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I am submitting herewith a thesis written by Jon P. Winkler entitled "An Evaluation of a Silicon Carbide/Alumina Ceramic Composite for Use in High Temperature Air Heaters." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Engineering Science and Mechanics.

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**AN EVALUATION OF A SILICON CARBIDE/ALUMINA
CERAMIC COMPOSITE FOR USE IN HIGH
TEMPERATURE AIR HEATERS**

A Thesis

Presented for the

Master of Science Degree

The University of Tennessee, Knoxville

Jon P. Winkler

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ABSTRACT

An evaluation of a ceramic composite tube for possible use in high temperature air heaters was conducted. A silicon-carbide/alumina composite ceramic tube which had been tested for 107 hours in a coal fired flow facility at a tube temperature of approximately 1149°C was evaluated by comparison of unexposed, exposed, and as-received samples of the material to determine changes in properties. The project included mechanical testing and microscopy. The mechanical testing consisted of compression and tension testing of circular rings of unexposed and exposed material. The microscopy included stereo, metallurgical, and scanning electron microscopy as well as microprobe analysis employing EDS and WDS analysis and x-ray mapping. Parameters such as Young's modulus, tensile and compressive strengths, porosity, and constituent distribution were determined to demonstrate any changes in the material due to exposure to high temperature. The strength of the material did not change significantly from the unexposed to the exposed, but the Weibull modulus was found to have decreased indicating a higher probability of failure. Porosity was found to have increased from as-received to unexposed and again from unexposed to exposed. Aluminum content was found to decrease in the same way from as-received to exposed.

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NOMENCLATURE

b	Width of ring (in)
m	Weibull modulus
n	Number of samples
P	Load (lbs)
P_f	Probability of Failure
ψ	Angle from vertical (rad)
Q	Stress magnification factor
r	Radius (in)
r_i	Inner radius (in)
r_o	Outer radius (in)
σ	Failure stress (psi)
σ_o	Characteristic stress (psi)
σ_u	Stress with no probability of failure (psi)
V	Volume of test sample (in ³)

CHAPTER I

INTRODUCTION

The need for greater efficiency in electricity producing plants has driven the development of magnetohydrodynamics (MHD). An MHD cycle would be added to steam turbine facilities so that the MHD cycle would be the first or topping cycle and the steam turbine cycle would be the last or bottoming cycle. With commercial MHD electric power plants it will be possible to increase the efficiency of the electricity producing process. The University of Tennessee Space Institute has a proof of concept (POC) magnetohydrodynamic coal fired flow facility in order to test the concept in a working environment.

An MHD topping cycle begins by burning coal which has been seeded with potassium salt to facilitate ionization of the exhaust gas. The exhaust plasma is accelerated to supersonic speeds and enters the MHD channel which is a duct with insulating and conducting wall sections. A magnetic field orthogonal to the flow direction is applied. The plasma is then slowed by Lorentz forces and an electrical current is generated at the end of the MHD topping cycle. The exhaust gas slows further and enters the steam turbine bottoming cycle [1].

Typical conversion efficiency of a steam turbine electricity plant is around 30 to 45 percent [2,3]. With the addition of MHD as a topping cycle, efficiencies up to 55 percent could be obtained. An MHD topping cycle typically has efficiencies in the range of 75 to 80 percent [2], hence, more of the power should be generated in the MHD topping cycle rather than in the steam turbine bottoming cycle.

In order to achieve the desired efficiency in the MHD cycle, one of two procedures must be employed. The first is to inject pure oxygen during combustion in order to achieve higher combustion temperatures which will yield better conversion efficiencies. The other is to preheat the combustor air through the use of high temperature air heaters (HTAH). Currently, there are no high temperature air heaters capable of producing air temperatures high enough to warrant their implementation, so the method of oxygen addition is presently being used. However, the high cost of oxygen production makes this method less desirable than high temperature air heaters.

There are two types of high temperature air heaters: regenerative and recuperative. Regenerative air heaters use a bed of rock, beads, or brick to form a matrix which is heated by the combustion exhaust. When an adequate temperature is reached, the exhaust gas is closed off and the combustor air opened. The air blows over the matrix and is heated before it enters the combustor. Unfortunately, this process requires that the combustor air and exhaust gas must be cycled on and off, so, to achieve a steady air flow to the combustor, several air heaters must be implemented [2].

The second type of high temperature air heater is the recuperative air heater. This method heats the combustor air directly by passing it through tubes which are in the exhaust gas flow before sending it into the combustor. This has the advantage of a steady airflow as opposed to cycling and hence needs only one air heater [2].

The procedure of passing air through the furnace requires a material for the tubes which will withstand the high temperatures and corrosive environment of the furnace and should have a high heat transfer coefficient. Possibilities for this material include high temperature superalloys. Most of these alloys are cobalt or nickel based, but there are other element bases such as niobium (Nb), rhenium (Re), and titanium (Ti) which provide good high temperature qualities as well. The liquidus temperatures for some common superalloys are given in Table 1 at a 50-50 weight percentage reference. If the operating temperature is set at 1649°C, then the only alloy of those in the table which satisfies this requirement is Nb-Re. This alloy is very expensive, so although feasible, it is unlikely for large scale production. Also, it would have to withstand the corrosive properties of the furnace, which is questionable due to the reactive nature of alloys. Therefore, other materials such as ceramics need to be investigated. All of the necessary properties seem to exist in ceramic materials, so ceramics have begun to be studied for high temperature air heaters as well as other high temperature applications.

Many ceramics and ceramic composites possess high strength and high corrosion and erosion resistance at elevated temperatures and moderate to good thermal conductivity. Because of these properties, there is an increasing interest in ceramics for a wide variety of applications ranging from aerospace to the automotive industry. Extensive testing is currently being conducted to evaluate the performance of ceramics in various environments at high temperatures. The research is important to determine the properties and behavior of ceramics in high temperature

Table 1. Table of liquidus temperatures for 50-50 wt.% superalloys [4].

Alloy	Liquidus Temperature
Titanium-Aluminum (Ti-Al)	1430°C
Niobium-Aluminum (Nb-Al)	1605°C
Nickel-Aluminum (Ni-Al)	1340°C
Cobalt-Niobium (Co-Nb)	1450°C
Niobium-Rhenium (Nb-Re)	2450°C

applications. The present project outlines one such effort to evaluate the performance of a ceramic composite tube in a coal fired MHD facility in order to determine any changes in the tube material after exposure to high temperature and a highly corrosive environment.

CHAPTER II

BACKGROUND

MHD Facility and Tube Placement

The proof of concept (POC) MHD facility at the University of Tennessee Space Institute is shown in Figure 1. The upstream test train consists of a vitiation heater, the combustor, circular aerodynamic duct, and the diffuser. The diffuser is followed by the radiant furnace which is the section of interest for HTAH studies [2]. The radiant furnace is approximately 4 feet square and 32 feet high. The walls are made of two 1/4 inch steel plates separated by a one inch gap which is filled with water. Each wall has approximately 50 instrumentation ports [2]. A recuperative high temperature air heater (HTAH) would be positioned in the radiant furnace, because the radiant furnace provides conditions comparable to an actual MHD facility and is adequate for testing HTAH materials [2]. The temperature conditions in the furnace range from approximately 1600°C to 1890°C, and velocities of approximately 12 m/s to 100 m/s have been measured depending on the location in the furnace [2].

The tube which was evaluated was placed in the furnace at level 3 (Figure 2) in a reducing environment with the velocity approximately 14 m/s. The gas temperature was approximately 1650°C, and the tube temperature was measured at approximately 1230°C [5]. The potassium seed in the coal used to ionize the exhaust gas significantly increased the corrosive nature of the slag. The tube was in direct contact with the high velocity slag at high temperature for approximately 107 hours.

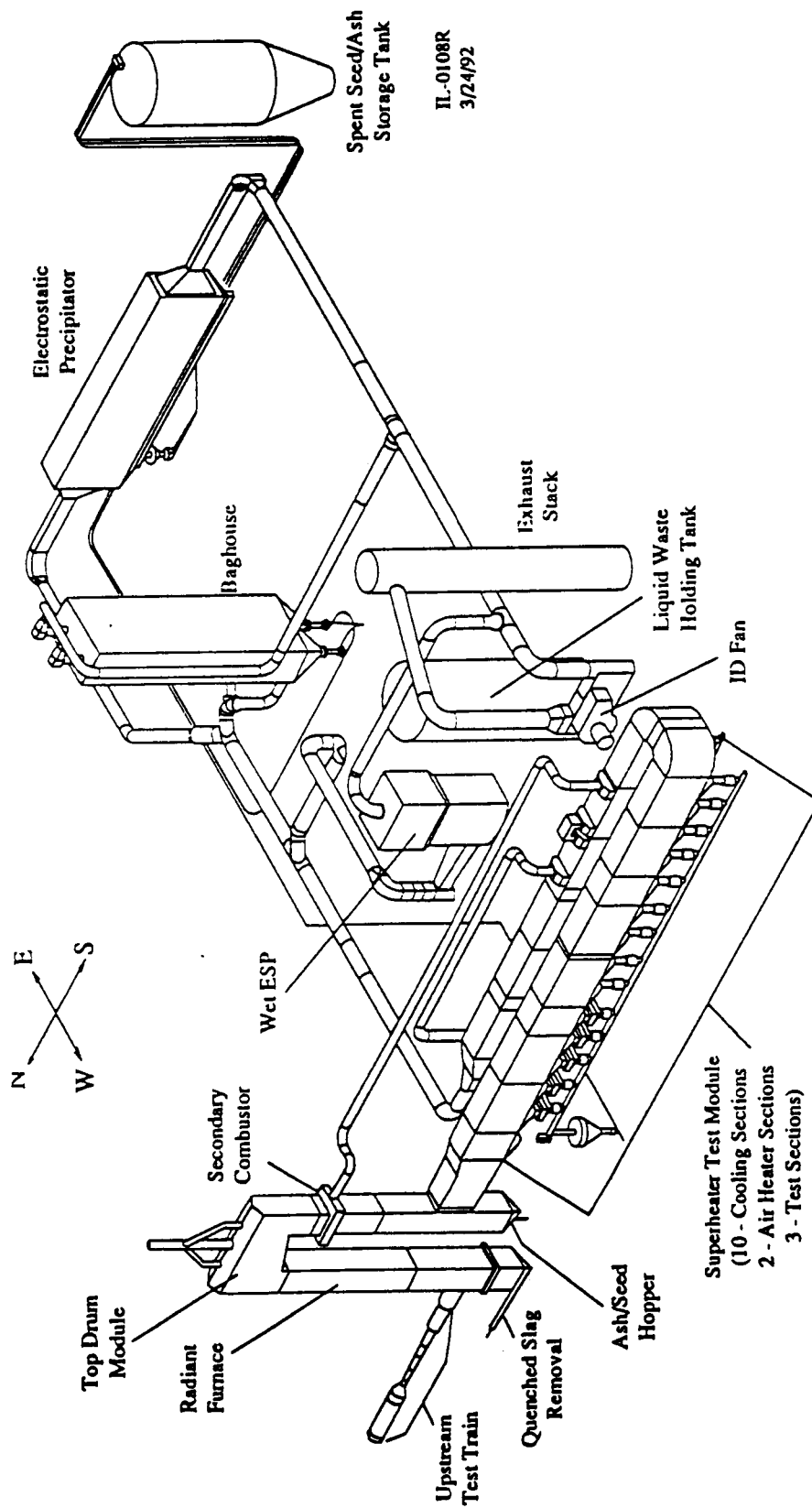


Figure 1. UTSI MHD Facility [1].

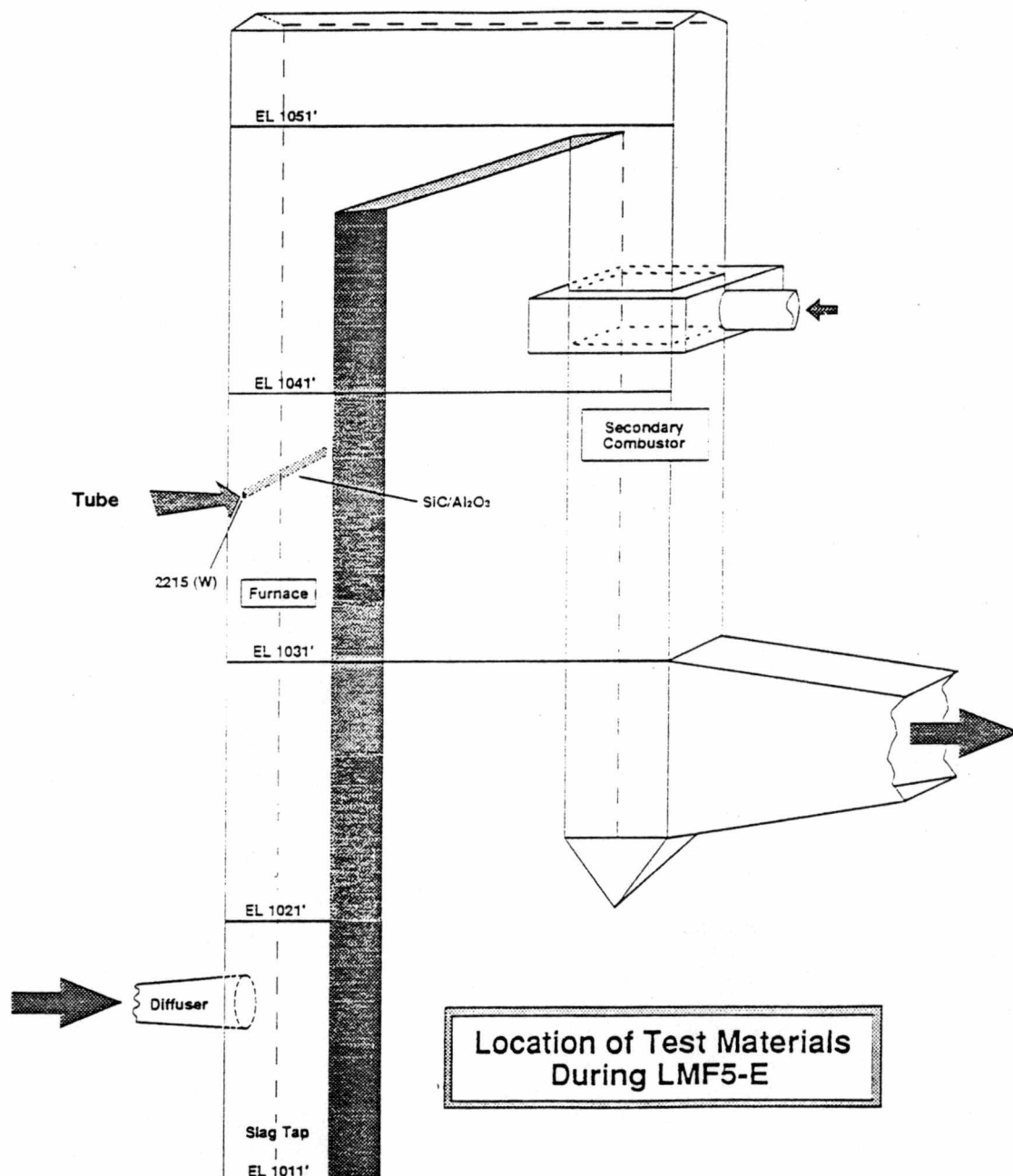


Figure 2. Diagram showing placement of tube in furnace [2].

Materials

Several ceramics have been considered for use in the high temperature air heaters. These may be classified into three types: monolithic, coated, and composite ceramics. Among the monolithic ceramics which have been tested at the UTSI facility are silicon carbide, magnesia aluminum spinel, zirconia, chrome magnesia, boron nitride, and alumina. The coated ceramics included silicon carbide, boron nitride, and alumina, all coated with zirconium oxide. These ceramics have been tested in the MHD facility with limited success [1]. Among the more successful for surviving the furnace environment were silicon carbide, alumina, and zirconia. Zirconia showed the highest resistance to corrosion and high temperature but had a low tolerance to thermal shock [2]. Properties of some of the ceramics being considered for high temperature air heater applications may be seen in Table 2.

The third type, ceramic composites, are becoming more common in many applications. These seem to have the highest success rate of the ceramics during testing in the furnace. Many of the composites consist of a ceramic matrix reinforced by a second ceramic to form a ceramic matrix composite (CMC). The reinforcement material can be in the form of fibers, whiskers, particles, and flakes (fibers are long single strands whereas whiskers are basically short fibers) and add strength to the final material. However, fiber and whisker reinforcement tend to be more directionally strong in the direction of the fiber orientation. Particulate and flake reinforcement provides a more isotropic material and hence more uniform strength properties in all directions.

Table 2. Thermal and mechanical properties of various ceramics [6,7,8].

	Si ₃ N ₄	Al ₂ O ₃	SiC	ZrO ₂
Crystal Structure	Hexagonal	Hexagonal	Cubic	Tetragonal
Density (kg/m ³)	3180	3980	3210	6100
Hardness (kg/mm ²)	2000	2100	2500	1300
Young's Modulus (GPa)	310	390	440	290
Poisson's Ratio	0.22	0.23	0.17	0.24
Tensile Strength (MPa)	350-425	300	175	45
Compressive Strength (MPa)	700-2750	3400	1400-3400	2000
Flexural Strength (MPa)	800-900	500-600	675	500-700
Thermal Conductivity (W/m K)	31	35	85-120	1.8
Specific Heat (J/kg K)	720	920	710	630
Coeff. of Thermal Expn. x10 ⁻⁶ (°C ⁻¹)	3.0	7.1	4.3	11
Melting or Decomposition Temp. (°C)	1900	2050	2830	2760

Components made of ceramics are formed in several ways. Most of these methods are still expensive, and methods are being developed and perfected to economically produce ceramic components. Methods include hot pressing, slip-casting, and injection molding to make a pre-form. The component may then be processed further by firing, sintering, or coating.

The tube which was evaluated in this study was a silicon carbide particulate/alumina matrix composite ($\text{SiC}_p/\text{Al}_2\text{O}_3$). The composition of the composite in volume percent was approximately 40% SiC, 50% Al_2O_3 and 10% Al. The 10% free aluminum was due to the synthesis method of the composite. The evaluated tube pre-form was formed using the slip casting process. This SiC preform was then placed on a block of aluminum and heated above the melting point of aluminum. The molten aluminum was drawn up into the preform by capillary action surrounding the silicon carbide particles and oxidizing at the same time in a controlled atmosphere. The incomplete oxidation of aluminum resulted in a mixture of alumina and free aluminum as matrix material around the silicon carbide particles.

CHAPTER III

PROCEDURE

Overview

The tube, which was obtained from a commercial source, was evaluated to determine any physical or chemical changes in the material due to exposure to the furnace. The evaluation included mechanical testing of exposed and unexposed samples, optical and scanning electron microscopy, and microprobe analysis of as-received, unexposed, and exposed samples. As-received samples were samples in the same condition as when they had been received from the manufacturer. Unexposed samples were samples which were involved in the furnace test but which were not in direct contact with the slag and furnace environment. The unexposed section was the part of the tube which was inserted in the wall of the furnace in order to hold the tube in the furnace (see Figure 3), so the temperature of the unexposed section was lower than that of the exposed section. Exposed samples were samples which had been in the furnace directly exposed to the slag and furnace environment during testing.

The strength of ceramic materials, unlike metals, is limited by the size of inherent flaws such as porosity, inclusions, microcracks, etc. These flaws can vary considerably in size and can be introduced into the material during processing, handling, machining, or while in service. Structural ceramics are generally brittle with low fracture toughness which imposes a severe restriction on the size of the critical flaw. In addition, stressed ceramic components fail according to the weakest

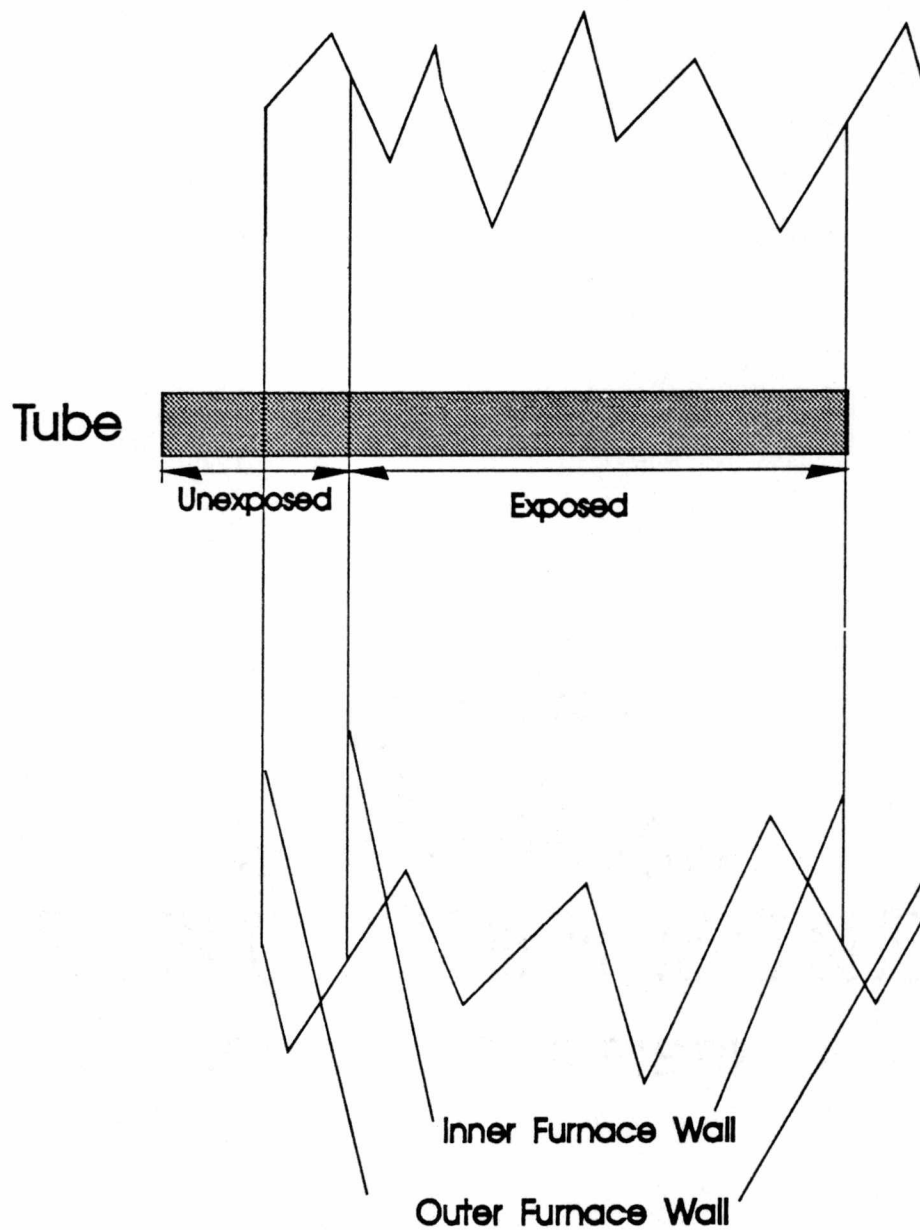


Figure 3. Diagram of tube in furnace wall showing unexposed and exposed sections.

link theory, which states that the failure stress is determined by the weakest point, which is the most critical flaw [9,10]. As a result, strengths tend to vary and require statistical description. For this purpose, the Weibull statistical method was chosen. Weibull statistical analysis is based on the weakest link theory and has been used extensively to determine the probability of failure of ceramics under load. The probability of failure in a ceramic can be related to the strength of a specimen through determination of the Weibull number which is estimated using the experimental strength values obtained from mechanical testing.

Mechanical Testing

The tube was cut so that the part of the tube which was outside the furnace and the part of the tube which was inside the furnace and exposed to the furnace conditions were separated (Figures 4-6). These parts were then further sectioned into rings approximately 10 mm in width ($\pm 2\text{mm}$) (Figures 7-8), and each ring was marked in order to identify the top of the tube while in the furnace. Six of these rings from both the exposed and unexposed sections were selected for testing under uniaxial tension and compression to determine their mechanical properties. An Instron testing machine (Figure 9) was used to perform both the compression and tensile tests.

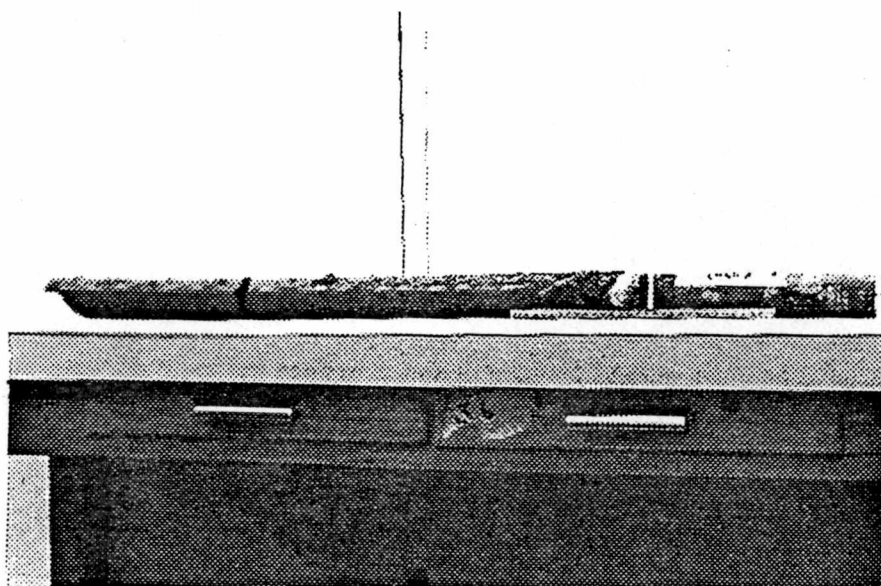


Figure 4. Sectioning of post-furnace tube.

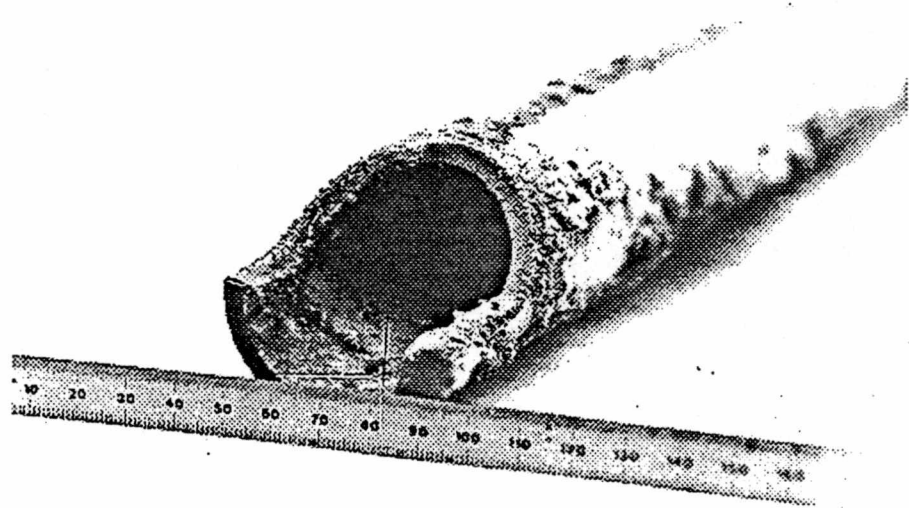


Figure 5. Tube end which was exposed to furnace.

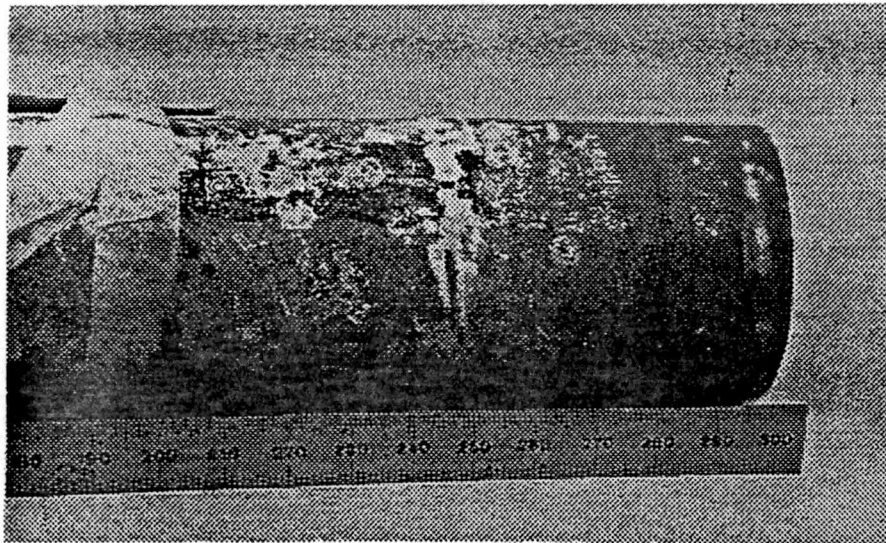


Figure 6. Unexposed end of the tube.

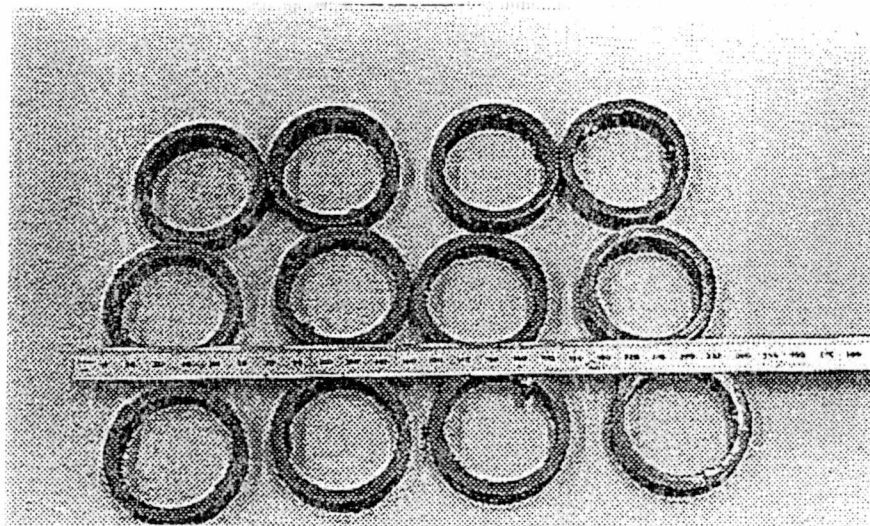


Figure 7. Ring sections for mechanical testing.

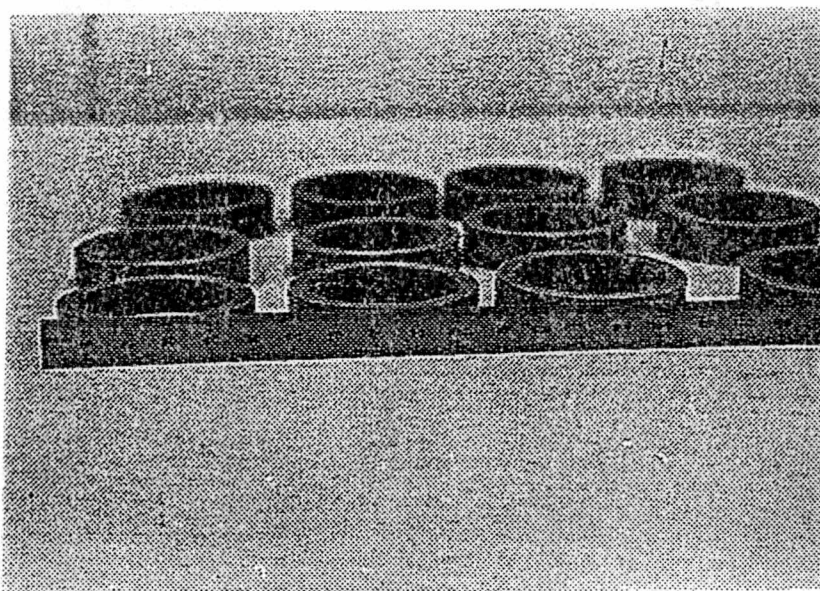


Figure 8. Ring sections for mechanical testing.

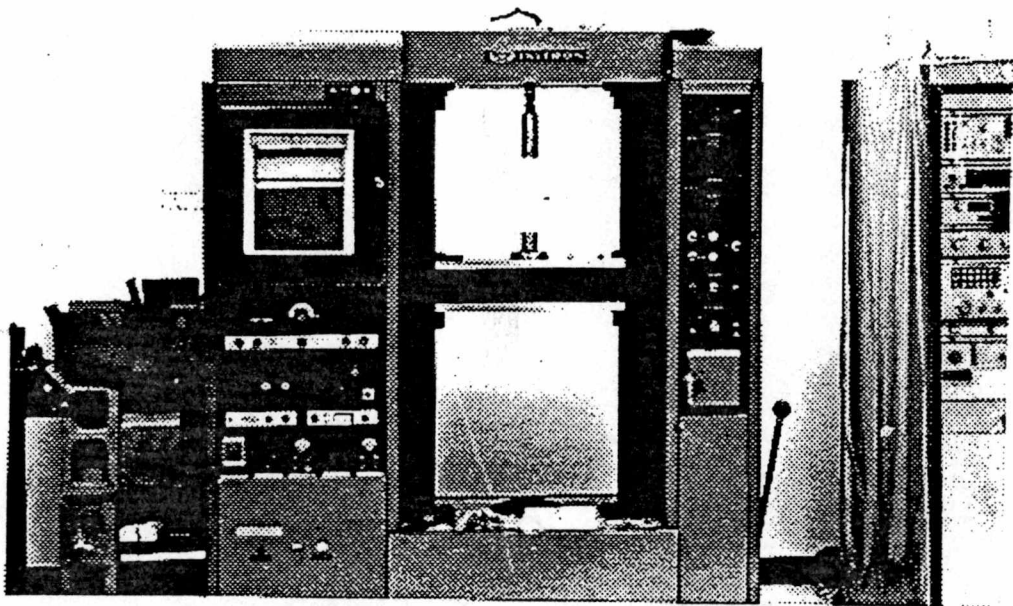


Figure 9. Instron machine used for compression and tensile testing.

In order to achieve uniform line contact during the uniaxial tensile testing, two steel pins (6mm diameter and at least 70mm long) were positioned diametrically opposite at the top and bottom on the inside surface of the ring. These pins were attached to a moving cross-head and a load cell by holding fixtures as shown in Figure 10. The ring was positioned so that the top of the ring corresponded to the top of the tube while it was in the furnace. This was done in order to determine the effect of the tube orientation in the furnace on fracture origin during testing. The compression testing sample was placed between two diametrically opposite flat plates which were connected to the moving cross-head and load cell. This arrangement also provided uniform line contacts between the sample and the plates (see Figure 11). Each ring was loaded under either uniaxial tension or compression at a strain rate of $2.07\text{e}^{-4}\text{s}^{-1}$. Load-elongation curves were recorded on an x-y recorder using the output from the extensometer. The typical curve shape for compression and tension plots is shown in Figures 12-13. These plots were then used to construct stress-strain curves. A ring modulus and fracture strength were then obtained from the stress-strain curves. The ring modulus obtained was calculated as stress over strain, or the Young's modulus, for a circular ring configuration. This information was further used to calculate the Weibull number.

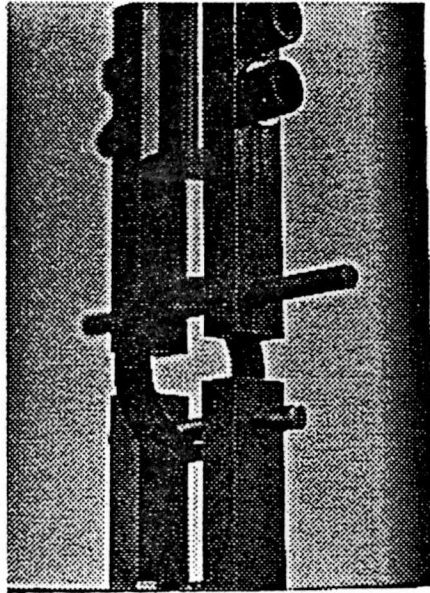


Figure 10. Device for holding sample in tensile testing.

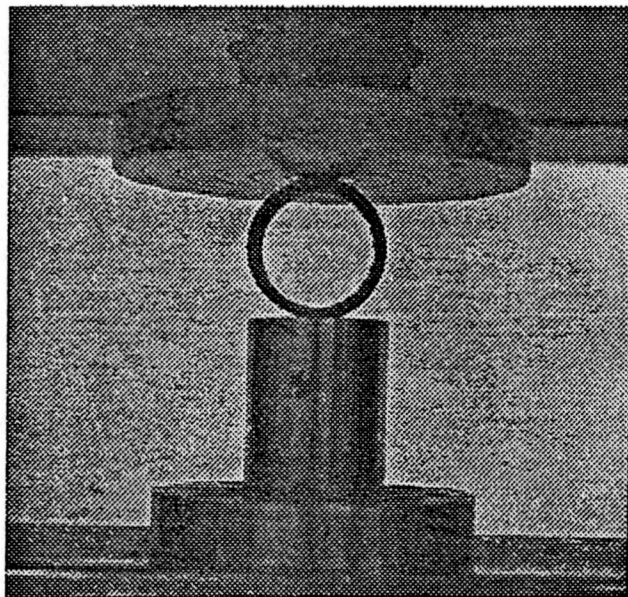


Figure 11. Plates used for compression testing.

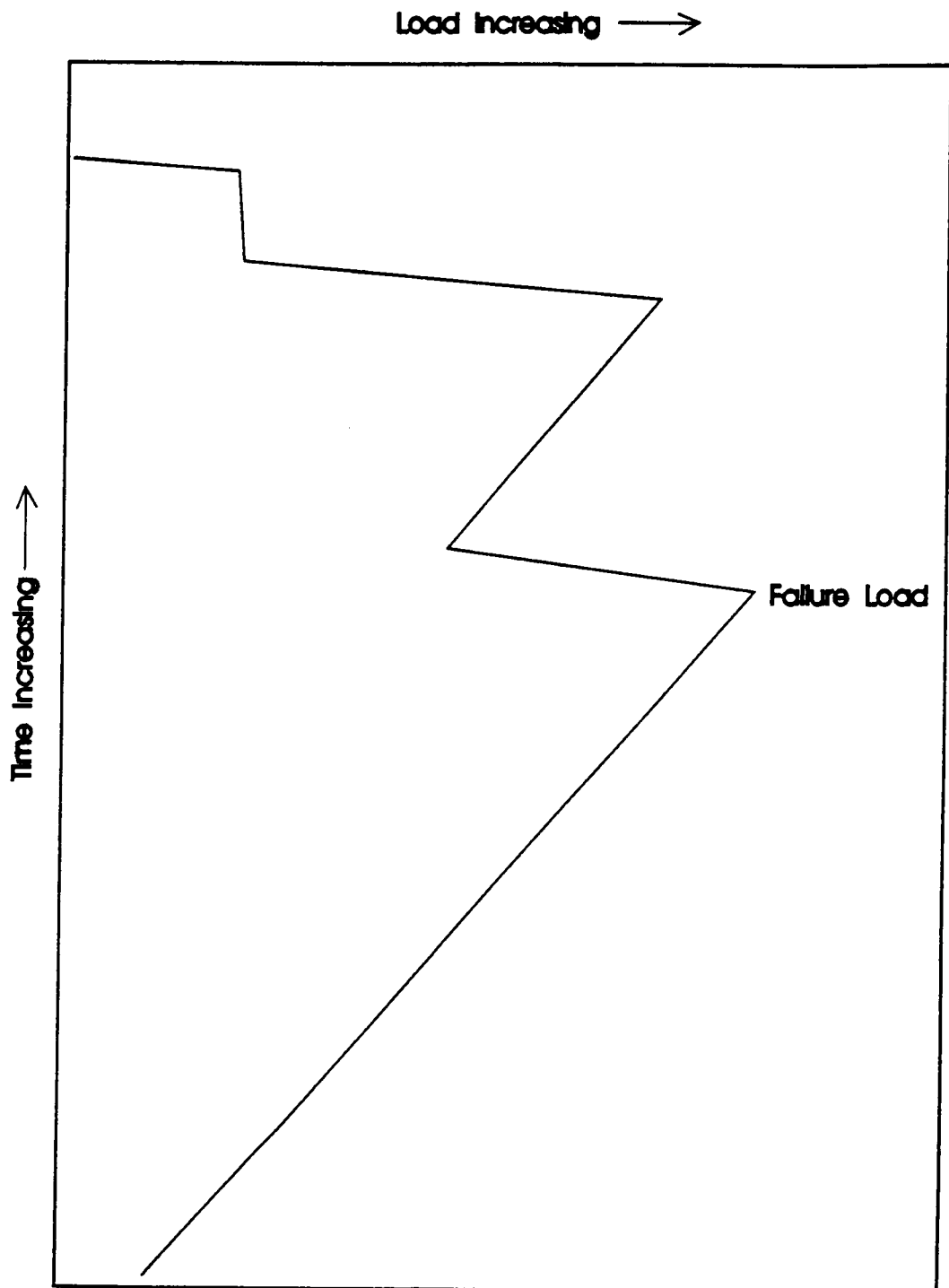


Figure 12. Basic curve shape of load vs. time for compression testing.

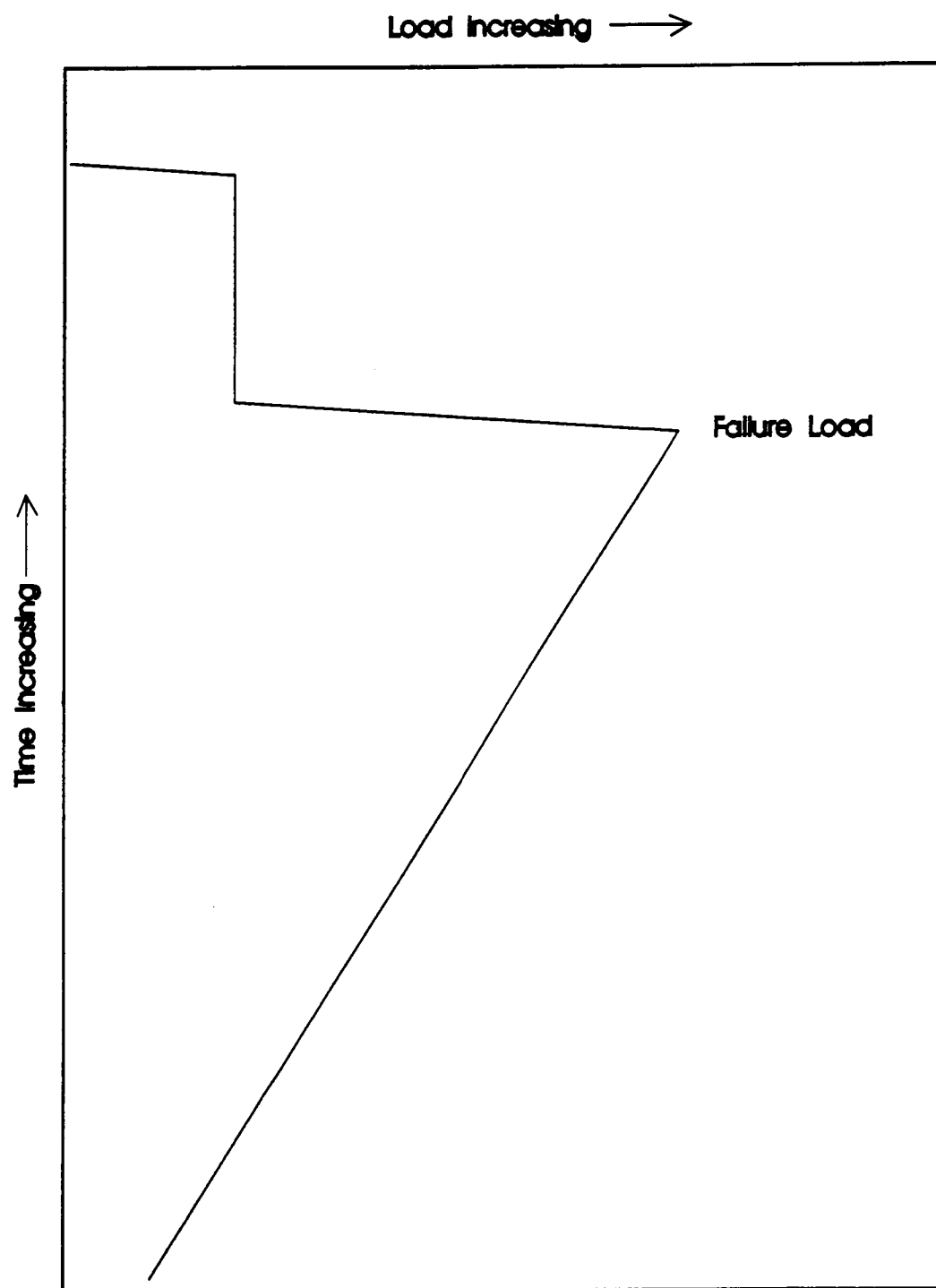


Figure 13. Basic curve shape of load vs. time for tension testing.

Microscopy

Physical features such as amount and distribution of porosity have strong influence on mechanical properties of ceramics. Optical and scanning electron microscopy for studying porosity, residual aluminum, and element distribution was performed on as-received, unexposed, and exposed samples. Samples were cut from unexposed and exposed tube sections and from an as-received coupon and mounted in epoxy. Each sample was metallographically prepared by polishing with a series of grit papers. The final polishing was done with a 3 μm diamond paste.

Quantitative elemental analysis was performed using a Cameca MBX microprobe with a light element detector for x-ray analysis using energy dispersive spectroscopy (EDS). The distribution of elements in the material was also studied quantitatively on the microprobe using wavelength dispersive spectroscopy (WDS) techniques. The microprobe was operated at 20-25 kV and around 30 nA beam current. The WDS analysis was conducted in an area mode with areas ranging from $8.2 \times 10^{-3} \text{ mm}^2$ to $11 \times 10^{-3} \text{ mm}^2$. The microprobe was initially calibrated for the WDS analysis by taking counts of chemically pure elements under similar settings. These counts were used to obtain the proper settings for optimum count response while recording the elemental x-ray maps.

Scanning electron microscope pictures were taken at 800x of the as-received, unexposed, and exposed samples and were enlarged to yield a magnification of approximately 2016x. These photographs were used to measure the area fraction of porosity by the point count method. The pores were traced onto a transparency

sheet which was overlaid with a calibrated grid [11]. The number of grid points which lay within the pores were counted and totalled. This value was then divided by the total number of grid points and multiplied by 100 to obtain the area percent of porosity.

Photographs taken with an optical microscope at an effective magnification of 525x were enlarged to yield a magnification of approximately 3238x. The point count method was employed to determine the volume percentage of free aluminum in the material samples. Characterization of topographical features of the fractured surfaces was conducted using both stereo and scanning electron microscopes.

CHAPTER IV

DISCUSSION

Mechanical Testing

The parameters which were obtained from the mechanical testing were the compressive strength and the tensile strength. The mechanical testing performed was not designed to produce an absolute measure of these parameters but rather to detect any difference in the parameters between the unexposed and the exposed material. These parameters were then used to calculate the ring modulus, strain, and Weibull number. Due to lack of the as-received material in tubular form, it was impossible to evaluate the material against the shipping standard. Therefore, the material was evaluated by comparing the section of the tube directly exposed to the furnace and the section which was unexposed. Because of the large temperature difference during testing of these two sections, the purpose of the mechanical testing was to determine any change in the mechanical properties of the material due to exposure to high temperatures.

The mechanical tests which were performed to evaluate the reliability of the material were conducted on a small number (six) of samples. Also, the unavailability of as-received material in the tubular shape forced the evaluation of the unexposed material against the exposed material. Therefore, the statistical analysis conducted on a relatively small number of unexposed and exposed samples does not truly reflect absolute values for the samples and is a qualitative indication of the changes in properties of the material after exposure to the high temperature furnace

atmosphere. Also, the magnitude of the error in the results is relatively high. An estimate of the Weibull number was needed since it was not known for the material after exposure to high temperatures. There are several methods of estimating the Weibull number for a brittle material. Among these are the least squares method, the method of maximum likelihood, and the method of moments. The least squares method was chosen for this estimate due to the ease of calculation and because the probable high error resulting from the small number of test specimens made a more precise estimate insignificant.

The equation for stress, σ , is given by

$$\sigma = \frac{P}{b * \pi * r_o} * Q, \quad (1)$$

where P is the load in pounds, b is the width in inches, r_o is the outer radius, and Q is the stress magnification factor for a circular ring which is non-dimensional [12].

The probability of failure, P_f , is given by

$$P_f = 1 - \exp \left[-V \left(\frac{\sigma - \sigma_u}{\sigma_0} \right)^m \right], \quad (2)$$

where m is the Weibull number, V is the volume of the material, σ is the failure stress, σ_0 is a characteristic stress, and σ_u is the stress below which there is no probability of failure [9,13]. The parameter σ_u was taken to be zero, and the average of the stress values for a given test was used for σ_0 . Then, this equation can be put in the form

$$y = \ln \ln \left(\frac{1}{1 - P_f} \right) = \ln \frac{V}{\sigma_0^m} + m \ln \sigma. \quad (3)$$

The graph of y versus $\ln \sigma$ is a straight line of slope m which can be seen in Figures 14-15 [13].

The least squares method uses this linear model to obtain an estimate for m which is given by

$$m = \frac{\sum_i y_i \ln \sigma_i - (1/n) \sum_i y_i \sum_i \ln \sigma_i}{\sum_i (\ln \sigma_i)^2 - (1/n) (\sum_i \ln \sigma_i)^2}, \quad (4)$$

where n is the number of samples tested and y_i is given by

$$y_i = \ln \ln \left[\frac{1}{1 - [i/(n+1)]} \right]. \quad (5)$$

The stress values, σ_i , must be ordered so that $\sigma_1 \leq \sigma_2 \leq \dots \leq \sigma_n$. Then the expected value of $P_f(\sigma_i)$ is $i/(n+1)$.

Using the least squares method, the estimate of the Weibull number obtained for the tension testing was 19.39 for the unexposed material and 7.02 for the exposed material, as seen in Table 3. For the compression testing the estimate was 8.69 for the unexposed material and 5.41 for the exposed material.

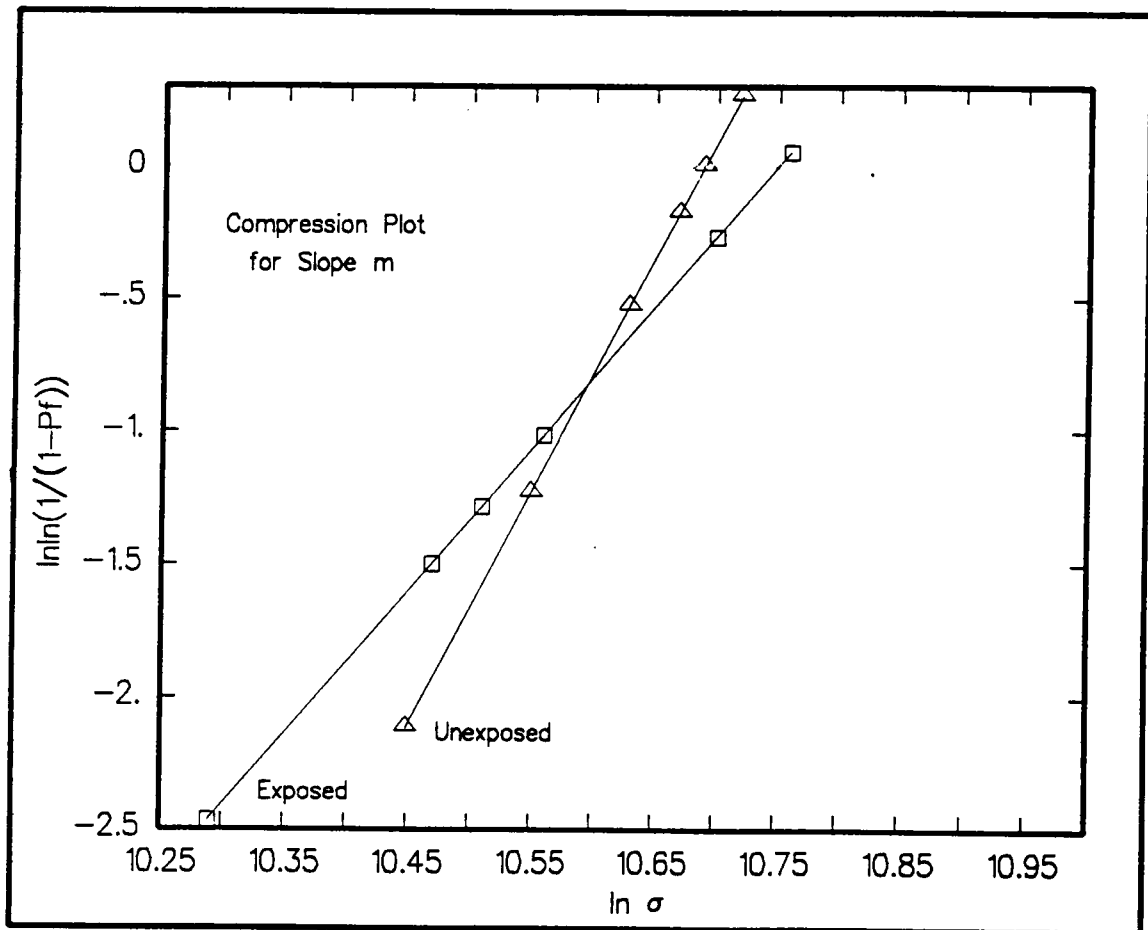


Figure 14. $\ln \ln(1/(1-P_f))$ vs. $\ln \sigma$ for compression. Slope is m.

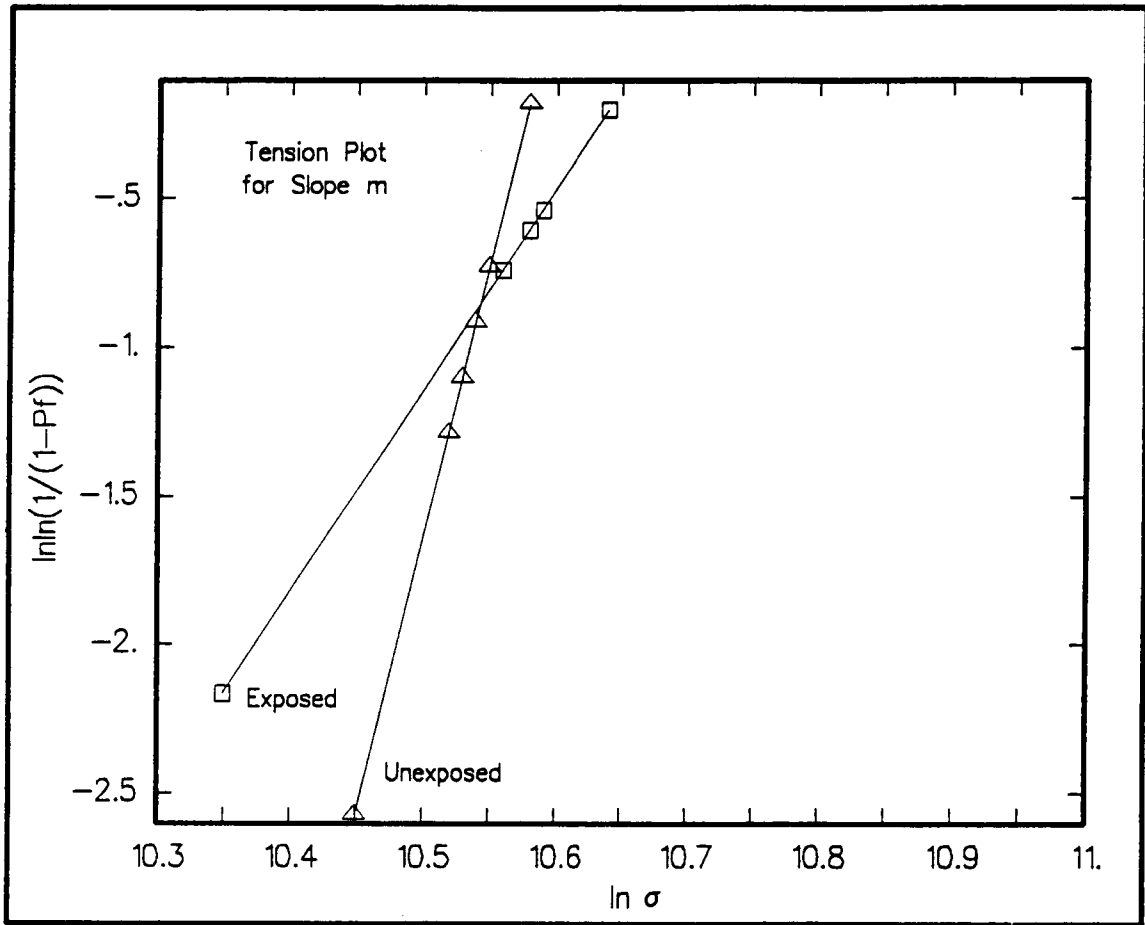


Figure 15. $\ln \ln(1/(1-P_f))$ vs. $\ln \sigma$ for tension. Slope is m.

Table 3. Calculated parameters used for Weibull number estimation.

σ (psi x 10 ³)																
Tension	Unexposed	-39.29	-38.08	-37.94	-37.45	-37.06	-34.61									
	Exposed	-41.87	-39.85	-39.52	-39.47	-38.49	-31.24									
Compression	Unexposed	34.45	38.18	41.24	43.23	44.12	45.47									
	Exposed	29.51	35.36	36.78	38.59	44.21	47.19									
P_f																
Tension	Unexposed	0.58	0.37	0.35	0.29	0.24	0.07									
	Exposed	0.58	0.46	0.44	0.43	0.38	0.10									
Compression	Unexposed	0.11	0.25	0.44	0.58	0.64	0.74									
	Exposed	0.08	0.20	0.24	0.30	0.53	0.66									
y																
Tension	Unexposed	-0.15	-0.76	-0.83	-1.09	-1.29	-2.61									
	Exposed	-0.15	-0.50	-0.56	-0.56	-0.74	-2.21									
Compression	Unexposed	-2.12	-1.22	-0.55	-0.14	0.03	0.30									
	Exposed	-2.47	-1.50	-1.28	-1.02	-0.29	0.07									
<table><tr><td>m</td><td>Tension</td><td>Compression</td></tr><tr><td>Unexposed</td><td>19.39</td><td>8.69</td></tr><tr><td>Exposed</td><td>7.02</td><td>5.41</td></tr></table>								m	Tension	Compression	Unexposed	19.39	8.69	Exposed	7.02	5.41
m	Tension	Compression														
Unexposed	19.39	8.69														
Exposed	7.02	5.41														
<table><tr><td>σ_0 (psi x 10³)</td><td>-37.91</td><td>39.86</td></tr></table>								σ_0 (psi x 10 ³)	-37.91	39.86						
σ_0 (psi x 10 ³)	-37.91	39.86														

A smaller number for the Weibull number represents a larger scatter in the data and therefore a higher probability of failure. In both the tension and compression testing, the Weibull number is less with the exposed material than for the unexposed material. This seems to indicate that the probability of failure of the material after exposure in the furnace is higher. However, this being a statistical analysis with only six samples of each material section for each test, the results must be viewed cautiously.

Equation 1 was used for both the tensile and compressive strengths. This was possible because the only difference between these two tests is that the load was negative for one of the tests and positive for the other. A negative load was used for the tensile testing and a positive load for the compression testing. The failure loads for each test may be seen in Table 4.

Table 4. Failure Loads and specifications of rings.

Tension Testing				
Unexposed Tube				
Sample #	Av. Width(mm)	Av.Thickness(mm)	Volume(mm ³)	Failure Load(lbs)
1	10.64	4.24	6842.48	282
2	10.97	4.29	7057.96	286
3	11.36	4.31	7305.61	293
4	11.13	4.18	7156.06	268
5	10.31	4.19	6631.82	282
6	10.94	4.11	7038.67	289
		Average =	7005.43 ± 217.10	283.33 ± 7.87
Exposed Tube				
1	10.90	4.62	7009.72	237
2	11.18	4.75	7288.22	307
3	9.97	4.70	6409.90	267
4	10.53	4.78	6774.94	307
5	10.17	4.64	6538.55	282
6	11.02	4.75	7085.30	303
		Average =	6834.44 ± 308.46	283.83 ± 25.56
Compression Testing				
Unexposed Tube				
1	11.13	4.26	7160.88	-335
2	11.31	4.20	7275.06	-358
3	11.08	4.21	7125.50	-318
4	10.84	4.24	6971.13	-288
5	10.58	4.22	6807.10	-325
6	9.43	4.36	6062.55	-226
		Average =	6900.37 ± 403.03	-308.33 ± 42.31
Exposed Tube				
1	11.54	4.86	7419.79	-355
2	11.05	4.61	7107.81	-363
3	12.54	4.81	8064.64	-321
4	1.05	4.66	7107.81	-227
5	10.61	4.63	6824.79	-285
6	11.58	4.95	7448.73	-285
		Average =	7328.93 ± 390.69	-306 ± 46.54

The equation for Q is given by

$$\begin{aligned}
 Q(\psi, r) = & 2A_0 - B_0 \left(\frac{r}{r_o}\right)^{-2} + \sum_{n=2,4,6}^{\infty} [2A_n(n+1)(n+2) \left(\frac{r}{r_o}\right)^n + \\
 & 2n(n-1)B_n \left(\frac{r}{r_o}\right)^{n-2} + \\
 & 2n(n+1)C_{nx}^n \left(\frac{r}{r_o}\right)^{-n-2} + \\
 & 2(n-1)(n-2)D_{nx}^{n-2} \left(\frac{r}{r_o}\right)^{-n}] n \cos(n\psi)
 \end{aligned} \tag{6}$$

where

$$A_0 = \frac{1}{2(1-x^2)}, \tag{7}$$

$$B_0 = -\frac{x^2}{1-x^2}, \tag{8}$$

$$A_n = \frac{1}{2R_n} \left(\frac{1-x^{2n}}{n} - \frac{1-x^{2n-2}}{n+1} \right), \tag{9}$$

$$B_n = \frac{1}{2R_n} \left(\frac{1-x^{2n}}{n} - \frac{1-x^{2n+2}}{n-1} \right), \tag{10}$$

$$C_n = \frac{x^n}{2R_n} \left(-\frac{1-x^{2n}}{n} + \frac{1-x^{2n-2}}{n+1} x^2 \right), \tag{11}$$

$$D_n = \frac{x^n}{2R_n} \left(-\frac{1-x^{2n}}{n} x^2 + \frac{1-x^{2n+2}}{n-1} \right), \tag{12}$$

$$R_n = (1-x^{2n})^2 - n^2 x^{2n-2} (1-x^2)^2, \tag{13}$$

ψ is the angle from the vertical axis, r_o is the outer radius of the ring, r_i is the inner radius of the ring, r is the radius of interest, and x is the ratio r_i/r_o [12].

The equation for stress breaks down as r approaches r_o . Therefore, the values used for the stress magnification factor were calculated at $r=r_i$. The values of stress magnification factors can be seen in Table 5. The corresponding table of stress values for both the compression testing and the tensile testing can be seen in Tables 6-7. The stress values for $r=20.5\text{mm}$ were used for calculations. The computer code used to generate the stress magnification values is found in Appendix A. If the ring were to be mapped out for either the compression test or the tensile test according to the stress values at particular radii and angles, a positive stress would indicate compression stressing in the ring at a particular point, and a negative stress would indicate tensile stressing. So, the point in the table where the stress changes from positive to negative represents a change from compression to tensile stressing. This is demonstrated in Figure 16.

The ring modulus is σ/ϵ or stress/strain. The strain is found by dividing the change in dimension by original dimension. Since the tests were uniaxial diametrical loading of a ring, the inside diameter was used as the original dimension for the calculation of the strain.

The ring modulus was calculated for both the compression and tension testing. As can be seen in Tables 8-9, the average ring modulus for the tension testing was -3.47e^6 and -3.71e^6 psi for the unexposed and exposed material respectively. The average ring modulus for the compression testing was 7.78e^6 and 5.24e^6 psi for the unexposed and exposed material respectively.

Table 5. Calculated stress magnification factors, Q.

r (mm)	$\psi=0,\pi$	$\psi=\pi/20$	$\psi=\pi/10$	$\psi=3\pi/20$	$\psi=\pi/5$
20.5	-174.88	-135.59	-89.50	-47.09	-7.94
21	-128.09	-101.23	-66.69	-34.54	-4.87
21.5	-87.14	-68.44	-44.88	-22.48	-1.81
22	-50.15	-37.10	-24.03	-10.91	1.20
22.5	-15.91	-7.77	-4.10	0.17	4.12
23	16.42	20.20	14.96	10.79	6.93
23.5	48.08	46.45	33.14	21.02	9.66
24	92.78	67.86	49.65	31.98	12.88
24.5	526.13	38.02	52.50	60.46	23.76
	$\psi=\pi/4$	$\psi=3\pi/10$	$\psi=7\pi/20$	$\psi=2\pi/5$	$\psi=9\pi/20$
20.5	26.98	56.80	80.79	98.36	109.08
21	21.58	44.17	62.35	75.67	83.79
21.5	16.62	32.36	45.02	54.30	59.96
22	12.00	21.22	28.64	34.07	37.39
22.5	7.64	10.65	13.69	14.84	15.92
23	6.49	0.55	-1.80	-3.53	-4.59
23.5	-0.55	-9.19	-16.06	-21.12	-24.28
24	-5.07	-19.24	-29.50	-37.43	-43.34
24.5	-16.80	-37.89	-38.72	-44.27	-63.79
	$\psi=\pi/2$	$\psi=11\pi/20$	$\psi=3\pi/5$	$\psi=13\pi/20$	$\psi=7\pi/10$
20.5	112.68	109.08	98.36	80.79	56.80
21	86.52	83.79	75.67	62.35	44.17
21.5	61.86	59.96	54.30	45.02	32.36
22	38.50	37.39	34.07	28.64	21.22
22.5	16.29	15.92	14.84	13.69	10.35
23	-4.95	-4.59	-3.53	-1.80	0.55
23.5	-25.36	-2.43	-21.12	-16.06	-9.19
24	-45.64	-43.34	-37.43	-29.50	-19.24
24.5	-72.67	-63.79	-44.27	-38.72	-37.89
	$\psi=3\pi/4$	$\psi=4\pi/5$	$\psi=17\pi/20$	$\psi=9\pi/10$	$\psi=19\pi/20$
20.5	26.98	-7.94	-47.09	-89.50	-135.59
21	21.58	-4.87	-34.54	-66.69	-101.23
21.5	16.62	-1.81	-22.48	-44.88	-68.44
22	12.00	1.20	-10.91	-24.03	-37.10
22.5	7.64	4.12	0.17	-4.10	-7.77
23	6.49	6.93	10.79	14.96	20.20
23.5	-0.55	9.66	21.02	33.14	46.45
24	-5.07	12.88	31.98	49.65	67.86
24.5	-16.80	23.76	60.46	52.50	38.02

Table 6. Calculated tension failure stress values in psi x 10³.

Unexposed Tube								
r (mm)	Sample #						AVE.	Std. Dev
	1	2	3	4	5	6		
20.5	-38.08	-37.44	-37.06	-34.61	-39.29	-37.94	-37.41	1.43
21	-27.89	-27.42	-27.14	-25.35	-28.78	-27.79	-27.40	1.05
21.5	-18.98	-18.66	-18.47	-17.25	-19.58	-18.91	-18.64	0.71
22	-10.92	-10.74	-10.63	-9.92	-11.27	-10.88	-10.73	0.41
22.5	-3.47	-3.41	-3.37	-3.15	-3.58	-3.45	-3.40	0.13
23	3.58	3.52	3.48	3.25	3.69	3.56	3.51	0.13
23.5	10.47	10.30	10.19	9.52	10.80	10.43	10.28	0.39
24	20.20	19.86	19.66	18.36	20.85	20.13	19.84	0.76
24.5	114.57	112.65	111.49	104.11	118.21	114.14	112.53	4.30
						AVE.	5.40	
Exposed Tube								
r (mm)	Sample #						AVE.	Std. Dev
	1	2	3	4	5	6		
20.5	-31.24	-39.47	-38.49	-41.87	-39.85	-39.52	-38.41	3.36
21	-22.88	-28.90	-28.19	-30.67	-29.19	-28.94	-28.13	2.46
21.5	-15.57	-19.66	-19.18	-20.86	-19.86	-19.69	-19.14	1.67
22	-8.96	-11.32	-11.04	-12.01	-11.43	-11.33	-11.01	0.96
22.5	-2.84	-3.59	-3.50	-3.81	-3.63	-3.60	-3.49	0.31
23	2.93	3.71	3.61	3.93	3.74	3.71	3.61	0.32
23.5	8.59	10.85	10.58	11.51	10.96	10.87	10.56	0.92
24	16.57	20.94	20.42	22.21	21.14	20.96	20.38	1.78
24.5	93.99	118.73	115.80	125.97	119.90	118.89	115.55	10.11
						AVE.	5.55	

Table 7. Calculated compression failure stress values in psi x 10³.

Unexposed Tube								
r (mm)	Sample #						AVE.	Std. Dev
	1	2	3	4	5	6		
20.5	43.23	45.47	41.24	38.18	44.12	34.45	41.11	3.78
21	31.66	33.30	30.20	27.96	32.31	25.23	30.11	4.44
21.5	21.54	22.66	20.55	19.02	21.98	17.16	20.49	1.88
22	12.40	13.04	11.83	10.95	12.65	9.88	11.79	1.08
22.5	3.93	4.14	3.75	6.47	4.01	3.13	3.74	1.16
23	-4.06	-4.27	-3.87	-3.58	-4.14	-3.23	-3.86	0.36
23.5	-11.89	-12.50	-11.34	-10.50	-12.13	-9.47	-11.30	1.04
24	-22.93	-24.12	-21.88	-20.25	-23.41	-18.27	-21.81	2.01
24.5	-130.05	-136.80	-124.07	-114.85	-132.73	-103.63	-123.69	11.36
						AVE. = -5.94		

Exposed Tube								
r (mm)	Sample #						AVE.	Std. Dev
	1	2	3	4	5	6		
20.5	44.21	47.19	36.78	29.51	38.59	35.36	38.61	5.80
21	32.38	34.56	26.94	21.61	28.26	25.90	28.28	4.25
21.5	22.03	23.52	18.33	14.70	19.23	17.62	19.24	2.89
22	12.68	13.53	10.55	8.46	11.07	10.14	11.07	1.66
22.5	4.02	4.29	3.35	2.69	3.51	3.22	3.51	0.52
23	-4.15	-4.43	-3.45	-2.77	-3.62	-3.32	-3.63	0.54
23.5	-12.16	-12.98	-10.11	-8.11	-10.61	-9.72	-10.61	1.60
24	-23.45	-25.04	-19.51	-15.66	-20.47	-18.76	-20.48	3.08
24.5	-133.01	-141.98	-110.65	-88.78	-116.09	-106.37	-116.15	17.45
						AVE. = -5.57		

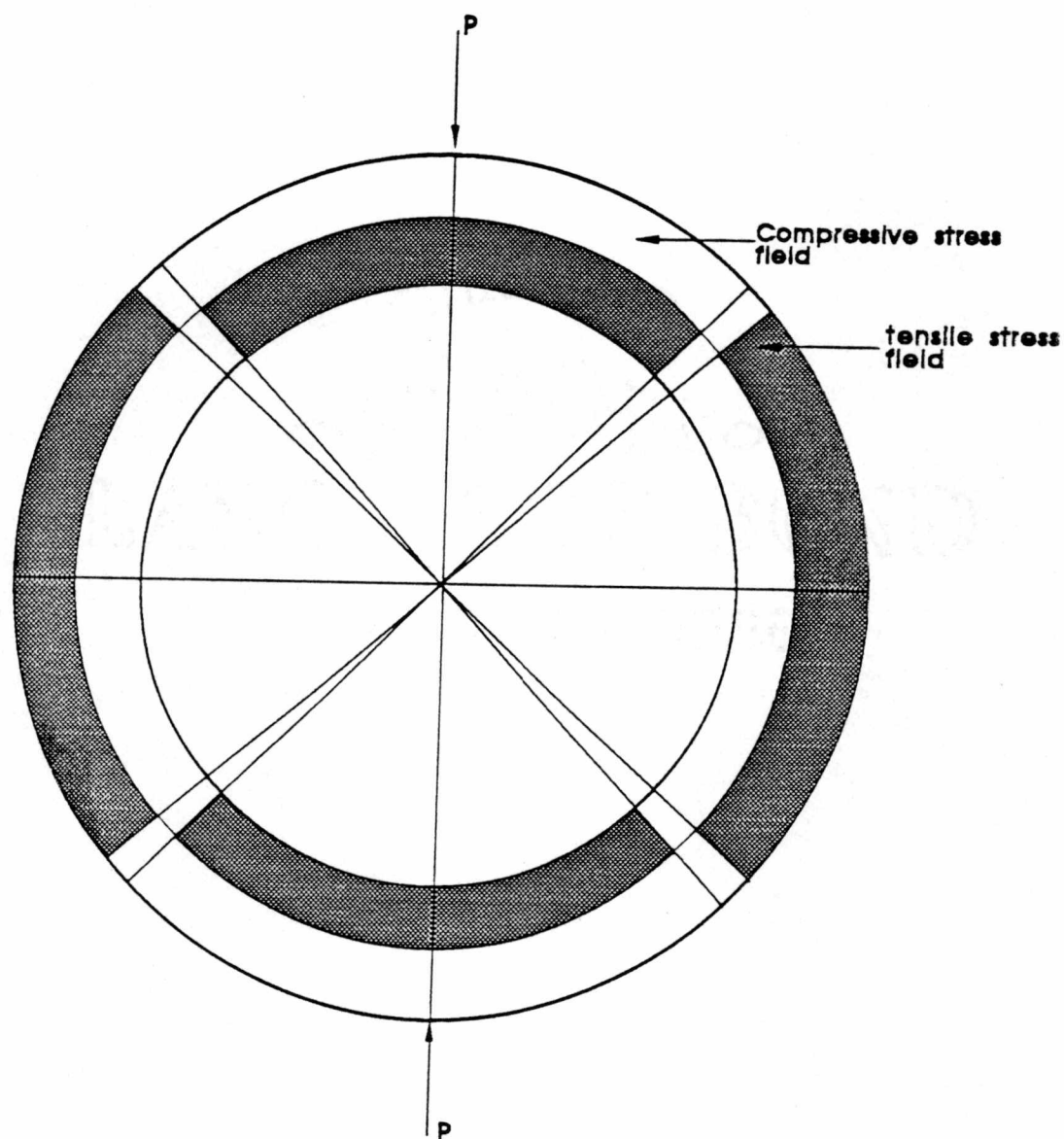


Figure 16. Schematic showing compressive and tensile stresses in a ring under uniaxial diametrical compression [12].

Table 8. Ring Modulus for tension testing.

Unexposed						
Sample #	# Blocks on recording paper	Time to failure (min)	Crosshead Displacement ($\times 10^{-3}$ in)	Failure stress at inner radius (psi $\times 10^3$)	Failure strain	Ring Modulus (psi $\times 10^5$)
1	37	0.74	14.80	-38.08	0.0091	-42.06
2	35	0.70	14.00	-37.45	0.0086	-43.62
3	39	0.78	15.60	-37.06	0.0096	-38.71
4	65	1.30	26.00	-34.61	0.0159	-21.83
5	42	0.84	16.80	-39.29	0.0102	-38.32
6	66	1.32	26.40	-37.94	0.0160	-23.65
AVE. =	47	0.95	18.93	-37.41	0.0116	-34.70
StDev =	13.03	0.26	5.21	1.43	0.0031	8.67
Exposed						
Sample #	# Blocks on recording paper	Time to failure (min)	Crosshead Displacement ($\times 10^{-3}$ in)	Failure stress at inner radius (psi $\times 10^3$)	Failure strain	Ring Modulus (psi $\times 10^5$)
1	33	0.66	13.20	-31.24	0.0082	-37.98
2	45	0.90	18.00	-39.47	0.0113	-34.97
3	32	0.64	12.80	-38.49	0.0080	-48.06
4	60	1.20	24.00	-41.87	0.0151	-27.78
5	53	1.06	21.20	-39.85	0.0132	-30.13
6	36	0.72	14.40	-39.52	0.0090	-43.75
AVE. =	43	0.86	17.27	-38.41	0.0108	-37.11
StDev =	10.51	0.21	4.20	3.36	0.0027	7.13

Table 9. Ring Modulus for compression testing.

Unexposed						
Sample #	# Blocks on recording paper	Time to failure (min)	Crosshead Displacement ($\times 10^{-3}$ in)	Failure stress at inner radius (psi $\times 10^3$)	Failure strain	Ring Modulus (psi $\times 10^5$)
1	21.24	0.42	8.50	43.23	0.0052	83.17
2	22.50	0.45	9.00	45.47	0.0055	82.39
3	21.60	0.43	8.64	41.24	0.0053	77.77
4	25.00	0.50	10.00	38.18	0.0061	62.60
5	21.37	0.43	8.55	44.12	0.0052	84.56
6	18.60	0.37	7.44	34.45	0.0045	76.48
AVE. =	21.72	0.43	8.69	41.11	0.0053	77.78
StDev =	1.89	0.04	0.76	3.78	0.0005	7.40
Exposed						
Sample #	# Blocks on recording paper	Time to failure (min)	Crosshead Displacement ($\times 10^{-3}$ in)	Failure stress at inner radius (psi $\times 10^3$)	Failure strain	Ring Modulus (psi $\times 10^5$)
1	30.28	0.61	12.11	44.21	0.0075	58.57
2	26.40	0.53	10.56	47.19	0.0066	71.28
3	30.00	0.60	12.00	36.78	0.0075	48.99
4	26.28	0.53	10.51	29.51	0.0066	44.70
5	34.50	0.69	13.80	38.59	0.0086	44.82
6	30.45	0.61	12.18	35.36	0.0076	46.28
AVE. =	29.65	0.60	11.86	38.61	0.0074	52.44
StDev =	2.79	0.05	1.12	5.80	0.0007	9.66

The difference in the ring modulus from the unexposed to the exposed material does not change as much in tension testing as it does in the compression testing. This may be explained partly by material properties. In general, ceramics are brittle materials which are weaker in tension than in compression. A change in the tensile strength is less likely to be detected due to the small values and lack of measurement precision. However, in compression, ceramics are generally very strong, giving larger values. Therefore, any change in the material which produces a change in the compressive strength is more likely to be noticed.

A summary of the parameters obtained may be seen in Table 10. The change in the Weibull number is significant for both tension and compression. However, the change in the ring modulus and strength is much more prevalent in the compression testing than in the tension testing.

Table 10. Summary of parameters from mechanical testing.

Tension	Ring Modulus (GPa)	Fracture Strength(MPa)
Unexposed	-23.92	257.9
Exposed	-25.59	264.8
Compression		
Unexposed	53.63	283.5
Exposed	36.16	266.2

Microscopy

The surface analysis was performed in order to detect and evaluate any microscopic changes in the material due to exposure to the furnace conditions. For the surface analysis, a small coupon of as-received material was obtained, so the material could be evaluated against the shipping standard. Each examination was performed on samples of the three material conditions: as-received, unexposed, and exposed.

The material was evaluated in a Cameca micro-probe using EDS and WDS analyses and x-ray mapping. The results of the EDS on all three materials confirmed that the major elements present were silicon and aluminum. Even though the micro-probe is equipped with a light element detector for energy dispersive x-ray analysis (EDS), in many instances, sample self-absorption effects significantly reduced the intensity of oxygen and carbon peaks. No other element was detected in a quantity sufficient to indicate anything out of the ordinary in any of the materials.

When the micro-probe was utilized for the WDS analysis and properly calibrated, detection of carbon was possible, although oxygen was still out of the precision range. Using WDS, x-ray maps of silicon, aluminum, and carbon were produced for each of the materials. These can be seen in Figures 17-28 with a corresponding point of reference marked for orientation. The silicon carbide particles can clearly be seen surrounded by the alumina matrix. However, it was not possible to separate out the alumina and the free aluminum with the micro-probe analysis.

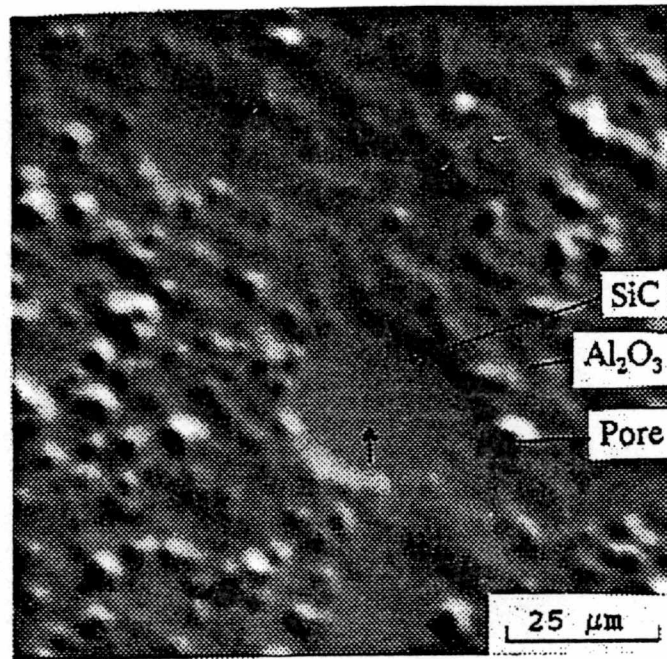


Figure 17. Photograph of x-ray map area in as-received sample.

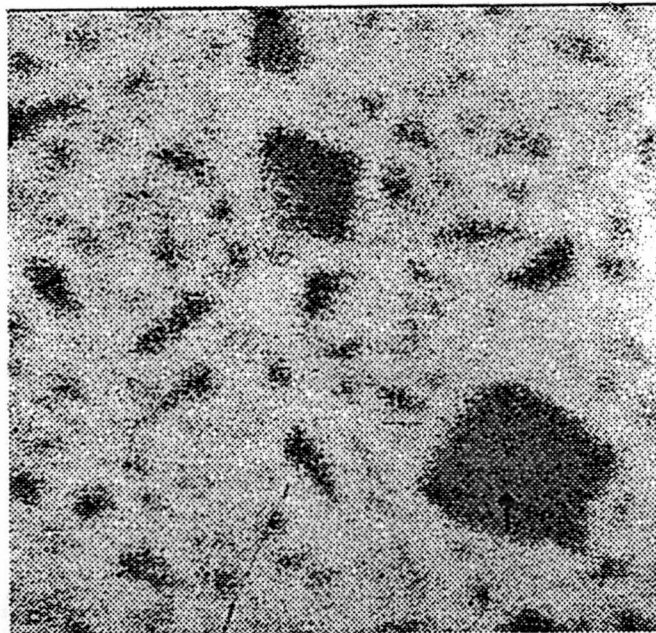


Figure 18. X-ray map of aluminum in as-received sample.

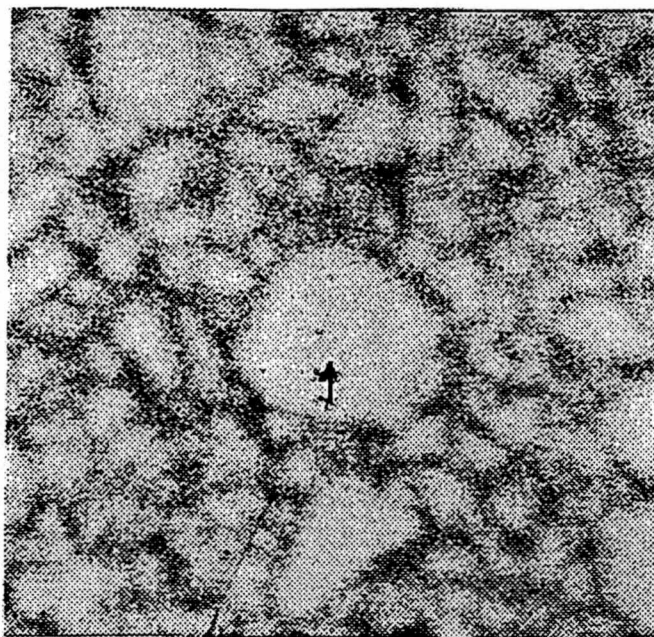


Figure 19. X-ray map of silicon in as-received sample.

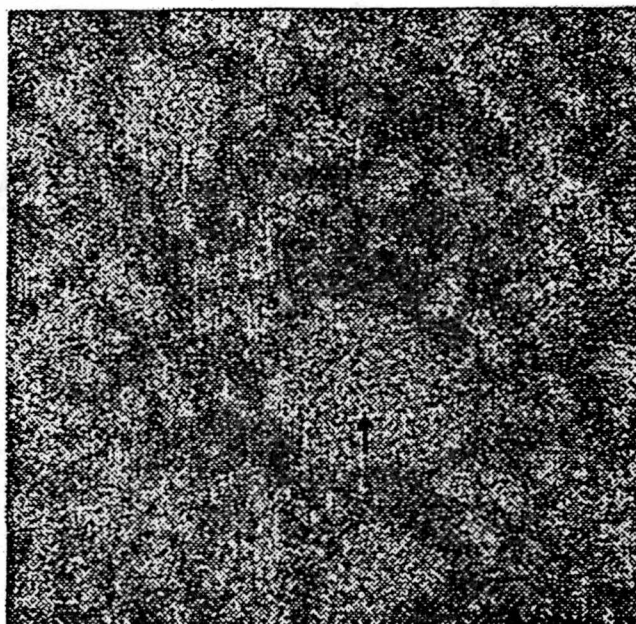


Figure 20. X-ray map of carbon in as-received sample.

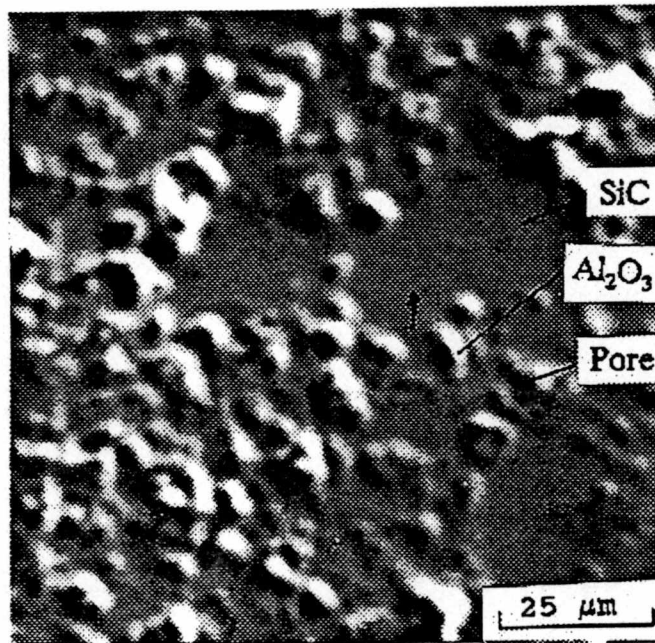


Figure 21. Photograph of x-ray map area in unexposed sample.

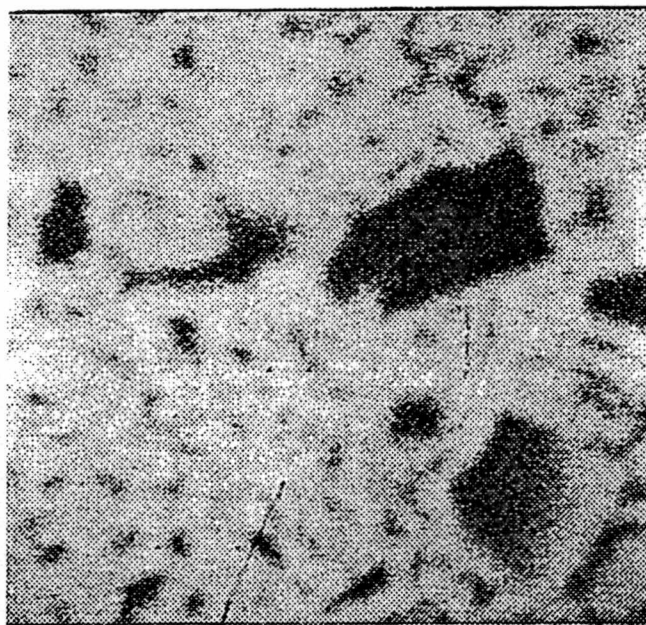


Figure 22. X-ray map of aluminum in unexposed sample.

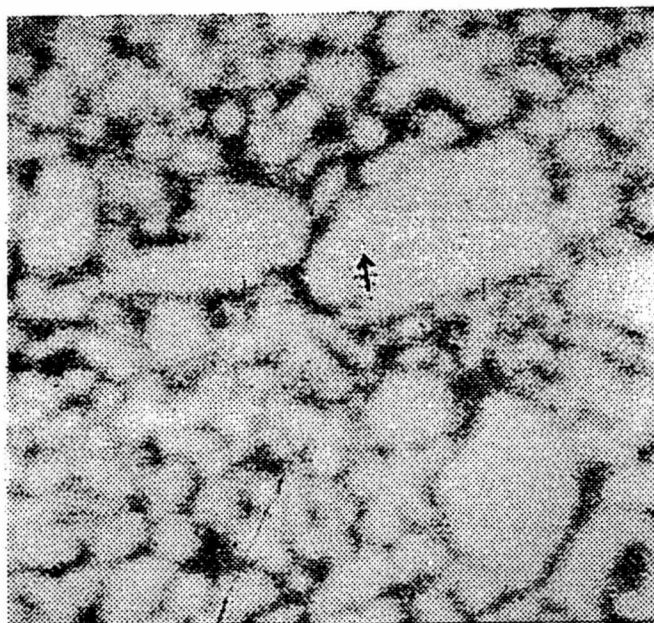


Figure 23. X-ray map of silicon in unexposed sample.

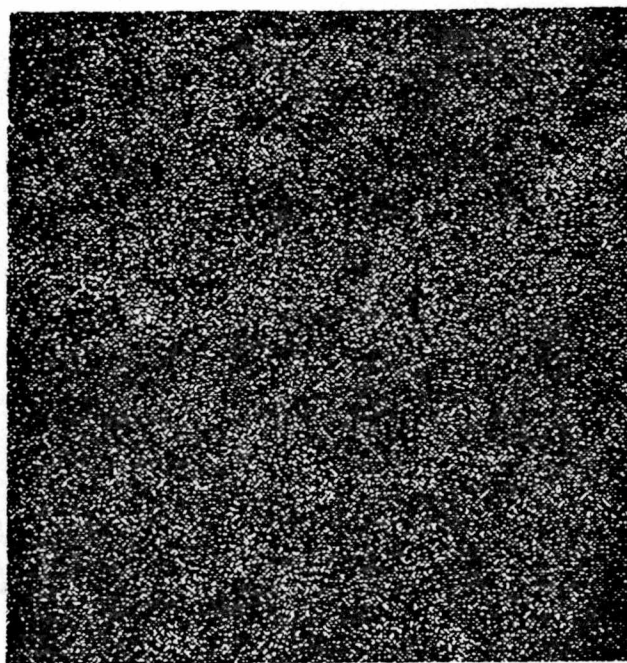


Figure 24. X-ray map of carbon in unexposed sample.

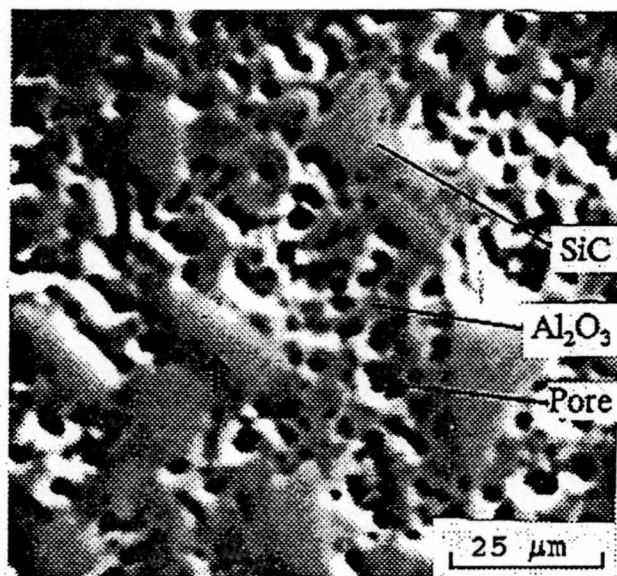


Figure 25. Photograph of x-ray map area in exposed sample.

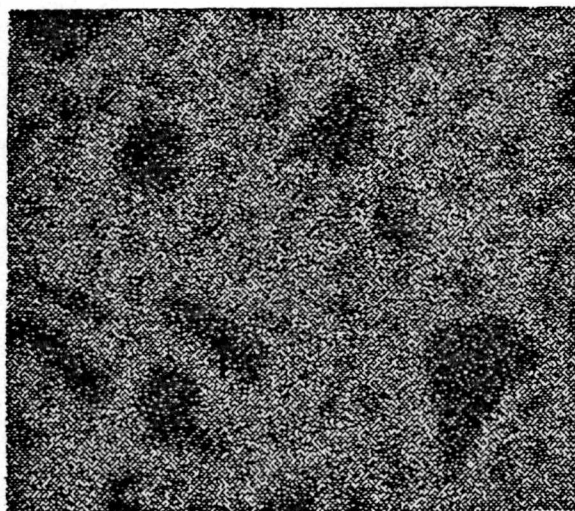


Figure 26. X-ray map of aluminum in exposed sample.

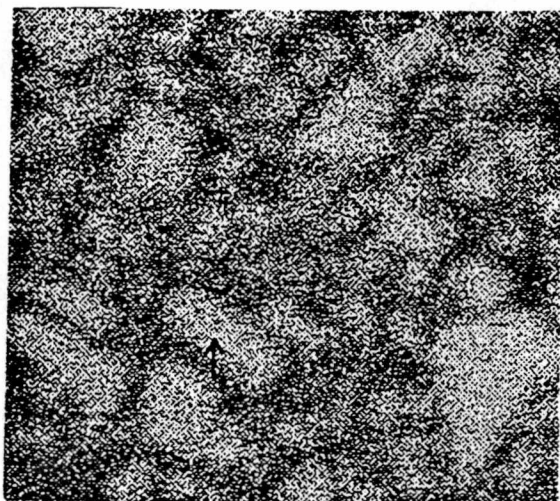


Figure 27. X-ray map of silicon in exposed sample.

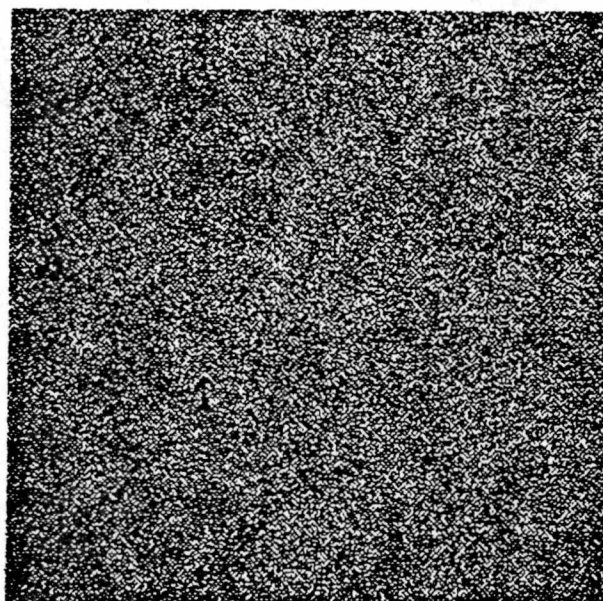


Figure 28. X-ray map of carbon in exposed sample.

The slag was also evaluated in the micro-probe, although not to the extent of the ceramic material. There were two types of slag. One was a hard greenish material and the other a yellowish material which broke into a coarse powder very easily. The major elements in both slag types detected using EDS analysis were potassium (K), silicon (Si), aluminum (Al) and calcium (Ca) (see Figure 29). The height of the peaks is not significant except to show what elements were present. When the yellowish slag was evaluated, a broad peak extending over a wide range of eV values (~10-19) was seen (Figure 30). Such a broad peak is an indication of either extremely fine grains or an amorphous structure comprised of various elements. The chemistry lab evaluated a sample of quenched slag (a combination of slag types from the furnace) which is of similar composition using weight analysis techniques for various elements. The list of these elements is given in Table 11. The % element column represents the amount of the element detected. The % oxide column represents the amount of the element in the most stable oxide form. It was assumed that all elements were in the most stable oxide form.

The slag had accumulated to varying thicknesses on the tube surface (Figure 31), and it appeared to adhere mechanically, since very little force was required to remove it from the tube. Once removed, the surface of the tube was visually inspected and appeared virtually identical to the unexposed surface indicating that no reaction had taken place between the slag and the tube. In addition, the dimensions of the tube had not changed significantly (Table 4) from the unexposed to the exposed sections.

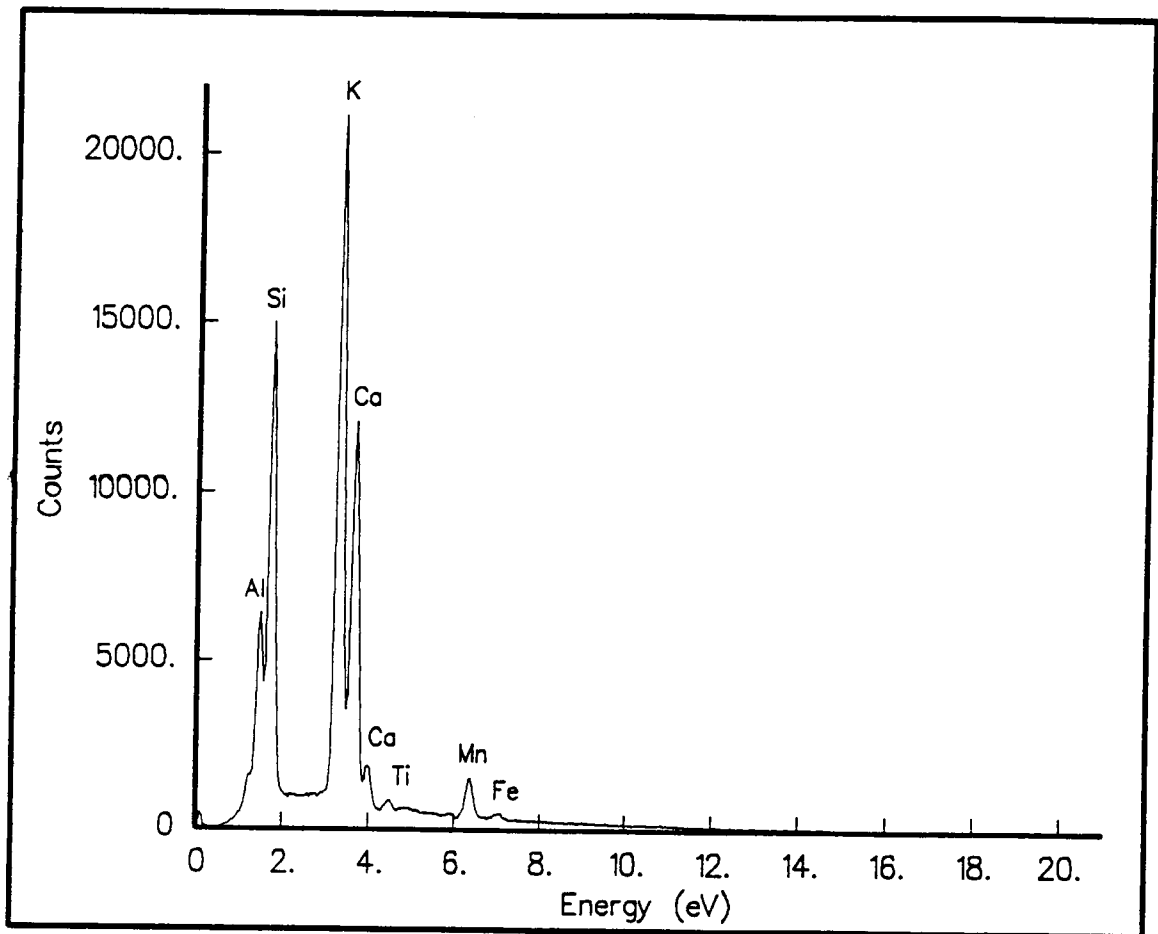


Figure 29. EDS plot of a typical slag.

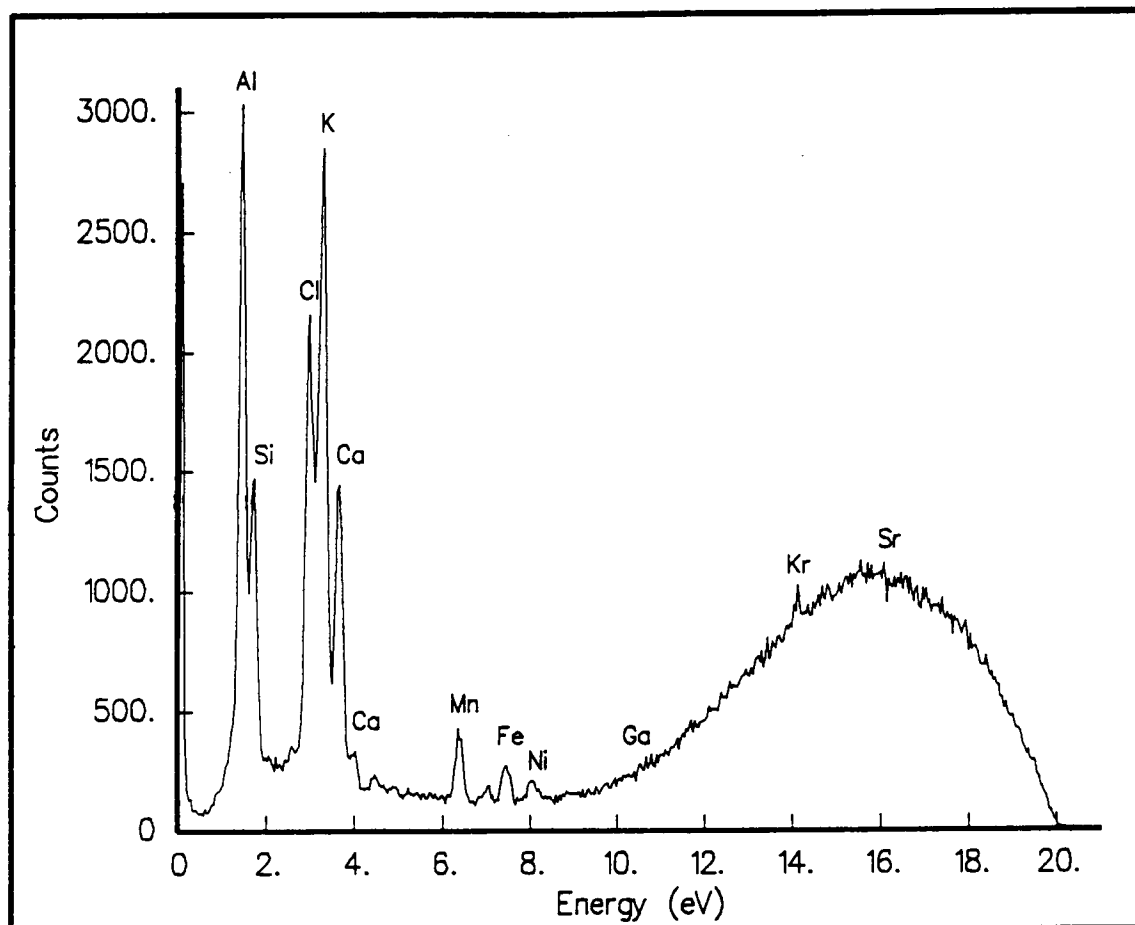


Figure 30. EDS plot of a yellowish slag.

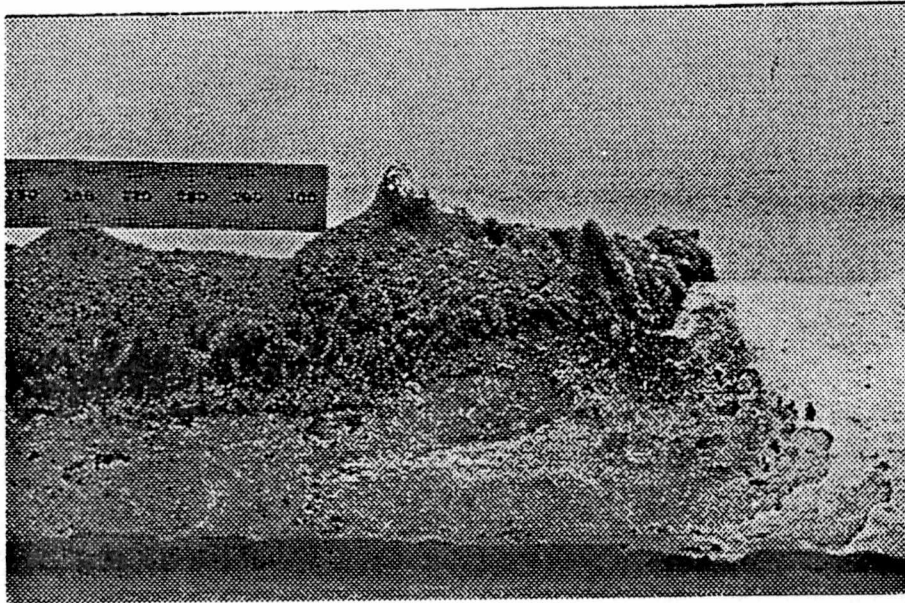


Figure 31. Combination of slag types on tube surface showing varying thicknesses of deposits.

Table 11. Chemistry Lab elemental analysis of quenched slag.

Elemental Analysis of Quenched Slag		
Element	% Element	% Oxide
Aluminum	9.77	18.47
Calcium	14.43	20.19
Chromium	0.05	0.07
Iron	6.63	5.19
Potassium	5.07	6.10
Magnesium	1.80	2.99
Sodium	0.19	0.26
Silica	21.30	45.56
Titanium	0.47	0.78
Total =		99.61

The micro-probe analysis also showed no depth of penetration of the slag and no new elements on the surface. Therefore, it can be concluded that no reaction occurred between the slag and the material of the tube.

Observation of the porosity in as-received, unexposed, and exposed samples was conducted using optical and scanning electron microscopy. In the as-received material, the porosity was very low. The porosity in the unexposed material appeared to increase as compared to the as-received, and the exposed material had even more porosity than the other two. This would seem to indicate an increase in material porosity when exposed to high temperatures.

The assumption was made that the porosity was isotropic throughout the material. This assumption seemed to be acceptable for the unexposed and exposed samples. However, the as-received porosity seemed to vary from quite evident to

almost none. Although the material was examined in several places and the porosity appeared to be fairly uniform, measurements were taken with one photograph for each sample of the materials. The results of these measurements showed that the porosity in the as-received material varied from approximately 1 volume % in some areas to greater than 5 volume %. The porosity of the unexposed material increased to approximately 18 volume %, and the porosity of the exposed material increased even further to approximately 23 volume %, as seen in Table 12. Area was calculated in grid points from the grid overlay and then translated to μm^2 .

Table 12. Porosity measurements of as-received, unexposed, and exposed samples.

Total Area = 37,541 gridpts. = $8961\mu\text{m}^2$			
	As-Received	Unexposed	Exposed
Area (gridpts)	732	6801	8726
Area (μm^2)	174.73	1623.40	2082.90
# Pores	59	202	275
% Area	1.95	18.12	23.24
Calculated Mean Pore Size (gridpts)	12.41	33.67	31.73
Calculated Mean Pore Size (μm^2)	2.96	8.04	7.57

This indicates that the porosity of the material increases when exposed to high temperatures. There are several possible explanations for this occurrence. First, as the temperature of the material increases, more of the free aluminum is likely to oxidize. As this happens, porosity will increase since the density of alumina (3980 kg/m^3) is higher than that of aluminum (2699 kg/m^3) [6,7,8]. Also, as the melting temperature of aluminum is reached ($\approx 660^\circ\text{C}$) and the aluminum melts, it is possible for it to move through the material in the same way the material was originally formed, through capillary action. Through this process, the aluminum can reach a surface of the tube and either drip off or boil off depending on the temperature of the environment. This process would obviously increase the porosity.

In order to examine the aluminum content, photographs were taken of each of the samples with an optical microscope and enlarged (see Figures 32-34). These pictures were then overlaid with a grid, and the aluminum percentage was evaluated in the same way as the porosity. The measurements were taken from two areas on each sample and averaged. The aluminum content was lower in the exposed material than in the unexposed or as-received. The unexposed was very close to the as-received (see Table 13). This seems to indicate that the temperature of the unexposed section of the tube was not high enough to cause loss of aluminum. A plot showing the aluminum content and porosity content can be seen in Figure 35.

Another possibility for the increase in porosity is a process called coalescing whereby the existing pores begin to grow together forming larger pores. The other

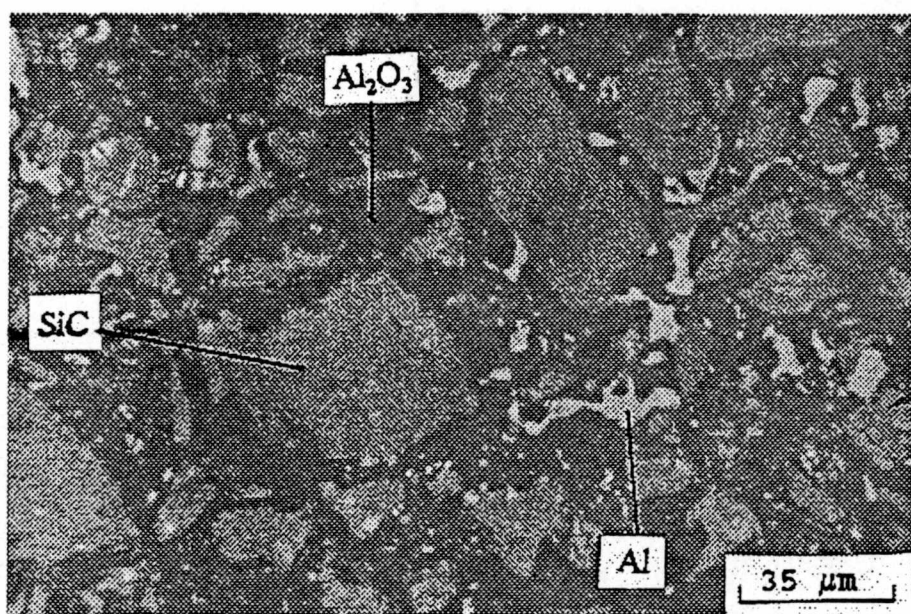


Figure 32. Optical micrograph of as-received sample.

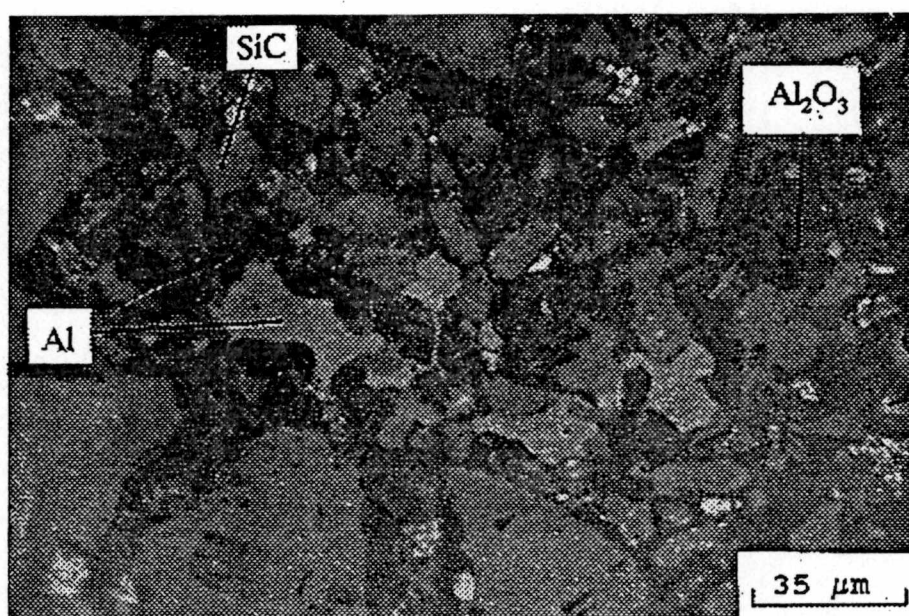


Figure 33. Optical micrograph of unexposed sample.

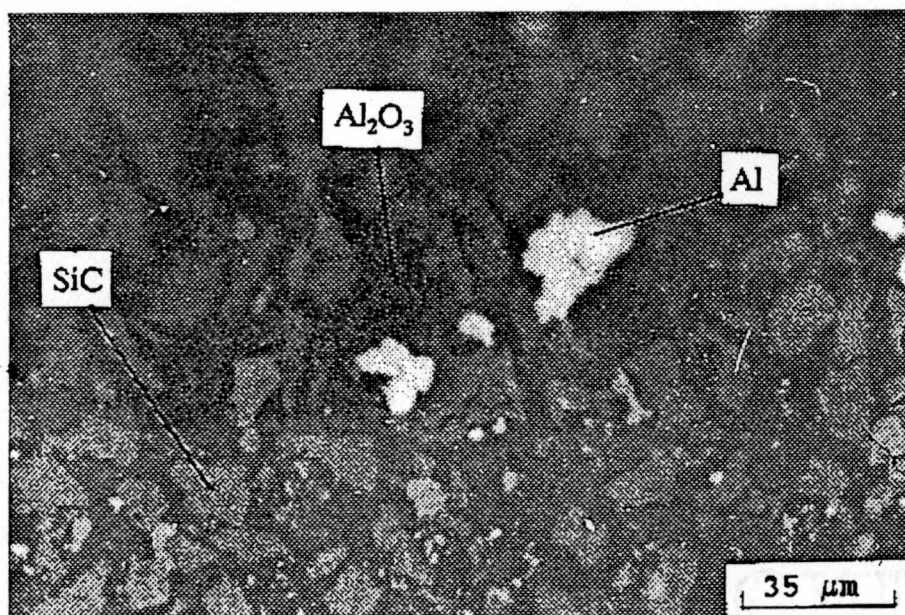


Figure 34. Optical micrograph of exposed sample.

Table 13. Aluminum measurements for as-received, unexposed and exposed samples.

Total Area = 22,351 gridpts. = 2082.54 μm^2						
	As-Received 1	As Received 2	Unexposed 1	Unexposed 2	Exposed 1	Exposed 2
Area (gridpts)	1227	1601	1388	1303	728	960
Area (μm^2)	114.36	149.21	129.36	121.44	67.85	89.47
# Pools	105	37	12	25	12	20
% Volume	5.49	7.16	6.21	5.83	3.26	4.30
Calculated Mean Pool Size (gridpts)	11.69	43.27	115.67	52.12	60.67	48.00
Calculated Mean Pool Size (μm^2)	1.09	4.03	10.78	4.86	5.65	4.47

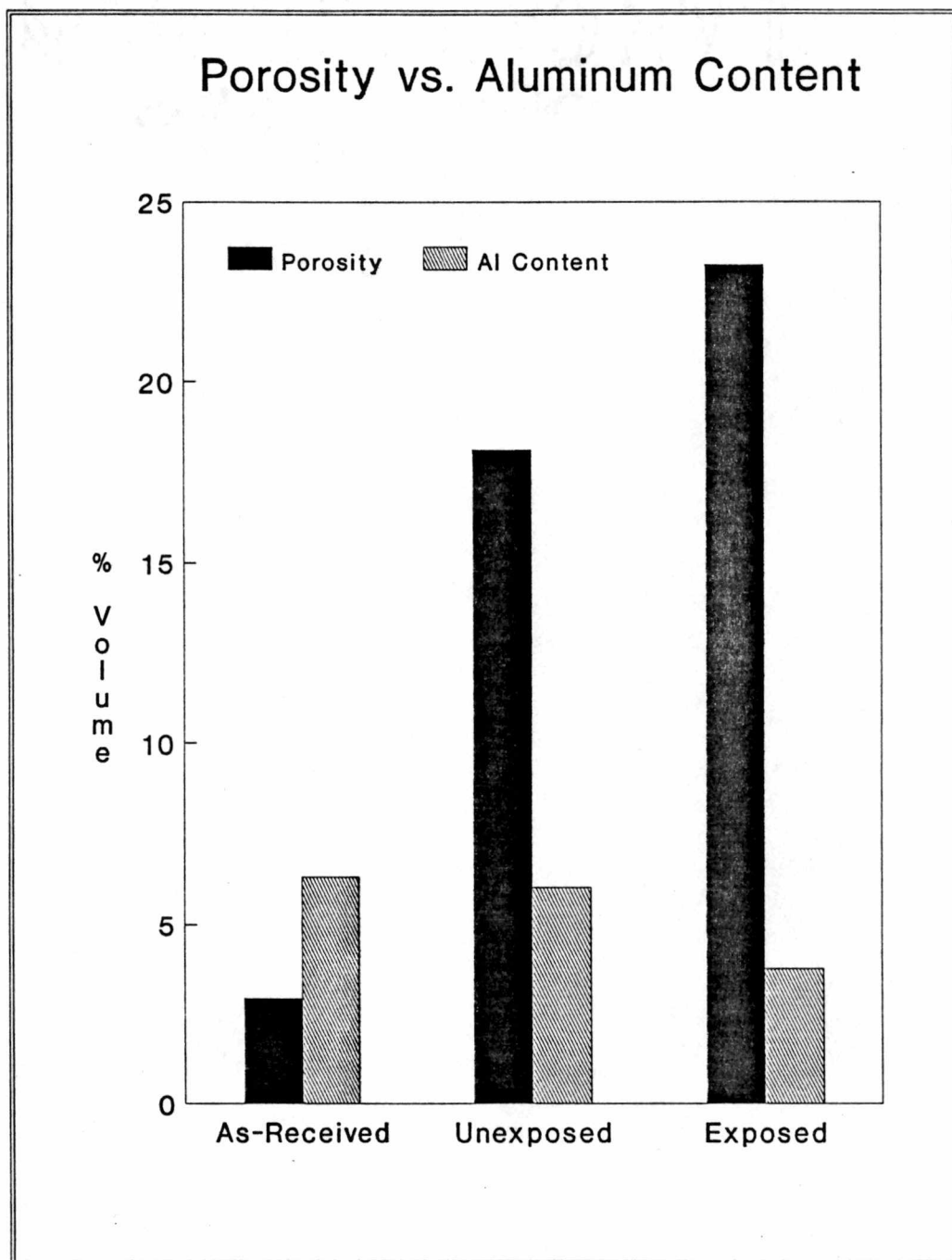


Figure 35. Plot of Al content and porosity content as measured.

possibility is that the material is actually deteriorating chemically. Silicon has a strong affinity towards oxygen thereby forming a thin layer of silicon dioxide (SiO_2) on the surface of the silicon carbide. When exposed to high temperatures, such a SiO_2 layer could react with Al_2O_3 forming a low melting eutectic (1587°C) according to the phase diagram illustrated in Figure 36. This low melting eutectic could in turn initiate any one or all of the previously mentioned mechanisms and result in the formation of pores.

A high possibility is a combination of processes. If the as-received material started with 10 volume % porosity and 10 volume % free aluminum, then if all the aluminum were removed leaving pores, that would yield a maximum porosity of 20 volume %, which is below the 23 volume % measured in the exposed material. Also, because there is still free aluminum in the exposed material, the porosity cannot be caused solely by the oxidation or vitrification of aluminum. Therefore, it is highly likely that the combination of two or more of these processes worked in the formation of the porosity.

The results of the microscopy indicate that no reactions with the slag took place and no corrosion or erosion occurred. The only property of the material which appeared to have changed significantly was the porosity. The porosity had increased from the as-received to the unexposed and again from the unexposed to the exposed. The exposed material still had free aluminum indicating that the porosity increase was caused by the combination of several processes including oxidation, flowing, or vitrification of aluminum, coarsening, and deterioration.

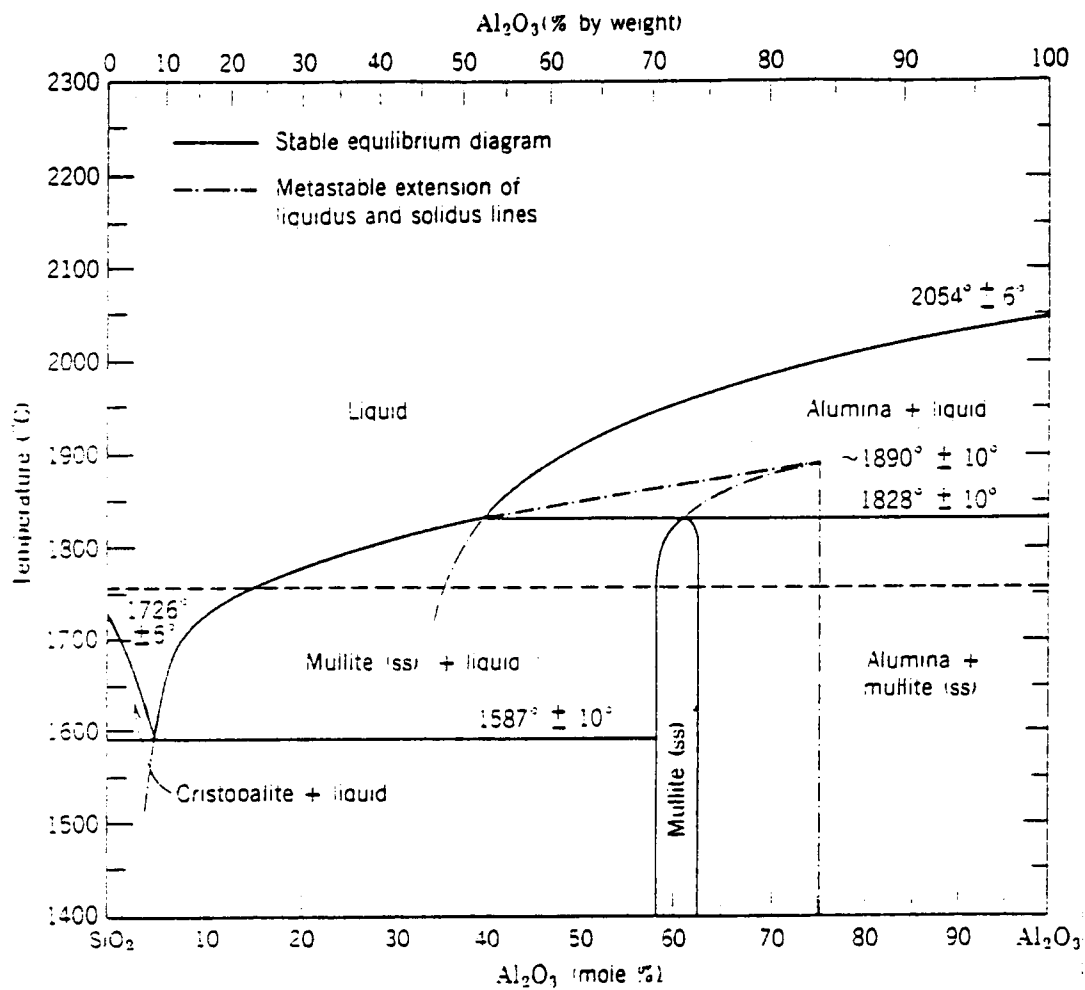


Figure 36. Phase diagram of SiO_2 and Al_2O_3 [14].

Furthermore, scanning electron micrographs of fracture surfaces of the rings tested in tension in compression for both the unexposed and exposed conditions can be seen in Figures 37-40. Overall fracture in all samples appears brittle (coarse surface) in nature as expected for ceramic materials. However, the surface does show striations in some regions indicating a localized ductile nature. This ductile nature may be due to the presence of residual aluminum in the composite. As previously mentioned, the variation of residual aluminum due to high temperature exposure is not substantial as is the case for porosity (Tables 12-13). This fact may be a possible reason for having small values of the Weibull number (m) (Table 3) for the exposed sample as compared to the unexposed, and strength values remaining the same within a small reasonable range respectively.

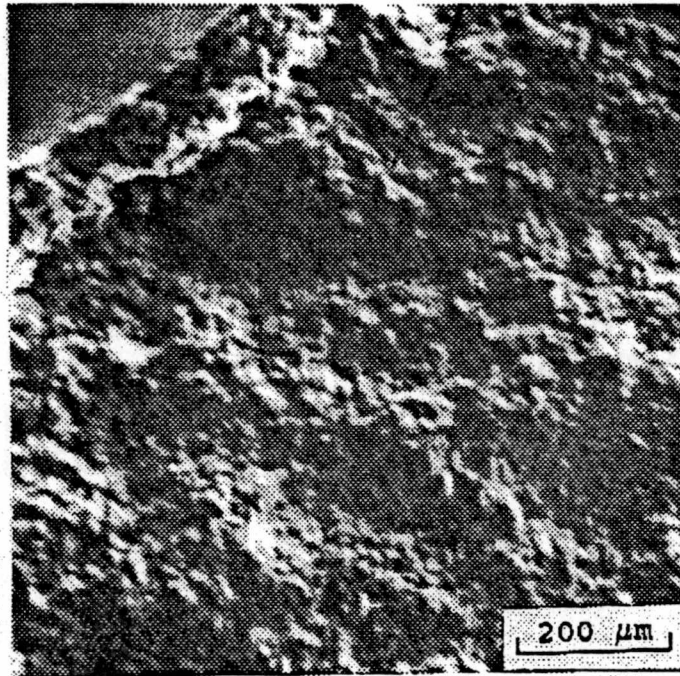


Figure 37. Scanning electron micrograph of unexposed compression fracture surface.

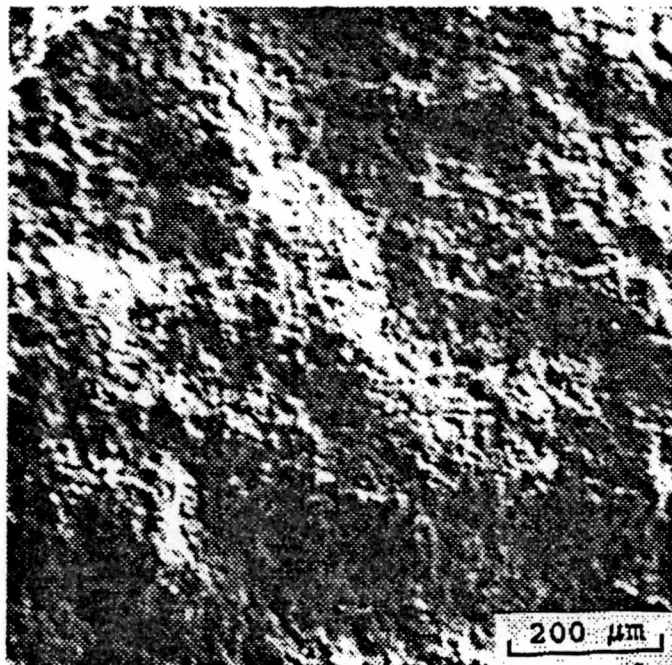


Figure 38. Scanning electron micrograph of exposed compression fracture surface.

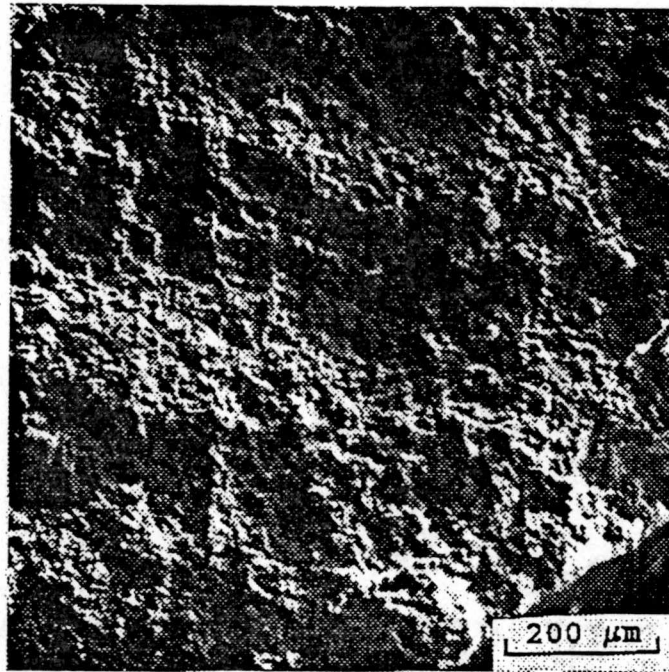


Figure 39. Scanning electron micrograph of unexposed tension fracture surface.

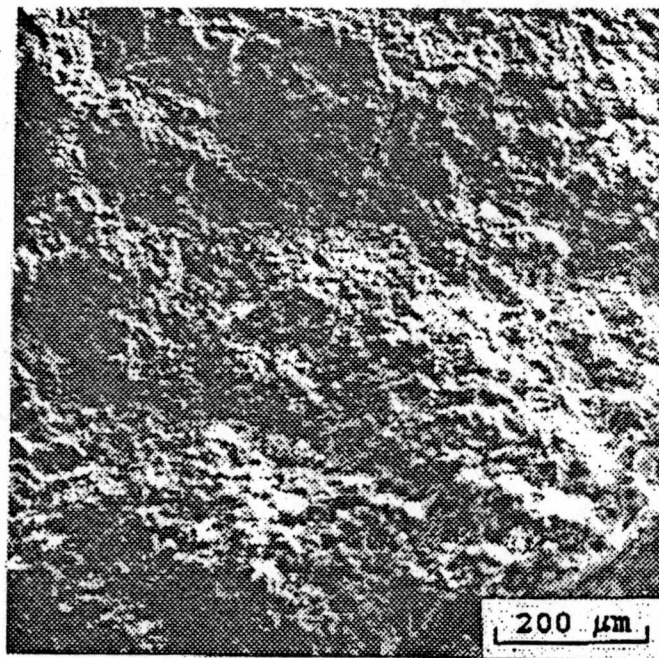


Figure 40. Scanning electron micrograph of exposed tension fracture surface.

CHAPTER V

CONCLUSION

Based on the analysis performed on this material in this study, it appears that the material is very promising for use in high temperature air heaters. It seems to have high strength characteristics as well as good resistance to corrosion and erosion. The most significant changes in the material from the as-received condition to the exposed condition were the increase in porosity and the decline of the aluminum content.

The analysis of the silicon carbide/alumina material used for high temperature air heater tubes can be summarized with the following results. First, the mechanical testing did not show a significant change in the strength of the material after exposure, and the orientation of the tube in the furnace did not affect fracture origin. However, the Weibull number decreased from the unexposed to the exposed material indicating a higher probability of failure and a wider spread of the data. The data found from the mechanical testing should be used with caution due to the extremely small test size. Second, the measurements of the tube indicated little or no corrosion or erosion by the slag. Next, the surface analysis showed that there was no reaction with the slag, and the major elements found in the samples were consistent with the as-received material. Finally, microscopic examination revealed an increase in porosity associated with the exposure temperatures. When the porosity was measured, it showed as-received approximately 2 volume %, unexposed approximately 18 volume %, and exposed approximately 23 volume % porosity.

Also, the free aluminum measurements showed as-received as 6.33 volume %, unexposed as 6.02 volume %, and exposed as 3.78 volume %.

Further research is necessary to determine the material properties at extended exposure to high temperatures. The porosity needs to be evaluated after exposure to various temperatures for various exposure times to determine if there is a limiting porosity value, or if the material will eventually deteriorate completely. The material also needs to be tested at higher temperatures to the point of deterioration in order to determine the limiting temperature conditions.

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and
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Appendices

Appendix A

Computer Code for Generating Stress Magnification Factors

```

program stress;
const
    ri = 20.5;
    ro = 25;

var
    ch:char;
    d,count,n,nn,num:integer;
    r,Qprev,si,si2,Ao,Bo,Rn,An,Bn,Cn,Dn,Q,Q1,Q2,Q3,Q4,Qa,Qsum,t,x:real;
    Q11,Q22,Q33,Q44,QQ,Qsum2,Qa2:real;

function power(x:real; m:integer):real;

begin
    t:=1;
    for nn:=1 to abs(m) do
        t:=t*x;
    if m<0 then
        power:=1/t
    else
        power:=t;
end;

procedure calculate;

begin
    x:=ri/ro;
    Ao:=1/(2*(1-sqr(x)));
    Bo:=-sqr(x)/(1-sqr(x));
    si:=2*pi/20;
    Qsum:=0;
    Qprev:=1;
    count:=0;
    n:=2;
    gotoxy(24,5);
    write('si=2pi/20=',si);
    while (n<=1800) and ((Qsum-Qprev<>0) or (count<=3)) do
        begin
            Qprev:=Qsum;
            Rn:=sqr(1-power(x,2*n))-n*n*power(x,2*n-2)*sqr(1-sqr(x));
            An:=(((1-power(x,2*n))/n)-((1-power(x,2*n-2))/(n+1)))/(2*Rn);
            Bn:=1/(2*Rn)*((1-power(x,2*n))/n-(1-power(x,2*n+2))/(n-1));

```

```

Cn:=power(x,n)/(2*Rn)*(-(1-power(x,2*n))/n+(1-power(x,2*n-2))/(n+1)*sqrt(x));
Dn:=power(x,n)/(2*Rn)*(-(1-power(x,2*n))/n*sqrt(x)+(1-power(x,2*n+2))/(n-1));
if (Cn<>0) or (Dn<>0) then
begin
  Q1:=(2*An*(n+1)*(n+2)*power(r/ro,n))*n*cos(n*si);
  Q2:=(2*n*(n-1)*Bn*power(r/ro,n-2))*n*cos(n*si);
  Q3:=(2*n*(n+1)*Cn*power(x,n)*power(r/ro,-n-2))*n*cos(n*si);
  Q4:=(2*(n-1)*(n-2)*Dn*power(x,n-2)*power(r/ro,-n))*n*cos(n*si);
  Q:=Q1+Q2+Q3+Q4;
end
else
  Q:=(2*An*(n+1)*(n+2)*power(r/ro,n)+
    2*n*(n-1)*Bn*power(r/ro,n-2))*n*cos(n*si);
Qsum:=Qsum+Q;
n:=n+2;
if Qprev-Qsum=0 then
  count:=count+1
else
  count:=0;
gotoxy(24,1);
write('Qsum=',Qsum);
end;
Qa:=2*Ao-Bo*power(r/ro,-2)+Qsum;
gotoxy(24,d mod 41 + 10);
writeln('r=',r,'  Qa= ',Qa);
end;
begin
  clrscr;
  for d:=41 to 49 do
  begin
    r:=d/2;
    Calculate;
  end;
end.

```

Appendix B

Test History of Tube

The tube which was evaluated was tested in the UTSI MHD facility in Tullahoma, TN during test number LMF5-D (LMF=Low Mass Flow) [2]. The test used Rosebud coal and was seeded with potassium. The tube was placed at level 3 in the radiant furnace. The test lasted for a period of 106.50 hours. After approximately 57 hours of testing, cold air was passed through the tube producing a thermal shock, and the tube broke [5]. This allowed slag to enter the interior of the tube. The tube was left in the furnace for the remainder of the test, after which the tube was removed from the furnace and submitted for evaluation.

VITA

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