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CERAMIC COMPOSITE COMBUSTOR CANS FOR EXPENDABLE TURBINE ENGINES

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Ceramic matrix composite fabrication by microwave assisted chemical vapor infiltration (MWCVI) has been demonstrated as a feasible, faster, and more economical technique as compared to conventional CVI. Two deposition systems, carbon and silicon carbide, were investigated using this processing technique. Densification rates over an order of magnitude higher have been achieved on small cylindrical Nicalon fiber preforms. Economic forecasts based on previous SiC deposition and this Phase I work shows a significant improvement in part cost as compared to conventional CVI work. A reduction in cost of a factor of 20 has been calculated. A larger, commercial microwave system has been designed for scaled up prototype part fabrication.

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CERAMIC COMPOSITE COMBUSTOR CANS
FOR EXPENDABLE TURBINE ENGINES

1.0 Introduction and Research Objectives

The use of advanced ceramic composite materials in projected markets has been plagued by the high cost of component materials (specifically fibers), and long processing times required to densify the fiber preforms. Innovative solutions to these problems have led researchers to investigate chemical vapor infiltration (CVI) processes which emphasize shorter, more economically favorable processing times. **Deposition techniques which show improvement over conventional isothermal CVI must be implemented into ceramic composite fabrication of key hardware, such as the composite combuster can for expendable turbine engines.**

The use of microwave-assisted CVI (MWCVI) processing of fiber reinforced ceramic composites is one such promising technique. The MWCVI process has the potential for low cost fabrication of ceramic composite components through substantial reduction of infiltration time. Ceramic Composites, Incorporated (CCI) proposed to demonstrate in Phase I that the use of microwave assisted CVI processing decreases the densification time over conventional isothermal CVI. The hardware chosen for this demonstration was a 3" outer diameter, 0.08" thick C/SiC combuster can for use in expendable turbine engines. The influence and relationship of power, temperature, microwave coupling, and system pressure on the densification are key technical objectives which will be discussed in later sections.

Another major objective of the Phase I research was to establish a preliminary assessment for a scaled-up MWCVI reactor for low cost fabrication of prototype components (Phase II) that have high commercialization potential (Phase III). Thus, the Phase I research included the demonstration and development of MWCVI processing, which offers substantial reductions in processing times and cost of ceramic composites.

1.1 Background on CVI Techniques

Chemical vapor infiltration (CVI) is one of the few processes capable of incorporating continuous ceramic fibers into a composite without mechanically, chemically, or thermally damaging the fibers. Most CVI processing today is performed using an isothermal-isopressure steady state process with process temperatures in the 900 to 1400°C regime and with absolute pressure in the multi-torr range (typically below 30 torr). CVI is a very time consuming process, typically requiring several hundred process hours for densification of composite sections of nominally 6 mm thickness. Relatively modest improvements have been made in the basic process through the incorporation of pressure cycling to enhance diffusional mass transport. A schematic of the five basic types of CVI is presented in Figure 1. Typical processing times for densification (to 90% dense) of a SiC/SiC composite material range from 100 to 250 hours of deposition, depending on the CVI type used.

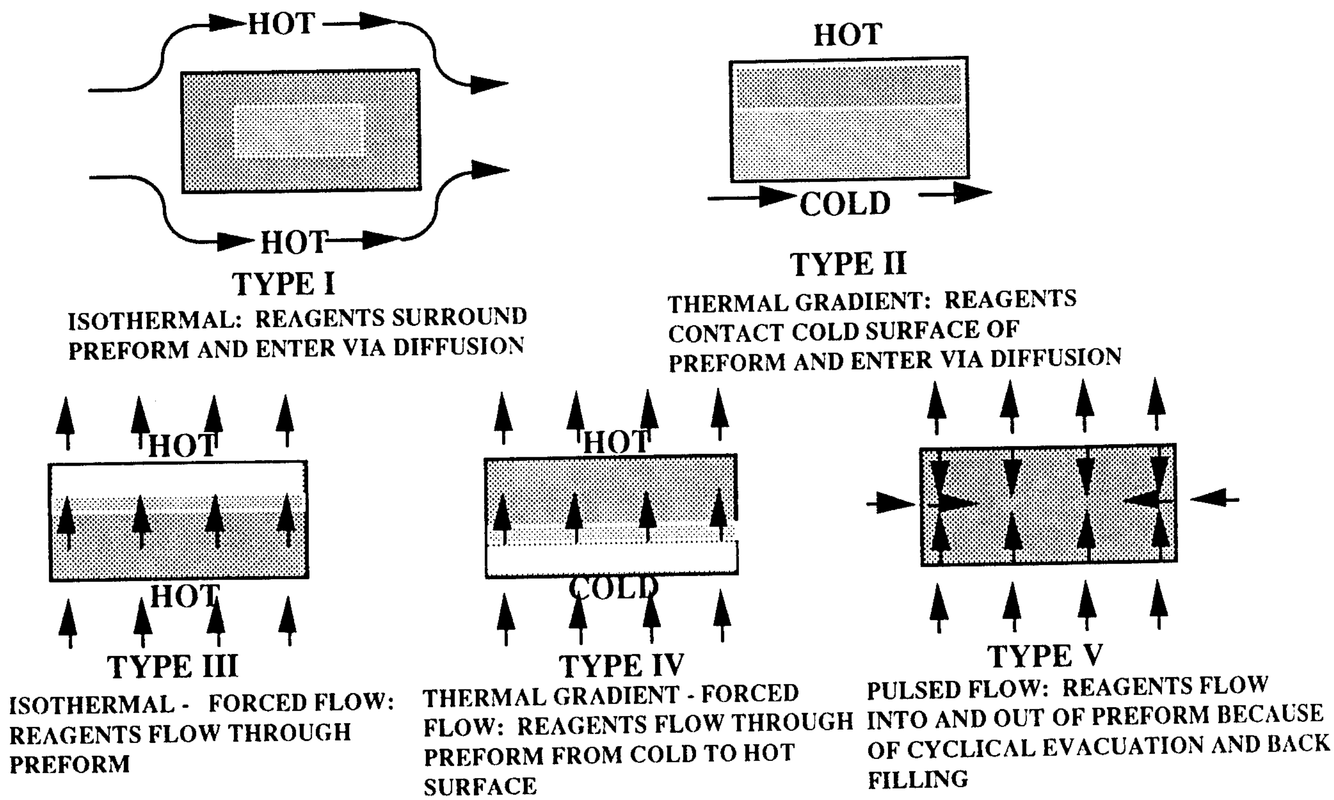

Figure 1. Five General Processes of CVI

Oak Ridge National Laboratories developed the forced flow thermal gradient (FFTG) CVI method in the early 1980's. In this technique, the fibrous composite preform, typically a disk, is trapped in a graphite fixture in which one major surface of the composite faces the furnace hot zone and reactant gases are forced through the opposing cool face. Hot face processing temperatures are typically 300°C higher than typical isothermal CVI process conditions. Process time reductions of an order of magnitude have been realized. However, adoption of this technique has been limited, perhaps due in part because of its complex, thermo-hydraulic behavior. Simple geometric shapes such as cylinders and plates have been produced. Conic sections, contoured closed end structures and structures with intersecting elements or multiple surfaces, such as skins and interior webs, are either difficult or impractical to process. The potential benefits of the FFTG process are also counterbalanced by the need to commit for each component that is being processed, a complete reactant and diluent gas feed system that is capable of operating under widely varying absolute pressure conditions.

The major drivers of CVI processing time are mass transport of reactants and by products within the porous composite structure and local thermal conditions. If thermal gradients are not imposed externally, as in FFTG, the surface of the preform, being closer to the radiant heating source, is normally hotter than the core, resulting in higher deposition rates in the surface region. Long processing times then result because processing is typically performed at the lowest thermal conditions feasible, in order to minimize this depositional gradient by lowering the overall reaction rate. The tendency for higher deposition at the surface of the preforms also limits the ultimate thickness of components that can be processed by CVI.

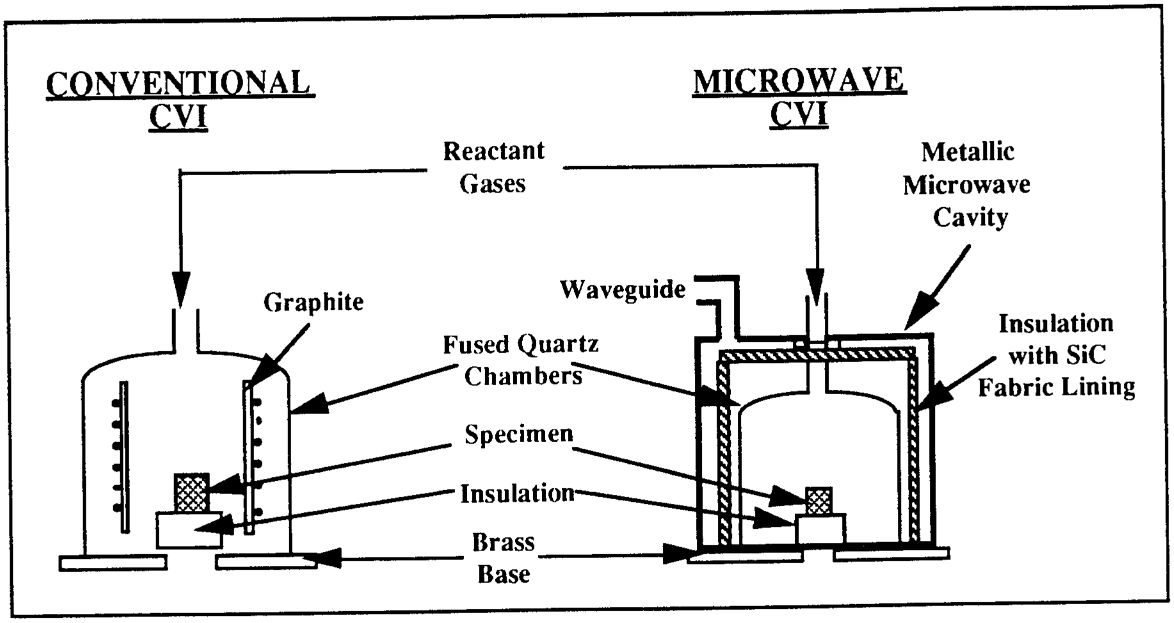

Figure 2. Conventional and Microwave Assisted CVI Schematics

The principal advantage of using microwave energy for CVI processing is that, with microwaves, heat is generated internally instead of originating from external heating sources. This phenomenon, together with radiative heat losses from the surface of the specimen, produces an inverse temperature gradient, i.e., the center of the specimen is hotter than the surface. It is this unique property that makes microwave heating an ideal source for CVI composite fabrication. By densifying the composite preform from the center out, rather than from the outside in as in the conventional process, higher processing temperatures can be used, thus shortening process time. In addition, higher densities will be possible even in thicker sections.

The initial MWCVI design is shown schematically in Figure 2, and can be compared to a conventional CVI furnace. The fused quartz reaction chamber that encloses the induction heating system in a conventional CVI reactor is placed inside a metallic microwave cavity. In addition, an insulated enclosure is used which can be lined on the inside with a microwave absorbing material. The result is that the preform and reactants inside the quartz chamber can be heated both by direct microwave absorption and by radiant heat from the enclosure lining.

1.2 Background on Combuster Cans

One potential test article for low cost CVI study is the thin walled cylindrical can combuster for use in expendable turbine engines. The development and demonstration of fiber reinforced ceramic composite combuster hardware has been one of the outstanding technical successes of the Integrated High Performance Turbine Engine Technology Program (IHPTET). Ceramic composite combustor hardware such as liners, cones and plates for can, annular and radial outflow combustor configurations have all been produced and have achieved high levels of performance in rig tests.

Ceramic composite components are likely to be early candidates for incorporation into expendable turbine engines because of their higher operating temperature capabilities and reduced cooling requirements, which provide greatly improved performance. However, expendable turbine engines are component cost sensitive, and the technical successes experienced in the IHPTET ceramic composite combuster program have been dominated by expensive chemical vapor infiltration of C/SiC and SiC/SiC composites.

Figure 3 shows a Garrett Turbine Engine Company combustor can produced using triaxial braiding of the Nicalon fiber tow followed by isothermal CVI of SiC. This combustor was successfully tested without attachment problems. Densification of the can via isothermal CVI required 160 hours of processing. Figure 4 shows a partially densified 3" diameter combustor can fabricated by conventional CVI at CCI.

2.0 Experimental Approach

The experimental approach involved the construction of the MWCVI system, modifying the design based on trial experiments, and the demonstration of the densification of fiber preforms, specifically a cylindrical combustor can, using the microwave technique. Investigation of processing parameters was completed in order to optimize the densification efficiency. The MWCVI equipment modifications are presented in the next section; this section presents the experimental approach including the experimental protocol and designed studies used to understand the densification.

The experimental protocol, schematically shown in Figure 5, involved the following steps:

1. Tube shaped samples were prepared by wrapping Nicalon cloth around a 1" diameter mandrel.

2. The mandrel was removed, and the cloth tubes were placed in a 600°C furnace in air for 1 hour to remove the polymer sizing.

3. The samples were then placed in the microwave cavity and heated in air; this was completed to burn any loose filaments from the surface to help minimize arcing due to the concentration of electrical field at loose fiber tips.

4. The initial weight was measured, and the sample was loaded into the microwave vacuum chamber.

5. The deposition was completed - typically all the reactant gases were flowing prior to sample heating.

6. The final weight was recorded following deposition, and, if desired, the density was measured.

Figure 3

A fully densified combustor can has previously been fabricated by isothermal CVI in 250 hours.

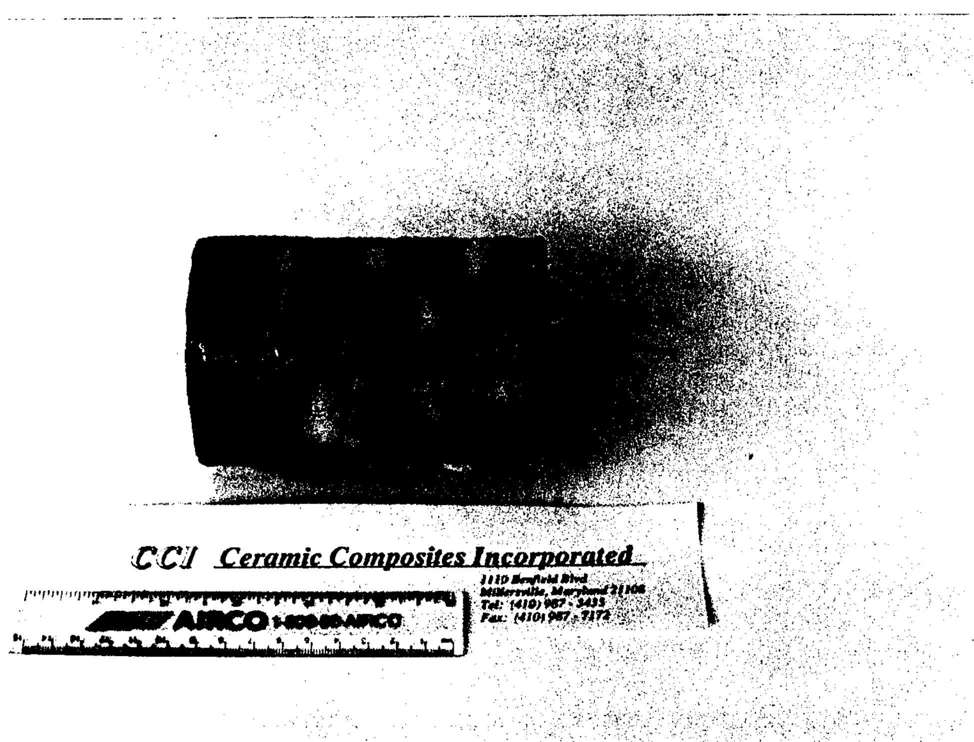

Figure 4

A partially densified (3" O.D.) combustor can fabricated by isothermal CVI is only 50% dense after 60 hours of processing.

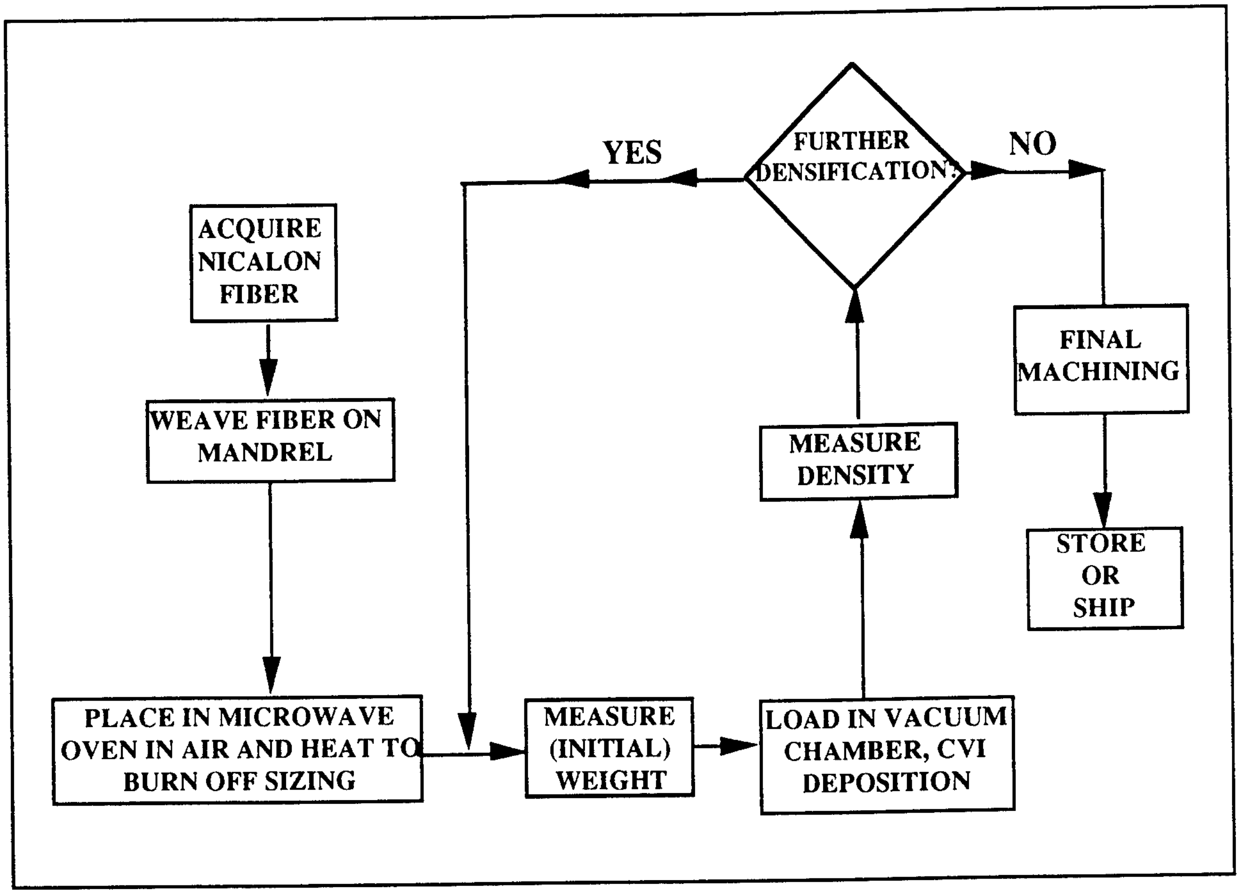

Figure 5. Protocol for Part Fabrication of SiC/SiC Composites

The density was measured by a conventional technique. The dry weight (D) was recorded, and the sample was then boiled in distilled water for 4 hours. Following cooling to room temperature, the suspended (S) and saturated (M) weights were measured. The calculations are summarized in Figure 6.

The carbon deposition was completed using the CH_4 + Ar reactant system. The SiC deposition was completed using methyltrichlorosilane (MTS), H_2, and Ar. A schematic of the SiC deposition system configuration is shown in Figure 7. The liquid MTS reagent is introduced using a heated flask with a H_2 + Ar carrier gas sweeping the MTS vapor into the reactor.

WEIGHT & DENSITY CALCULATIONS

<u>PROCEDURE:</u>

1. Determine Bulk Volume from Dimensions if Possible — V_B
2. Determine Dry Weight — D
3. Boil H O (4hrs) dilute to R.T.
   Determine Suspended Weight — S
4. Wipe Down w/ moist Rag
   Determine Saturated Weight — M

<u>CALCULATIONS:</u>

SG = Specific Gravity = $\frac{D}{D-S}$

BD = Bulk Density = $\frac{D}{V_B}$

CBD = Calculated Bulk Density = $\frac{D}{M-S}$

Open Porosity = $\frac{M-D}{V_B}$

Calculated Open Porosity = $\frac{M-D}{M-S}$

Figure 6 Density Calculation Procedure for CMC's

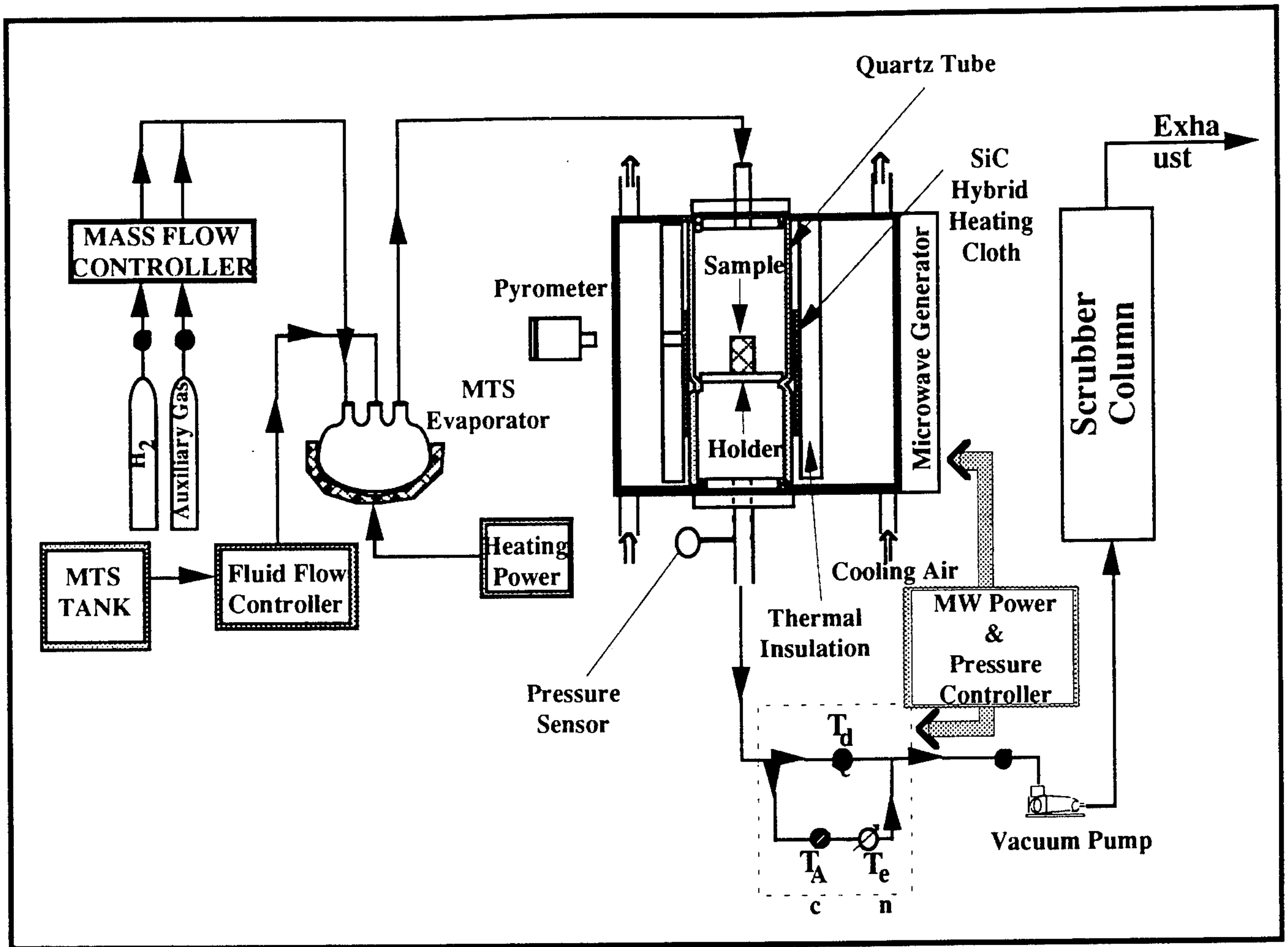

Figure 7. MWCVI Schematic for SiC Deposition

3.0 Microwave-Assisted Chemical Vapor Infiltration System

The experimental development of the microwave assisted CVI process required extensive modification of a Litton commercial 2.6 kW microwave oven. The development of a MWCVI system that would permit independent control of the on-off MW power and simultaneous yet independent control of reactant pressures required extensive equipment modification and development. The ability to independently control the microwave power settings, the duration of on and off cycles, the reactant gas pressures and timing sequences will permit optimum process protocols to be developed for a specific matrix material and part geometry.

3.1 Pulsed Power - Pulsed Pressure Protocols

The innovative MWCVI technique consists of the following sequential steps: introduction of the reactant gases; maintenance of gas pressure while microwave power is increased; and reduction of pressure and power to accelerate evacuation of the by-product gases. A summary description of the individual process parameters is given in Table 1 along with a description of the process timing steps shown in Figure 8.

Initially, the preform is pre-heated by microwave energy to a temperature T1, lower than the critical reaction temperature Tc, and the reactant gases are introduced into the reaction chamber. These gases enter the chamber at pressure P_h and fill the porous volume of the preform. This pressure is held for a short period to allow gas concentrations to reach equilibrium inside the preform.

In the next step, when the microwave power is applied, radiative losses keep the surface cooler than the interior, thereby increasing the rate of deposition in the interior relative to exterior regions of the composite. After the heating and reaction portion of the cycle is completed, the microwave energy is switched off to decrease the temperature below Tc. This dramatically reduces reaction rates which, in turn, enhances the evacuation of the by-product gases. Simultaneous reduction in reactant pressures below pressure P_1 also assist the evacuation process. Low temperature T_1 is necessary to prevent unwanted deposition at surface or near surface regions during the reactant gas introduction at the start of the next cycle. The previous forced evacuation of by-products also promotes diffusion of reactant gases into the interior of the preform on each successive cycle.

Microwave energy is dissipated preferentially at the surface of glossy materials, enabling the deposition and densification process to proceed outward from the core of the preform. As long as the preform surface porosity remains open, uniform densities are achievable. The proper pulsing sequence enables uniform densification to occur at the highest rate for a specific matrix composition and component geometry.

As can be seen from Table 1, a series of processing experiments are required to optimize the pulsed MW, pulsed reactant CVI process for a specific material system and component application. To build a system capable of conducting the MWCVI process optimization studies the following equipment development activities were completed.

3.2 Microwave CVI Chamber Modification and Construction

The manual on-off time settings on the 2.6 kW microwave oven required by passing this electronic circuitry with a timer that could set anywhere between 100% on to 100% off. This enabled any pulsed on-off cycle such as 75% on - 25% off to be automatically preset.

The installation of the CVI vacuum system involved cutting holes in the microwave oven to accommodate the quartz tube reaction chamber. Next metallic end plates with O-ring seals were installed at the end of the quartz tube to prevent microwave leakage and hold the vacuum in the quartz tube. A photograph of the quartz tube - O-ring seal at the upper end of the chamber is shown in Figure 9. Ceramic thermal insulation was placed around the quartz tube. A SiC cloth was bonded to the inside wall of the center section of the insulation. The SiC fabric acted as a susceptor that permitted heating of the chamber with microwave energy from room ambient for ceramic fiber preforms with poor microwave absorption properties. A photograph of the entire MWCVI system showing the ceramic insulation around the quartz chamber is depicted in Figure 10. Pressure variation of the vacuum system was accomplished via the use of a throttle value, an Electro-Pneumatic Activated Ball Valve (EPABV) and a relay circuit. A schematic of this setup is shown in Figure 11. Reactant gas lines of methane and argon carrier gas with appropriate mass flow contollers were hooked to the inlet of the quartz chamber. Initial check-out runs of the vacuum pressure system and pulsed microwave system were made using a 1" diameter x 1.5" high cylindrical Nicalon preform with a wall thickness of 0.15". A photograph showing the overall system with reactant lines is seen in Figure 12.

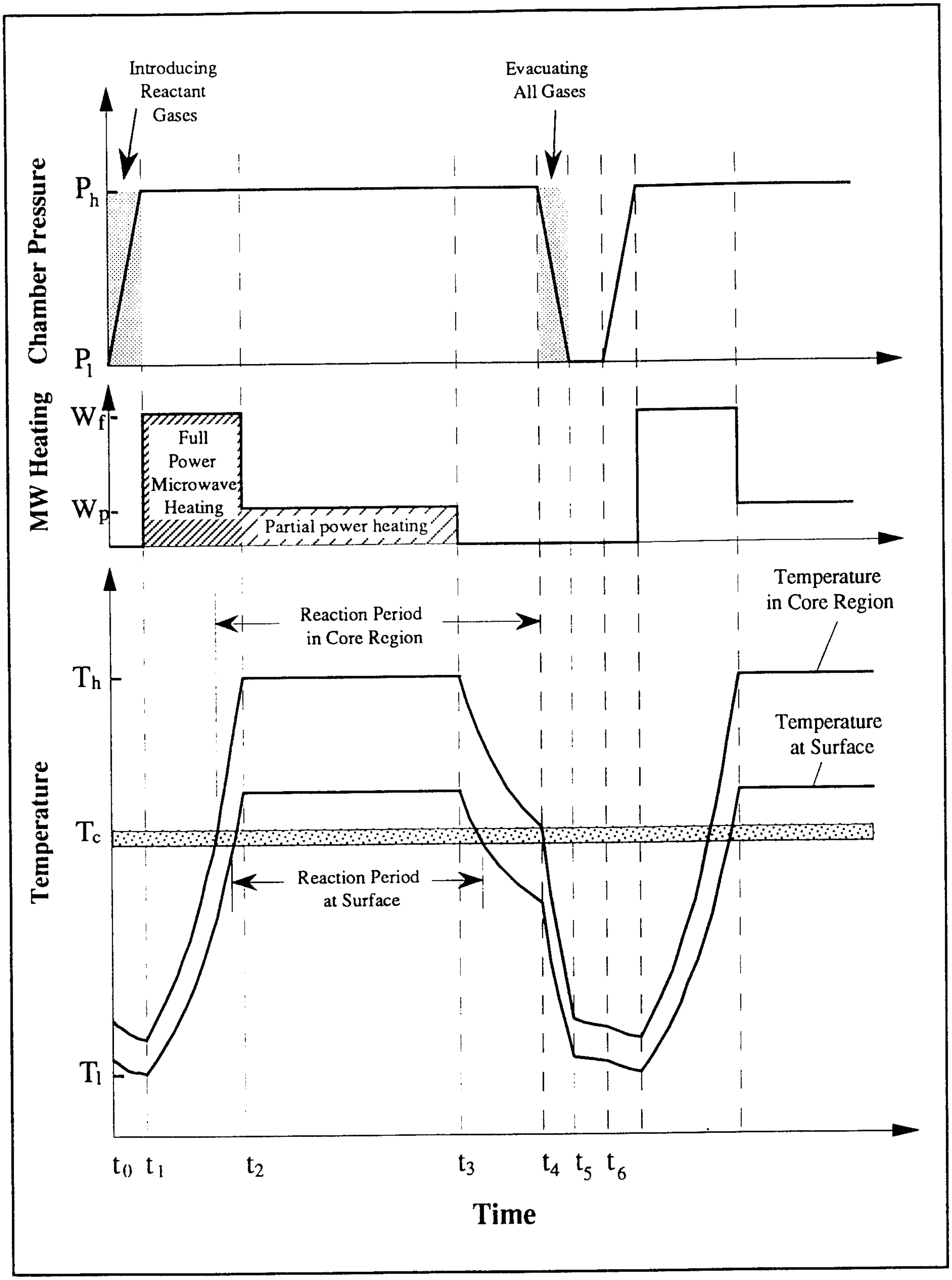

Figure 8. Timing Protocol for Microwave CVI

Table 1 List of the Processing Parameters in MWCVI

Paramete r	Description	Value (Range)
W_f	Power of full power MW heating	Given & limited by equipment
W_p	Power of Partial power MW heating	Automatically set by the temperature control loop
τ_{in}	$\tau_{in} = t_1 - t_0$; Time interval of gas introduction	To be determined in pressure rise & fall experiments
τ_{fp}	$\tau_{fp} = t_2 - t_1$; Time Period of full power MW heating	Automatically set by the temperature control loop
τ_{pp}	$\tau_{pp} = t_3 - t_2$; Time period of partial power MW heating	<u>ADJUSTABLE PARAMETER</u> High cutoff limit is determined in the activation energy experiment
τ_d	$\tau_d = t_4 - t_3$; Time interval between end of MW heating and beginning of gas introduction	Automatically set by the temperature control loop
τ_{out}	$\tau_{out} = t_5 - t_4$; Time interval of evacuation of gases	To be determined in pressure rise & fall experiments
τ_{wait}	$\tau_{wait} = t_6 - t_5$; Waiting time interval between two cycles	To be estimated as short and effective as possible
P_h	Equilibrium high chamber pressure during reaction period	<u>ADJUSTABLE PARAMETER</u> Lowest value is limited by the plasma formation during MW heating
P_l	Equilibrium low chamber pressure during gas evacuation	As low as possible, but is limited by the vacuum system and gas flow
T_h	Highest temperature in core region at reaction period	<u>ADJUSTABLE PARAMETER</u> The higher T_h, the higher the reaction deposition rate; It is limited by the degradation temperature of the preform fiber
T_l	Lowest temperature at surface at non-reaction period	To be estimated and it should be lower than the critical reaction energy
T_c	Critical temperature of the chemical reaction	Characteristic temperature of the chemical reaction

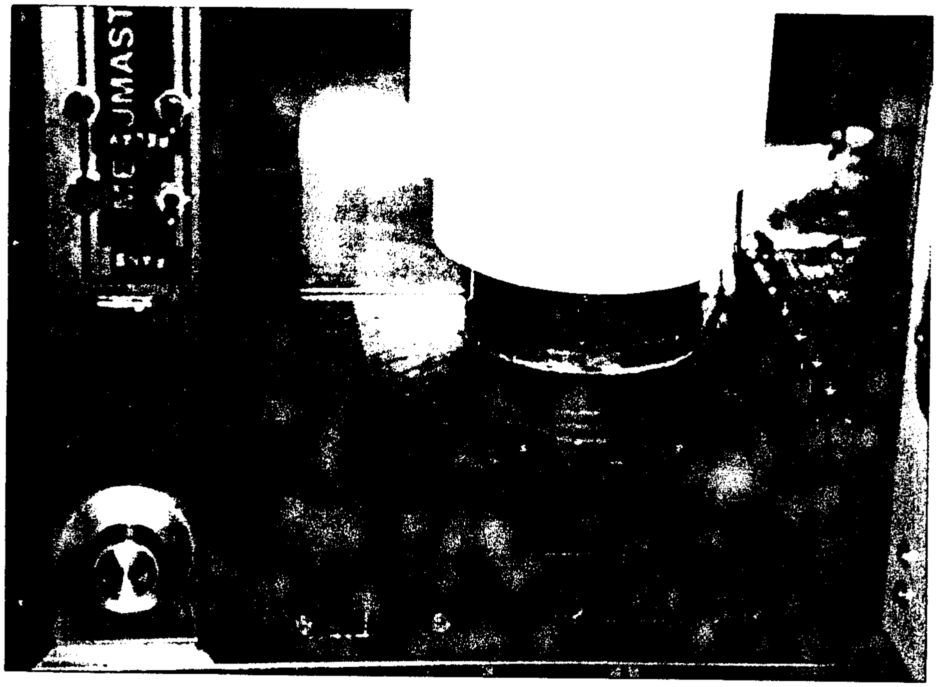

Figure 9

O ring at the top of the quartz chamber for the MW CVI furnace.

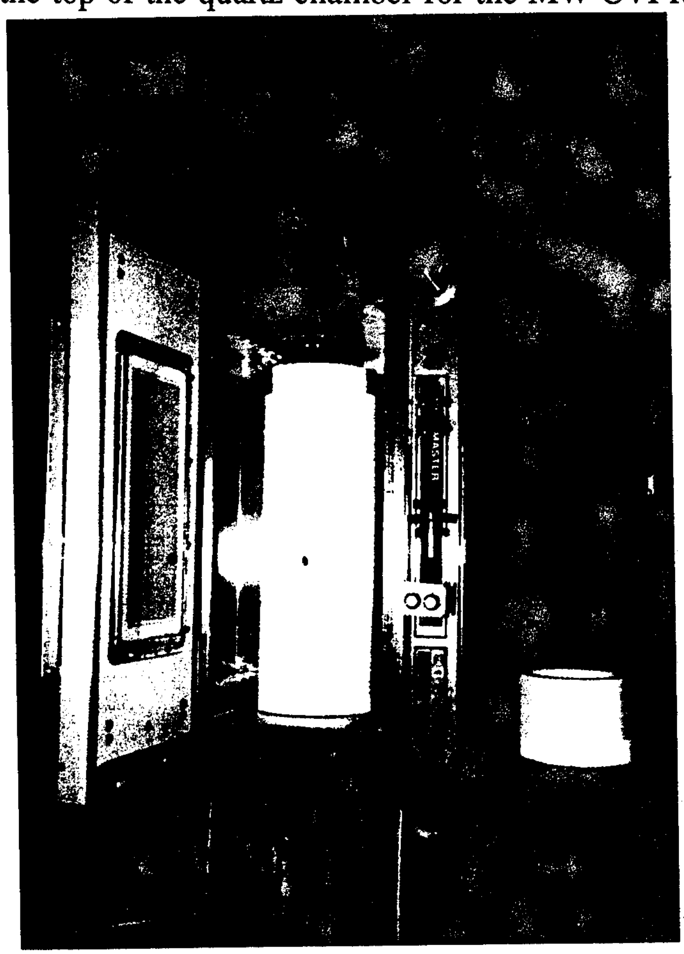

Figure 10
Quartz chamber with surrounding ceramic insulation for MW CVI.

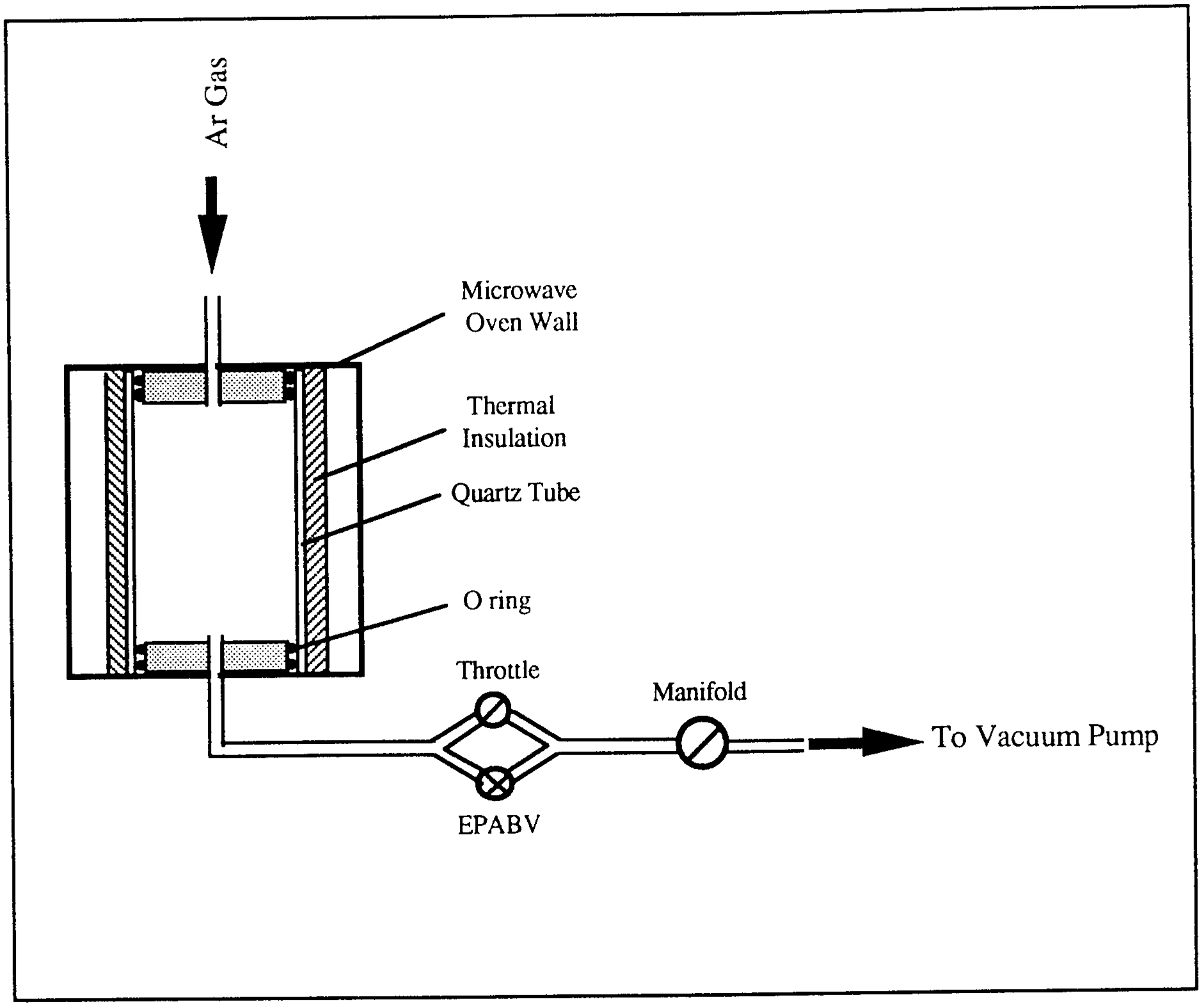

Figure 11. Schematic Illustration of Vacuum Test of MWCVI System

3.3 Initial Pulsed Microwave-Pulsed Reactant Tests

<u>Highlights of the initial test runs were as follows:</u>

The lowest pressure of the system was achieved (1.6 torr) with the EPABV closed, and the throttle and manifold open. The vacuum leak rate was 12.6 torr/hour when the throttle was closed. The rate was 10.0 torr/hour with the throttle open and the manifold closed, respectively.

With argon gas alone flowing into the microwave chamber at 0.5 SLMP, and throttle and mainfold vacuum valves opened, the pressure of the system was 5.0 torr. When the microwave power was switched on, an argon plasma resulted. The formation of the plasma inhibited heating of the SiC preform, since the majority of the microwave energy was absorbed by the plasma. The argon flow rate was then increased to 3 SLPM (the vacuum pressure was about 10 torr) but the plasma still persisted. The manifold valve was partially closed and the chamber was slowly increased to determine what pressure was required to eliminate the plasma. When the chamber pressure reached about 400 torr, the plasma could no longer be sustained. During the next experimental test run, the MW system abruptly quit after 30 minutes.

Subsequent investigation led to the discovery that a fire sensor had overheated and shut down the electronic control circuitry. Since the commercial microwave ovens were never designed for long duration heating runs, a forced air cooling systems had to be designed and installed. A schematic of the air ventilation system is shown in Figure 13.

This air ventilation system consists of two air channels located on the left and right sides of the quartz tube/thermal insulation tube. The cross sectional areas of the air cooling channels were 9.875" x 2.125" and 10.125" x 2.75", respectively. Four corresponding slots on the microwave oven wall were cut for inlets and outlets of the two forced air channels. The cut slots were covered with perforated metal sheets to prevent the leakage of microwave energy. The two channels were connected to a powerful air blower through a specially designed and fabricated connector. The photograph in Figure 14 of the rear of the MWCVI furnace displays the blower motor and the metal cooling chambers at the top of the furnace. The blower has a free air flow rate of about 760 CFM to prevent overheating of the MW chamber during extended CVI runs. A subsequent three hour run using methane demonstrated that forced air cooling system protected the fire sensors and microwave electronics.

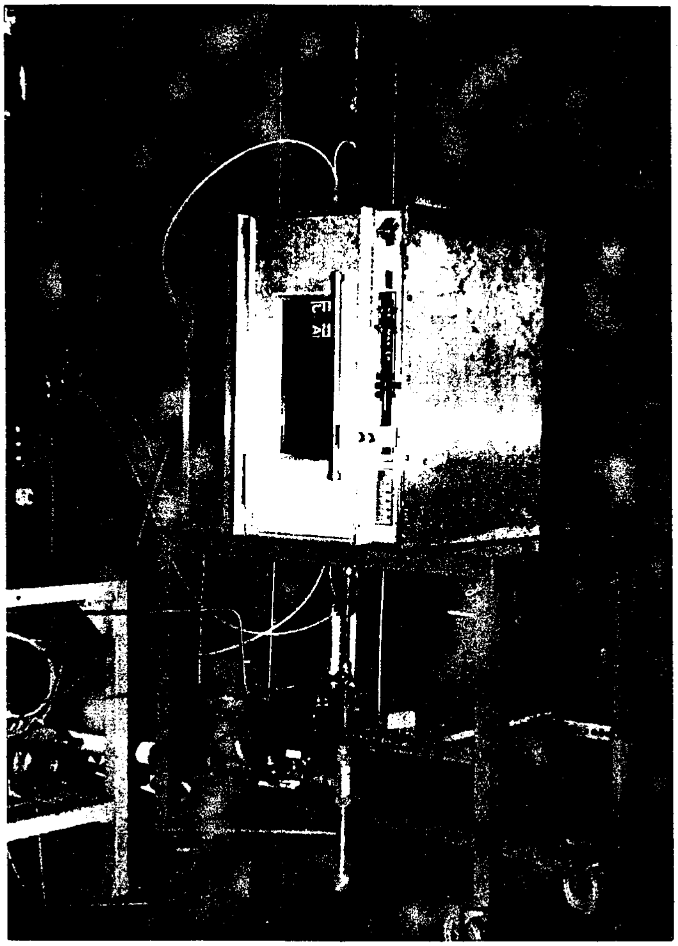

Figure 12

MW CVI system with reactant gas lines and exhaust plumbing

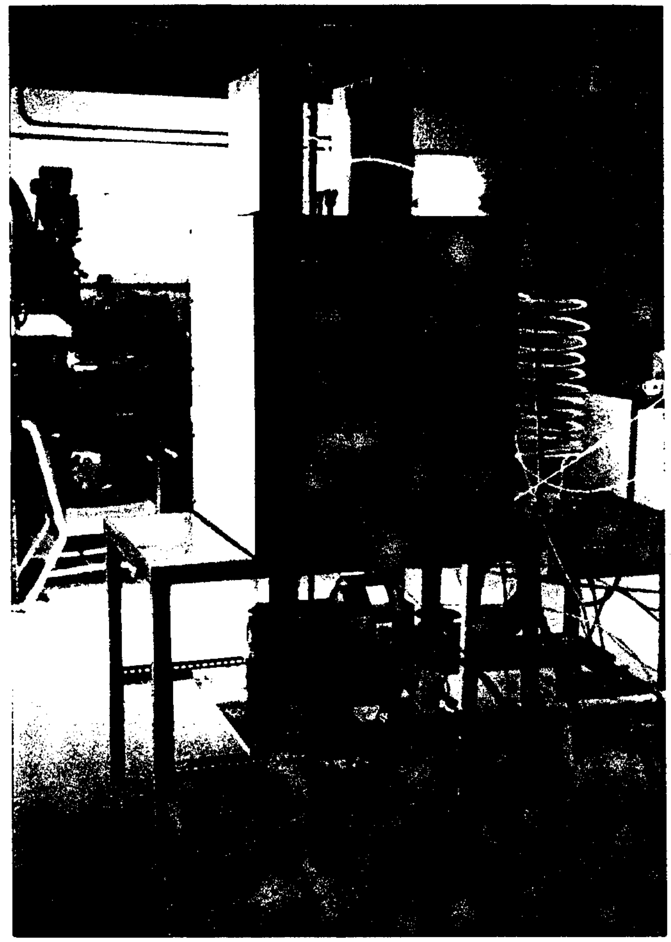

Figure 14

Blower and metal cooling channels at top of MW CVI furnace.

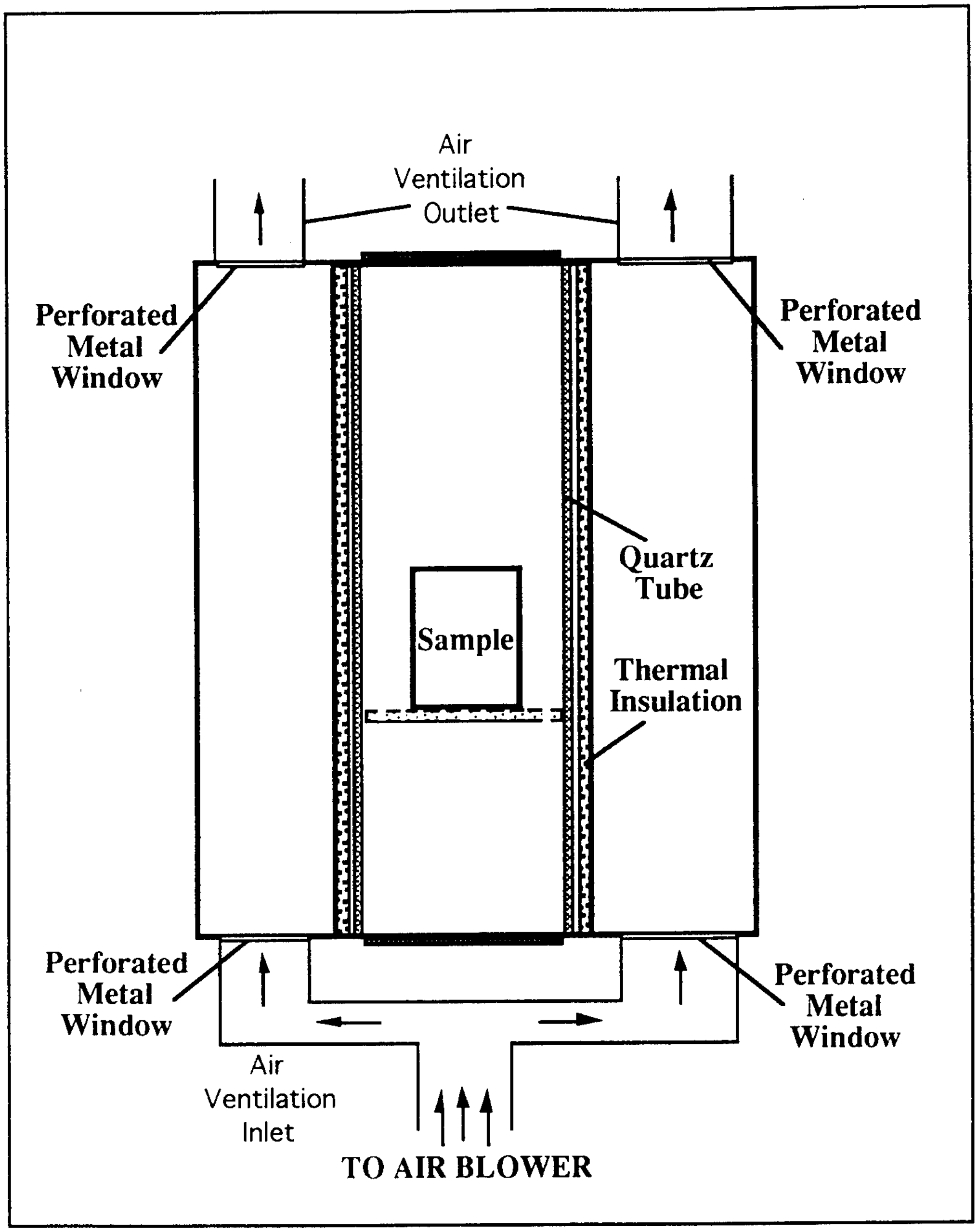

Figure 13 Schematic Illustration of Air Ventilation System in MWCVI.

4.0 Densification Results

The majority of experimental work during the Phase I effort involved the modification/development of the microwave processing unit. Once the iterative equipment development was complete, two major processing studies were completed in order to demonstrate the improved densification rate projected for microwave processing.

Four tubes of Nicalon cloth (1" diameter x 3" long) were infiltrated with carbon via the decomposition of methane. The infiltration was completed at 600 torr, for time periods of 1, 2, and 4 hours. The temperature of the inside wall of the preform was measured using an optical pyrometer, and was approximately 800°C. The deposition experiments were complicated, however, due to a noticeable decrease in temperature during the process. This was a result of sample stage coupling to the microwave power. This problem was also observed when studying power cycling of the preform. Several different stage materials were used to minimize the coupling effect, including Nextel fiber tow, mullite, and finally porous alumina plate. The results using the porous alumina were superior; elimination of silicon and other impurities from the sample stage material is essential for reduction of the undesired coupling. Identification of other materials, including sapphire, quartz, and pure alumina fiber tow (Nextel 610) was completed for future exploration.

The densification of the carbon/Nicalon composite was negligible for the short processing times investigated. The measured increase was approximately 1%/h. The stiffness of the tube, however, was markedly increased, indicating that the deposition was increasing the structural integrity of the tubes. Since carbon was not the preferred system of study, the experimental work shifted to Nicalon/SiC composite densification studies.

4.1 SiC Deposition/Densification

The feasibility of economical MWCVI processing of SiC/SiC material was demonstrated as shown in the Figure 15 densification comparisons. Sample densification rates of 2-5%/h were demonstrated as compared to the 0.1-0.2 %/h achieved by conventional CVI. The faster deposition rate (5%/h) was achieved on a higher porosity preform (with a 22% initial density).

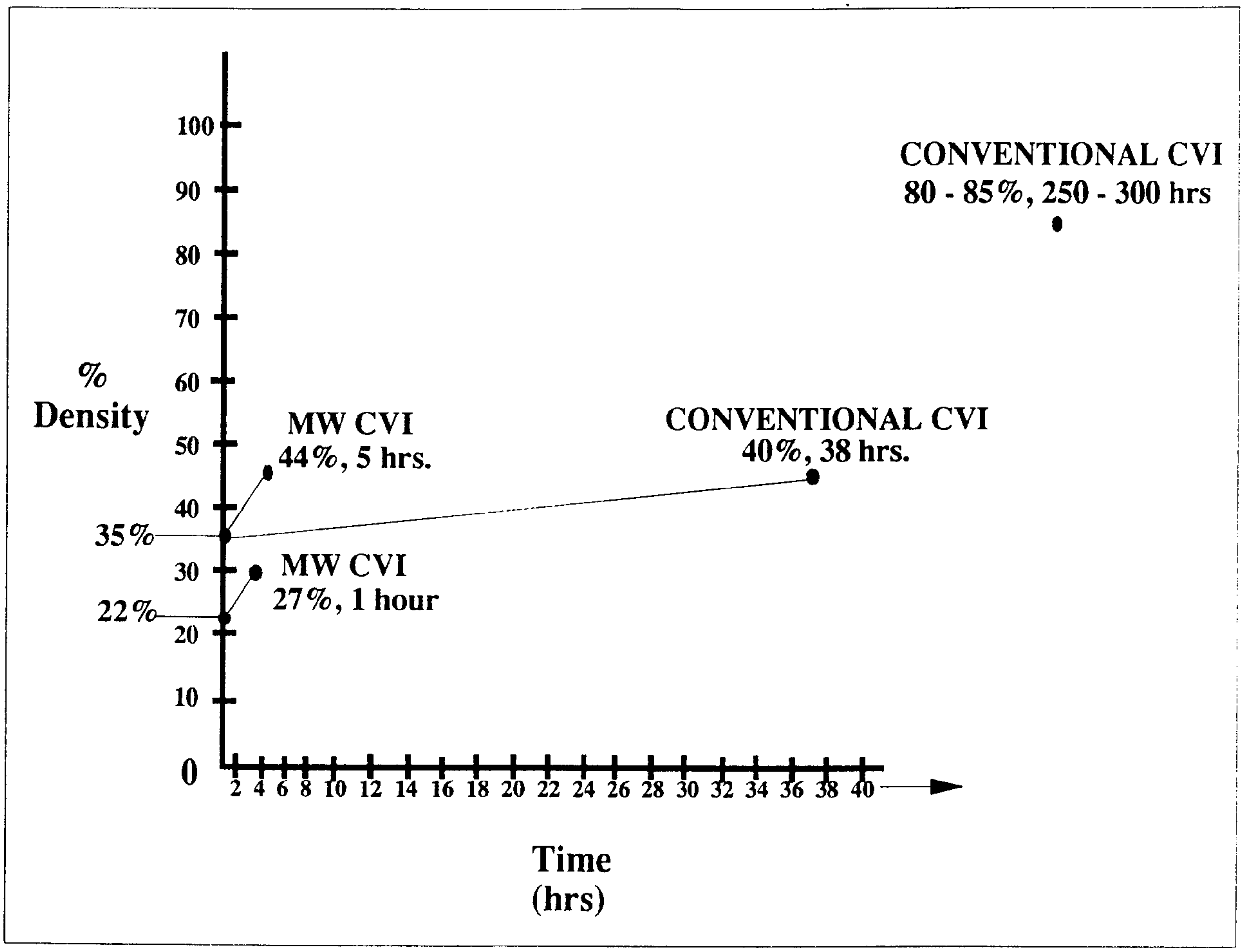

Figure 15. Densification Rates for SiC/SiC Materials by MWCVI and Conventional CVI Processing.

As observed with the carbon system, the interior wall of the Nicalon cloth was approximately 50-100°C hotter than the exterior surface as measured by optical pyrometer. Sample heatup time was 10 minutes, and was completed with the reactants already passing through the deposition chamber. The heatup was completed at 400-600 torr since the presence of argon previously had resulted in plasma formation at pressures below 400 torr. During the deposition, the total flow rate was nearly 1 l/min.

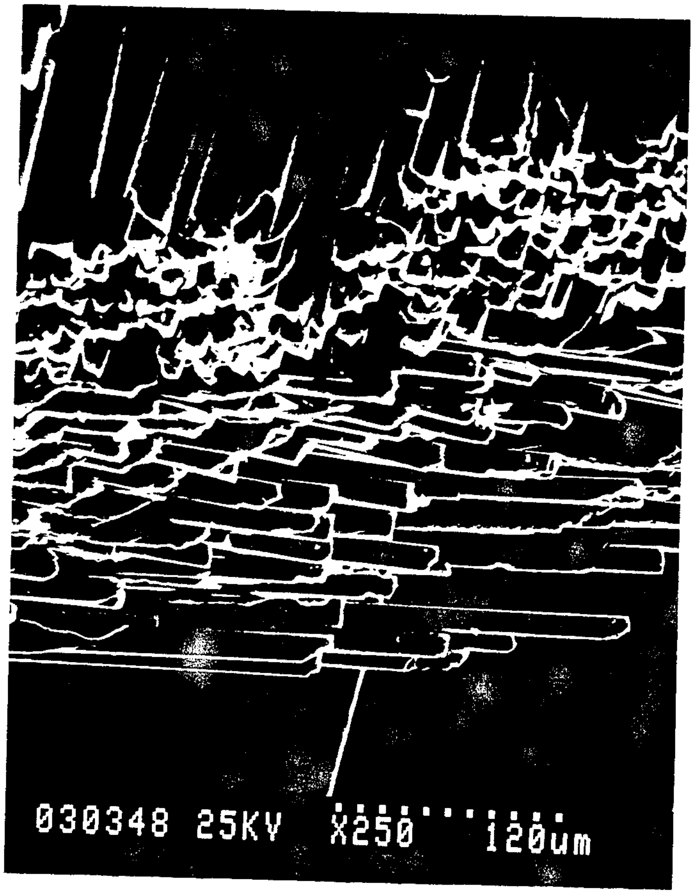

Figure 16

Cross-section micrograph of a conventional CVI fiber preform after 40 hours of SiC deposition. Typical densification times are 200-250 hours.

Figure 17

Cross-section of a MWCVI fiber preform taken from the interior wall of a tubular geometry. The preform section was densified in 5 hours. The remaining cross-section (outside wall) was not coated.

There was no observed deposition during the process. The sample remained at temperature until the stage material would begin to couple with the microwave field. A gradual decrease in temperature followed at that point. After removing the test cylinder, the first noticeable difference was in the stiffness of the part. The interior core of the sample appeared to be significantly densified. A comparison of this density increase is shown in Figures 16 and 17. Figure 16 is a cross section of a conventional CVI preform after 40 hours of infiltration; there is only a minor fraction of densification observed for this preform. Figure 17 is the interior wall of a cylinder (with a total thickness of 150 microns) that was completely densified in 5 hours. The exterior portion of the cylinder thickness was uncoated, and therefore did not hold together for SEM analysis. This performance (inside-out densification by MWCVI) is one of the key advantages over the isothermal approach. No machining is required to open diffusion paths for the reactant due to exterior surface overcoating.

The appearance of the cylinder also matched the SEM observed photos. The interior surface was gray and appeared coated with SiC; the exterior surface appeared to be uncoated and actually unaffected by the coating process. For longer processing times, it is expected that complete (100%) densification will be attained. In the future, for a more accurate density profile, epoxy mounted, polished cross sections of the CVI parts will be completed.

4.2 Economic Analysis

The operating conditions, along with conventional CVI processing parameters are shown in Table 2. The reduction in time, reagent and power consumption can be used as a basis for economic justification of this processing technique. Recent work which evaluated the economic fabrication of SiC fiber production using a similar processing chemistry and technique to this SiC/SiC work has identified the significant cost parameters for advanced ceramic production: initial fiber cost and reagent (MTS) cost. A comparison of the costs for CVD of SiC tow are given in Table 3 showing the high percentage of raw material cost for ceramic composite fabrication.

Table 2. CVI Processing Comparison: Microwave vs. Conventional SiC/SiC

	MWCVI	CONVENTIAL CVI
Furnace Heat-Up	10 mins	2-3 hours
Burn Off Sizing	10 mins	1-2 hours Conventional Furnace
Pyrocarbon Fiber Coating	3 hours	20 hours
SiC Matrix Densification	30 hours	160 - 200 hours
Cooldown	15 mins	3 - 4 hours
Reactant Pressures	600 torr	5 torr
Flow Rates	1ml/min	6ml/min
Reactant Gas Consumption Ration Per Unit Time	1	7

Table 3. Economic Analysis and Factors of SIC Tow Fabrication

ITEM	CONTRIBUTION TO FIBER; FIBER COST (%)
Methyltrichlorosilane	50.8
Carbon Tow Substrate	28.4
Personnel	9.0
Hydrogen	6.3
Calcium Oxide for Scrubber	3.1
Equipment	1.6
Facility and Utilities	0.6

Table 4. Economic Forecast of MWCVI

	MWCVI	CONVENTIONAL CVI	RATIO OF C-CVI/MWCVI
Processing Temperature	600 torr	5 torr	/
Temperature	1050°C	950°C	/
Heat-Up	10 mins	2 hours	12
SiC Densification	30 hours	200 hours	6.7
Pyrocarbon Densification	3 hours	20 hours	6.7
Cool Down	15 mins	4 hours	16
Engineering/Job	12 hours	40 hours	3.3
Heat and Cool Down Gas Consumption Ratio Per Unit Time	Ar: 0.5 SLPM H_2: 0.2 SLPM	AR: 5 SLPM H_2: 10 SLPM	10 50
Runtime Gas Consumption Ration Per Unit Time	MTS: 1ml/min Ar: 0.4 SLPM H_2: 0.4 SLPM	MTS: 6ml/min Ar: 10 SLPM H_2: 4.5 SLPM	6 25 11.3
Electric Power Per Unit Time	7Kw	50Kw	7
Water Usage Ratio	1	6	6
Hazardous Waste Disposal Ratio	1	6	6
Nicalon Fiber $ Ratio	1	1	1
Mandrel $ Ratio	3	1	0.33

MWCVI shares several common economic advantages to the SiC tow process. First, the microwave equipment is a cold-walled process -- the reagent deposits SiC only on the hot surfaces as compared with hot wall systems. Using microwave heating provides only one hot inside surface of the preform. Second, the deposition of SiC using MWCVI has been demonstrated at high pressure (600 torr). This fact allows for a higher reagent concentration to be used in the gas phase, providing more material which can be deposited. Growth rate increases as temperature and reagent concentration increases. The final advantage is the higher temperature operation of the MWCVI process; the inside-out heating prevents any overcoating of the surface which blocks diffusion paths for the reagent in conventional CVI.

4.3 Conceptual Design for MWCVI Scaleup

The conceptual design of a large scale microwave reactor for CVI has also been completed during the Phase I effort, and is also described in detail in the Phase II proposal. The cavity design and scaleup factors are described next.

Microwave energy has been applied to materials processing in such areas as sintering of ceramics, joining of ceramics, curing of organic matrix composites, encapsulation of integrated circuits, chemical vapor deposition, drying and sintering of ceramic sols and chemical vapor infiltration.[1] In all cases the most critical demand placed on a microwave applicator is that it must provide a uniform processing environment. Specifically, the microwave field pattern must be either uniform or its configuration tightly controllable. The central features of most system designs are concepts that focus on meeting this requirement.

At a low level of technical complexity, this objective is *approached* in a conventional, over moded, home microwave oven. The dimensions of the cavity are such that it is capable of supporting 20 to 30 different electromagnetic modes.[2 3] Each standing wave pattern in the cavity is called a "mode." This nomenclature is the same as that used to discuss the patterns of a vibrating string stretched between two points. If there are many modes, and if equal microwave power is carried by each mode, then the spatial distribution of power is more uniform than it would be if there were only one mode or if most of the power were being carried by a single mode. Conventional microwave ovens are designed to support many modes. The generic name for such a cavity is a "multimode" cavity. They incorporate a "mode stirrer" to improve the distribution of microwave power among those modes. However, this solution is never entirely satisfactory for several reasons. First, there are only a finite number of modes that can be excited. Thus, field homogeneity can only be approximated. Second, the fact that microwave power has to be introduced into the cavity at some point rather than simply appearing uniformly distributed within the cavity, assures that even with a large number of modes, field isotropy is not achieved. Third, the object being processed disturbs the field pattern. Even for cylindrical samples circumferential process

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1 Sutton, W. H., "Microwave Processing of Ceramic Materials," Amer. Ceram. Soc. Bul. **68**, 376 (1989).

2 Turner, R. F. B., et al ,"On the Counting of Modes in Rectangular Cavities," J. Microwave Power **19**, 198 (1984).

3 Puschner, H., Heating With Microwaves: Fundamentals, Components and Circuit Techniques (New York, Springer-Verlag, 1966), pp. 176 - 182.

nonuniformity is not uncommon. That is, one side of a cylinder is processed at a different rate than the other side.

At a considerably higher level of technical complexity, the goal of uniform microwave processing can be approached by using a variable frequency microwave source to feed a conventional rectangular microwave cavity.[4] In this scheme, the mode distribution is continuously varied by continuously changing the frequency of the microwave source. It is easy to show that a considerable increase in uniformity can be achieved by varying the frequency over a relatively small band. This improvement has been demonstrated by comparing the heating profiles produced in a conventional microwave oven with that produced in a system that sweeps the frequency from 4.5 to 5.5 GHz. The results are impressive. However, commercialization of the technique faces several serious obstacles. First, the maximum power available in this system is only 200 W. The resulting power density is far below that needed to process most commercial objects like heat exchanger tubes. Second, even such low power variable frequency microwave sources are costly and high power sources prohibitively so. For example, the cost of a 200 watt traveling wave tube amplifier is in the tens of thousands of dollars without including the necessary oil cooling or power supply, and when completed would still provide too little power to implement many important microwave processing techniques. Higher power can be achieved but at considerable costs. Gyrotrons and high power klystrons are obvious possibilities but costs typically are measured in multiples of $100k. Third, the FCC has designated 2.45 GHz ± 50 MHz and 915 MHz ± 15 MHz as the only two frequency bands for industrial and medical applications. Thus, although the swept frequency approach has research merit in that the fundamentals of microwave materials processing can be studied at low power, such instrumentation will not reach the "factory floor." In an effort to improve on this situation, TA&T performed a paper design study of a 500 W system that would work in a narrow band around the designated 2.45 GHz. The source was a relatively inexpensive <u>tunable</u> coaxial magnetron. But it was determined that the power supplies would drive the cost of the system to more than $10,000/kW. *On the other hand, since microwave oven technology is so mature, microwave power can be supplied at a <u>fixed</u> frequency of 2.45 GHz for less than $500/kW!*

4.4 Rationale For The Proposed Approach

In a very large number of processing environments of interest to TA&T, nearly all samples to be CVI or sol-gel processed possess cylindrical symmetry - rocket combustors and heat exchanger tubes, for example. As one might suspect, the cylindrically symmetric field uniformity needed for uniform processing of these geometries is extremely difficult to achieve in the rectangular geometry of a conventional microwave oven cavity. While improvements can be obtained by sweeping the frequency, this cannot be considered a viable commercial option for the reason outlined above.

An excellent alternative is a multimode cylindrical cavity. It is a highly attractive method because it would exploit the natural symmetry of most sample geometries and thereby reduce the problem of creating process uniformity. In particular, circumferential processing nonuniformity would be virtually eliminated, while radial processing nonuniformity would be symmetric around the sample. As discussed below, this residual

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4 Johnson, A. C., et al, "Use of a Variable Frequency Microwave Furnace for Large-area, Uniform Processing," Am. Ceram. Soc. Symposium Proc. (1993).

processing nonuniformity would be much easier to eliminate in a cylindrical cavity than in a rectangular cavity.

The microwave modes referred to above are designated as either transverse electric, TE_{mnp}, or transverse magnetic, TM_{mnp}. For TE modes in a cylindrical cavity, the electric field vector points predominantly in the radial direction with the magnetic field vector pointing predominantly in the axial direction. For TM modes the role of the electric and magnetic field vectors is reversed. The subscript m corresponds roughly to the number of half wavelengths around a mean circumference inside the cavity, the subscript n corresponds roughly to the number of half wavelengths in the radial direction, and the subscript p corresponds to the number of half wavelengths along the axis of the cylinder. For example, Table 5 lists the modes possible in a cylindrical cavity 16" in diameter and 16" long and in one that is 4" longer and excited by microwave

Table 5. Two Sets Of Cylindrical Cavity Modes

16" dia. x 16" long	16" dia. x 20" long
TE_{025}	TE_{032}
TE_{232}	TE_{232}
TE_{134}	TE_{135}
TE_{225}	TE_{026}
TM_{125}	TE_{127}
TM_{224}	TE_{218}
	TE_{118}
	TM_{225}
	TM_{126}
	TM_{027}
	TM_{018}

radiation in a ± 50 MHz band centered on 2.45 GHz.[5] [6] It can be seen that the number of modes nearly doubles for a 25% increase in length. Further combinations can be derived from the expression for the resonant frequency of a cylindrical cavity:

$$(f_oD)^2 = \left(\frac{cp}{2}\right)^2\left(\frac{D}{L}\right)^2 + \left(\frac{cu_{m,n}}{\pi}\right)^2 \qquad (1)$$

where f_o is the frequency in Hz, D and L are the diameter and length in cm, c is the speed of light in cm/s, p is an integer, and $u_{m,n}$ is either the n^{th} root of the Bessel function $J'_m(u_{m,n}) = 0$ for TE modes, or the n^{th} root of $J_m(u_{m,n}) = 0$ for TM modes. For TE modes p = 1,2,3, ... and for TM modes p = 0,1,2,3, ...

As the cavity is lengthened or its diameter increased, the possible number of modes may also increase, although not monotonically, with one of the dimensions. Therefore, the microwave field uniformity also increases. But since the cavity volume would be decreasing, the available power density also would decrease. Thus, improvements in processing uniformity can be had at a cost in processing power density

5 Ginzton, E., Microwave Measurements (New York, McGraw-Hill Book Co., 1957), pp. 354 - 358.

6 Jackson, J. D., Classical Electrodynamics (New York, John Wiley & Sons, Inc.,1962), pp. 252 - 255.

available at the sample. The dimensions used in the example above correspond to a cavity volume equal to that of a commercial microwave oven currently being successfully used by TA&T for microwave CVI and sol-gel processing. The 25% increase in length resulting in a more uniform field distribution could well offset the corresponding 25% decrease in the power density. In the extreme, very high power density is available in *single* mode cavities, but good uniformity is available only over a small volume. Therefore, a design trade-off is needed. During the proposed effort, expression (1) will be used to maximize field uniformity consistent with maintaining high power density.

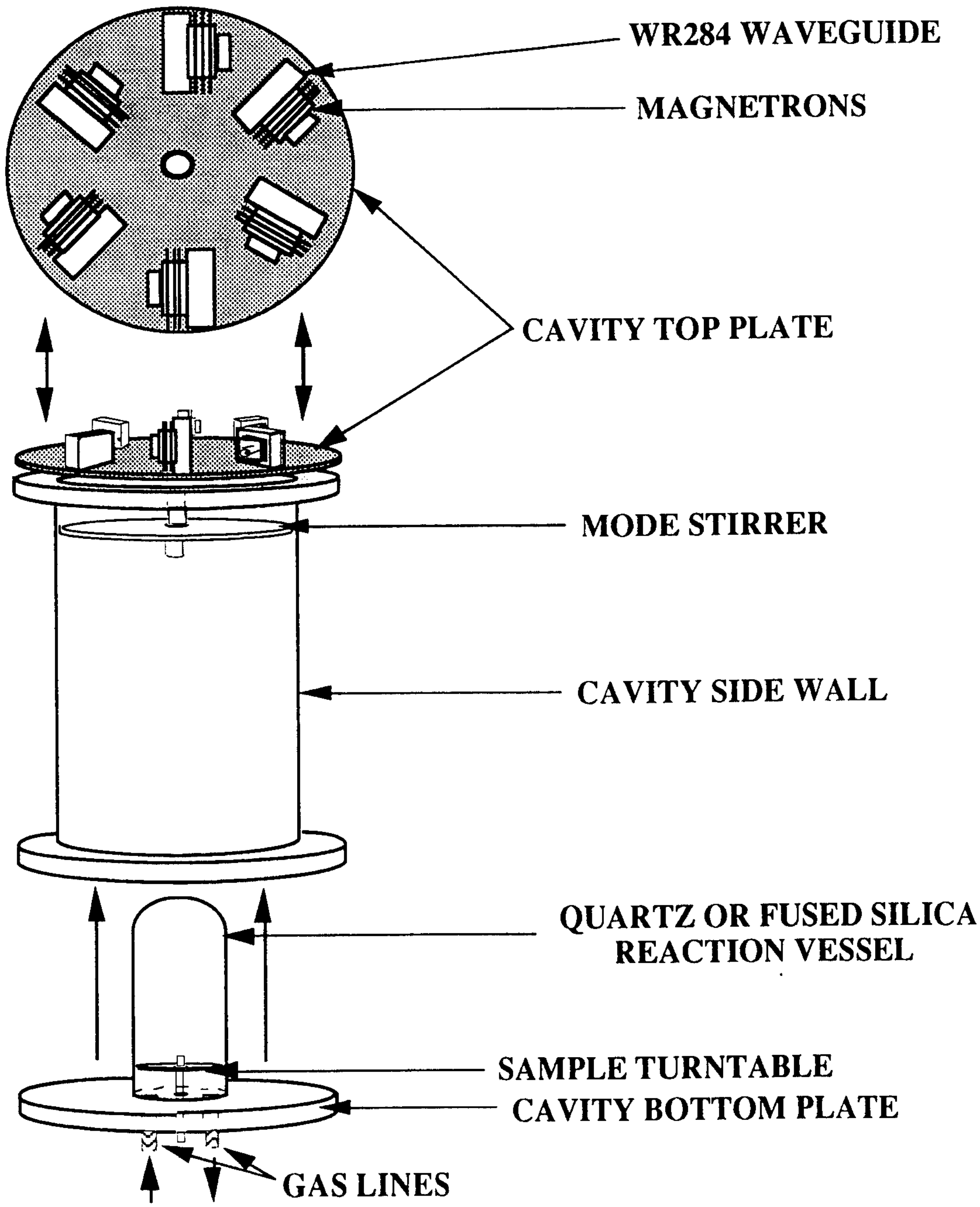

Figure 18. Fundamentals Of Proposed Multimode Cylindrical Cavity

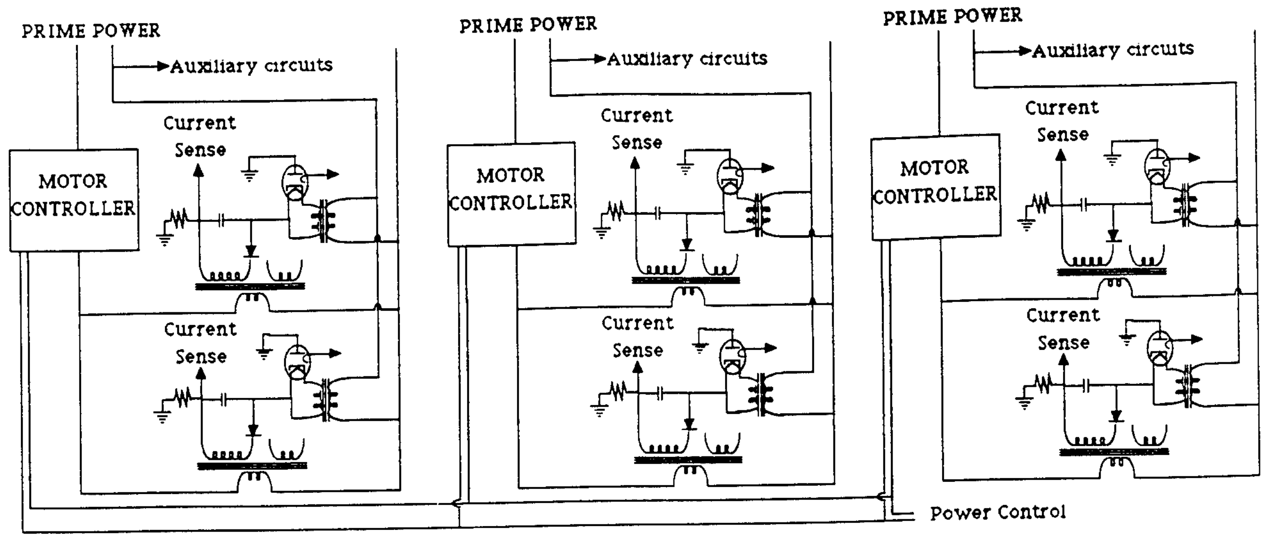

Figure 19. Schematic Of Control System

4.5 Design Concept

A proposed cylindrical cavity design concept is shown in Figure 18. A basic schematic of the high voltage electronics is shown in Figure 19. All microwave components would be inexpensive, readily available or off-the-shelf items currently being used in commercial microwave ovens. Six short sections of WR284 waveguide would feed six waveguide openings in the cavity from one end. To each waveguide section would be coupled a single, inexpensive (under $100) 1-1.5 kW magnetron. A single mode stirrer would be located near the top plate inside the cavity. Voltage would be supplied to each magnetron by the following, off-the-shelf components: 1) a filament transformer capable of isolating the filament at 4000 - 6000 volts below ground; 2) a power transformer capable of providing about 0.5 amperes at 4000 Vrms; 3) a diode bridge and filter capacitor. These values corresponds to the usual 70% prime-to-microwave power conversion efficiency of magnetrons. The primaries of the power transformers would be connected together in pairs, as shown in Figure 19, and prime power to each pair supplied through a SCR motor controller. The control lines from the three motor controllers would be connected together and used for both microwave power control and feedback stabilizations.

This configuration is preferred because, except for the motor controllers, it would employ the inexpensive components of a conventional restaurant-quality microwave oven. The motor controllers also would be inexpensive since they would be small, of low current operation and require no special cooling consideration. TA&T already has implemented this entire power control scheme for use with the two magnetrons driving the rectangular cavity currently being used for its CVI and sol-gel processing. It is very important to note that this approach is far more cost effective at achieving 6-9 kW of microwave power than one utilizing a single magnetron, water cooling, and a 9-13 kW high voltage power supply. The cost of the power supply alone would make this latter approach prohibitive. Primary power would be supplied from a

440 three phase line with buck-boost autotransformers to provide the three 110 volt 40 ampere lines needed to operate the high voltage supplies, filament supplies, blowers, interlocks, etc.

4.6 System Refinements (Hardware)

The three motor controllers would be used to vary the anode voltage of the six magnetrons from zero to maximum (4000 - 6000 V) by varying a control D.C. voltage from 0 - 10 V. This could be adjusted manually or by means of a voltage derived from some sensor. In particular, TA&T has used the voltage difference between the output of a two-color IR pyrometer and a fixed set point to maintain constant temperature. An improved approach would be one in which the pyrometer output was digitized using an analog-to-digital converter and a computer used to compare the digital value with a preset digital value. A proportional-integral-differential (PID) control scheme then could be implemented in software. TA&T is developing this approach for the considerably more complicated tasks of stabilizing the temperature of a substrate during CVD diamond deposition and the substrate in a molecular beam epitaxy machine. TA&T would transfer its control technology to the proposed program for the purpose of temperature control during microwave CVI processing. Once the computer is in place, data logging and gas flow control are natural additional refinements. TA&T is also developing these techniques for its diamond deposition and has completed several control modules.

4.7 System Refinements (Modeling/Software)

A cylindrical geometry was selected for the cavity because many of the cylindrical electromagnetic modes exhibit the same symmetry as the majority of the samples to be processed. Thus, uniform heating should be easier to achieve than if a cylindrical sample were being processed in a cavity of rectangular geometry. True homogeneous processing is possible only if the radiation in the cavity is isotropic. The greater the number of modes, the more nearly this isotropic condition is realized. As mentioned above, the larger the cavity the greater the number of modes that could be excited, but the smaller the resulting power density. Therefore, in a cavity of practical design, there can be only a limited number of modes possible, and so only a limited degree of microwave field uniformity and processing homogeneity. The residual inhomogeneities must be dealt with in some other way.

One method is to rotate the sample about its main axis. This will smooth out the effects of azimuthal inhomogeneities. It is a method frequently used in materials processing of cylindrical samples -- sputter deposition of drill bits, for example -- and as shown in Figure 18, sample rotation capabilities will be incorporated into the proposed cavity design. This leaves axial and radial field inhomogeneities. They will be reduced by incorporating cylindrically symmetric dielectric structures around the sample. These structures may consist of ceramic rings, and array of ceramic posts or even graphite susceptors. The effects of such structures must be calculated and the effect of the sample accounted for because the sample itself considerably alters the field configuration of the empty cavity.

A relatively straightforward method to accomplish this is the Transmission Line Matrix (TLM) method.[1] It provides a simple algorithm for modeling the electromagnetic fields in complex structure. The method is applied by dividing the region of interest into many small intervals much as is done in applying the finite element method. But in the TLM method, the spaces between nodes are treated as real transmission line segments of length DL and propagation delay Dt. Electromagnetic propagation is then treated as a combination of two processes: incident voltage pulses traveling towards a node, and pulses that are scattered by one node to travel toward and be incident on an adjacent node. The two processes are combined in a scattering matrix that is used to describe the dynamics of the entire network. In this way it is possible to model the complete three-dimensional microwave cavity.[2] This model has been used to predict the heating pattern of lossy objects in conventional microwave ovens.[3] An object of known complex dielectric factor was placed in an oven. The field pattern in the material was calculated by the TLM method. Infrared thermographic images of the heated object were in excellent agreement with the calculated field patterns showing increased temperature where the field intensity was highest. The strength of the method was further demonstrated by accurately calculating shifts in the resonant frequency of the cavity upon loading it with a dielectric object.

This method will be used in the Phase II effort to determine the shape and orientation of dielectric structures that will increase electromagnetic field homogeneity in the presence of the sample. A computer program will be used that will permit rapid modeling of field patterns as the structure configurations and geometries are changed. These objects then will be fabricated and used to improve processing uniformity.

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1 Desai, R. A., et al ,"Computer Modelling of Microwave Cooking Using the Transmission-line Model," IEE Proceedings **139**, 30 (1992).

2 El-Sayed, E. M., and Morsy, M. N., "Use of Transmission-line Matrix Method in Determining the Resonant Frequencies of Loaded Microwave Ovens," J. Microwave Power **19**, 65 (1984).

3 De Leo, R., et al ,"TLM Techniques in Microwave Ovens Analysis: Numerical and Experimental Results," International Conference on Computation in Electromagnetics (London, 1991) p. 361.

5.0 Conclusions and Potential Applications

The Phase I effort resulted in several accomplishments which met the technical objectives and expectations of the MWCVI concept. First, a conventional 2.6 kW microwave oven was modified for CVI operation, including the fabrication of a reaction chamber, gas introduction, vacuum equipment, extensive air and water cooling, and appropriate susceptor and insulation. A pulsed power, pulsed pressure system was added to increase the processing flexibility of the system, and provide enhanced densification rates of the fiber preforms.

Once the system was fabricated, a series of experiments were completed which studied the deposition/densification of carbon and silicon carbide matrix materials. **An order of magnitude improvement in densification rate over conventional CVI techniques was observed.** The preform geometry studied was similar to combustor can geometry for expendable turbine engine assemblies. Therefore, the Phase I work successfully achieved its main technical objective: the demonstration of an improved, economical CVI method for ceramic matrix composite fabrication.

Several potential markets exist for the immediate insertion of the MWCVI technology. These markets include, but are not limited to combustor cans, turbine blisks, heat exchanger tubes, and radiant burners. The potential for higher operation temperatures and improved efficiency utilizing components fabricated from CMC materials is expected to increase, specifically when economical processes are developed for production.