-inches	28				Up to 79,000	Up to 79,000	N/A
Density	lb/ft^3	197	163	192	188	162	169	202
Porosity	Percent		~19			15	14	Theoretical 0
Gas Permeability			High perm.		Gas Tight			
Hardness: Test Type	--	Knoop		Knoop 100	Knoop			KHN 100
Values	As Specified	2800		2500	2500			2740
Strength: Tensile	KSI							
Compressive				260	150 @ RT			119 @ RT
Flexure (Rupture)		50 @ RT 50 @ 1222°F	18 @ RT 18 @ 1832°F	33 @ RT 30 @ 2372°F	23 @ RT	6.2 @ RT 6.3 @ 2462°F	5.6 @ RT 3.4 @ 2462°F	
Fracture Toughness	$KSI\sqrt{in}$	2.3 @ RT 5.8 @ 1832°F			2.8 @ RT 6.8 @ 2192°F 4.6 @ 2462°F			
Moduli: Elasticity	10^6 PSI	59.4 @ RT 54.4 @ 1832°F	30	54	53 @ RT 48 @ 2192°F			34 @ RT
Shear		25.6 @ RT 24.5 @ 1832°						
Bulk								
Rigidity								
Poisson's Ratio	--	.15 @ RT .11 @ 1832°F			.16			
Sonic Velocity	10^3 ft/sec							
Maximum Use Temperature	°F			2800	2462 - 3000	3200	3200	3200
Thermal Expansion: (Linear Coefficient) R.T.	10^{-6}/°F	1.33	2.67	2.7				
RT - 1112°F					2.78			
RT - 1832°F					2.5			2.5
RT - 2552°F		2.61				2.61	2.61	
Thermal Conductivity @ R.T.	$Btu/hr/ft^2/ft/°F$	52.3	25		49.5			25.
1112°F		29.7			15.1			
1832°F				22.9		(9.42)	(8.75)	
2552°F								10.
Heat Capacity	Btu/lb	.29 @ RT .6 @ 1832°F	.27	.29 @ 1800°F		.51	.51	
Resistance to: Thermal Shock								
Thermal Cycling								
References	--	8, 9, 10, 17, 22	7,11,21,22	7, 11, 22, 26	8, 9, 17, 18, 22	19	20	23

TABLE II (Continued)

RECUPERATOR MODULE
MATERIALS PROPERTIES EVALUATION

Material Number	Units	11	12	14	15	16	18	22	26
Material		RBN	Sioxyn	Vistal	AD-85	AD-94	Mullite	CD-1	Zirconia
Supplier		Airesearch	Norton	Coors	Coors	Coors	Coors	Coors	Coors
Chemistry	--	Si_3N_4 Reaction Bonded	Si_2ON_2	Polycrystalline Al_2O_3, 99.9% Pure	85% Al_2O_3, impure	94% Al_2O_3, impure	75% Al_2O_3, 25% SiO_2	Cordierite	ZrO
Color	--	Blue Grey	Light Grey	Translucent White	White	White	Light Buff	Light Grey	White
Grain Size	Micro-inches			(600-1800) 800 Ave.	(80-500) 240 Ave.	(80-1000) 480 Ave.			
Density	lb/ft^3	171	125	250	212	227	177	150	352
Porosity	Percent								
Gas Permeability				Gas Tight	Gas Tight	Gas Tight		Gas Tight	
Hardness: Test Type	--	- - -	- - -	Rockwell 30N	Rockwell 45N	Rockwell 45N	- - -	Rockwell 45N	- - -
			Knoop 100	Knoop	KHN 100	KHN 100			
Values	As Specified	- - -	- - -	85	73	78	- - -	70	- - -
			1000	2190	9.8 GPa	11.1 GPa			
Strength: Tensile	KSI	19		32 @ RT 15 @ 1832°F	22 @ RT	28 @ RT 15 @ 1832°F			
Compressive		>100	35	370 @ RT 70 @ 1832°F	280 @ RT	305 @ RT 50 @ 1832°F	80 @ RT		
Flexure (Rupture)		35	7 @ RT 6 @ 2550°F	41 @ RT 25 @ 1832°F	43 @ RT	51 @ RT 20 @ 1832°F	27 @ RT 22 @ 1832°F	24 @ RT 17 @ 2102°F	
Fracture Toughness	KSI$\sqrt{in}$	1.79 @ RT		4.4 @ RT	3.0 @ RT	3.4 @ RT	1.34 @ RT		3.98 @ RT
Moduli: Elasticity	10^6 PSI		11	57	32	41	22.5		
Shear				23.5	14	17			
Bulk				34	20	24			
Rigidity				23					
Poisson's Ratio	--			.22	.22	.21			
Sonic Velocity	10^3 ft/sec			32.5	26.9	29.2			
Maximum Use Temperature	°F		2800	3452	2552	3092	3092	2372	
Thermal Expansion: (Linear Coefficient) R.T.	10^{-6}/°F		2.4	3.61	2.94	3.5	2.06	0.22	
RT - 1112°F									
RT - 1832°F				4.72	4.0	4.39	2.78	1.22	
RT - 2552°F									
Thermal Conductivity @ R.T.	Btu/hr/ft^2/ft/°F			.25	8.4	10.4	2.37		
1112°F					(3.2)	(3.8)			
1832°F			3.0	4.52					
2552°F									
Heat Capacity	Btu/lb		.29 @ 1800°F	.38 @ 212°F .54 @ 2552°F	.40 @ 212°F	.38 @ 212°F	.32 @ 212°F		
Resistance to: Thermal Shock									
Thermal Cycling									
References	--	25	26	12,14,15,24	15, 24	15, 24	16	13	

data. In all cases, specific product values are reported when available. The drop in thermal conductivity from room temperature to 3000°F seems to be general with all materials.

Fracture toughness values are difficult to obtain by standard techniques. Furthermore, correlation of values for different accepted techniques is not good for brittle ceramics. Terra Tek's recently developed technique noted earlier was used in obtaining consistent fracture toughness data. These tests were run on as many materials as could be obtained from suppliers. In the case of KT and Sintered Alpha, the manufacturer's data had a large spread of values for different techniques. K_{Ic} room temperature values for Sintered Alpha ranged from a low of 2.6 Ksi$\sqrt{m}$ for the surface crack bend method to a high of 4.9 Ksi$\sqrt{m}$ for a single edge notched beam test.[33] Terra Tek measurements provided a value of 2.3 Ksi$\sqrt{m}$. Values provided in Table II are Terra Tek values at room temperature. Elevated temperature data are from the other references noted in Table II.

An important element in the materials evaluation is the use of these properties to calculate the thermal stresses and engineering safety margin in the design calulations. Also, the change in strength as measured by flexure tests (discussed later in the Performance Evaluation section) served to evaluate each material.

Performance Test Furnaces and Glass

Two types of furnaces were used to test materials performance: one for thermal cycling and thermal shock and the other for long-term exposure effects in the glass and combustion exhaust environments.

The thermal shock and cycling furnace consisted of a refractory lined steel chamber used as a "glory hole" in glass blowing operations. It is normally cycled between room temperature and 2200°F on a daily basis. Full temperature excursions occur in about 30 minutes. Figure 1 shows some test samples in the rear of the furnace chamber.

A two-furnace facility was designed to permit testing in a combustion product exhaust and glass furnace exhaust at the same time. This permitted greater flexibility in the test program, more materials, and a shorter time frame. The dimensions of the furnace and flue dictated the physical dimensions of the material samples to be tested in the exhaust environment. The front-to-back cross section in Figure 2 shows a detail of the sample insertion area of the flue.

The exhaust environment furnace designs were basically the day tank furnaces normally used at Millcreek Glass for art glass production. Modifications in materials and operations were made to accommodate the special test requirements which differed from those of the normal day tank.

Probably the two most important factors in determining the environment under consideration are the temperature and the composition of the glass being melted. At Millcreek, the glass used is between standard soda-lime and borosilicate glasses in composition, and contains some components that do not appear in most standard compositions. Furthermore, because the temperature at which glass is drawn from a furnace is 400°F to 800°F below the temperature at which it is melted, a day tank type of furnace such as Millcreek uses spends much of its time at a lower temperature than the operating temperature of a continuous furnace. It was decided that these differences implied that the research work was not compatible with Millcreek's production furnaces. Therefore, the new furnaces were designed and constructed for project use to

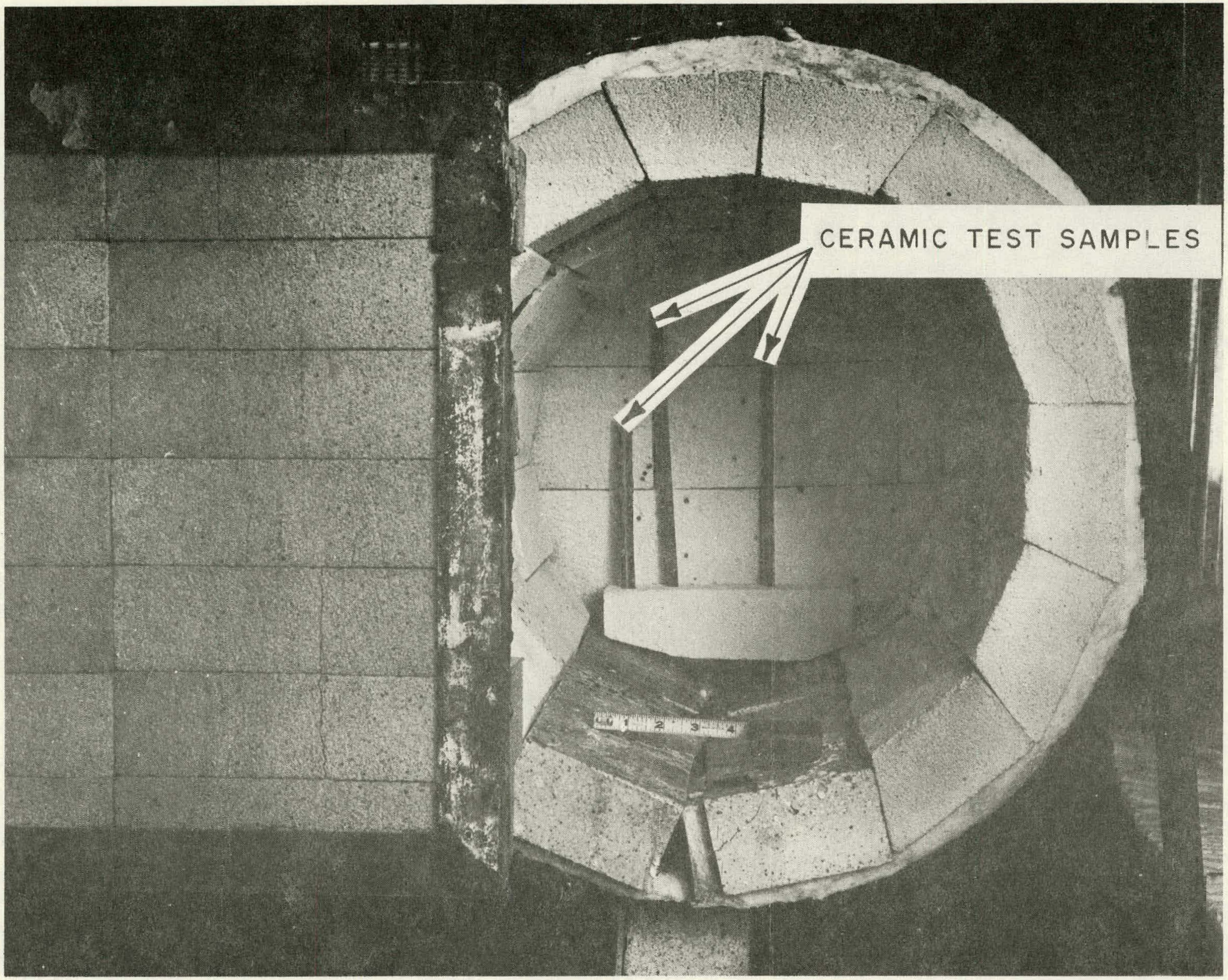
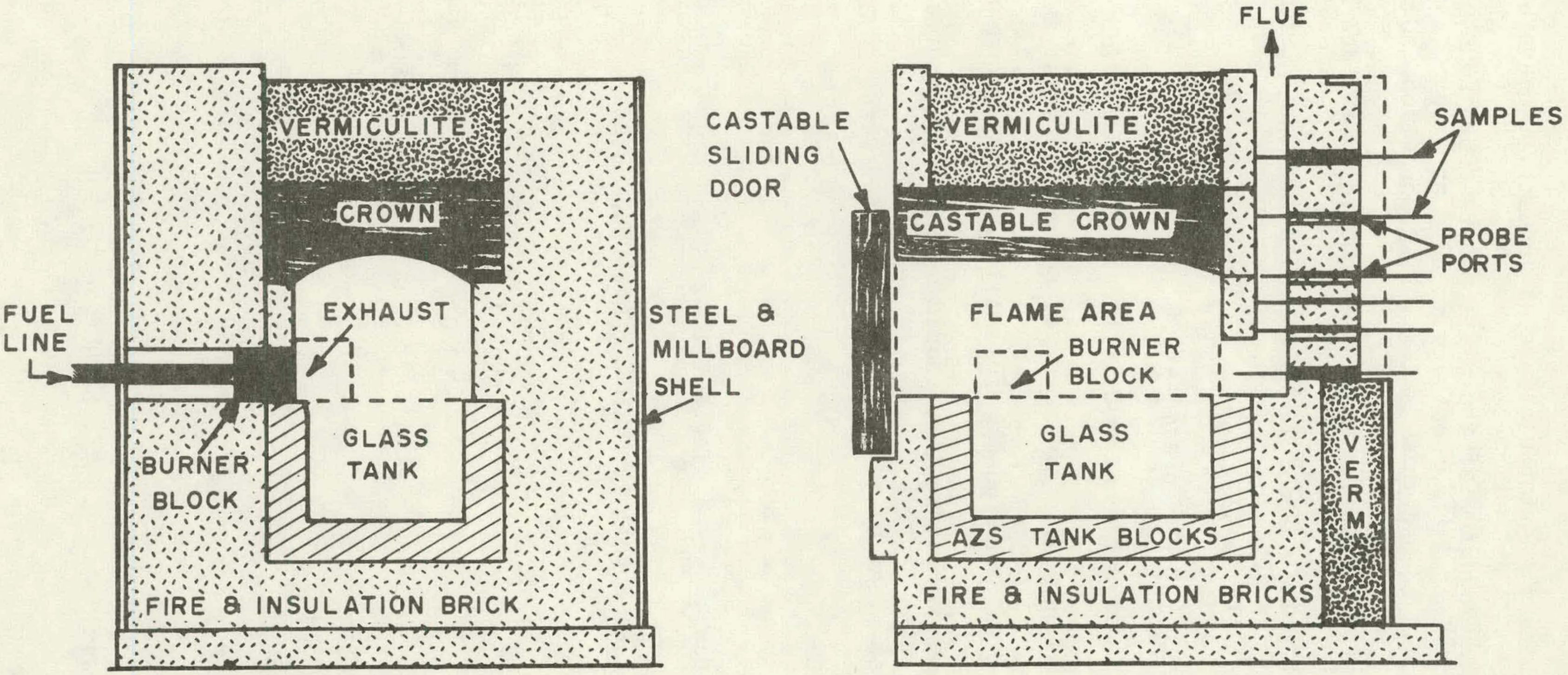


Figure 1. The thermal cycling furnace. Samples are upright in the rear. This furnace is used as the "glory-hole" during glass blowing. It is cycled from room temperature to over 2000°F very rapidly on a daily basis.



SIDE-TO-SIDE CROSS SECTION

FRONT-TO-BACK CROSS SECTION

Figure 2. Special day tank type furnaces used at Millcreek Glass for material tests.

contain standard industry glass formulations at industry temperatures.

Because easily the majority of the glass produced by industry is a soda-lime type, it is prudent to use this as the test composition. Glass formulation ERDA 1, which was developed to match the industrial furnace atmospheres, was subsequently modified to DOE 2 formulation to provide a better match to the final glass product (Table III).

Although the glasses are representative of soda-lime glass used in the plate glass and container glass industries, any one plant in the glass industry would probably not use barium, cryolite, and borax at the same time. These additions are used individually in small quantities in many soda-lime formulations and it was felt that they should be included to give the exhaust all the components that might be found in any operating plant.

TABLE III

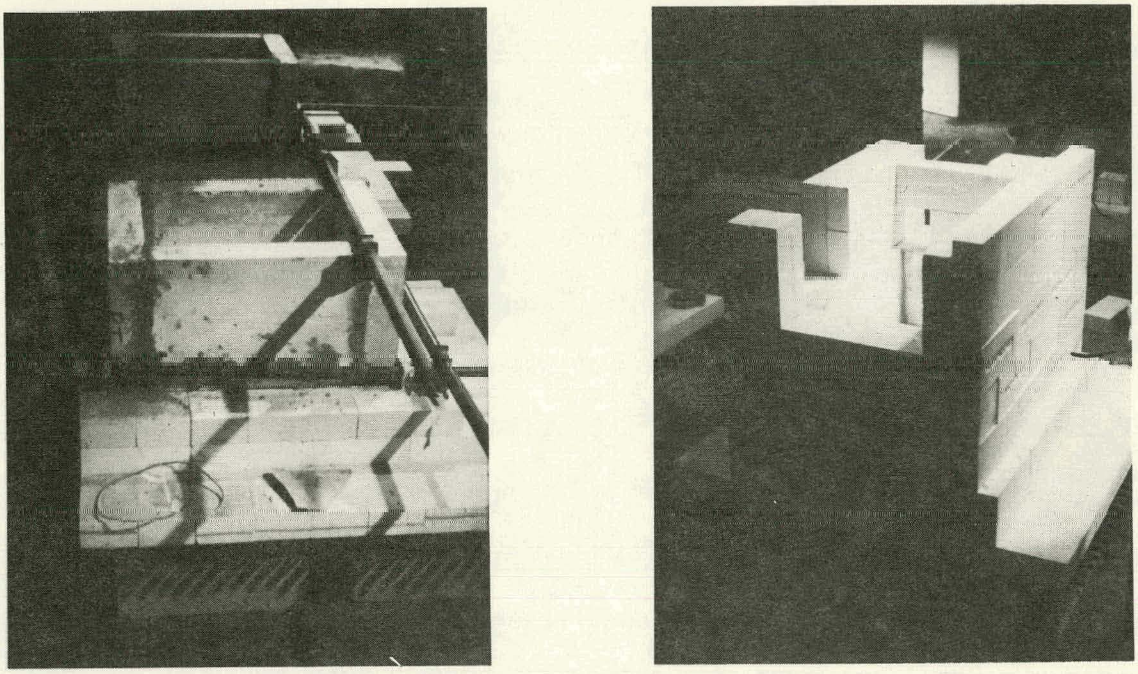
GLASS FORMULA COMPARISON
(Pounds)

	ERDA 1	DOE 2
SiO_2	63.0	65.0
Na_2CO_3	22.0	21.0
Kingman Feldspar	10.0	-
F_4Kona Spar	-	9.0
$CaCO_3$	18.0	12.0
Dolomite	9.0	8.0
$BaCO_3$	0.7	0.5
Cryolite	1.0	0.2
Anhydrous Borax	0.7	0.1

Figure 3 shows the details during furnace construction. The tiles that formed the glass melt tank are an AZS fused-cast material from Carborundum Corporation called Monofrax S-3. These and a comparable product made by Corhart are the industry standard for soda-lime tank blocks. The sidewalls of the furnace above the glass level are Clipper D.P. "Super Duty" firebricks which contain over 40% alumina and about 50% silica. The roof is made from a Babcock and Wilcox castable 141A high alumina castable refractory. These high alumina materials differ markedly from those used in continuous tanks where silica blocks would be the norm. The reason for this choice is that silica blocks have large thermal expansion coefficients at temperatures below approximately 1800°F. This makes them difficult to heat in a small furnace without spalling. Further, if modifications are required in a furnace during testing, silica blocks are a problem because they are extremely difficult to cool and reheat with enough control to prevent collapse. Exterior insulating bricks provide structural support and thermal insulation. Granular Vermiculite completed the insulation requirements. These chosen materials are equivalent to materials that Millcreek Glass has used successfully in the past and there was no question that they would serve successfully under test conditions.

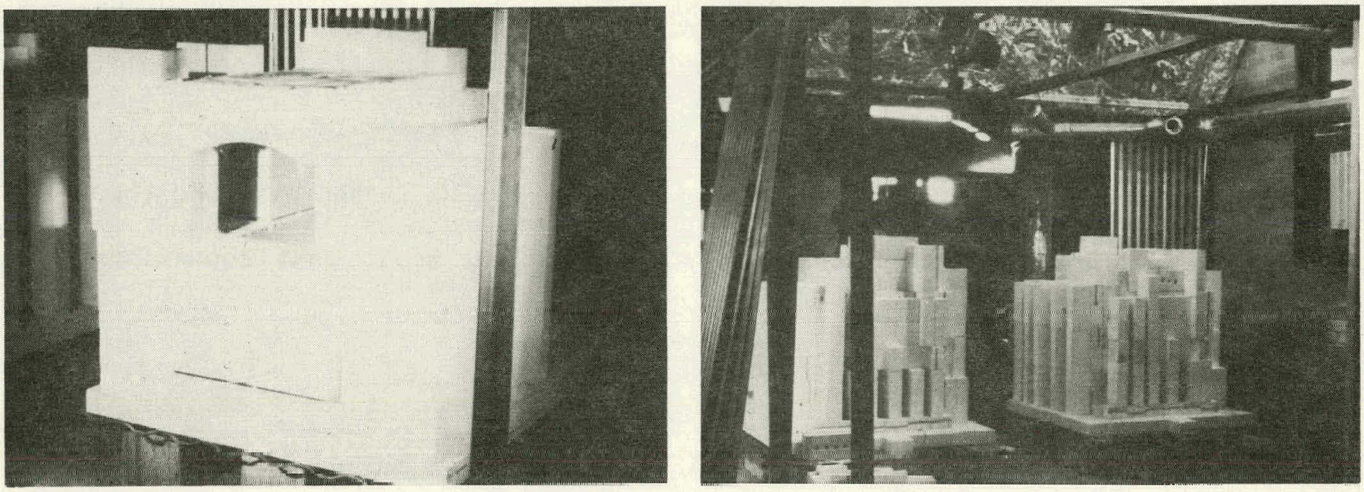
Performance Tests in an Exhaust Environment

A furnace exhaust environment can be characterized by three elements: 1) thermal conditions, 2) combustion products which are related to fuel and fuel/air ratios, and 3) furnace product wastes. While not all conditions for all potential furnace applications could be tested in this project, it was possible to test materials in two different conditions. A pure combustion product atmosphere (where no glass was ever melted in the test furnace) was



Tank blocks in place

Fire brick in place



Crown and insulation brick in place

Two furnaces prior to final enclosure

Figure 3. Test furnaces shown during progressive construction stages.

established in one of the two material test furnaces. This exhaust contained Co_2, O_2, N_2, and H_2O as principal components after the air and natural gas combustion. This condition provided baseline data on material performance in a high temperature combustion product atmosphere which can be related to expected performance in any furnace. The other of the two test furnaces produced soda-lime glass during the entire test and the exhaust contained the by-products of glass melting in addition to the combustion products.

An absolute performance is difficult to predict from any single test because of the great variability in conditions from one glass furnace to another and even from one exhaust port to another. To overcome this difficulty, a widely accepted method of measuring relative performance for refractory materials has been developed and is in general use.[34] This method consists of placing the refractories directly in the exhaust stream which tends to accelerate time dependent effects. Multiple materials are tested simultaneously and the relative effects provide a measure of likely relative performance in practice. By using this test method, investigators can identify the best materials after a relatively short test period.

Test samples were fabricated from rods and tubes of candidate materials and inserted into both exhaust flues transverse to the gas flow at a range of temperatures from 2000°F to 2850°F.

It was desirable to maintain constant conditions during the tests and duplicate as closely as possible conditions expected in an industrial glass furnace. To do this, the following measurements were used as controls: An Omega model "S" sheathed, ungrounded thermocouple (Pt vs. Pt 10% Rh) periodically monitored temperature profiles of the flue sample area. An Orsat gas analyzer model 621 A.31:30 monitored exhaust chemistry for O_2, CO_2, and CO.

Except during short periods of burner failure, flue conditions were maintained oxygen rich with exhaust O_2 kept between zero and plus 5 percent. Rockwell model 321 CFH natural gas meters monitored fuel consumption on each furnace.

Furnace temperatures were kept constant at a high melt temperature. The glass formulation ERDA 1 was used for several months and then changed to DOE 2 to improve the final glass product match with industry glass. To duplicate chemistry, timing, and charge carryover conditions, the day tanks were charged with additional glass batch at frequent intervals, generally three or four times per day. Glass melt was removed as necessary to prevent overflow.

Material test specimens were preferably long cylinders or rods. Some materials, however, were not readily available in this shape and had to be sliced from long tubes. Some tubes were small enough to be used intact. Exposure cross sections were typically 2.5 inches long and 0.375 to 0.500 inches wide.

Before insertion in the furnace, material samples were photographed, measured, and weighed. Figure 4 shows the sample port area with some samples in place. Archive samples were also preserved for chemical analysis and other material properties tests to follow. During the furnace test period, samples were periodically removed, photographed, inspected, and evaluated. Figure 5 shows examples of archive and exposed test samples. Severely degraded materials were replaced with other materials because furnace size and temperature profiles did not permit testing of all material candidates simultaneously at all desired temperature ranges.

With the furnaces, conditions essentially like those in the flue of a continuous soda-lime tank were achieved. Continuous furnace conditions were

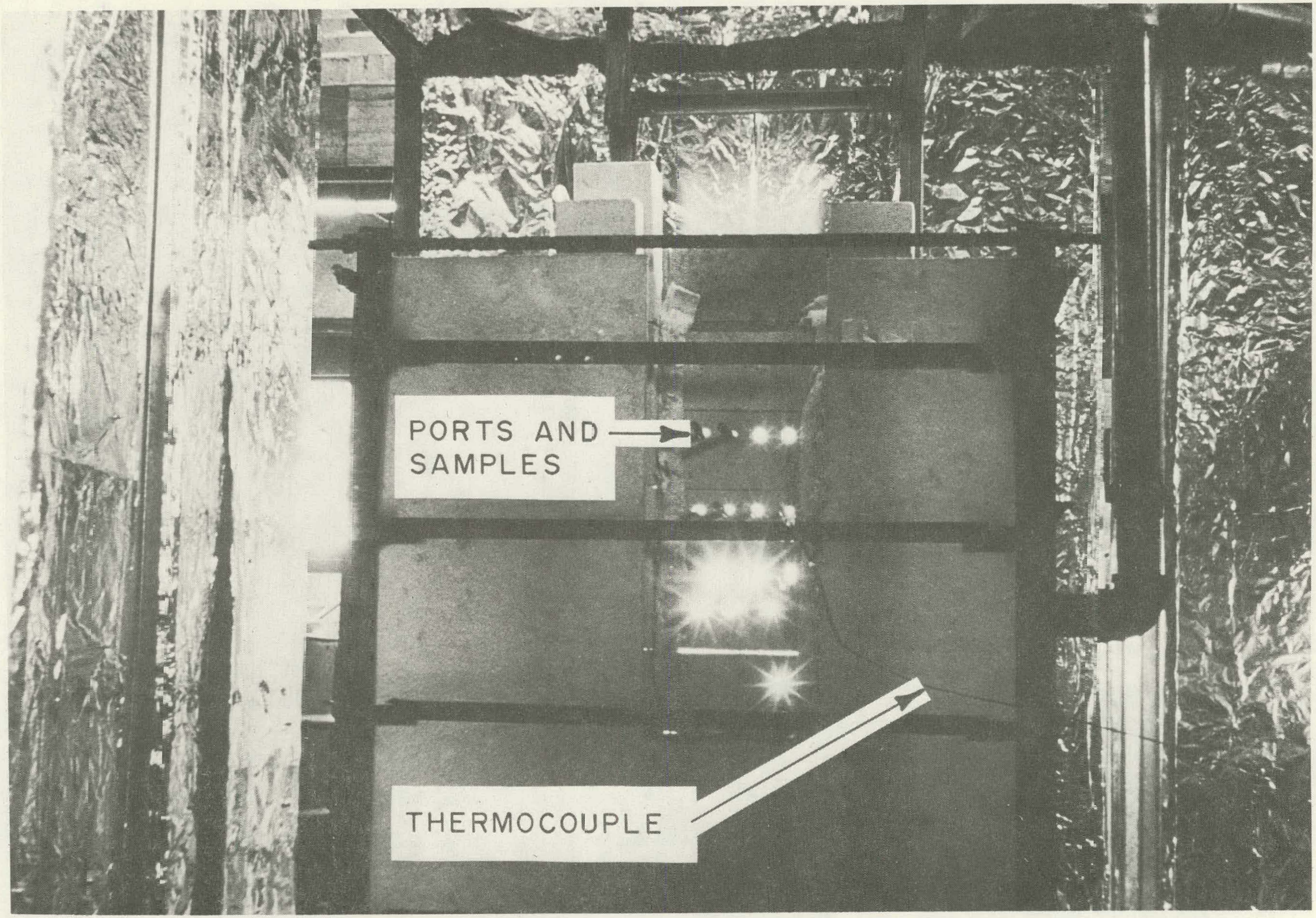


Figure 4. Test furnace during operation. At this point not all ports are occupied with samples.

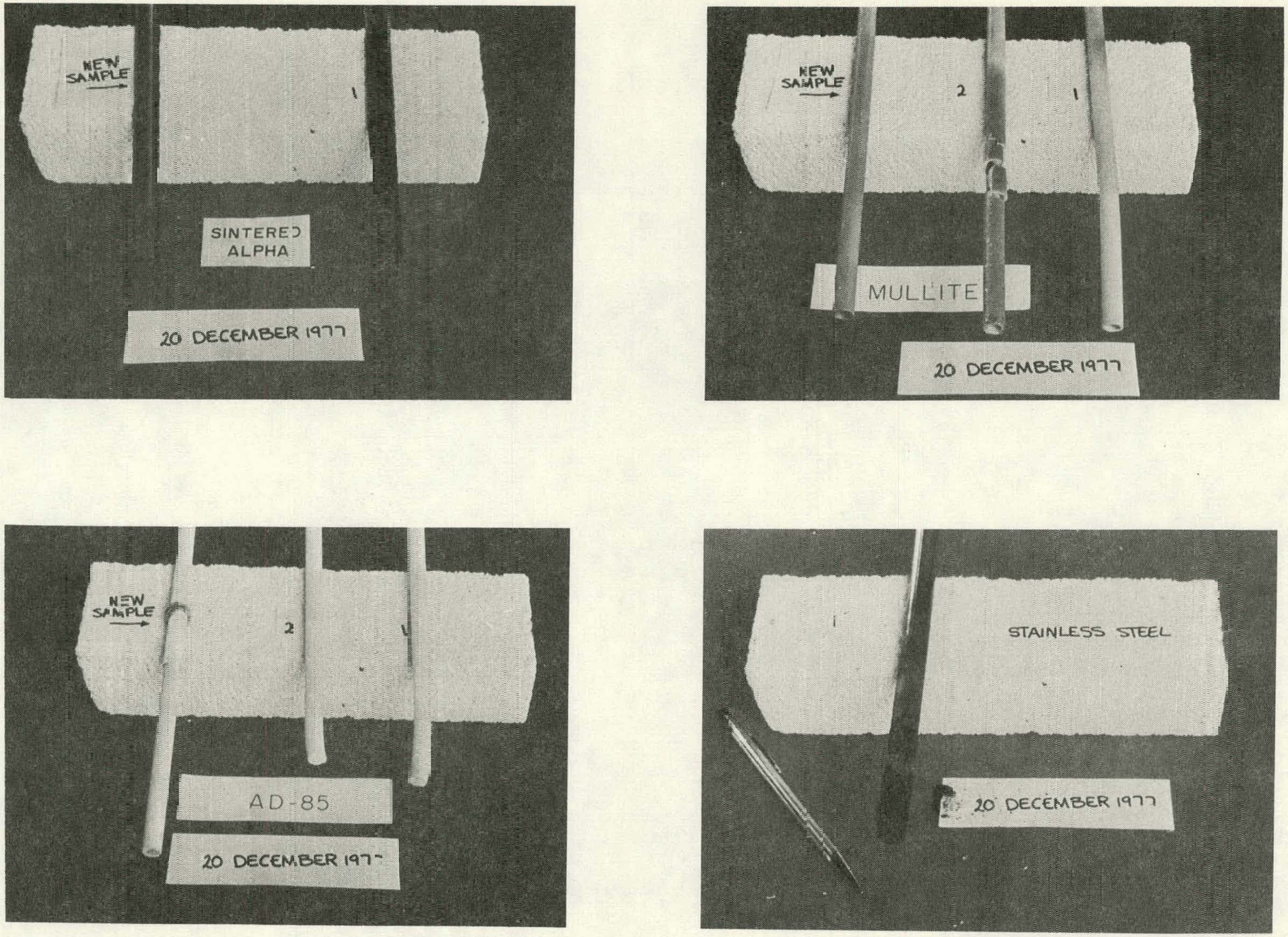


Figure 5. Furnace test samples. Unexposed samples are identified as New. Samples exposed to glass furnace exhaust are identified by the number 2. Samples exposed to combustion product exhaust are identified by the number 1.

further duplicated by spacing of "charging" to give a batch carryover into the flue comparable with what might be found in a large tank.

One cannot expect that the conditions in the flue of a small day tank type of furnace will be identical at all times to a given flue in a given large continuous furnace. But, in fact, so much variation exists between one large furnace and the next, and even between the various exhaust ports in a given furnace, that the control in the test conditions allows results as meaningful as those achievable in a larger installation.

This variability makes the time effect difficult to assess. This means that a comparative approach must be relied upon for results analysis. If one material performs significantly better than another in one environment over a given span of time, then it should also be better for another essentially similar environment over a longer time span. This approach is commonly and successfully used in the glass refractories industry.[34]

Furnace testing of materials samples began in November 1977, and continued until March 1978. Furnace temperatures varied between a low of 2360°F, for a short period during which a burner problem appeared, and 2955°F. The average was very close to 2800°F. The average temperature for the hottest sample was 2800°F and the lowest test temperature was approximately 2100°F. While one furnace never melted any glass, small amounts of glassy condensate did appear on some samples. The source of this condensate was apparently derived from kiln materials during firing.

Thermal Cycling Tests

For some possible applications of the recuperator such as a remelt furnace, an amount of thermal cycling may be experienced. To determine material degradation due to thermal cycling, the glass blowing "glory hole" furnace was used. The furnace cycled from room temperature to above 2000°F in less than one-half hour. Temperatures were typically held for several hours. Cooling to 200°F occurs in less than one-half hour. This cycle was typically made once a day. Because of limited space, only representative materials were tested. These were Sintered Alpha, Si_3N_4, Vistal, AD 998, and KT.

After 80 cycles, no failures occured in Sintered Alpha, AD 998, or KT. Although a small hairline longitudinal fracture did appear in the Vistal sample, it did not propagate entirely and the sample remained intact. Si_3N_4 performed well but was withdrawn from the test before completion because of poor performance in other tests and was replaced in this test by AD 998.

Performance Evaluations

As indicated earlier, performance of the module candidate materials in both combustion and glass furnace exhausts were evaluated on a relative basis. Tables IV and V list observation results for the glass furnace exhaust and combustion product exhaust respectively.

As can be seen by comparing observations from Tables IV and V with expected material limits noted in Table II, in several cases furnace samples deformed and failed after continuous exposure at temperatures significantly below the manufacturer's quoted service temperature. Thus, quoted service temperatures may be valid for short exposures and not long-term use. A failure example is AD 94 which was supposed to be good to 3100°F but which sagged under no-load conditions at 2650°F. The suspected reason is that the 6 percent impurities are in a continuous phase and represented a significant por-

TABLE IV

MATERIAL SAMPLE OBSERVATIONS

FURNACE I (WITHOUT GLASS)

Approx. Ave. Temp.	Material and Sample #	Date Inserted	Date Observed	Observations
2800	CVD SiC:1	4 Nov.	21 Dec.	Glazed with some foam. Apparent decrease in diameter over part of exposed length.
			28 Feb.	Some decrease in diameter on cooler part of exposed length.
2650	AD 94:1	3 Nov.	11 Nov.	Slumping slightly. Moved to cooler area at 2550°F.
2650	NC 430:1	16 Nov.	21 Dec.	Glazed. No dimensional deterioration.
			28 Feb.	Milky coating. Virtually no dimensional deterioration.
2650	Mullite:1	3 Nov.	11 Nov.	Easily bent at temperature. Greatly reduced resistance to thermal shock. Withdrawn.
2650	Vistal:1	15 Nov.	21 Dec.	Iron staining from sample above. No dimensional deterioration.
			28 Feb.	Loss of "Waxy" surface. No dimensional deterioration.
2650	Zirconia:1	4 Nov.	25 Nov.	Hot end slumped slightly. Severe multiple fracturing in intermediate region inside flue wall. Withdrawn.
2650	Sinteredα:1	∿10 Dec.	21 Dec.	Slight glazing. No dimensional deterioration.
			15 Jan.	No change. Moved to area at 2600°F to make room for AD 998 sample.
2650	AD 998:1	15 Jan.	23 Feb.	No apparent deterioration.
2600	Sinteredα:1	15 Jan.	28 Feb.	Milky coating. No dimensional deterioration.
2600	Cordierite:1	3 Nov.	7 Nov.	Hot end slumped, then fell off. Withdrawn.
			28 Feb.	Recovered end curled and badly deteriorated.

TABLE IV (Continued)

MATERIAL SAMPLE OBSERVATIONS

FURNACE I (WITHOUT GLASS)

Approx. Ave. Temp.	Material and Sample #	Date Inserted	Date Observed	Observations
2550	NC 400:1	4 Nov.	21 Dec.	Some glazing and foam. No dimensional deterioration.
			28 Feb.	No apparent dimensional deterioration.
2550	Vistal:1	4 Nov.	11 Nov.	No change observed. Moved to hotter area at 2650°F.
2550	AD 85:1	4 Nov.	11 Nov.	Severe slumping. Withdrawn.
2550	AD 94:1	11 Nov.	21 Dec.	Iron staining from another sample. No dimensional deterioration.
			28 Feb.	Same as 21 Dec.
2450	Si_3N_4:1B	4 Nov.	20 Dec.	Sample broken upon extraction for observation. Possible flaw apparent. Possible slight deterioration of exposed surface.
2450	KT:1B	4 Nov.	21 Dec.	Glazed. No dimensional deterioration.
			28 Feb.	Possible slight rounding of sharp edge.
2300	Mullite:3	11 Nov.	25 Nov.	Darkening of surface, crystalline development on one side. Greatly reduced resistance to thermal shock. Withdrawn.
2300	Cordierite:3	11 Nov.	25 Nov.	Exposed side of sample darkened, blistered and expanded causing sample to curl upwards. Withdrawn.
2250	Si_3N_4:3B	28 Nov.	21 Dec.	Glazed. Little if any dimensional deterioration.
			31 Jan.	Sample broken easily upon extraction. Break very similar to sample Si_3N_4:1B.
2250	CVD SiC:3	28 Nov.	21 Dec.	Glazed. No dimensional deterioration.
			28 Feb.	Same as 21 Dec.
2100	Cordierite:4	28 Nov.	21 Dec.	Slight edge deterioration. Surface darkened.
			28 Feb.	Surface darkened. Edge slightly rounded.
2100	Stainless 426	22 Nov.	20 Dec.	Severe scaling. Withdrawn.

TABLE V

MATERIAL SAMPLE OBSERVATIONS

FURNACE II (WITH GLASS)

Approx. Ave. Temp.	Material and Sample #	Date Inserted	Date Observed	Observations
2800	CVD SiC:2	4 Nov.	21 Dec.	Glazed with some foam. Apparent decrease in diameter over part of exposed length.
			13 Jan.	Sample eroded into two parts. Most erosion at or near supporting wall where glass condensate is apparent.
2800	Sinteredα:2	13 Jan.	30 Jan.	Sample eroded almost into two parts. Erosion limited to zone at supporting wall where material temperature is well below 2800°F.
2800	KT:4	31 Jan.	10 Apr.	Slight decrease of diameter inside supporting wall at much lower temperature.
2650	AD 94:2	3 Nov.	11 Nov.	Slumping slightly. Moved to cooler spot at 2550°F.
2650	NC 430:2	16 Nov.	21 Dec.	Glazed. No dimensional deterioration.
			28 Feb.	Sample circumference decreased at sample wall zone of lowered temperature.
2650	Mullite:2	3 Nov.	11 Nov.	Easily bent at temperature. Crystalline development in ceramic body. Greatly reduced resistance to thermal shock.
2650	Vistal:2	15 Nov.	21 Dec.	Surface no longer feels waxy. No dimensional deterioration.
			10 Apr.	Slight slumping. Diameter decreased 0.02 inches at supporting wall zone only.
2650	Zirconia:2	4 Nov.	25 Nov.	Severe multiple fracturing in intermediate temperature region inside flue wall. Withdrawn. Sample broken upon withdrawal and exposed portion lost.
2650	Sinteredα:2	9 Jan.	13 Jan.	Sample moved to hotter area at 2800°F after expiration of CVD SiC:2.
2650	AD 998:2	15 Jan.	22 Feb.	Slight slumping, longitudinal cracking, distortion of tip.
2600	Cordierite:2	3 Nov.	7 Nov.	Hot end slumped, then fell off, Withdrawn.
2600	KT:2	13 Jan.	2 Mar.	Slight edge deterioration. Slight decrease in circumference 0.5 inches inside support wall.

TABLE V (Continued)

MATERIAL SAMPLE OBSERVATIONS

FURNACE II (WITH GLASS)

Approx. Ave. Temp.	Material and Sample #	Date Inserted	Date Observed	Observations
2600	Si_2ON_2:1	~7 Nov.	25 Nov.	Surface covered with foam. Decrease in volume of exposed material. Sample subsequently broken in port. Exposed portion not extractable.
2550	NC 400:2	4 Nov.	21 Dec. 28 Feb.	Some glazing and foam. No dimensional deterioration. Some decrease in circumference at support wall zone.
2550	Vistal:2	4 Nov.	15 Nov.	No change observed. Moved to hotter spot at 2650°F.
2550	AD 85:2	4 Nov.	9 Nov.	Severe slumping. Withdrawn.
2550	AD 94:2	11 Nov.	21 Dec. 28 Feb.	No dimensional deterioration. Decrease in circumference at support wall zone.
2450	Si_3N_4:2	4 Nov.	20 Dec. 13 Jan.	Glazed with some foaming initially Slight surface deterioration. Sample broken easily upon extraction. Break and coloration very similar to sample Si_3N_4:1B.
2450	KT:2	4 Nov.	21 Dec.	Glazed. Slight decrease in diameter of exposed length. Move to hotter spot at 2600°F on 13 Jan.
2250	Si_3N_4:4	28 Nov.	21 Dec.	Major decrease in diameter over entire exposed length. Withdrawn.
2250	Sinteredα:3	13 Jan.	31 Jan.	Major disappearance of material over exposed length.
2250	KT:3	31 Jan.	10 Apr.	Major decrease in circumference of exposed material.
2100	AD 85:3	10 Jan.	10 Apr.	Glass apparently combining into the surface on lower side.
2100	Cordierite:5	10 Jan.	10 Apr.	Sample warped upward with some deterioration of lower side.
2100	Mullite:4	10 Jan.	10 Apr.	Slight disapperance of material from lower side.
2100	Vistal:4	10 Jan.	10 Apr.	Material coated with a glaze on lower side. Glaze easily breaks off.

tion of the material strength. When these impurities failed after long-term exposure at temperatures below the limit for pure Al_2O_3, the entire structure was weakened. This points out clearly the need for more accurate, long-term high temperature data on module materials.

Furnace performance tests in a combustion product atmosphere and a soda-lime glass furnace atmosphere have shown a number of materials to be unsuitable at any temperature above 2000°F and others to have operating limits below 2800°F.

Silicon nitride was dramatically affected in the glass exhaust at 2200°F with close to 0.1 inch reduction in sample radius in two months. Although KT and NC 430 were thought to be similar, visual differences are apparent in the respective samples with NC 430 appearing to have the faster deterioration. These differences may be due to a greater free silicon content in NC 430. These differences, however, may also be related to sample configuration differences. Vistal changed color slightly but does not appear to have changed character.

Following exposure in the flues, samples were tested at room temperature for flexure strength (Table VI). Sample size limited the test sample population to one for the exposed materials and two to three for the archive material. In some cases sample deterioration prevented testing entirely.

Furnace performance evaluations by Dr. Ivan Cutler of the University of Utah are reproduced in the Appendix.

Net results from all material evaluation and performance efforts, including those categorized as design calculations, cost projections, and fabrication studies, are discussed in the Conclusions section.

TABLE VI

STRENGTH CHANGE AFTER FURNACE EXPOSURE

Sample No.	Furnace	Temperature (°F)	Ratio of Exposed Material Strength to Archive Strength	Strength Loss or Gain (Percent)
Sintered Alpha 1	Combustion	2650	0.94	-6.
NC-400-1	Combustion	2550	1.03	+3.
NC-430-1	Combustion	2650	0.83	-17.
Kt-1	Combustion	2450	0.72	-28.
Kt-2	Glass	2450	0.66	-34.
Si_3N_4-1	Combustion	2450	0.23	-77.
Si_3N_4-3	Combustion	2250	0.47	-53.
Vistal-1	Combustion	2650	1.06	+6.
Vistal-2	Glass	2650	0.86	-14.
AD-85-1	Combustion	*	0.13	-87.
AD-94-1	Combustion	2550	0.72	-28.
AD-94-2	Glass	2550	0.47	-53.
AD-998-1	Combustion	2650	0.81	-19.
AD-998-2	Glass	2650	0.99	-1.
Mullite-1	Combustion	2650	1.26	+26.
Mullite-2	Glass	2650	0.03	-97.
Cordierite-3	Combustion	2300	0.13	-87.
Cordierite-4	Combustion	2100	0.40	-60.

*Broken hot end sat at bottom of flue. Temperature history uncertain but known to exceed 2650°F.

MODULE FABRICATION EVALUATION

High temperature ceramics can be fabricated in many complex shapes at a wide variety of costs. Some materials are readily formed in a number of ways depending upon the dimensions, shape, and required tolerances in the end product. Other materials can only be formed in a single or limited fashion. Fabricability and cost are complex functions of size, shape, tolerances, and material. These factors and their importance to the ceramic recuperator are discussed below.

Fabrication Methods

Ceramics are first formed as greenware and then fired in a kiln. In all cases, final truing can be done on the fired product to improve tolerance control, but this is costly and should be avoided. Numerous greenware forming methods are available but not all can be used on all ceramics.

a) Extrusion

This process involves forcing a semisolid mix of raw ceramic material and suitable binders through an extrusion die. The extruded greenware is then cut and fired.

b) Slip Casting

In this process a very fluid mix of water (usually), perhaps some binders, and raw ceramic material is poured into an absorbent mold (usually plaster of paris). As the water is drawn off, a semidry "cake" forms on the mold wall. Continued water withdrawal results in a thicker "cake". After the desired wall thickness is achieved, the excess fluid is poured out and the green "cake" is dried and removed from the mold for later firing. Wall thicknesses are very limited and porosity is usually high in this process.

c) Pressing

Variations exist in the method wherein a powder, perhaps with some binders, is compressed into a mold. This is sometimes done hot or cold, depending upon the material requirements. Pressure can be applied uniaxially with die rams or isostatically with gas or fluids and a flexible membrane surrounding a mandril. Very high densities and material strengths can be achieved this way. Porosities are usually less than 5 percent.

d) Injection Molding

This process needs a multipiece mold forming a complete negative of the desired product. A viscous fluid is then injected into the void volume under pressure. This results in a very uniform product as compared with slip casting and has a greater capability for complex shapes than many other methods. It is not possible, however, for all of the candidate materials.

e) Machining

Any method of forming a billet can be used to create a machinable preform. Final product is machined in the green state before firing.

f) Chemical Vapor Deposition

In this method, a thin film is chemically deposited from a vapor phase material at ceramicising temperatures. This method is used to coat a suitable preformed substrate, usually either a carbon form or another silicon carbide product, with pure silicon carbide. The result is a very dense, non-porous surface, highly resistant to attack.

g) Two-Piece Construction

It is possible to join or weld pieces together to form complex shapes. In the case of slip casting, some joints can be made in the green state using the same "slip" as the welding material. Some materials can be welded by reaction bonding during and even after the main firing process.

Kiln Limitations

Kilns are expensive to build and operate and prototype fabrication is limited to existing equipment. This is especially restrictive for materials that are batch fired.

Another factor is the kiln furniture. Because of shrinkage (which for some materials is as great as 20 percent), heat transfer considerations, and firing ductility, it is difficult to support an object in a kiln without introducing deformations during firing. The more complex the shape, the harder it is to support during firing and the greater the constraints on kiln utilization.

Tolerance Control and Fabricability Limits

Many potential sources of malformation exist between the initial forming and final product stages which can affect tolerances. End product truing is expensive and should be avoided wherever possible, but need not be completely excluded. The approach suggested from this work is to develop a design capable of working with the tolerances normally achieved in the fired product. This reduces the waste or reject ratio at all steps and results in minimum product cost. The tolerance control varies for each candidate but generally can be kept to one percent overall.

Suppliers were surveyed to define the fabricability limits of the identified candidate materials. Recent literature on fabricability of large alumina parts for fusion research is consistent with these results.[35]

In Table VII a "yes" indicates that a given condition or method was found to be in use in the industrial firms surveyed. A "no" indicates a material is not or cannot be fabricated by the noted technique. Most materials can be formed in more than one way and it is difficult to anticipate which method will be most economical in a high-production situation. However, it is readily concluded that reasonable sizes and shapes are technically achievable for all prime candidates.

X-rays of silicon nitride test rods revealed imperfect cores of either unreacted materials or inclusions of contaminants. In one case a sample failure is partly attributable to such an imperfection. In casting or forming large shapes, such variations are often difficult to control and fabricability with good quality control is difficult to assess until actual experience is acquired. This points out three needs for prototype test efforts:

a) Good quality control

b) Materials properties tests on samples taken from production-run parts

c) Careful examination of failed service parts to detect fabrication abnormalities.

TABLE VII

EVALUATION OF FABRICATION TECHNIQUES FOR RECUPERATOR MODULE MATERIALS

	Sintered Alpha	KT	NC 430	NC 400 with CVD coat	Vistal	AD 998	AD 94	Si_3N_4 Reaction Bonded	Cordierite
Forming methods									
Extrusion	No	No	No	No	No	No	Yes	No	Yes
Slip cast	2	No	Yes	Yes	No	Yes	No	Yes	Yes
Cold pressed	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Hot pressed	N/A	N/A	No	No	No[3]	No	No	No	No[3]
Injection molded	Yes	Yes[4]	No	No	No	No	No	Yes	No
2 piece construction	2	Yes	Yes	Yes	Limited	No	No	Yes	No
Machine green billet	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Chemical deposition	No	No	No	Yes	No	No	No	No	No
Present oven or kiln limits									
Length	1'	3'	25"	25"	6"	7"	19"	2'	2'
Mass	No limit	No limit	No limit	No limit	No limit	No limit	No limit	>11 lbs.	No limit
Potential oven or kiln limits									
Length	3	3'	3'	3'	6"	7"	19"	>2'	2'
Mass	No limit	No limit	No limit	No limit	No limit	No limit	No limit	>11 lbs.	No limit
Maximum wall thickness[5]									
Slip cast	0.25 to 0.5"	N/A	5/8"	5/8"	N/A	0.25"	N/A	1"	6
Pressed, hot or cold	No limit	1"	1"	1"	0.5"	N/A	No limit	1"	6
Injection molded	0.25"	1"	N/A	N/A	N/A	N/A	N/A	1"	6
Machined	No limit	1"	N/A	N/A	N/A	N/A	No limit	1"	6
Chemical deposition	N/A	N/A	N/A	.05" coat	N/A	N/A	N/A	N/A	N/A
Tolerance control without final machining	1%	<1%	1-2%	1-2%	1%	3%	1.5%	<0.1%	1%
Furnace shrinkage	13%	1%	0%	0%	>25%	~18%	~16-20%	0.1%	14%
Present delivery times for complex parts	3-6 mos.	10-12 wks.	12-14 wks.	16-18 wks.	12 wks.	12 wks.	12 wks.	8-12 wks.	12 wks.

1. Only considered for complex shape required by module design.
2. Present capability not proven but near term development expected.
3. Not by Coors.
4. Compression/transfer molding.
5. Limit may be due to firing or forming limits or both.
6. Thick wall not normally manufactured and some fabrication development would have to precede.

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MODULE DESIGN ANALYSES

This project is aimed at testing, improving, developing, and evaluating the specific Millcreek Glass Recuperator Design. Some design variations may have large effects on thermal stresses. This portion of the effort did not attempt to investigate the effects of design variations but rather concentrated on the basic design to prove the concept viability. It would certainly be wrong to assume that the configuration tested thus far is the only configuration to be considered as the final working design. Design variations may have significant effects upon fabrication, heat transfer, and economics but affect the technical viability very little.

Thermal Stress Analysis

A stress analysis was performed by imposing an operating thermal profile condition upon the recuperator. This results in differential thermally induced strains with resulting stresses which can be readily calculated. These stresses are related to the properties of elasticity (E) thermal expansion (σ) and temperature gradients (ΔT_p). The maximum stress is defined as follows:

$$S_{max} = C\ E\sigma\Delta T_p$$

Where C is a constant containing all geometric factors. This equation can now be used to obtain a materials engineering safety factor for a given set of thermal conditions by dividing this value by the materials strength. This also denotes a relative resistance to mechanical failure for this given constrained geometry when the safety factors are compared for different materials at similar conditions. Table VIII presents the relative engineering safety factors to thermal stress for the candidate materials for a given set of thermal conditions. For the most part, these values are encouraging since ΔT_p of 250°F is much larger than expected in a working ceramic recuperator.

TABLE VIII

RELATIVE THERMAL STRESS RESISTANCE

Material	E[1] 10^6 psi	σ[2] 10^{-6}/°F	ΔTp °F	S_0 ksi	Reported Strengths		Safety Factor Flexure/0.64 S_0
					Tensile ksi	Flexure ksi	
Sintered Alpha	59.4	1.33	250	7.3	-	50	10.8
NC 400	30	2.67	250	7.4	-	18	3.8
NC 430	>30	>2.67	250	>7.4	-	33	7.0
KT	53	2.73	250	13.6	-	23	2.6
Refrax 20	40[3]	2.5[3]	250	9.2[3]	-	6.2	1.1
Refrax 50	40[3]	2.5[3]	250	9.3[3]	-	6.6	1.1
CVD-SiC	34	2.5	250	7.8	-	>20[3]	4.1
Si_3N_4	24[3]	1.7[3]	250	3.8	19	35	14.4
Vistal	57	3.61	250	19	32	41	3.4
AD 85	32	2.94	250	8.7	22	43	7.7
AD 94	41	3.5	250	13.2	28	51	6.1
Mullite	22.5	2.06	250	4.3	-	27	9.8
Cordierite	>20[3]	1.22 @ 1800°F		>2.3 @ 1800°F	-	24	16.3

1. This is for room temperature because E at elevated temperatures decreases rapidly near the melt temperature. Future analysis would show E and S_0 as functions of T and ΔTp.

2. This is the average value near room temperature. This value tends to increase with temperature.

3. Estimated value.

Heat Transfer

Heat transfer assessments must be made primarily on the basis of calculational efforts. The heat transfer assessments, performed prior to the work reported herein, were updated on the basis of new and more reliable properties obtained from this work. Based upon the preliminary design concept and thermal calculations the following minimum criteria for economical operation were established:

a) Recovery of heat at 2300°F and a required effectiveness of 0.8 or greater limits ΔT to 500°F between hot and cold gases, where ΔT is the average temperature difference between the hot and cold gases across the recuperator.[36]

b) Turbulent air flow is probably required to maintain $\bar{h}_{ce}$ and $\bar{h}_{ca}$ in a range of about 17 BTU/hr-ft^2°F. The coefficients $\bar{h}_{ce}$ and $\bar{h}_{ca}$ are the net surface heat transfer coefficients for gas to solid for the exhaust and air stresses respectively. These coefficients include the effects of surface texture and areal multiplication because of fins and other heat transfer enhancement mechanisms (surface extenders).

c) For a thickness of 0.5 inch, the wall material unit thickness heat transfer coefficient, K/L, should be 22.5 BTU/hr-ft^2°F/ft or better.

d) A three-year payback is desirable as stipulated in the PON.

Results now available indicate these criteria will be met or exceeded. These are:

a) Heat transfer sections can be reduced in thickness from 0.5 inch to 0.25 inch or less (0.04 ft to 0.02 ft). Materials considered in this project are available in dense, fine grained forms and can be fabricated in thin dimensions. Further, designs can reduce structural load on the heat transfer wall by having the cylinder wall take the load. For some materials, 0.25 inch is a maximum wall thickness because of fabrication limitations. Heat conductivities are generally higher than 0.9 BTU/hr-ft^2°F/ft and range from about 3 BTU/hr-ft^2°F/ft for alumina at 2800°F to 30 BTU/hr-ft^2°F/ft for SiC at 1100°F. Thus, K/L might range from 144 to 1440 BTU/hr-ft^2°F/ft rather than the minimum of 22.5 used in earlier calculations.

b) With a higher value of thermal conductivity, K, for the module material, the necessary value of ΔT can either be reduced permitting recovery of heat at higher temperatures or the recuperator length can be reduced for lower cost.

c) Economy of scale is anticipated such that for a size increase of 89 percent fabrication cost may only increase by 30 percent.

These three factors are expected to improve heat transfer by a factor of three over the 3400 BTU/hr-ft^2 of interface cross section area determined in pretest calculations resulting in a projected heat transfer of 10,000 BTU/hr-ft^{2}IA. (IA refers to the interface area and not to the gas-solid surface area.)

MODULE DESIGN TESTS

Cold Flow Tests and Analysis

The cold flow tests required a model which duplicated a large enough portion of the flow channels to provide for accurate pressure drop measurements. Pressures were measured well away from each end to avoid the large end effects that occur at the entrance and exit of the flow channel. High density polyethylene and lucite were used for the machined model. These materials machine to very smooth and low-friction surfaces. To obtain a rough surface for comparison of texture effects, a monolayer of sand was formed on the polyethylene surface.

A testing assembly (Figure 6) in the College of Engineering at the University of Utah was used to perform the cold flow tests. The air supply consisted of a motor connected through a continuously variable transmission to an impeller blower. The exhaust from this blower passed through a pressure orifice chamber and then through the test configuration. The mass flow of air was measured using the pressure difference, ΔP_o, across the orifice in the pressure orifice chamber using the standard relationship developed by the University for this equipment. A thermal anemometer was used to measure peak air velocity. This velocity measurement verified the square root relationship between velocity and pressure drop so that reliable extrapolations could be made into the regions above the flow limits of the blower and also at low flow rates where ΔP_o in the orifice chamber was too small to measure accurately.

Configurations tested included several variations in size and aspect ratios. One configuration was tested smooth and roughened with a monolayer of sand on the interface surface. Results of the flow tests are shown in Figure 7 in terms of mass flow rate versus internal recuperator pressure drop.

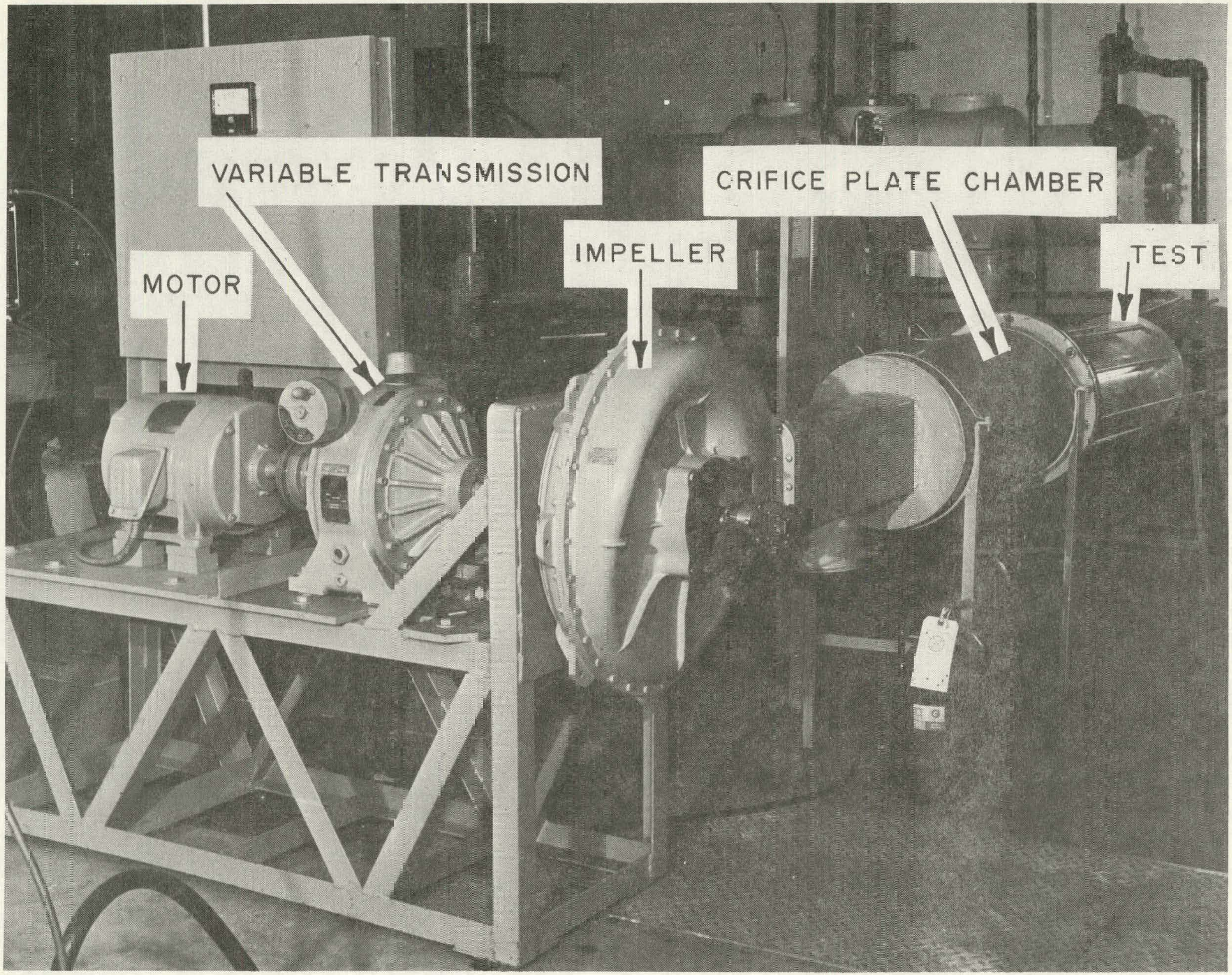

Figure 6. Cold flow test assembly.

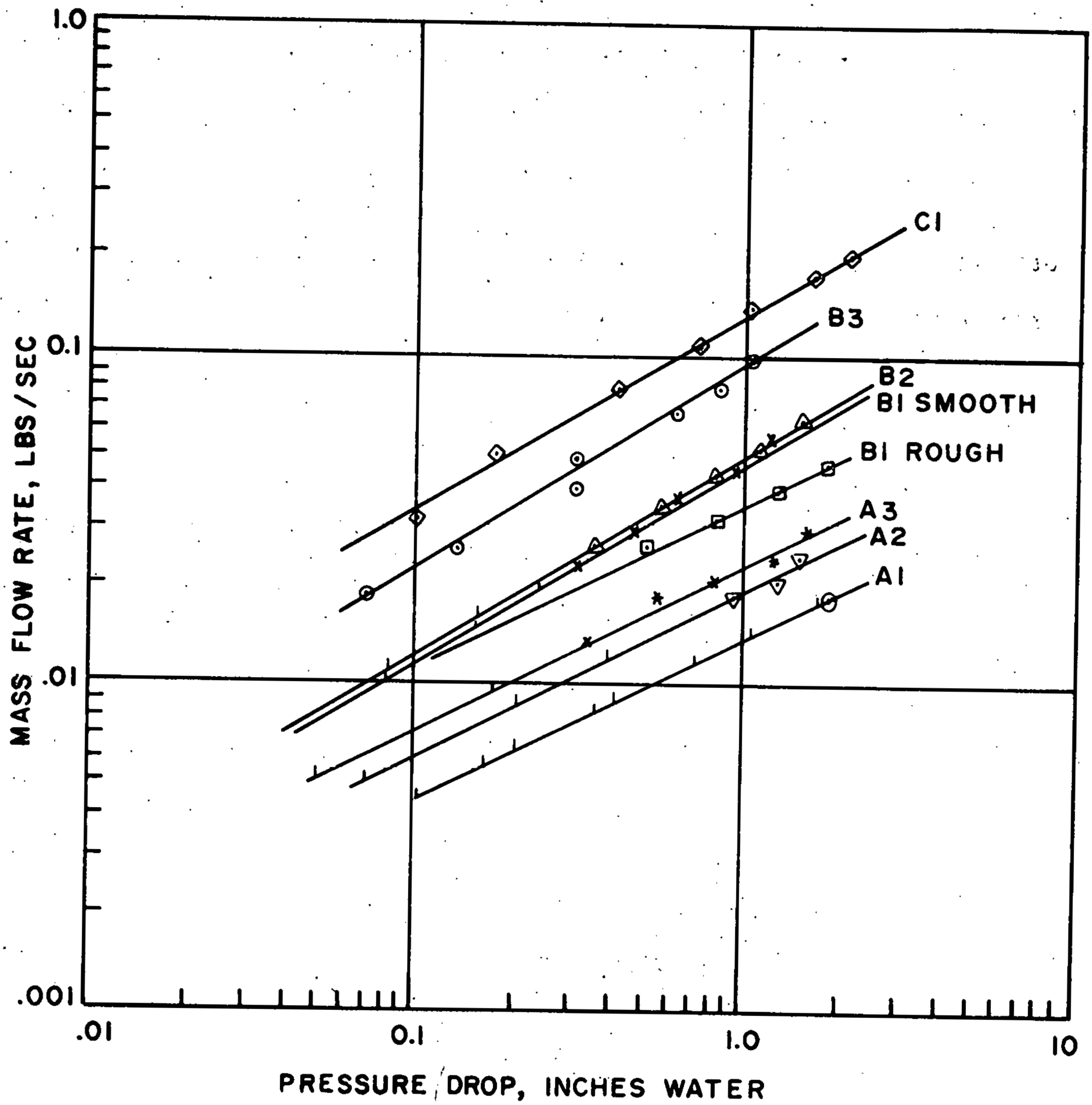

Figure 7. Cold flow test data showing air mass flow rate as a function of pressure drop in the recuperator. Data below 0.01 lbs per second is inferred from peak velocity data and is indicated by tick marks on the curves.

The mass flow rates at a given ΔP_R (Figure 7) can be used to predict mass flow rates for untested geometries. Figure 8 shows the data at a ΔP_R of 1 inch of water for all configurations with an aspect ratio of 1.0. Predicted mass flow values for 8-inch and 12-inch units at a ΔP_R of 1.0 inch of water and an aspect ratio of 1.0 are shown by the dashed symbols of Figure 8.

Table IX is a summary of the data and results of an analysis to determine optimum aspect ratio and size for a furnace rated at about 1.6 million BTU per hour unrecuperated with an effective pressure drop of 1 inch of water in the recuperator. A key to this analysis is to recognize that all configurations must result in the same net pressure drop and total mass flow as dictated by the given furnace operation. Local gas contains 875 BTU/SCF. At a 20 percent excess of air, each ft^3 of gas results in 13 ft^3 of exhaust at standard conditions or about 0.54 lbs of exhaust per second. From the data and these criteria, and recuperator core length (ℓ), the number of tubes or channels (Nr), and the total channel length (L), are defined. Using these data and geometry considerations, a normalized gas transit time (T) and a total heat exchange surface area (S_H) are calculated.

The product, T S_H, is the heat recovery factor and is a measure of the heat transfer capability for that size and configuration. The higher the value, the more effective and efficient the configuration.

Relative cost effectiveness requires normalizing to manufacture cost. Most suppliers report projected fabrication costs on a per pound basis. V_m is the volume of ceramic material needed in each case assuming constant heat exchange wall thickness but a cylinder wall thickness which varies linearly with tube radius. $V_m/T \cdot S_H$ provides a measure of the relative cost effectiveness per pound or RCEP, with a lower number indicating a lower cost per heat recovery unit for a greater cost effectiveness. Figure 9 shows a plot of

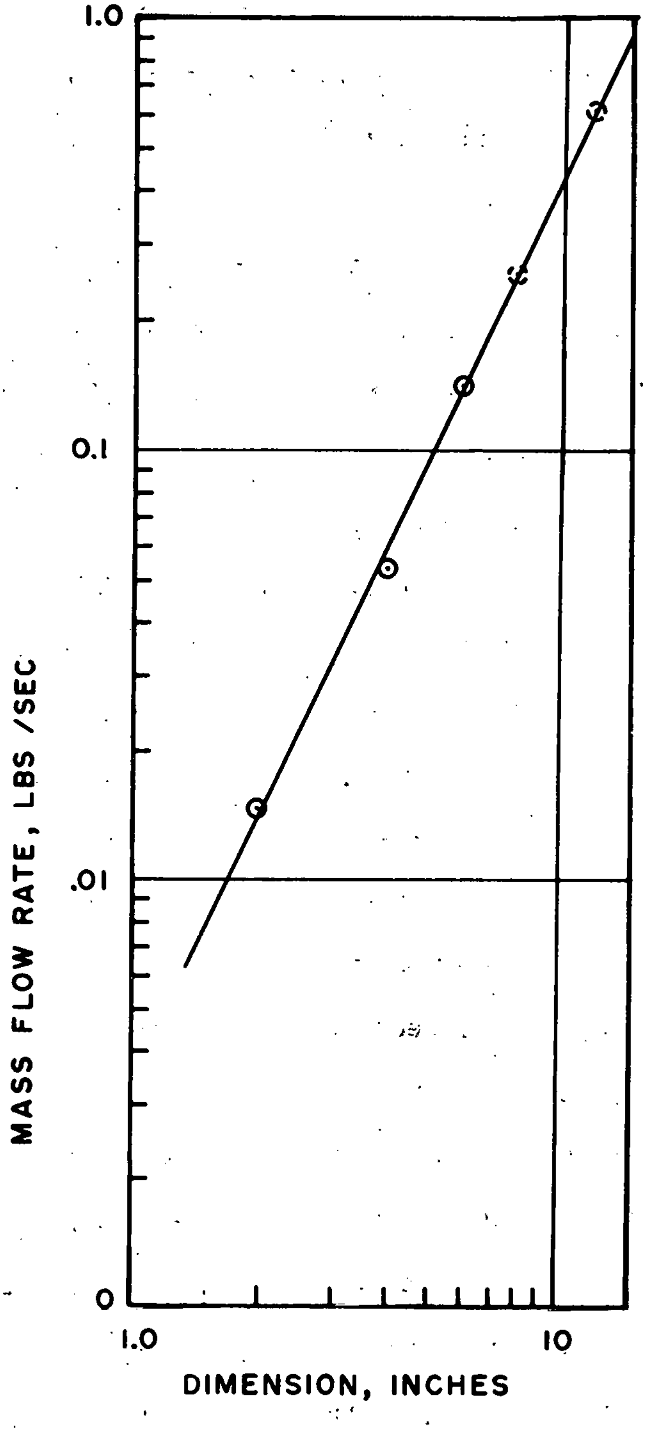

Figure 8 . Mass flow rate for various dimensions and aspect ratios of 1.0. Values at 8 inches and 12 inches are inferred.

TABLE IX

SUMMARY OF COLD FLOW TEST ANALYSES

Parameter	Description	Units	A1	A2	A3	B1S	B1R	B2	B3	C1	D1	E1
V_o	Test Unit void volume	ft^3	0.0089	0.0154	0.0231	0.0626	0.0601	0.0751	0.1101	0.2684	0.6593	2.2715
s_H	Test Unit heat transfer surface	ft^2	0.2864	0.2740	0.3164	1.0290	1.0255	1.0504	1.1091	2.4131	4.3254	9.8642
-	Length per ft^2 of s_H	ft	3.5	7.3	9.4	1.46	1.46	1.9	2.7	1.24	0.92	0.608
-	Volume per ft^2 of s_H	ft^3	0.0311	0.0562	0.0724	0.0609	0.0585	0.0713	0.0991	0.1109	0.1524	0.230
ℓ	Length of ΔP Test section	ft	0.67	1.33	2.0	1.0	1.0	1.33	2.0	2.0	2.67	4.0
$\dot{M}$	Mass flow rate at $\Delta P_R = 1.0$ inch water	lbs/sec	0.0142	0.0190	0.0234	0.048	0.036	0.051	0.099	0.137	0.25	0.58
Nr	Number of ΔP Test Units to supply 0.54 lbs/sec	---	38.4	28.7	23.2	11.4	15.0	10.7	5.5	4.0	2.21	0.9
L	Total core length of recuperator = $\ell \cdot Nr$	ft	25.7	38.3	46.5	11.4	15.0	14.2	11.0	7.9	5.9	3.76
S_H	Total heat exchange surface = $L \cdot s_H / 1.5\ell$	ft^2	7.3	5.3	5.0	7.8	10.3	7.5	4.1	6.4	6.3	6.2
V_T	Total recuperator void volume	ft^3	0.23	0.29	0.36	0.48	0.60	0.53	0.40	0.71	0.97	1.42
T	Exposure time = $\rho V_T / \dot{M} Nr$ (at STP conditions)	sec	0.056	0.070	0.087	0.116	0.146	0.129	0.097	0.172	0.235	0.344
$T \cdot S_H$	Heat recovery factor	sec ft^2	0.41	0.37	0.44	0.90	1.50	0.97	0.40	1.10	1.48	2.13
V_m	Volume of scaled module material	ft^3	0.49	0.45	0.54	0.28	0.43	0.48	0.39	0.53	0.66	0.89
CEP	Cost per heat recovery unit ($V_m / T \cdot S_H$)	---	1.20	1.22	1.23	0.31	0.29	0.49	0.98	0.48	0.45	0.42

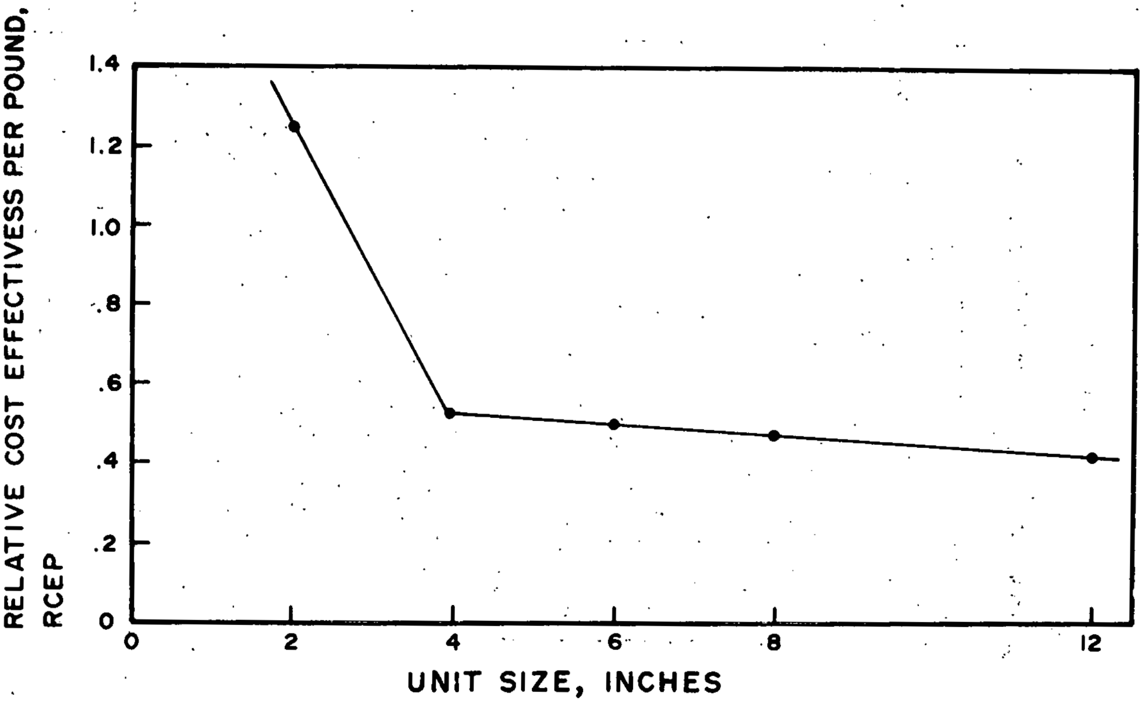

Figure 9. Relative cost effectiveness per pound. RCEP shown as a function of diameter for an aspect ratio of 1.0.

RCEP as a function of diameter.

One model was run with both a smooth and a rough textured surface. Values of RCEP indicate a rough surface improves the cost effectiveness slightly. For considerations related to fouling, an aspect ratio of less than one is probably not desirable unless dimensions greater than 8 inches are used because the height of the flow chamber becomes too small and subject to plugging. The A units data indicates no advantage to a small aspect ratio; however, the B units data indicates a clear advantage of two times or greater for the smaller aspect ratios tested. There is no clear explanation for these differences at the two diameters. The shape of the plot in Figure 9 indicates that an optimum unit size is at least 4 inches.

Final optimum geometries will also depend upon heat transfer effects not considered in this analysis and cost factors currently unavailable. Such considerations may favor the smaller dimensions because of fabrication limitations. Hence, a prototype recuperator should be sized between 5 and 10 inches.

Test Module Fabrication

Testing of test modules under operating conditions verifies the thermal stress calculations and material properties evaluations. The determination of operating limits for a selected test material also normalizes the thermal stress calculations and provides an absolute as well as relative means of evaluating all candidates. As it turned out, however, the test modules proved so durable that they could not be broken by the laboratory attainable thermal conditions.

From the proven primary candidate materials, a KT from Carborundum was selected for the test modules on the basis of performance, cost, and deliverability. Six test units were fabricated at the factory from a pattern hand-fabricated at Terra Tek and Millcreek Glass. A rubber mold was made from the pattern and used to cast the greenware. The flexibility of this mold permitted tolerance deviations which, although not detrimental to this test, would be unacceptable for a working recuperator. A more rigid mold would overcome this difficulty. Several surface blemishes also occurred on some test modules during fabrication. These were not aggravated during the tests and post-test analysis of modules at the factory showed them to be superficial. Avoidance of such blemishes for production units is assured by the supplier.[37]

The test module design matched as much as possible the design recommended for the projected prototype tests.

Thermal Stress Tests

Testing of units was accomplished by placing them into the flue of a day tank glass furnace. An air injection system established a cold side temperature which could be accurately controlled and operating conditions covering a wide range of temperature differentials could be simulated.

Furnace limits required 30-hour heat-up and 30-hour cool-down periods during each test run. After heat-up was achieved, air injection to the cold side was begun and increased until the desired gas ΔT was reached. These conditions were maintained for an hour or more to insure that a stable condition had been reached and that test temperatures simulated long run conditions. The test units were then cooled with the furnace and inspected for deterioration and other effects.

The test configuration resulted in a large ΔT_B at the bottom or exhaust entry zone. Because of heat transfer under coflow conditions, the ΔT_T at the top or exhaust exit zone was about one-third or less of the ΔT_B. This provided opportunity to test unit behavior over a wide range of ΔT's in a single test run. It was fully expected that some deterioration might occur at the greatest attainable ΔT_B and a definite limit on ΔT could be defined. This was not the case, however. In the final test, a ΔT_B of 1200°F and a ΔT_T of 300°F were achieved with no evident deterioration of any kind related to thermal stress.

During this test, gas entrance hot and cold temperatures into the test stack were 2400°F and 1200°F respectively. Exit temperatures were 1950°F and 1650°F. Average ΔT then was about 750°F.

Thermal Shock Tests

The test units were subjected to a rapid thermal shock to determine the shock stability of the module design. A unit at room temperature was inserted into a furnace (the "glory hole") which was at 2000°F. The unit and furnace equilibrated at about 2300°F in 10 minutes. The unit was then rapidly removed from the furnace and placed on the floor at room temperature. Although some audible sounds were emitted upon cooling, no evidence of any failure was detected. The unit was examined visually and with a dye penetrant, again without evidence of any stress or shock failure.

JOINT DESIGN AND MATERIAL EVALUATION

A modular design permits easier cleaning and replacement, and thus significantly reduces the lifetime cost. Furthermore, a nonpermanent joint simplifies fabrication and construction with attendant savings. Such joints, however, need to be made reasonably airtight to reduce gas loss and interpath leakage. Yet they must endure high temperature exhaust conditions with a minimum of deterioration and "welding" to modules. Three potential joint types were initially identified.

a) Mechanical

A mechanical joint would require matching and perhaps intermeshing surfaces. Fabrication and tolerance control would be extremely expensive for a pure mechanical joint with the necessary leakage requirements.

b) Nonreactive Seals

Material of a flexible or pliable nature can adjust to minor tolerance and expansion variations which are inherent in module fabrication or which might result from furnace exposure, making the joint area less expensive to construct. Temperature requirements limit the selection to ceramic fibers and cloths.

c) Reactive Seals

This would include materials which would bond modules together in a more or less permanent joint. Two types of reactive joints suggested by others are a viscous glass joint[38] (which may result naturally after use in some parts of the recuperator) and a brazing or welding of modules as performed by Norton on SiC[39] (this may also occur naturally for some candidate materials).

The concept of a permanent seal between modules is somewhat contrary to the idea of modularity and replacement ease. This was pursued briefly for several materials, however, because it was not initially clear that airtight permanent joints would not be required.

From the attainable cost and ease of fabrication standpoints, a combination mechanical and nonreactive seal is recommended for future efforts. Four materials were identified as potential joint seal materials: Kaowool, Saffil, 3-M Fiber and Fiberfrax.

Joint seal materials were tested in an operational configuration because of the need to evaluate the possible interaction of the seal and module material at temperature. Seal materials were fabricated into gaskets and inserted between test modules during the thermal stability tests of those modules. These tests are discussed in more detail under the Thermal Stress Tests section. Materials experienced thermal conditions, compressive loads, and exhaust atmospheres. Exposed materials were examined visually and manually for evidences of material degradation, chemical alteration, interaction with modules and condensate capture. Tests were done on Kaowool, 3-M fibers, and Fiberfrax.

Performance tests on all joint seal materials indicate their acceptability for use in the prototype model. Because known temperature limits were not exceeded in the tests, published data is the basis for defining the useful temperature ranges noted in the Conclusions section below.

ECONOMICS

The economics of this recuperator design is determined by efficiency or heat recovery, life expectancy, operating and maintenance costs, and, of course, initial fabrication and installation cost.

Heat Recovery and Value

A principal factor is the net heat transfer rate potentially attainable at the expected operating conditions of 10,000 BTU/hr-ft^{2}IA. (IA is the interface cross section area as opposed to the extended surface area.) A survey of local and national fuel rates indicates commercial natural gas to currently average about $2.06 per million BTU. Thus, at today's gas rates a module stack of one square foot IA can be expected to recover about $180.00 in energy cost per year.

Fabrication and Installation

A cost analysis was performed on several prime candidates (Table X). The costs and module weights are conservative. This assures realisticly achievable values and, if manufacturers' cost projections are fully realized, the economics will be even better. The analysis was done for a 9-inch unit. A 9-inch recuperator unit, at a cost of $10.00 per pound, would cost about $180.00 to $240.00 per ft^2 of heat transfer area. A 12-inch unit cost is projected at $150.00 per square foot of heat transfer area. [40]

Installation is relatively simple and the cost of joint seal material is minor. Joint seals and lift mechanism costs are estimated to be 20 percent of the recuperator module costs. Fiber and brick insulation materials and installation are estimated to be 40 percent of the cost of the modules based

TABLE X

COST ANALYSIS

	Si_3N_4	Vistal	AD 998	KT/Sintered Alpha	Cordierite
Unit Weight, lbs.	3.55	5.22	5.20	4.10	3.13
Material Density (relative)	2.73	3.99	3.98	3.15	2.43
Est. Raw Material Cost, $/lb	2 to 4	30	2 to 4	2.5 to 5	1 to 2
Test Units (6)					
Est. delivered cost, $	10,000	NA	1200 to 2400	6000 to 10,000	1500 to 3000
Cost/Units, $	1667	NA	200 to 400	1000 to 1667	250 to 500
Cost/lb. $/lb.	470	NA	50 to 100	320 to 530	100 to 200
Projected Costs					
Raw material, $/lb.	1	30	1 to 2	2 to 4	2 to 4
Forming, $/lb	3 to 6	5	1 to 2	2 to 4	2 to 4
Firing, $/lb.	6 to 12	10	.5 to 1	2 to 4	1 to 2
Reject rate	1.3:1	2:1	2:1	2:1	2:1
Cost/lb.	13 to 25	90	5 to 10	12 to 24	10 to 20
Fabrication Methods	Slip cast, reaction bonded in N_2 atmosphere.	Disc cold pressed and machined in green state or Iso-pressed, then fired in normal atmosphere	Slip cast and fired in normal atmosphere.	May be dry pressed, injection molded, slip cast or possibly extruded then fired.	Dry pressed and machined in green state, then fired.

on engineering estimates. Net fabrication and installation costs of industrial size modules becomes $150.00 x 1.6 or $240.00 per square foot of heat transfer area.

Operation, Maintenance, and Life Expectancy

The specific design promises potential for ease of removal and cleaning. Based on fouling seen in this work, it is anticipated that less than 25 percent of the units would have to be replaced over a 3-year payback period. Operation, maintenance, and cleaning are estimated to be less than 15 percent per year or 45 percent of the capital cost for 3 years. Thus, it is estimated these costs will add no more than about 70 percent of the fabrication and installation cost to the overall recuperator cost, or a total of about $170.00 per square foot IA.

Economic Payback

The sum of the recuperator costs for a 3-year period is $410.00. The return is $180.00 per year or $410.00 in 2.3 years. Thus the payback is about 2.3 years. (Because this payback period depends upon the cost of money and other factors not controlled in this study, payback could vary between 2 and 3 years.)

In making these calculations, three other factors should be recognized that affect the economics. First, it is recognized that in the present inflationary state energy costs may inflate faster than product costs which would enhance the economics. Second, it is assumed that the metal recuperator section at the lower temperature range would also be economically self-sustaining. Third, the reduced cost of pollution control equipment resulting from lower fuel consumption will further improve the economic gain.

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CONCLUSIONS

Summary of Results and Material Evaluations

The accomplishments to date have exceeded early expectations in that more potential recuperator materials have been identified than thought possible and module behavior under operating conditions has exceeded minimum stability requirements. Specificly, the results are as follows:

- A recuperator module of prototype design has been fabricated and tested at and above expected thermal stress conditions with complete success. Thermal stress calculations indicate safety factors of up to 16 for some candidates.
- Fabrication of complex parts made of high temperature ceramics is indicated as possible for all good candidate materials. Fabrication of six modules made from KT silicon carbide was achieved. On the basis of test module fabrication, near-zero reject rates can be expected for at least some candidate materials.
- Materials have been identified which can meet the requirements for operation in a glass furnace exhaust as verified by tests in the required environments. These materials are cordierite, mullite, and alumina below 2300°F and high purity alumina and four types of silicon carbide above 2300°F.
- Economic assessments indicate a payback period of 2.3 years in an industrial installation exclusive of tooling and process development costs.
- Expected tolerance control and proven joint material behavior provide confidence in achieving unit joint behavior which insures low leakage.

- No limitation or problem has been identified which would prohibit successful demonstration and commercialization of this basic design for a high temperature range recuperator.

These results clearly indicate the advantages of proceeding with a prototype test of this design which would include operation and testing of a complete recuperator on a day tank size glass furnace.

Table XI presents the summary conclusions for all material evaluations based upon material properties, material performance, cost, fabricability, calculated heat transfer, and availability.

Recommended Prototype Design

On the basis of efforts discussed herein, a recommended design for prototype test modules and recuperator configuration has been established.

All tests except one cold flow test were conducted on test pieces with flat, smooth heat transfer surfaces so that heat capture area matched interface cross section area. As discussed above, surface texturing, finning, and other mechanisms for improving gas to solid heat transfer rates may be included in future test and working designs now that the basic module stability has been established to study the time dependent benefits for design optimization efforts. Table XII shows the materials recommended for use in the prototype test. These final recommendations are consistent with other contemporary results.[41]

Details of the recommended test plan for prototype tests accomplished in this effort have been prepared and forwarded to the sponsor under separate cover.

TABLE XI

MATERIAL EVALUATION SUMMARY

MATERIAL			PRINCIPAL CHARACTERISTICS	EVALUATION RESULTS
NO.	NAME	SUPPLIER		
1.	Sintered Alpha-SiC	Carborundum	Fully dense with good thermal and mechanical properties. Still in development stage.	Fracture toughness as tested less than manufacturer specification but still adequate. Furnace performance at high temperature excellent. Some corrosion and material loss evident at about 2250°F probably correlates to glass condensation temperature. No significant change in strength after exposure. Can be fabricated in required shape. Prime candidate for high temperature prototype module.
2.	NC 400-SiC	Norton	Porous and permeable to gas. Good mechanical and thermal properties and readily fabricated.	Furnace behavior good except at about 2250°F where corrosion is evident. No change in mechanical strength evident after exposure. Material could serve as substrate for CVD-SiC but this would complicate fabrication and performance is unproven. Secondary candidate for high temperature prototype module.
3.	NC 430-SiC	Norton	Dense with some free silicon in matrix. Good mechanical and thermal properties and readily fabricated.	Furnace behavior good at high temperature. Some corrosion evident at about 2250°F from condensing glass. A 10% to 20% strength loss apparent after exposure probably due to loss of free silicon from matrix. This reduction not significant for stress requirements though. Prime candidate for high temperature prototype module.
	Refel	Refel	Very similar to NC 430. Not tested.	
4.	KT and Super KT SiC	Carborundum	Dense with good thermal and mechanical properties. Some free silicon. Readily fabricated.	Behavior in furnace similar to other SiC products. Corrosion more noticeable in glass furnace than in combustion furnace but still low except at about 2250°F. Strength after exposure shows a 30% reduction probably due to loss of free silicon; however, final strength well above minimum requirements. Prime candidate for high temperature prototype module.
5.	Refrax 20 SiC bonded with Si_3N_4 CN 130 SiC bonded with Si_3N_4	Carborundum Norton	Some porosity and large grain sizes. Low strengths characteristic of all low cost refractory grade materials.	Low strengths prevented furnace sample preparation and indicate likely inadequate use performance. Material rejected on a strength basis for prototype module.
6.	Refrax 50 SiC bonded with Si_2ON_2 NC 163-SiC bonded with Si_2ON_2	Carborundum Norton	Charateristics similar to Refrax 20 and CN 130 respectively.	Low strength prevented furnace sample preparation and indicate probable inadequate performance in use. Material rejected on a strength basis for prototype module.
7-8	CVD-SiC	M.T.C.	A fully dense, chemically vapor deposited skin on carbon or NC 400 substrate.	Furnace behavoir shows no advantage over other silicon carbides. Cost improvements not forseeable. Secondary candidate for high temperature prototype module.
9.	NC 127-SiC bonded with Fe_2O_3	Norton	Similar to other coarse grain silicon carbide refractories.	Iron content makes its usefulness doubtful because of the coloring effect of iron on glass. Material rejected as candidate for prototype module.
10.	NC 132 Hot Pressed Si_3N_4	Norton	High density with good thermal and mechanical properties but expensive to fabricate. Not tested in favor of candidate 11.	Material rejected as candidate for prototype module. Cost reductions could not be shown.
11.	RBN-101 Si_3N_4 reaction bonded	Garrett AiResearch	High density with good thermal and mechanical properties. Less expensive than hot pressed form.	No corrosion except at about 2250°F apparently due to condensing glass. Corrosion at 2250°F was excessive. Imbrittlement with loss of strength occurred in shaft away from hot end at about 2000°F. Hot exposed end suffered 50% to 80% loss of strength. Material rejected on basis of strength failure as candidate for prototype module.

TABLE XI (Continued)

MATERIAL EVALUATION SUMMARY

MATERIAL			PRINCIPAL CHARACTERISTICS	EVALUATION RESULTS
NO.	NAME	SUPPLIER		
12.	Sioxyn, Si_2ON_2	Norton	Fine grained high density material also used as a binder for SiC.	Low strength hampered furnace sample preparation and indicated probable poor use performance. Sample broke during early furnace tests. Material loss noted prior to break at 2600°F. Material rejected as candidate for prototype module.
13.	Sialon	Univ. of Utah	Potential inexpensive material with good properties. Not available in large forms. Not tested.	Material not available for prototype module.
14.	Vistal Al_2O_3	Coors	Polycrystalline Alumina with full density, good mechanical properties and fair to good thermal properties. Fabrication in large shapes not proven. Transparant to IR to some extent.	Excellent behavior in furnace at all temperatures and no corrosion evident at any temperature. No loss of strength after exposure. Although, an apparent longitudinal crack in one sample showed up, it did not result in any failure nor did it affect the flexure strength parallel to the crack plane. Secondary candidate at all temperatures for prototype module.
15.	AD 94 Al_2O_3	Coors	94% pure Alumina with good mechanical and fair to good thermal properties.	Strength loss at less than manufacturers specified maximum use temperature. Slumped at 2650°F but held up at 2550°F. Strength loss of 30% to 50% following extended thermal exposure at 2550°F. Secondary candidate for medium temperature range for Phase II.
15a.	Ad 998 Al_2O_3	Coors	A higher purity alumina with characteristics similar to AD 94 and selected for testing when AD 94 showed less temperature survival than expected.	Behavior essentially the same as AD 94 in the furnace but less loss of strength after exposure. A 20% post exposure strength loss occurred in the combustion furnace but no strength change was evident in the glass furnace sample. However, distortion occurred where glass condensed on the sample. Primary candidate for medium temperature for prototype module.
16.	AD 85 Al_2O_3	Coors	An 85% purity Alumina with fair mechanical and thermal properties.	Material slumped severely at 2550°F apparently due to behavior of the 15 percent impurity. Material rejected in favor of AD 998 and AD 94 for prototype module.
17.	AH 1948 Al_2O_3	Norton	Low density Alumina of coarse grain materials. Low strength charateristics. Not tested.	
18.	Mullite	Coors, McDaniel	Complex material of full density. Fair to good mechanical strength. Poor to fair thermal properties. Inexpensive and easily fabricated.	Easily bent at 2650°F. Crystalline development and total loss of strength with fracturing in glass atmosphere. Post exposure strengh of combustion furnace sample increased 25%. Glass furnace samples had no strength post exposure. Behavior satisfactory at lower temperatures. Primary candidate for medium temperature for prototype module.
19.	Mulnorite	Norton	Refractory grade mullite of low density. Porosity too high. Not tested.	
20.	Mullite R	Coors		
21.	Fuzed Cast AZS	Corning	Dense material of known durability in glass contact. Expensive to form. Not tested due to expense.	Recent developments may provide a cheaper way to fabricate complex shapes. Should be reconsidered as possible candidate for prototype module.

TABLE XI (Continued)

MATERIAL EVALUATION SUMMARY

MATERIAL			PRINCIPAL CHARACTERISTICS	EVALUATION RESULTS
NO.	NAME	SUPPLIER		
22.	Cordierite CD-1	Coors	Inexpensive and easily fabricated. Very low thermal expansion and stress. Maximum temperature limited to about 2300°F.	Post exposure strength dropped by 87% above 2300°F and dropped 60% above 2100°F. Condiserable deformation and blistering above 2100°F. Secondary candidate for medimum temperature for prototype module.
23.	MgO		A successful checker material but not availabe in dense complex forms. Not tested.	
24.	$MgO \cdot Cr_2O_3$		A successful checker material but not available in dense complex forms. Not tested.	
25.	$MgO \cdot Al_2O_3$	Coors	High resistance to alkalis but otherwise no real advantage over Al_2O_3. Testing withheld in favor of Alumina.	Future evaluation recommended.
26.	ZrO Zirconia	Coors	Very good mechanical properties but low thermal conductivity. Known phase transition occurs with time at singular temperature.	Behavior good at high temperature but failure of sample always occurred at point of transition temperature. Nature and extent of effects indicates a rather broad temperature range for this effect over extended exposure times making it an unreliable material. Material rejected for prototype module.
27.	CaO bonded Castable refractory		Very inexpensive.	Difficulty in forming consistent samples and apparent low strengths resulted in materials rejection for testing. Materials rejected for prototype module.
28.	Phosphate bonded Castable refractory		Very inexpensive.	
29.	BeO Beryllia		Very good conductivity with good strength.	Material rejected for Phase I testing due to potential health hazards.
30.	Platinum and other high temperature metals.		Expensive. Good material properties. Fabrication usually difficult.	Cost considerations cause rejection of these materials for Phase I testing.
31.	BN Boron Nitride		Excellent material properties but expense appears too high.	Not selected for testing in Phase I but should be reconsidered if future production costs can be decreased.
32.	3M Fibers Saffile	3M Saffile	Available as loose fibers and woven cloth and tape. Good high temperature capability.	Tested to 2500°F or greater. Cloth showed a tendency to become more rigid during exposure but this is not deleterious to use. No other effects apparent. Prime candidate for high temperature joint seal.
33.	Fiberfrax	Carborundum	Available in wide variety of forms including preformed gasket. Manufacturer limits use to about 2300°F.	Tested to about 2500°F with no apparent effects. Prime candidate for medium temperature joint seal.
34.	Kaowool		Available in loose pack and in several temperature grades.	Tested to about 2500°F. Some charring noticed on exposed areas after 72 hours. Secondary candidate for joint seal - medium temperature.

TABLE XII

MATERIAL CANDIDATES FOR HIGH TEMPERATURE RECUPERATOR USE

MODULE MATERIALS		
<1700°F	1700°F-2300°F	>2300°F
Stainless, steel, 309 446	Cordierite Mullite Low purity Alumina	Vistal or High Purity Alumina Sintered Alpha KT CVD NC 430

JOINT SEAL MATERIALS		
<1700°F	1700°F-2300°F	>2300°F
Fiberfrax Kaowool	Fiberfrax Kaowool	3-M Ceramic Fiber (62% Alumina, 14% Boria, 24% Silica) Saffile

ACKNOWLEDGEMENTS

Successful completion of this work required the skill and effort of many people and their contribution is gratefully acknowledged. The following deserve special recognition. Phillip Hastings and Stephen Rosenblatt of Millcreek Glass performed furnace design and construction and conducted material exposure tests. Lynn Barker of Terra Tek conducted fracture toughness tests. Dr. Weldon Wilkening of Terra Tek designed the special test frame apparatus and conducted the flexure strength tests on exposed material samples.

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APPENDIX

Ceramic Evaluation

Dr. Ivan Cutler, University of Utah ceramicist and project consultant, examined material samples after furnace exposure and provided the following evaluation.

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Report from Dr. Ivan B. Cutler to Terra Tek

April 1, 1978

EVALUATION OF SAMPLES SUBJECTED TO COMBUSTION, AND GLASS FURNACE ATMOSPHERES

TERRA TEK, INC., Salt Lake City, Utah

Mechanism of Failure of Refractory Materials

In the refractory testing for use in glass tank recuperators, the mechanisms of failure are important for our consideration. There are really three mechanisms of failure that can be examined with the materials supplied to me. One mechanism of failure concerns oxidation. Non-oxide materials such as silicon carbide and silicon nitride are useful at high temperatures because they develop a silicon dioxide protective layer. At low temperatures this protective layer is a glass; at high temperatures above about 1200°C or 2100°F, the glass film will crystallize to cristobalite. It still remains protective, however, in spite of its crystallization. Because the rate of oxidation is controlled by the silica film on the outside of these non-oxide materials, anything that would change the silica film will change the rate of oxidation. The rate of oxidation has been shown to increase somewhat in a water vapor atmosphere for example, because the water actually changes the viscosity of this glass film on the outside of the silicon carbide or silicon nitride. Other materials that change viscosity of silica glass also accelerate the rate of oxidation. In the presence of vapors of alkalies that are present in glass tanks, the rate of oxidation is predicted to increase and it certainly does. In these tests the specimens clearly show that the rate of oxidation is accelerated by the presence of vapor species that come out of the glass tank.

These accumulate on the surface, decrease the viscosity of the glass, accelerate oxidation, and hence accelerate corrosion. It should be noted, however, that in spite of the corrosion all of the silicon carbide and silicon nitride samples showed very good creep resistance. That is, they did not deform under their own weight when subject to the combustion atmosphere or the glass atmosphere. This means that they did not suffer from penetration of any of these alkalies in between the crystals or grains of silicon carbide or silicon nitride. Likewise, they are very creep resistant on their own accord, the property that is well recognized in the ceramic industry.

Among the oxides which would include aluminum oxide, mullite, and cordierite, the mode of or mechanism of failure is one of creep which may be accelerated a great deal by penetration of the glass or vapor species in between the crystals in the oxide. Creep is easily noted by deformation under the weight of the specimen itself. Creep type failures are also easily observed by high temperature strength testing.

For the purposes of the tests that have been performed to date, creep failure is easily observed in the deformation of samples in the hot zone both in combustion atmospheres as well as in glass atmospheres. Creep that is accelerated in the presence of a glass tank atmosphere is ordinarily due to the penetration of the glass constituents as they condense out on the refractory and penetrate along grain boundaries into the oxide type materials. This provides a low viscosity glassy material at grain boundaries that easily accelerates any deformation due to the action of gravity on the specimen involved.

Observations of Samples Submitted for Examination

On the silicon carbide and silicon nitride samples, it was readily apparent that a maximum accumulation of corrosive constituents from the glass tank occurred at some intermediate temperature. Saying this in a different way, the volatile constituents that come from the glass tank or a dust that may come from the glass tank from adding batch to the glass tank tends to accumulate at some intermediate temperature. At the very high temperatures the alkalies are so volatile that they do not remain in contact with the carbides and nitrides. To some extent this is also observed on the oxides as well. It would be interesting to be able to predict the temperature at which maximum accumulation occurs. I can only estimate from S-α3 and silicon nitride sample 4 that the maximum accumulation occurs at approximately 2200°F. At temperatures below this there appears to be very little corrosion accelerated by the glass. At temperatures above this the corrosive agents appear to boil out and a corrosion, although more severe than in a combustion atmosphere, is less severe than it is at this intermediate temperature.

Even in the combustion atmosphere there is obviously some iron oxide that is available in the vapor as can be observed from the colored oxide film present on the oxidized samples. This iron oxide undoubtedly accelerates corrosion to some extent but it certainly appears to be minimal. Most of the silicon carbide samples as well as silicon nitride samples, appear to be able to withstand the combustion atmosphere in the absence of the volatile glass constituents.

The high purity alumina samples, even to the AD-94, apparently can withstand the attack of the glass constituents. This is characteristic of aluminum oxide which has a good record for withstanding corrosive atmospheres in glass tanks. I could detect a little creep or bending in the AD-94. The AD-85, of course, which is sintered with the aid of considerable magnesium, calcium silicate glass shows a great deal of deformation and probably could not be used at such high temperatures as 2550°F without severe deformation. Inasmuch as there are many refractory products available with reasonably high purity aluminum oxide above 98% Al_2O_3, it would appear to me that these materials would be adequate to temperatures in the neighborhood of 2600°F. Perhaps the more difficult problem of long term operation of a recuperator would be the continual condensation of the glass constituents mainly alkalies that would take place at an intermediate temperature eventually plugging or restricting the flow of gases and decreasing the heat transfer coefficients to the point of making recuperative operation very difficult. Compared to the alumina, the mullite and cordierite were inadequate. The mullite, in particular, showed a great deal of penetration of glass atmosphere constituents. Not only did the penetration occur and, undoubtedly, change the viscosity and creep characteristics of the mullite, but it also changed the coefficient of thermal expansion as evidenced by the multitude of cracks that occurred in the penetrated portion of the mullite.

Even the iron from the combustion atmosphere was sufficient to alter the cordierite at the higher temperatures. It may be that we are above the eutectic in the cordierite system (Magnesia-alumnina-silica). Cordierite is normally not used at such high temperatures as 2600°F. On the other hand, it is very sensitive to iron as shown by cordierite sample 3 which showed some creep and shrinkage on the hot end where iron would absorb and produce a lower melting liquid. Evidently the cordierite cannot be used above about 2100°F as shown by sample 4.

Summary of Observations

Although I have made detailed observations on each sample elsewhere, general conclusions can be drawn that show without doubt the accumulation of alkalies and other volatile constituents on the refractory materials that is maximized at some intermediate temperature near 2200°F. This accumulation accelerates oxidation of carbides and nitrides and renders them inadequate for long term operation of a recuperator at these temperatures. Of the oxides, it is apparent that aluminum oxide withstands creep and penetration better than any of the other oxides. Both mullite and cordierite are sensitive to the penetration of alkalies and other volatile constituents available from glass furnace atmospheres.

ADDENDUM TO ECONOMICS SECTION

Phase I Final Report: High Temperature Range Recuperator

The calculation of economic pay out in the Phase I Final Report: High Temperature Range Recuperator was made on a basis of the heat recovered by the recuperator as having a value equivilent to methane with the same combustion content. This was done so as to separate the recuperator economics from the end-use system economics. This approach, however, severely underestimates the true economic picture for the recuperator. This addendum has been prepared to clarify the calculations and elaborate upon the saving potential. The earlier calculations are two conservative for the following two reasons.

1. The recuperated heat is in the form of usuable hot gas while the methane heat must be converted through some firing process to generate the sensible heat. These firing processes are usually about 65 to 70 percent efficient. Thus the recuperated heat value should be multiplied by 1.4 to 1.6 before calculating the economic value based on methane cost.
2. The most likely use of recovered heat is to preheat combustion air for the source furnace. The interaction between the furnace and recuperator is such as to reduce fuel requirements by more than the simple heat recovery calculation.

The above approaches are independent and cannot be added. They are treated separately below.

Calculation of Economic Return from Sensible Heat Value

In the referenced text, the heat recovery is estimated at 10,000 Btu/hr-ft^{2}IA. Since this is in the form of sensible heat we can equate it to the heat content of methane which can be converted to sensible heat at about 70 percent. Thus at \$2.06 per MCF of methane (one MCF of methane contains about 10^6 Btu), the sensible heat recovery is valued at

$$\frac{10^4 \text{ BTU}}{\text{hr}} \times \frac{365 \times 24 \text{ hr}}{\text{year}} \times \frac{\$2.06/10^6 \text{ BTU}}{0.7 \text{ eff.}} = \$258.00/\text{year}$$

Using the same simple payback approach we have a net fabrication and installation cost of \$240.00/ft^2IA and an annual operating cost of \$57.00. Thus the payback (neglecting interest and rate of return) is about 15 months instead of the 2.3 years calculated in the main text.

Calculation of Economic Return from Preheated Air Usage.

Two modes of operation are possible to make use of preheated combustion air. These are 1) reduction of fuel with constant furnace output and 2) increased furnace output with constant fuel.

1) Possible Fuel Savings

A. Fuel Reduction

A typical furnace configuration using a recuperator is shown in Figure A1.

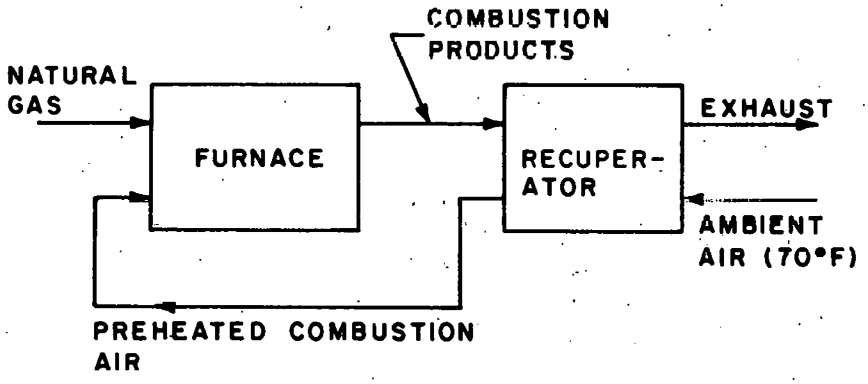

Figure A1. Typical furnace recuperator configuration.

In a glass furnace, the flame temperature is typically around 1700°C. Furnace exit temperatures usually range from 1500 - 1600°C. (Reference: S. R. Scholes, Modern Glass Practice, 7th Ed., Cahners Books, 1975, Pg. 156, 160.)

For this analysis we use the following technique.

a) Assume natural gas to be primarily methane.

b) Assume burner to be adiabatic.

c) Calculate required methane flow per unit heat load in furnace for several air preheat temperatures (for constant flame temperature and furnace exit temperature).

The combustion equation is:

$CH_4 + (X)(2)(O_2 + 3.76\ N_2) \rightarrow CO_2 + 2H_2O + (X)(7.52)N_2 + (X-1)\ 2O_2$

where X = excess air

For an Adiabatic Flame:

$$\hat{H}_R = \hat{H}_P$$

where $\hat{H}$ denotes the total enthalpy of products and reactants (on a molar basis)

$$\hat{H}_P = (\sum_i \eta \hat{h}_i)_{Products}$$

$$\hat{H}_R = (\sum_i \eta \hat{h}_i)_{Reactants}$$

where:

η = Stoichiometric coefficients in combustion equation

h_i = Enthalpy of each compund in combustion process

The product properties of combustion at the set flame temperature are

T = 1700°C (use 3600°R to avoid interpolation of values)

P ≅ 14.7°PSIA

$\hat{h}_{CO_2}$ = -129,801.5 BTU/lb mole

$\hat{h}_{O_2}$ = 25,448.8 BTU/lb mole

$\hat{h}_{N_2}$ = 24,144.9 BTU/lb mole

$\hat{h}_{H_2O}$ = -72,696.2 BTU/lb mole

The reactant properties at different preheat temperatures are shown in Table A1.

TABLE A1

Reactant Enthalpies as a Function of Temperature

TEMP. (°R)	$\hat{h}_{O_2}$ (BTU/LB MOLE)	$\hat{h}_{N_2}$ (BTU/LB MOLE)	$\hat{h}_{CH_4}$ (BTU/LB MOLE)
537	0	0	
1,000	3,362.4	3,248.4	-32,179
1,500	7,292.0	6,919.4	
2,000	11,438.9	10,804.9	
2,500	15,718.3	14,860.0	
2,560	16,239.1	15,355.8	

Equating $\hat{H}_P$ to $\hat{H}_R$ and solving for X for several preheat temperatures we have;

$$(1)(-32,179) + 2(X)\,[\hat{h}_{O_2} + 3.76\,\hat{h}_{N_2}]$$
$$= (1)(-129,801.5) + 2(-72,696.2)$$
$$+ 7.52\,(X)(24,144.9) + (X-1)\,2\,(25,448.8)$$

and:

$$293,912.5 = 232,467.25\,X - 2X[\hat{h}_{O_2} + 3.76\,\hat{h}_{N_2}]$$

or

$$X = \frac{293,912.5}{232,467.25 - 2[\hat{h}_{O_2} + 3.76\,\hat{h}_{N_2}]}$$

Then X versus t for selected values of t is shown in Table A2.

TABLE A2

Excess Air vs. Preheat Temperature

TEMP. (°R)	X
537	1.26
1,000	1.460
1,500	1.772
2,000	2.29
2,500	3.29
2,560	3.48

The required methane flows normalized by furnace heat load can now be calculated. In the furnace, the furnace heat load (losses to ambient + heat input to material) is given by:

$$Q_{Furnace} = \overset{o}{M}_{Products} \; Cp_{Products} \; \Delta T_{Products}$$

where:

$Q_{Furnace}$ = Furnace Heat Load (BTU/HR)

$\overset{o}{M}_{Products}$ = Mass Flow Rate of Products (LBM/HR)

$Cp_{Products}$ = Specific Heat of Products (BTU/LBM°F)

$\Delta T_{Products}$ = Temperature Drop of Products in Furnace (°F)
(Burner Temp. - Exit Temp.)

Solving for $\overset{o}{M}_{Products}/Q_{Furnace}$ we obtain

$$\frac{\overset{o}{M}_{Products}}{Q_{Furnace}} = \frac{1}{Cp_{Products}\ \Delta T_{Products}}$$

From the Stochiometry of the combustion equation we have

$$\overset{o}{M}_{CH_4} = \overset{o}{M}_{Products} \times \frac{\text{No. Moles } CH_4}{\text{No. Moles Products}} \times \frac{\hat{M}_{CH_4}}{\hat{M}_{Products}}$$

Where:

$\overset{o}{M}_{CH_4}$ = Mass Flow of Methane (LBM/HR)

$\hat{M}_{CH_4}$ = Molal Mass of CH_4 (LBM/LBMOLE)

$\hat{M}_{Products}$ = Molal Mass of Products (LBM/LBMOLE)

or

$$\frac{\overset{o}{M}_{CH_4}}{Q_{Furnace}} = \frac{1}{(Cp_{Products}\ \Delta T_{Products})}\ \frac{\text{No. Moles } CH_4}{\text{No. Moles Products}}\ \frac{\hat{M}_{CH_4}}{\hat{M}\ \text{Products}}$$

In order to evaluate this equation we must make a determination of product properties. The specific heats at an average furnace temperature, 1600°C are:

<u>CO_2</u>

$\hat{C}p$ = 6.214 (cal/gMOLE °K) + 10.396 x 10^{-3} (cal/gMOLE °K^2)(T)

\- 3.545 x 10^{-6} (cal/gMOLE °K^3)$(T)^2$

= 13.249 (cal/gMOLE °K)

<u>H_2O</u>

$\hat{C}p$ = 7.256 (cal/gMOLE °K) + 2.298 x 10^{-3} (cal/gMOLE °K^2)(T)

\+ 0.283 x 10^{-6} (cal/gMOLE °K^3)$(T)^2$

= 12.553 (cal/gMOLE °K)

$\underline{N_2}$

$\hat{C}_p$ = 6.524 (cal/gMOLE °K) + 1.250 x 10^{-3} (cal/gMOLE °K^2)(T)

\- .001 x 10^{-6} (cal/gMOLE °K^3)$(T)^2$

= 8.862 (cal/gMOLE °K)

$\underline{O_2}$

$\hat{C}_p$ = 6.148 (cal/gMOLE °K) + 3.102 x 10^{-3} (cal/gMOLE °K^2)(T)

\- .923 x 10^{-6} (cal/gMOLE °K^3)$(T)^2$

= 8.72 (cal/gMOLE °K)

The Molal weights are:

$\hat{M}_{CH_4}$ = 16.042 LBM/LBMOLE

$\hat{M}_{Co_2}$ = 44.01 LBM/LB MOLE

$\hat{M}_{H_2O}$ = 18.016 LBM/LBMOLE

M_{N_2} = 28.016 LBM/LBMOLE

$\hat{M}_{O_2}$ = 32.0 LBM/LBMOLE

Then the product properties and fuel requirements are as given Table A3 and Table A4.

TABLE A3

Product Properties

EXCESS Air (X)	No. MOLES Products	X_{CO_2}	X_{H_2O}	X_{N_2}	X_{O_2}	$\hat{C}_p = \Sigma X_i \hat{C}p_i$ (CAL/GMOLE°K)	$\hat{M} = \Sigma X_i \hat{M}_i$ (LBM/LBMOLE)
1.26	12.995	0.077	0.154	0.729	0.040	9.763	27.867
1.46	14.899	0.067	0.134	0.737	0.062	9.642	27.995
1.77	17.850	0.056	0.112	0.746	0.086	9.509	28.134
2.29	22.801	0.044	0.088	0.755	0.113	9.364	28.290
3.29	32.321	0.031	0.062	0.765	0.142	9.207	28.458
3.48	34.130	0.029	0.059	0.767	0.145	9.187	28.468

TABLE A4

Fuel Requirements for Furnace Production

AIR PREHEAT Temp. (°F)	EXCESS AIR (X)	$\dfrac{\overset{\circ}{M}_{CH_4}}{Q_{Furnace}}$ $\dfrac{LBM/HR}{BTU/HR}$
77	1.26	3.516×10^{-4}
540	1.46	3.106×10^{-4}
1,040	1.77	2.625×10^{-4}
1,540	2.29	2.087×10^{-4}
2,040	3.29	1.495×10^{-4}
2,100	3.48	1.420×10^{-4}

These results are best illustrated by plotting normalized fuel requirements as a function of preheated air temperature as shown in Figure A2.

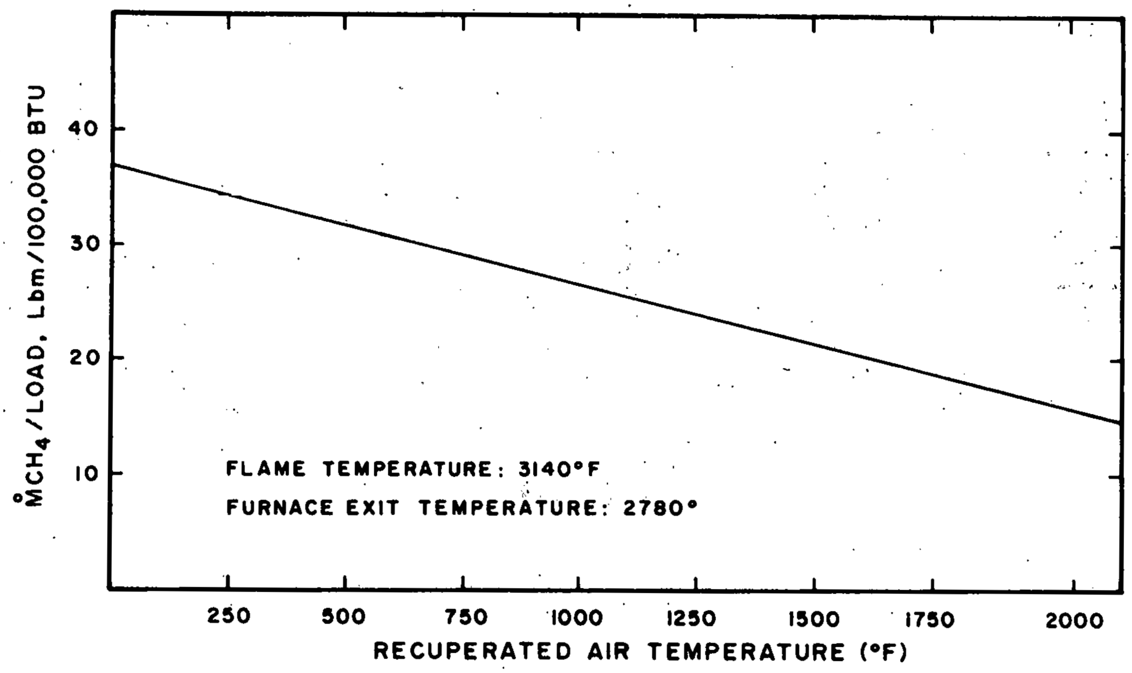

Figure A2. Graphic display of fuel requirements vs. preheat temperature based on 100,000 BTU/hr furnace.

These results are dramatic in showing reduction in fuel requirements. For example for a 100,000 BTU/hr furnace using recuperated precombustion air at 2100°F, the fuel savings from Figure A2 are:

$$\frac{35.2 \text{ lb } CH_4/hr - 14.2 \text{ lb } CH_4/hr}{35.2 \text{ lb } CH_4/hr} = 60\%$$

For 1100°F the fuel savings are

$$\frac{35.2 - 25.5}{35.2} = 28\%$$

These results are supported by operating experience in the glass industry where a 30% decrease in fuel requirements is commonaly achieved with precombustion air heated to approximately 1100°F (Private Communication, Foster Harding, Johns-Manville, Denver, Colorado).

To further confirm that these savings are possible it must be established that there is enough heat available in the furnace exhaust flow sufficient to preheat the combustion air to 2100°F. The goal of the high temperature project is to recover 70% of the energy in the exhaust stream. In terms of temperatures then

$$0.7 = \frac{(T_{Exhaust} - T_{Outlet})}{(T_{Exhaust} - T_{Ambient})}$$

For $T_{Exhaust} = 2780°F$

and $T_{amb} = 70°F$ then it is required that

$T_{Outlet} = 883°F$

The available heat in the exhaust products is

$$Q_{Exhaust} = \overset{o}{M}_{Products}\ Cp_{Products}\ \Delta T_{Products}$$

The required heat input to the air is:

$$Q_{Air} = \overset{o}{M}_{Air}\ C_{Pair}\ \Delta T_{Air}$$

$$Q_{Exhaust} \text{ must be } > Q_{Air}$$

the condition required then is:

$$\frac{\overset{\circ}{M}_{\text{Products}}\ \ Cp_{\text{Products}}\ \ \Delta T_{\text{Products}}}{\overset{\circ}{M}_{\text{Air}}\ C_{\text{Pair}}\ \Delta T_{\text{Air}}} \text{ must be} > 1$$

$$\frac{(34.130 \times 28.468) \times (0.323) \times (2780 - 883)}{(33.130 \times 28.97) \times (0.24) \times (2100 - 70)} = 1.27$$

Thus preheating to 2100°F is possible.

B. Economic Value of Fuel Savings

The estimated savings achieved by a recuperator recovering 2100°F preheat air compared to an unrecuperatred furnace are 60%. For a typical 240 ton per day glass furnace the fuel usage is about 10 x 10^6 BTU per ton unrecuperated. Annual savings at constant production are then

$$240 \times 365\ \frac{\text{tons}}{\text{year}} \times 6 \times 10^6\ \frac{\text{BTU}}{\text{Ton}} \times \frac{\$2.06}{10^6\ \text{BTU}} = \$1{,}083{,}000/\text{year}$$

Using the recuperator size requirements (conservative) for 70% recovery of stack gas we have

$$\frac{3.5 \times 10^6\text{BTU}}{\text{ton}} \times 240\ \frac{\text{ton}}{\text{day}} \times \frac{\text{day}}{24\ \text{hrs}} \times \frac{\text{ft}^2\text{IA}}{10{,}000\ \text{BTU}} = 3{,}500\ \text{ft}^2\text{IA}$$

and savings per unit ft^2IA is

$$\frac{\$1{,}083{,}000}{3{,}500\ \text{ft}^2\text{IA}} = \$309.00/\text{ft}^2\text{IA/year}$$

This is a somewhat better economic picture than the methane combustion heat calculation in that the simple pay back period is just under 12 months.

2) Possible Increased Furnace Loading at Constant Fuel Usage

A. Increased Production Potential

The previous fuel savings calculations show detailed problem formulation that will not be repeated here. For the present analysis we will use the following technique.

a) Calculate stoichiometry required for 1700°C flame temperature with no air preheat.

b) Calculate flame temperature as a function of air preheat temperature using previously calculated stoichiometry.

c) Calculate increased furnace loading possible using higher flame temperatures.

The combustion equation now is based upon a given 26% excess O_2.

$$CH_4 + 2.52\,(O_2 + 3.76\,N_2) \rightarrow CO_2 + 2H_2O + 9.48\,N_2 + 0.52\,O_2$$

The $\hat{h}_{reactants}$ are as given in previous calculations.

The enthalpy of reactants as a function of temperatures is shown in Table A5.

TABLE A5

Reactant Enthalpy vs. Temperature

T(°R)	H_R (BTU/LBMOLE)
537	- 32,179
1,000	7,073
1,500	51,760
2,000	99,026
2,500	148,233
2,560	154,243

The enthalpy of the products versus flame temperature then is shown in Table A6.

TABLE A6

Product Enthalpy vs. Flame Temperature

T(°R)	$\hat{h}_{CO_2}$	$\hat{h}_{H_2O}$	$\hat{h}_{N_2}$	$\hat{h}_{O_2}$	H_P
3,600	-129,801.5	-72,696.2	24,144.9	25,448.8	- 33,067
4,000	-123,981.1	-67,747.2	27,599.9	29,081.0	17,294
4,400	-118,107.4	-62,682.4	31.083.6	32,759.9	68,655
4,800	-112,187.6	-57,520.8	34,590.0	36,483.5	119,655
5,000	-109,212.5	-54,908.9	36,350.3	38,361.2	145,518

The following graph illustrates this relationship.

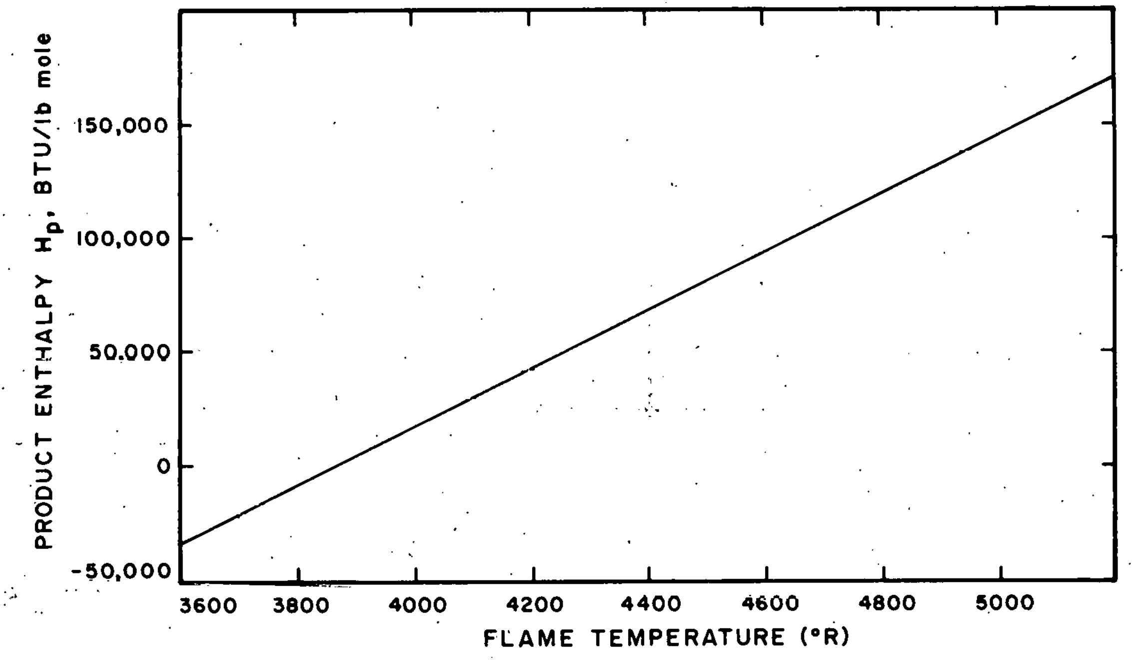

Figure A3. Product enthalpies displayed graphically.

Equating $H_R = H_P$, using Figure A3 and Table A5, the flame temperature vs. air preheat temperature is found to be as shown in Table A7.

TABLE A7

Flame Temperature vs. Preheat Temperature

AIR PREHEAT TEMP. (°R)	FLAME TEMP. (°R)
537	3600
1,000	3920
1,500	4260
2,000	4640
2,500	5030
2,560	5080

For constant flows and stoichiometry, the possible furnace loading is a ratio of the temperature differences (assuming constant specific heats):

$$q_{\substack{\text{Unrecuperated}\\ \text{Furnace}}} = \overset{\circ}{M}_{\text{Products}}\, Cp_{\text{Products}}\, (T_{\text{Flame unrecup.}} - T_{\text{Outlet}})$$

$$q_{\substack{\text{Recuperated}\\ \text{Furnace}}} = \overset{\circ}{M}_{\text{Products}}\, Cp_{\text{Products}}\, (T_{\text{Flame Recup.}} - T_{\text{Outlet}})$$

$$\frac{q_{\substack{\text{Recup}\\ \text{Furnace}}}}{q_{\text{Unrecup.}}} = \frac{T_{\text{Flame Recup.}} - T_{\text{Outlet}}}{T_{\text{Flame Unrecup.}} - T_{\text{Outlet}}}$$

For a furnace outlet temperature of 2780°F the product ratios are given in Table A8. The inverse of this case could be to maintain a constant loading rate and decrease the total fuel-air flowrate. For this mode the data presented in Table A8 would represent the ratio:

$$\frac{\text{fuel consumption unrecuperated furnace}}{\text{fuel consumption recuperated furnace.}}$$

TABLE A8

Increased Furnace Loading for Increased Flame Temperature

$T_{\text{Air Preheat}}$ (°R)	Loading recup. furnace/loading unrecup. furnace
537	1.0
1,000	1.89
1,500	2.83
2,000	3.88
2,500	4.97
2,560	5.11

In practice, materials considerations limit the maximum flame temperature and furnace redesign would probably be necessary to ensure the large ΔT's between the flame and the furnace exit appears as energy input to the glass rather than heat loss to the surroundings. A possible operating mode would be to increase flame temperature to the material limits of burners and furnaces and then increase the excess air ratio to maintain temperatures and optimize the through put for a given furnace site.

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