MICROWAVE SINTERING OF CERAMICS

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ABSTRACT

An overview of microwave sintering is presented and the basic theory for the design of impedance applicators is derived. Examples of applicators for ribbon materials and rods are developed and sintering results are presented. The problems imposed by radiation loss and plasma breakdown are examined and their solution by use of thermally reflecting high pressure cavities are described. A basic system for sintering with feedback control of surface temperature is presented.

INTRODUCTION

Recent experiments with microwave ultrarapid sintering (1) have demonstrated that smaller grain size and higher densities are possible in comparison with ordinary kilns. In addition, through-put is greatly increased and successful ceramics have been produced in minutes instead of hours as required by conventional methods.

Multimode and single mode applicators have been used to supply energy to the work piece. Multimode applicators, similar to microwave ovens, tend to produce variable and unpredictable results. Single mode applicators are more predictable and thus more capable of reliable design. The goal is to design the applicator so that a desired stable final temperature can be obtained. In single mode applicators, the field solution is often exact and when the object can be represented as an impedance, the design problem is particularly simple, and we call these "Impedance Applicators". This procedure is illustrated in the following examples.

SHEET APPLICATORS

The simplest applicator, used to sinter ceramic tapes or thin sheets, consists of a rectangular waveguide with a sheet of green ceramic passing through the waveguide with the sheet perpendicular to the major axis of the guide (2). The material is backed by an adjustable short circuit.

The input impedance of the applicator can be easily calculated, and since the sample is the only absorbing material, it absorbs all power entering the applicator. The problem then reduces to calculating the stable operating temperature from the heat balance equation.

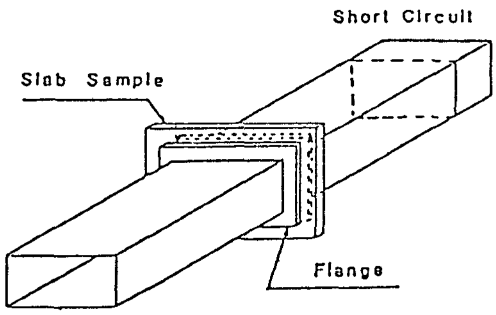

Figure 1. Sheet Applicator

ROD APPLICATORS

An impedance applicator for rod shaped samples is based upon a section of rectangular waveguide (3).

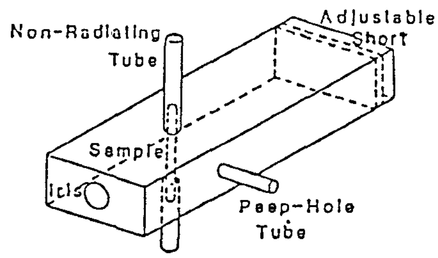

Figure 2. Rod and Tube Applicator

Here, a ceramic rod is inserted through beyond cutoff tubes into the waveguide along a line perpendicular to the broad face. The sample is backed by an adjustable short circuit and the applicator is tuned by an adjustable iris in front of the sample. By adjustment of the short circuit and the iris, the applicator can be matched and all available power dissipated in the sample. Since the rod can be represented as a shunt admittance, the input impedance can be calculated, and by means of the heat loss equation, the stable operating temperature can be predicted.

Since temperatures are quite high, upwards of 2000 C for sintering SiC, heat loss is primarily by radiation. The heat loss varies as T^4 and thus large increases in power are required to achieve significant increases in temperature. To alleviate this problem, we have been investigating impedance applicators which reflect radiant thermal energy back onto the surface of the ceramic (4).

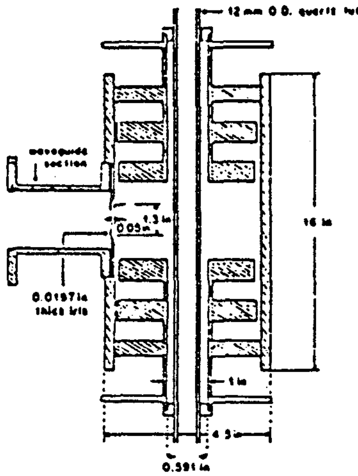

Figure 3. Cross Section of the TE_{111} Cavity

This need has led to the development of high temperature applicators using circular cylindrical geometries with the rod shaped sample proceeding along the axis of the applicator. A TE_{111} applicator was built with mirror smooth silvered walls to provide thermal reflection. The applicator is constructed with adjustable dumbbell short circuits. To prevent arcing at high power, the diameter of the first step of the dumbbell is reduced. Since the power at the second step is much less, the diameter can be safely increased. The applicator is resonated by asymmetric movement of the end walls and the coupling is varied by symmetric adjustment which shifts the iris position with respect to the interior field. This cavity has undergone cold tests, and the measured Q implies that there is very little loss in the end walls. Hot tests with SiC verify the ability to achieve a match at sintering temperatures and demonstrate the higher temperatures achieved by the thermally reflecting walls. Other useful results of this design are higher surface temperatures and more uniform heating.

As the required temperature increases, more power is required and an upper limit is imposed by plasma generation. To inhibit plasma formation and achieve higher temperatures, the applicators are pressurized to 150 At. Current experiments are proceeding at these pressures in nitrogen-hydrogen mixtures.

SINTERING SYSTEM

All measurements of input impedance and surface temperature during sintering are performed with the following apparatus.

A Raytheon Mod PGM 100 source capable of 800 watts at 2450 MHz is connected to a circulator to present the magnetron with a matched load. The side arm of the circulator is connected to a calorimeter to observe reflected power. Since the

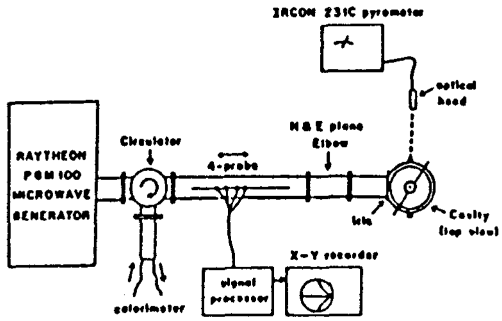

Figure 4. Measurement Setup for Microwave Heating Experiments

calorimeter response is slow compared to changes in the heating process, instantaneous impedance data is provided by a four point probe detector for observing the input impedance of the applicator and displaying the impedance contour on a Smith Chart. In this manner, dynamic variations in impedance can observed. This arrangement is far more efficient and informative than the industrially common reflectometer alternative. The surface temperature is monitored by IRCON radiometers focused on the sample through beyond cut-off tubes in the walls of the applicator.

At this time, successful design is hampered by the lack of conductivity and permittivity data as a function of temperature at 2450 MHz. To measure these parameters, the rectangular applicator was operated in inverse fashion to relate the constitutive parameters to the input impedance of the applicator (5). The temperature dependent parameters of Al_2O_3 were determined over the sintering temperature range.

ACKNOWLEDGEMENT

This research was supported by the Electric Power Research Institute under Project 2730-01 and by the Northwestern University Materials Research Center operated under NSF, DMR-85-20280.

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